HIMACROW
Harmonized Personnel Training for Macro and Microscopic Metallographic Examination of Structural Materials

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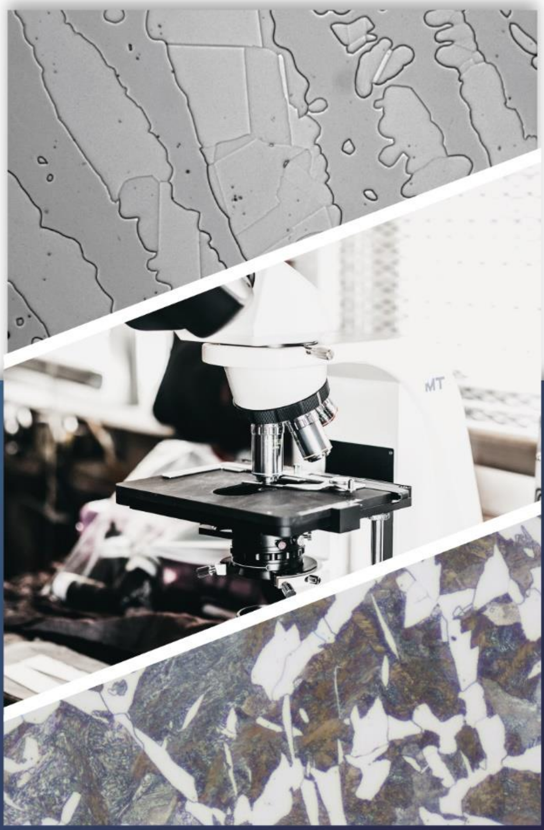

Educational Materials for HIMACROW Training Course

For the Personnel with the responsibility for **Macroscopic** and **Microscopic** Metallographic Examination of Base Materials and their Joints Made by Welding and Allied Techniques **(Standard & Comprehensive Levels)**.

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Introduction

Metallography is the study of the microstructure of metallic alloys. It can be more precisely defined as the scientific discipline of observing of the spatial distribution of the grains, constituents, inclusions or phases in metallic alloys, in relation to their properties, over the whole temperature range.

One of the first approaches dates back to the about 1860s, when Henry Clifton Sorby (10 May 1826 – 9 March 1908), used on the metals some techniques of petrography and observe their structure. Now was possible to associate the chemical, physical and mechanical characteristic of a metal or metallic alloy with its metallographic structure, for the metallurgy, it was the transition from empirical to real science. Nonetheless, the iron and steel industry processes of 120-130 years ago not allow to obtain metal alloys with a low inclusions or impurities control; therefore, the characteristics mentioned above were often random and not easily reproducible. Consequently, the development of metallography techniques occurred in the last 50-60 years, after the development of iron and metal industry.

Today the metallographic technician and analyser finds relevant space in industry, within metallurgical laboratories, quality control, research and development; these figures require multifaceted knowledge in various scientific and industrial sectors, they must have a good knowledge of the metallurgy of metal alloys and their weldability.

This document aims to provide both practical and theoretical guidance for metallographic technicians, analyst, engineer and laboratory manager.

The main metallographic tests can be divided in two big family: macrographic and micrographic examinations. The first one provides macroscopic information, for example welding imperfections, while the second one provides microscopic information, for example the type of microstructure, grain size, inclusions content and so on.

Macrographic examination generally finds its place in the following activities:

- Certification of welding procedure and welding personnel.
- Optimization of welding process.
- Quality control during manufacturing.
- Failure analysis.

On the other hand, the Micrographic examination in the following applications:

- Optimization of welding process.
- Quality control during manufacturing.
- Evaluation of long-term operating life for some kinds of alloys.
- Failure analysis.

- Research and development.

The preparation techniques, the analysis tools and the information we can obtain by metallographic examinations will be described in the following chapters.

Fig. 1.1 - Henry Clifton Sorby (10 May 1826 – 9 March 1908).

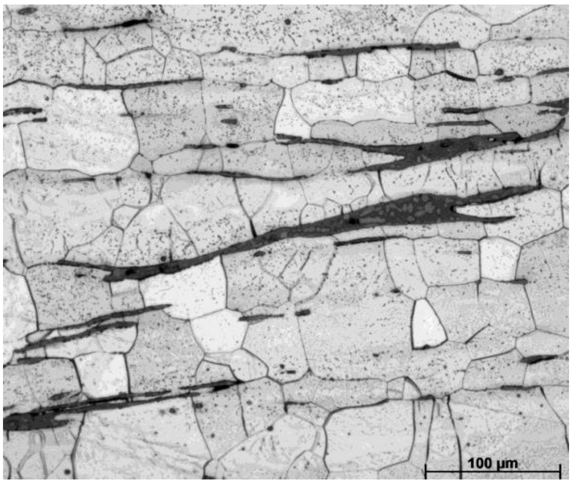


Fig. 1.2 – micrographic detail of a steel taken from a railway bridge of 1880.

1. CU1: Introduction to Metallographic Examination

A very important part of material testing is the metallographic examination. The chemical composition and macrostructure and microstructure of the materials determine the material's properties. The metallographic examination includes the knowledge of the metallographic specimen preparation and the examination methods, light microscopy, image analysis and etc. The metallographic specialist needs to know the important rules and regulations covering occupational safety.

1.2. The types of metallographic examination; work safety conditions during the preparation of specimens for metallographic examinations

Occupational safety and health are important in a metallographic labs considering the use of machines, chemicals, plastics, etc. The work in the laboratory involving machines, chemicals, etc., makes it imperative that all relevant rules and regulations covering occupational safety are followed. The laboratory having functions like a cut-off, specimen preparation, and etching involves a number of potential dangers. The most important precautions regarding safety and health are mentioned in the following, keeping in mind that not all potential dangers or necessary safety and health measures are mentioned. All materials stored in the laboratory must have a Material Safety Data Sheet (MSDS). A MSDS is designed to provide both laboratory personnel and emergency personnel with the proper procedures for handling or working with a particular substance. MSDSs include information such as physical data, health effects, first aid, reactivity, storage, disposal, protective equipment, and spill/leak procedures. These are of particular use if a spill or other accidents occur.

1.2.1. Sampling safety

Eye protection and gloves should be used when working with machines and tools. Abrasive cut-off machines will have a protective shielding for the operator against cooling fluid, pieces from a broken cutting wheel, etc. According to safety regulations, the machine can only be started when the protective case is closed. When cutting with bakelite or rubber wheels, fumes develop, and at larger machines, the cutting chamber should be connected to an exhaust system.

1.2.2. Mounting safety

With hot compression mounting, the main danger is the high temperatures used in the mounting presses. Gloves should be used for handling of the hot mounts, and when filling mounting material such as bakelite powder into the press, inhalation of dust should be avoided.

At cold mounting, there are several potential dangers. The cold mounting resins are potentially dangerous to health, and therefore all handling should be done with rubber (latex) gloves to avoid skin contact and the mixing and hardening shall take place under a smoke (gas, fumes) enclosure. When using a furnace to accelerate the hardening of epoxy, gloves should also be used. MSDS (Materials Safety Data Sheet) information should be available for all mounting resins.

Some cold mounting materials:

Epoxy

This material, both resin and hardener, can damage the skin, causing allergic reactions. The material should never come in contact with skin and the vapours should not be inhaled. All work with epoxy should take place under a smoke enclosure using personal protective equipment (PPE specified in the MSDS). It is recommended that containers and stirring pins are disposed after mixing. The epoxy is not dangerous when it has fully hardened, therefore no protection is needed for handling the sample.

Acrylics and Polyesters

Mixing should always take place under a smoke enclosure and gloves should be used when handling these materials to avoid inhalation and skin contact.

1.2.3. Mechanical preparation safety

The operator should be using a laboratory coat, gloves, protective glasses and masks in order to avoid all injuries. During the manual preparation, when using a grinding/polishing disk, precautions should be taken. The operator needs to use a well-mounted sample with a suitable size for manual preparation. The semiautomatic preparation machines are basically safe to use. If a specimen is slung away from the specimen holder during rotation, the specimen will be kept inside the machine, and the speeds of the disks and holders are relatively low. To avoid contamination and impurities, especially when using grinding disks and abrasive paste, that create a very fine sludge, which is difficult to remove, thus rubber or plastic gloves should be used. For large machines using a flammable alcohol-based lubricant, the alcohol fumes can be rather strong, and the machine should be placed in connection to a fume exhaust system. A better solution might be to change the lubricant to a water-based type. Fully automatic systems are totally enclosed and will not operate if the protective shields are not closed.

1.2.4. Electrolytic Polishing and Etching safety

The most serious potential danger at electrolytic polishing and etching is the mixing, handling, use, and storing of electrolytes. During the polishing process, precaution should be taken to avoid heating the electrolyte which may cause a fire or even an explosion. It is important to keep a correct level of electrolyte in the container because a lower level of electrolyte might generate air pockets in the polishing chamber causing a spark between the anode and cathode, which can lead to the ignition of the solution in the electrolyte. For these types of operations there should be used a laboratory coat, gloves, and protective glasses to avoid injuries.

1.2.5. Etching safety (toxic and flammable materials)

The etching procedure should be performed using gloves and eye protection under a smoke enclosure. The most serious potential dangers are connected to the mixing, handling, and storing of the chemicals used for etchants, as well as for the electrolytes. The user should have sufficient information on all the chemicals used from training and Material Safety Data Sheets (MSDS), which should always be available in the laboratory. Etchants/electrolytes are mostly solution mixtures of different materials mixed in the laboratory. During the mixing, very dangerous reactions can be established such as: fire, explosion, and dissolution, creating dangerous substances and/or fumes. As a rule, the mixing should always start with the water (alcohol) and then mix the other ingredients into this. A special procedure, however, should often be followed to obtain the necessary safety, and certain combinations of chemicals are known to be dangerous. Information on this must be available in the laboratory. The most common materials for metallography labs are:

Ethanol: Ethanol used in a laboratory has the following Hazards Identification: Organic solvents may be absorbed by inhalation and ingestion and cause permanent damage to the nervous system. The liquid may irritate the skin, the eyes and the respiratory tract.

Acetic Acid: Extreme care should be taken when mixing with other acids like perchloric acid and nitric acid. Mixtures of acetic acid and perchloric acid should be avoided.

Chromic Acid: Very strong oxidation substance, a very strong reaction with organic or easy-to-oxidize material and it cannot be safely mixed with acetic acid and most organic liquids, such as alcohols or glycerol. POISONOUS!

Nitric Acid: Strong oxidizing acid, creating gases with many organic materials and metals. Do not store solutions of more than 5% nitric acid in ethanol.

Perchloric Acid: Very aggressive acid that can cause serious explosions at higher concentrations. Very dangerous in contact with metals that oxidize easily, such as bismuth. Contact with organic material should also be avoided; if absorbed in an organic material the material must be placed in water. Organic material should never be used to wipe up perchloric acid.

Picric Acid: Aggressive oxidizer and should always be stored in wet condition. In dry conditions, the risk of explosion is high. Storage should be cool and safety conditions for fire outbreak should be put in place.

Toxic materials are substances that, when inhaled or ingested can lead to death or serious health problems.

Several toxic substances that are often used in a metallographic laboratory are:

- Metal based chemicals: Be, Pb, Cr, Cu, Ag, Sn, Zn, Ni (not ZnO_2): Poisonous in liquid or vapours.
- Chlorinated Carbohydrates: These solvents are very dangerous to inhale.
- Cyanides: These react strongly with acids, even the CO_2 content in humid atmospheric air gives a reaction. Very poisonous.
- Hydrofluoric Acid, Anhydrous: In concentrations higher than 0.5 % of the acid is very dangerous, making very serious tissue damage. Attacks almost all metals and glass. Very dangerous.
- Mercury: Chemicals bond on mercury and mercury vapours are poisonous.
- Methanol: Poisonous.
- Oxalic Acid (Anhydrous) (Dihydrate): In concentrations higher than 5 %, oxalic acid is poisonous.
- Phosgene (Carbonyl Chloride): Very dangerous to the respiratory system.

Flammable liquids are used in metallographic preparation for electrolytic polishing, lubrication, etching, and cleaning.

The number of flammable liquids stored in the laboratory should be kept at a minimum and carefully protected against heat and possible ignition. All rules and regulations should be followed carefully when handling flammable liquids. Heating in open containers should be done with special care, using a smoke enclosure and special electric heating plates.

All containers with dangerous materials should be marked, the examples of the danger symbols are shown in Table 1.1.5.1.

Table 1.1.5.1. Symbols of the dangers

Category of danger	Symbol of danger
Explosive	

Oxidizing	
Extremely flammable	
Corrosive	
Irritating	
Toxic	

Disposal can be a problem and should be done according to rules and regulations by local authorities.

References:

[1.1.1] Bramfitt B. L.., Benscoter A. O.: Metallographer's Guide, Practices and Procedures for Irons and Steels. 1st. Ed. ASM International, Materials Park, USA 2002. 169–244.

[1.1.2] McCall J. L.., Mueller W. M.: Metallographic Specimen Preparation, Optical and Electron Microscopy 1st. ed. Plenum Press, New York, USA 1973. 1–348.

[1.1.3] Vander Voort G. F.: Metallography Principles and Practice. 4th. ed. ASM International, Materials Park, 2007. 1–258.

[1.1.4] Kay Geels: Metallographic and Metallographic Specimen Preparation, Light Microscopy, Image Analysis and Hardness Testing ASTM International, West Conshohocken, USA 2007. 1-761.

1.3. Methods of specimen preparation for macro- and microscopic examination

The specimen preparation steps are shown in Figure 1.2.1.

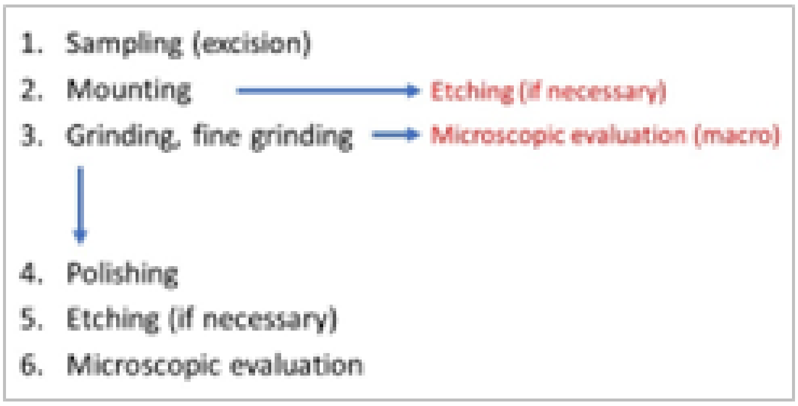


Figure 1.2.1. The macroscopic and microscopic specimen preparation flow

1.3.1. Sampling

When examining welded joints, it is advisable to follow the recommendations of the respective standards. During the cutting process, it is important to ensure that the material structure of the sample is not altered. Therefore, cutting should not cause the specimen to heat up or undergo plastic deformation, as both can lead to changes in the material structure and false interpretations. The processing can be performed using mechanical cutting or machining. Typically, metallographic specialists employ a diamond abrasive cutoff wheel with adjustable speed and water cooling as well as mechanical / electric saw with a cooling system in order to perform the sample cutting. For thin sheets and foils, cutting with scissors is also an option. However, in specific cases, laboratories use specialized cutting techniques, as an example, for materials with high levels of hardness, the most common processes are electro erosion or water jet cutting. The sheared surface will inevitably undergo deformation, so it is necessary to further process the surface by grinding.

1.3.2. Mounting

Mounting refers to encapsulating the machined sample or small-sized specimen in a polymeric material or other compounds. Depending on the type of resin used and the nature of the sample, cold or hot mounting methods can be applied. The primary objective here is to ensure that neither the structure nor the chemical composition of the sample is altered during the mounting process [1, 2, 3]. In cold mounting, a two-component resin is typically used. During the curing of the resin, exothermic reaction occurs, it does not cause structural changes for metals and their alloys. To prevent failure, it is necessary to follow the recommended specifications provided by the manufacturer. Hot mounting refers to a process of embedding a metal sample in a thermosetting plastic resin while using heat and pressure. It provides a stable and uniform environment for examining the internal structure and properties of metals and alloys. Caution should be taken in the case of a small, brittle samples or those that have a low melting point.

1.3.3. Marking

Marking of metallographic specimens is essential for a proper identification of the specimen that ensures that the metallographic specialist uses the correct sample for analysis. The marking should not in any way influence the microstructure of the surface to be examined; therefore, care should be taken before marking is done. It should also be taken into consideration that marking is written in an area where it is not removed and/or damaged during the preparation process.

Marking with Waterproof Ink

It is recommended to be done on the resin or as far as possible from the analysis area. Caution should be taken to avoid removing or partially whipping it. Marking ink or pencil can be used as an alternative.

Identification Tag

Usually, the tag is placed on the backside of the specimen and in the case of embedded samples they should be inserted in a reasonably transparent resin, as well as on the outside of the specimen/mount. Caution should be taken in order to prevent removing it during cleaning in water and alcohol.

Engraving

One of the most widely used methods of marking is vibration engraving. A very hard vibrating needle induces a visible deformation of the surface of the mount/specimen. This method will stress the material layer below the surface, and it should not be used for thin specimens like foils. Electro engraving gives less damage to the surface but can only be used on electrically conducting materials. The engraving on a clean and smooth surface, is done through the melting/evaporation of metal, caused by a high-temperature spark. Manual engraving, using a chisel, is also possible for most materials that do not have high hardness values.

Stamping

Stamping a number or code in the specimen is possible but not necessarily recommended because it can be easily removed during handling or processing.

1.3.4. Cleaning

It is important that the environment in which the process takes place is free of impurities and the operator’s hands are clean.

Considering that the preparation process often includes different granulations of grinding paper (grit 80 to 5000), polishing cloth and diamond suspension (1 to 3 µm), which are progressively changed, it is evident that cross-contamination can occur thus it is recommended to use each grit paper just once, because larger grains from an earlier step will produce cross-contamination.

Cleaning Before Start of Preparation

Failure to clean thoroughly can prevent cold and hot compression mounting resins from adhering to the specimen surface. Also, oxidation and other contamination should be removed unless these products are to be examined. As an example of coarser cleaning methods, like shot blasting and wire brushing might damage the surface, therefore it should be considered whether this might influence the final result of the preparation.

Cleaning During and After Preparation

In principle, the specimen and the specimen holder (fixed specimens) should be cleaned between every step in the grinding and polishing process, but at wet grinding on SiC paper, using a constant flow of water, a careful cleaning can be limited to after the last grinding step. When grinding on other media, like rigid composite disks, and at polishing, cleaning between each step is absolutely necessary. There are, however, some materials or some constituents in materials that will be preferentially attacked by water (zinc coatings on steel, lead inclusions in machining steels, etc.), therefore, water must be avoided completely in the latter preparation steps.

Cleaning by Hand

For cleaning most metals and other materials without pores, cracks, etc., manual cleaning is the most effective method. The specimen or specimen holder is held under running lukewarm water and rinsed with a soft brush or cotton ball dipped in water with a detergent. In cases of soft materials and after the last polishing step, a cotton ball is used to clean the prepared surface. The cleaning is finished with the spraying of ethylene alcohol from a spray bottle on the prepared surface and possibly wiping the surface before it is taken for drying. Ethanol containing a denaturation additive can be used as long as the additive does not leave a film on the specimen. In the case of specimens with pores, cracks, etc., and mounts with a gap between mounting material and specimen, ultrasonic cleaning is recommended, except for fragile materials.

In the case of polymer test samples, the cleaning is suggested to do with distilled water because ethanol can solve some polymer materials.

Ultrasonic Cleaning

Ultrasonic cleaning is efficient and relatively fast, removing dirt from pores, gaps, cracks, etc. The specimen or specimen holder is placed in a tank with a liquid, water with a detergent, alcohol, or an organic solvent like acetone. In cases of dirt difficult to remove, weak acids and basic solutions can be used. A transducer vibrates under the bottom of the tank, with 20–40 kHz creating gas bubbles (cavitation) in the liquid. These bubbles implode and a rubbing effect occurs which removes the dirt. The dirt contaminates the liquid and therefore a specimen must be rinsed, as mentioned above, before drying. Ultrasound penetrates glass so it is possible to place one or more beakers in the tank with water with a detergent around the beakers. When the specimens are only placed in the beakers, in a suitable liquid, this liquid can easily be exchanged when contaminated. Cleaning usually takes 0.5–1 min. Some soft, very porous, and brittle materials should not be cleaned ultrasonically or only for 10–30 s. Using specimen holders, the whole holder can be placed in the tank.

1.3.5. Drying

Drying should take place in a stream of mild air. It is important that the layer of alcohol is not dried on the prepared surface but is blown away and evaporates from the sides of the specimen/mount. Air can be supplied from a fixed hair dryer type apparatus allowing both hands to be free to clean the specimen with soft cotton wool and alcohol. Compressed air can be used as well, in which case clean, dry, oil-free air must be secured. A hairdryer is recommended for the finished specimen to avoid possible oil drops from the compressed air.

1.3.6. Mechanical surface preparation, Grinding

The surface of the sample is ground using progressively finer abrasive wheels. The abrasive material used can be silicon carbide-based or diamond grinding discs. Abrasive materials are commercially available in various grit sizes ranging from P60 to P4000. Abrasive materials with grit sizes of P2000 to P4000 are considered polishing agents as well. Whether manual or automated grinding is employed, during each grinding step, the sample is rotated 90 degrees to remove the previous grinding scratches. Thus, grinding eliminates the previous grinding scratches with perpendicular and finer scratches (see Figure 1.2.2). Grinding is performed with a continuous water supply, where the water helps remove loose particles, debris, and heat generated by friction.

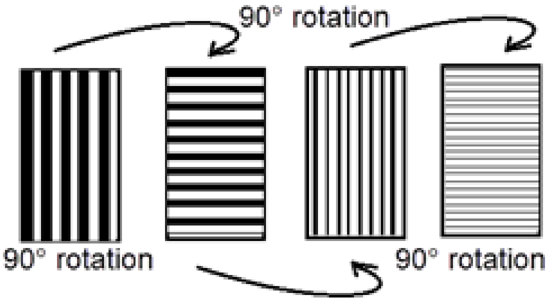


Figure 1.2.5.1. The surface preparation by grinding

1.3.7. Mechanical surface preparation, Polishing

The next step is to polish the surface prepared to the desired level of smoothness. Polishing can be performed through mechanical, chemical, or electrolytic methods. Mechanical polishing is carried out using a polishing disc or a polishing cloth. The material of the polishing cloth can be natural or synthetic. For polishing can used, a polishing paste or a distilled water suspension containing aluminium oxide, magnesium oxide, or diamond particles ranging from 0.05 to 15 µm in size, which should be compatible with the material and the hardness of the polishing cloth. During manual polishing, the sample should be moved in a circular motion opposite to the direction of rotation on the polishing disc. After polishing, the surface is washed with distilled water followed by alcohol. The applicability of alcohol should be checked for non-metallic samples. The surface is then dried with warm, blown air. Once the surface is polished, macroscopic examinations can be conducted. At 50 times magnification, certain cracks, welding defects, and inadequate fusion become visible.

1.3.8. Electrolytic polishing

Electrolytic preparation is a fast and efficient method for metallographic preparation, providing a deformation-free surface, whereas mechanical grinding and polishing tend to leave a thin deformation layer at the surface. Using the electrolytic polishing equipment, the specimen is configured as the anode in a suitable electrolyte, and the material is removed by controlled dissolution of the surface of the specimen. Electrolytic polishing can be followed by an electrolytic etching process to bring forth contrasts in the microstructure of the specimen.

1.3.9. Etching

More detailed examinations are conducted by etching the surface following the recommendations of ISO 16060. The etchant should be selected according to the quality of the material being examined. The etchant selectively attacks the sample surface to varying degrees at grain boundaries and grain surfaces, making them visible under a microscope. Altered microstructures resulting from heat treatment can also be effectively examined, especially in the case of tool steels. Color etching techniques provide more information and enhance recognition. The etchant forms a stable-colored film on the sample surface, which can be an oxide, sulphide, chromate, or complex compound. Strict adherence to the mixing order and ratios is essential during etchant preparation. For chemically resistant materials, electrolytic etching can be used. In conventional etching, the sample surface is immersed in the etchant and gently moved. The etching time also depends on the microscope resolution, with longer durations required for lower-resolution microscopes and shorter durations for higher-resolution microscopes. After etching, the sample is rinsed thoroughly with water, followed by rinsing with alcohol, and then dried with warm blown air. Some common etching materials are collected in Table 1.2.8.1.

Table 1.2.8.1. The most used etchants

Name	Etchant	Application area
Nital	97 ml ethyl alcohol; 3 ml 69% nitric acid	Unalloyed and low-alloy steel
Pikral	100 ml ethyl alcohol; 4 g picric acid	Unalloyed and low-alloy steel
Hydrochloric acid-iron chloride	960 ml ethyl alcohol; 20 ml 35% hydrochloric acid; 50 g iron(III) chloride	Copper and its alloys
Keller	950 ml distilled water; 25 ml 69% nitric acid, 15 ml 35% hydrochloric acid; 10 ml 40% hydrofluoric acid	Titanium and its alloys, aluminium and its alloys
Kroll	10 ml 40% hydrofluoric acid; 30 ml 69% nitric acid; 960 ml distilled water	Aluminum and its alloys
Oxalic acid solution	100 ml water (H2O) and 10 g oxalic acid (C2H2O4)	Stainless steel

References:

[1.2.1] Bramfitt B. L.., Benscoter A. O.: Metallographer's Guide, Practices and Procedures for Irons and Steels. 1st. Ed. ASM International, Materials Park, 2002. 169–244.

[1.2.2] McCall J. L.., Mueller W. M.: Metallographic Specimen Preparation, Optical and Electron Microscopy 1st. ed. Plenum Press, New York, 1973. 1–348.

[1.2. 3] Vander Voort G. F.: Metallography Principles and Practice. 4th. ed. ASM International, Materials Park, 2007. 1–258.

[1.2. 4] EN ISO 17639:2022 Destructive tests on welds in metallic materials — Macroscopic and microscopic examination of welds 2022.

[1.2. 5] ISO 15614-1:2017 Specification and qualification of welding procedures for metallic materials — Welding procedure test — Part 1: Arc and gas welding of steels and arc welding of nickel and nickel alloys.

[1.2. 6] ISO/TR 16060:2003 Destructive tests on welds in metallic materials — Etchants for macroscopic and microscopic examination, 2003.

1.4. Macroscopic examination and its application in welding technology

In this chapter, we will explore the significance of macroscopic examination in the field of welding technology. Welding is a widely used joining process in various industries, and the quality and integrity of welded joints play a crucial role in the performance and reliability of structures and components. Macroscopic examination provides valuable insights into the overall structure, defects, and quality of welds, enabling engineers and technicians to assess weld integrity and make informed decisions regarding their suitability for specific applications.

1.4.1. Overview of Macroscopic Examination

In this subchapter, we will provide an in-depth overview of macroscopic examination and its importance in welding technology. We will discuss the purpose and objectives of macroscopic examination, its advantages over other testing methods, and how it complements other inspection techniques, such as non-destructive testing and microscopic examination.

Macroscopic examination, also known as visual inspection, is a fundamental technique used in welding technology to evaluate the quality, integrity, and workmanship of weldments and welded joints. It involves the direct observation and assessment of the weld surface and adjacent areas using the naked eye or magnification tools.

Definition and Purpose of Macroscopic Examination

The purpose of macroscopic examination is to identify and analyze the overall characteristics, defects, and workmanship of welds. It aims to ensure that the welds meet the required standards, specifications, and performance criteria. This examination method plays a critical role in weld quality control, enabling the detection and evaluation of both visible and hidden issues that may affect the structural integrity, functionality, and reliability of welded components. Macroscopic examination holds significant importance in welding technology for several reasons:

Quality Assurance: Macroscopic examination allows for the assessment of weld quality and compliance with industry standards and specifications. It helps identify defects, discontinuities, and deviations from acceptable criteria, ensuring that welds are structurally sound and meet the desired performance requirements.

Early Detection of Defects: Visual inspection enables the early detection of defects in welds, such as cracks, porosity, lack of fusion, incomplete penetration, undercutting, and excessive reinforcement. Detecting these issues at an early stage allows for prompt corrective actions, reducing the risk of further damage or failure.

Cost-Effectiveness: Macroscopic examination is a relatively simple and cost-effective method compared to other advanced testing techniques. It provides valuable information about the overall condition of welds, helping in the decision-making process regarding further inspections or necessary repairs.

Complementing Non-Destructive Testing (NDT): Macroscopic examination complements other inspection techniques, including non-destructive testing methods such as ultrasonic testing, radiography, and magnetic particle inspection. It helps in identifying potential areas of concern for more detailed evaluation using NDT methods, thus optimizing the inspection process.

Visual Documentation: Macroscopic examination allows for the visual documentation of welds and their characteristics. Photographs, videos, or sketches taken during the examination serve as essential records for reference, analysis, and communication among welding inspectors, engineers, and stakeholders.

Macroscopic examination offers several advantages in the evaluation of welds:

Real-time Observation: Visual inspection provides real-time assessment, allowing immediate identification of visible defects and anomalies.

Cost-Effective: Macroscopic examination is a relatively inexpensive and accessible method that requires minimal equipment and training.

Wide Applicability: It can be applied to various weld types, sizes, and materials, making it versatile in different welding applications.

Non-Destructive: Macroscopic examination does not cause any damage to the tested specimens or structures.

However, it is essential to acknowledge the limitations of macroscopic examination:

Surface Sensitivity: It primarily focuses on the surface characteristics of welds, limiting the detection of subsurface or internal defects.

Subjectivity: Interpretation of visual inspection results can be subjective, depending on the inspector's experience, skill, and visual acuity.

Macroscopic examination plays a crucial role in weld quality control by providing valuable information about the overall integrity and workmanship of welds. It helps ensure that welds meet specified requirements, including geometry, dimensional tolerances, appearance, and the absence of visible defects. By combining macroscopic examination with other inspection techniques, such as non-destructive testing and microscopic examination, a comprehensive assessment of weld quality can be achieved. This integrated approach enhances the reliability, performance, and safety of welded structures, components, and systems.

In the European Union (EU), macroscopic examination in welding technology is guided by various standards and guidelines that provide requirements and recommendations for the inspection process. Some of the key EU standards related to macroscopic examination in welding include:

EN ISO 5817 - Welding - Fusion-Welded Joints in Steel, Nickel, Titanium, and Their Alloys (Quality Levels for Imperfections): This standard provides criteria for evaluating the macroscopic imperfections in fusion-welded joints. It defines different quality levels and acceptance criteria based on the type and size of the imperfections observed during macroscopic examination.

EN ISO 17637 - Non-Destructive Testing of Welds - Visual Testing of Fusion Welded Joints: This standard specifies the requirements for the visual testing of fusion-welded joints. It outlines the general principles, equipment, procedures, and acceptance criteria for macroscopic examination to ensure proper inspection of weld surfaces and the identification of visible defects.

EN 970 - Non-Destructive Examination of Fusion Welded Joints: This standard provides guidelines for the visual examination of fusion-welded joints. It covers the general principles, equipment, techniques, and acceptance criteria for macroscopic examination to assess the surface conditions, weld profiles, and the presence of visible defects.

EN 1290 - Destructive Tests on Welds in Metallic Materials - Macroscopic Examination of Welds: This standard specifies the requirements for macroscopic examination of welded joints through destructive testing methods. It details the procedures for cutting, preparing, and examining macro sections of welds to evaluate their quality and to identify any defects or discontinuities.

These standards, among others, provide guidance on the macroscopic examination techniques, acceptance criteria, and documentation requirements in welding technology within the European Union. Compliance with these standards ensures consistent and reliable evaluation of welds, promotes quality control, and supports the safe and efficient use of welded structures and components.

Importance of Macroscopic Examination in Welding Technology

Macroscopic examination plays a crucial role in welding technology, providing valuable insights into the quality, integrity, and reliability of welded joints. This inspection method involves the visual inspection and evaluation of weld surfaces and macrostructures to detect and assess various features, defects, and discontinuities. The importance of macroscopic examination in welding technology can be understood through the following aspects:

Quality Control and Assurance: Macroscopic examination serves as a fundamental tool for quality control and assurance in welding. It allows for the identification and evaluation of visible imperfections, such as cracks, lack of fusion, porosity, and undercut, which may compromise the structural integrity and performance of welded joints. By detecting and addressing these defects early on, macroscopic examination helps ensure that welds meet specified quality standards and regulatory requirements.

Weld Integrity Assessment: The macroscopic examination provides valuable information about the overall integrity of welded joints. It enables the inspection of the weld profile, including the shape, size, and reinforcement, which are crucial for achieving the desired joint strength and load-carrying capacity. By assessing the macrostructure, including grain structure, segregation, and heat-affected zone (HAZ) characteristics, macroscopic examination helps evaluate the mechanical properties, such as hardness, toughness, and susceptibility to cracking, and ensures that welds are suitable for their intended applications.

Defect Detection and Localization: Macroscopic examination allows for the identification and localization of defects in welded joints. Visual inspection of the weld surface and cross-sectional macrostructure helps identify common defects like weld discontinuities, porosity, lack of fusion, incomplete penetration, and undercut. By accurately characterizing these defects, macroscopic examination assists in determining their severity, extent, and potential impact on the performance and service life of welded structures.

Weld Repair and Evaluation: Macroscopic examination provides critical information for assessing the need for weld repair and evaluating the effectiveness of repair procedures. By identifying defects and their root causes, macroscopic examination helps in planning and executing appropriate repair strategies to rectify the observed issues. It also enables post-repair examination to ensure that the repaired weld meets the required quality standards and retains its structural integrity.

Complementary Technique: Macroscopic examination complements other inspection techniques used in welding technology, such as non-destructive testing (NDT) methods and microscopic examination. While NDT methods, like radiography, ultrasonic testing, and magnetic particle inspection, provide valuable information about internal defects and discontinuities, macroscopic examination focuses on the surface features and visible defects. Additionally, microscopic examination, including metallographic analysis and microscopy, provides detailed information about microstructural characteristics. By combining macroscopic examination with other inspection techniques, a comprehensive evaluation of welded joints can be achieved, enhancing the overall assessment of weld quality and integrity.

Macroscopic examination is of paramount importance in welding technology. It enables quality control, integrity assessment, defect detection, and repair evaluation. By utilizing this inspection method, welders, welding inspectors, and engineers can ensure the production of high-quality welds that meet the required standards and specifications. Moreover, macroscopic examination plays a crucial role in enhancing the safety, reliability, and performance of welded structures and components in various industries, including construction, manufacturing, transportation, and energy sectors.

Advantages and Limitations of Macroscopic Examination

Visual Inspection: Macroscopic examination allows for direct visual inspection of weld surfaces and macrostructures. This enables inspectors to identify surface irregularities, weld profile deviations, and visible defects with the naked eye. Visual inspection provides real-time feedback, allowing for immediate assessment and decision-making.

Cost-Effective: Macroscopic examination is a relatively cost-effective inspection method compared to sophisticated non-destructive testing techniques. It requires minimal equipment and can be performed by trained personnel using basic tools such as magnifying lenses, inspection mirrors, and adequate lighting.

Accessibility: Macroscopic examination is accessible and can be applied in various environments, including field inspections and on-site evaluations. It does not require complex setups or extensive preparation, making it suitable for on-the-spot inspections.

Complementary to Other Techniques: Macroscopic examination complements other inspection techniques, such as non-destructive testing (NDT) methods and microscopic examination. It provides a surface-level assessment that complements the internal defect detection capabilities of NDT methods. Combining macroscopic examination with other techniques enhances the overall inspection and evaluation of welded joints.

Quick and Immediate Results: With macroscopic examination, inspectors can obtain instant results during the inspection process. Defects and irregularities are readily visible, allowing for prompt decision-making regarding acceptance or rejection of welds. This immediate feedback enables timely corrective actions and reduces potential delays in the welding process.

Limitations of Macroscopic Examination:

Surface-Only Inspection: Macroscopic examination is limited to the surface features and visible defects of welded joints. It may not provide insights into internal defects or microstructural characteristics that can affect the long-term performance of the weld. To overcome this limitation, complementary techniques such as non-destructive testing and microscopic examination are necessary.

Subjectivity: Visual inspection relies on the experience, expertise, and interpretation skills of the inspector. The assessment of welds can be influenced by individual judgment, which may introduce some level of subjectivity into the evaluation process. To minimize subjectivity, standardized inspection procedures and criteria should be followed.

Limited Detection Sensitivity: Macroscopic examination may have limited sensitivity in detecting certain types of defects, especially those that are small, subsurface, or not visually apparent. Some defects, such as microcracks or localized porosity, may not be readily detectable by macroscopic examination alone.

Surface Condition Influence: The accuracy and effectiveness of macroscopic examination can be affected by the condition of the weld surface. Surface contaminants, coatings, or surface roughness can obscure the visibility of defects and may require surface preparation or cleaning before inspection.

Inability to Determine Internal Integrity: Macroscopic examination cannot assess the internal integrity of welded joints. It does not provide information about the soundness of welds within the material, including the presence of internal cracks, lack of fusion, or incomplete penetration. To evaluate internal integrity, complementary techniques such as radiography, ultrasonic testing, or other non-destructive testing methods are required.

Role of Macroscopic Examination in Weld Quality Control

Defect Detection: Macroscopic examination enables the detection of visible defects in welded joints. It allows inspectors to identify discontinuities, such as cracks, porosity, lack of fusion, incomplete penetration, and undercut, among others. By examining the macrostructure of the weld, inspectors can determine the presence and severity of defects that can compromise the mechanical properties and performance of the joint.

Weld Profile Evaluation: Macroscopic examination helps evaluate the geometry and profile of the weld. It allows inspectors to assess factors such as weld bead shape, size, and consistency, as well as the alignment and penetration of the joint. Deviations from the specified weld profile can indicate issues with welder technique, welding parameters, or joint fit-up, which can impact the overall quality and strength of the weld.

Acceptance or Rejection of Welds: Based on the findings from macroscopic examination, inspectors can make informed decisions regarding the acceptance or rejection of welded joints. Acceptance criteria, as defined by industry standards and specifications, provide guidelines for determining whether a weld meets the required quality and performance standards. Macroscopic examination helps assess welds against these criteria and provides objective evidence for acceptance or rejection.

Weld Repair Evaluation: In cases where defects are identified, macroscopic examination aids in evaluating the effectiveness and integrity of weld repairs. It allows inspectors to assess the quality of repair welds, ensuring that they address the original defects and meet the required standards. By conducting macroscopic examination after repairs, inspectors can verify that the repaired joints are sound and fit for service.

Documentation and Record-Keeping: Macroscopic examination contributes to the documentation and record-keeping process in weld quality control. Inspectors record their observations, findings, and assessments during the examination, providing a permanent record of the weld inspection. This documentation serves as evidence of compliance with quality control procedures and facilitates traceability and accountability in the welding process.

Quality Assurance and Compliance: Macroscopic examination is an integral part of quality assurance programs in welding. It ensures that welded joints meet the specified quality requirements, industry standards, and customer specifications. By performing macroscopic examination, manufacturers can demonstrate compliance with regulatory requirements and enhance customer confidence in the integrity and reliability of their welded products.

Process Improvement: Macroscopic examination also plays a role in process improvement and continuous quality enhancement. By analyzing the findings from macroscopic examination, manufacturers can identify recurring defects, evaluate the root causes, and implement corrective actions. This feedback loop helps optimize welding procedures, improve workmanship, and minimize the occurrence of defects in future welds.

1.4.2. Macroscopic Features of Welded Joints

This subchapter will focus on the different macroscopic features that can be observed in welded joints. We will examine common weld profiles, such as fillet welds, butt welds, and groove welds, and discuss the specific characteristics and variations associated with each type of joint. Through visual inspection and measurement techniques, we can evaluate the geometry, dimensions, and overall appearance of welds, providing insights into the welding process parameters and joint configuration.

The metallographic examination of welded joints includes macroscopic analysis and microscopic analysis. Metallography has been described as both a science and an art. Traditionally, metallography has been the study of the microscopic structure of metals and alloys using optical metallographs, electron microscopes or other surface analysis equipment. More recently, as materials have evolved, metallography has expanded to incorporate materials ranging from electronics to sporting good composites. By analysing a material’s microstructure, its performance and reliability can be better understood. This, metallography is used in materials development, incoming inspection, production and manufacturing control, and for failure analysis; in other words, product reliability.

The metallographic examination is an integral part of the investigation of the quality of welded joints, which involves, among others:

- identification of macro-microscopic defects;
- examination of the metallographic structure in all area’s characteristic of welded joints;

- hardness determination in the base metal, ZIT and weld;
- determination of the proportion of structural constituents and determination of the average grain size;
- classification by size of the grains of the structure;
- determination of other stereometric quantities of the structure (degree of phase dispersion, degree of deformation, degree of orientation, etc.).

Depending on the size of the structural constituents, the examination is carried out by optical or electronic microscopy at magnifications of up to 160,000:1.

Metallographic analysis is used for:

- verification of basic materials;
- assimilation of new welding materials;
- verification of the welding technologies applied to the manufacture of some products;
- checking the opportunity to apply the post-weld heat treatment;
- analysis of the causes of some accidents and others.

From the metallographic examinations, information is expected regarding:

- the existence of fragile constituents in ZIT and welding;
- indication of welding defects both in nature and in geometric size and number;
- finding the hardness peaks of the characteristic zones;
- complete structural characterization of the base metal, the ZIT and the weld;
- the nature and distribution of secondary phase.

Macroscopic examination is a visual inspection method used to examine the surface of a weld or metal object to identify any visible defects or irregularities. It involves the use of the naked eye or a magnifying lens to observe the weld or metal surface and identify any surface discontinuities, such as cracks, porosity, lack of fusion, undercutting, and surface contamination.

In welding technology, macroscopic examination is an essential quality control tool that helps to ensure that welding processes and procedures are carried out correctly and that the final welded product meets the required quality standards. It is used to examine the surface of a weld after it has been completed to check for any visible defects. It is particularly useful in welding technology because it allows for the detection of surface defects that may not be visible to the naked eye. This helps to identify potential problems early on, allowing for corrective action to be taken before the final product is completed. Additionally, macroscopic examination can help to identify the cause of any defects, allowing for the development of more effective welding procedures. Also, it can be used to evaluate the quality of the base metal before welding. By examining the surface of the base metal, any surface irregularities or defects can be identified, which may affect the quality of the weld. This can help to prevent welding defects from occurring and ensure that the final product meets the required quality standards.

In addition, macroscopic examination can be used to evaluate the effectiveness of a welding procedure or technique. By examining the surface of the welded joint, the quality and consistency of the weld can be assessed, and any areas of weakness or defects can be identified. This can help to improve the welding process and develop more effective welding procedures. Furthermore, it can be used for forensic analysis in cases of welding failure or accidents. By examining the surface of the weld or metal object, the cause of the failure or accident can be identified, which can help to prevent similar incidents from occurring in the future.

Overall, macroscopic examination is an important tool in welding technology that helps to ensure the quality and safety of welded products. It allows for the detection and correction of defects early on, ensuring that the final product meets the required quality standards and is safe for use. It allows for the detection and correction of defects early on, improves welding processes and procedures, and helps to prevent welding failures and accidents.

Introduction to Macroscopic Features

Macroscopic features are the observable characteristics of welded joints that can be examined visually without the aid of magnification. These features provide valuable insights into the structure, quality, and performance of the weld. By carefully examining the macroscopic features, inspectors and engineers can assess the integrity of the joint and identify any potential issues or deviations from the desired specifications. The macroscopic features of welded joints encompass various aspects, including weld bead morphology, fusion zone, heat-affected zone (HAZ), and base material. Let's explore these features in more detail:

Weld Bead Morphology: The weld bead morphology refers to the physical appearance and shape of the deposited weld metal. It can vary depending on the welding process, filler material, and joint configuration. Common weld bead morphologies include convex, concave, flat, or irregular profiles. The geometry and uniformity of the weld bead provide insights into the welding technique, heat input, and the degree of penetration achieved during the welding process.

Fusion Zone: The fusion zone is the region where the base metal and the weld metal have undergone complete melting and mixing. It represents the area where the fusion of the base metal and the filler material has occurred. The fusion zone should exhibit proper fusion and penetration, ensuring a sound and strong joint. Examination of the fusion zone helps determine the extent of fusion, the absence of defects, and the overall quality of the weld.

Heat-Affected Zone (HAZ): The HAZ is the portion of the base metal adjacent to the fusion zone that has been subjected to heat during welding. The heat input can cause metallurgical changes, such as grain growth, phase transformations, and changes in mechanical properties. The size and characteristics of the HAZ are influenced by factors such as welding parameters, material composition, and cooling rate. Examining the HAZ allows for the evaluation of potential microstructural changes and the presence of heat-related issues, such as excessive grain growth or hardness variations.

Base Material: The base material, or parent material, refers to the metal being joined by welding. Macroscopic examination of the base material provides a reference for assessing the integrity of the weld. It involves evaluating the base material's composition, grain structure, surface condition, and any pre-existing features or defects that may affect the weld quality. Comparing the macroscopic features of the base material with those of the weld can help identify discontinuities, deviations, or mismatches.

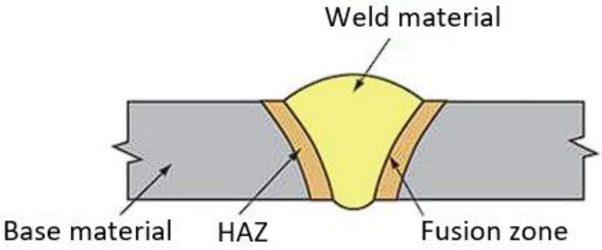


Fig. 1.3.1: Components of welded joints. Source: https://www.kobelco-welding.jp/education-center/medium/medium02.html

Macroscopic examination allows for a comprehensive analysis of these features by visually inspecting the weld joint and its surrounding areas. It provides a macro-level understanding of the weld quality, such as the extent of fusion, proper reinforcement, absence of cracks, and overall conformity to the desired specifications. The interpretation of macroscopic features requires a trained eye and an understanding of the welding process, materials, and applicable standards. Additionally, it is important to consider the specific requirements of the welded component or structure, as different applications may have unique criteria for assessing macroscopic features. This information serves as a basis for evaluating the weld's compliance with specifications, identifying any potential issues or deviations, and making informed decisions regarding the acceptability of the joint.

Weld Profiles and Joint Configurations

In welding technology, the weld profile and joint configuration play a significant role in determining the strength, integrity, and performance of the welded joint. The weld profile refers to the shape and dimensions of the welded cross-section, while the joint configuration describes the arrangement and geometry of the joint to be welded. Understanding these aspects is essential for assessing the quality of the weld and ensuring its suitability for the intended application.

Weld setup and joint Profiles

Fillet Weld: A fillet weld is a common type of weld profile used to join two surfaces that are approximately perpendicular to each other. It forms a triangular-shaped cross-section and is often used for lap joints, T-joints, and corner joints. The size of the fillet weld is specified by its leg length and throat thickness.

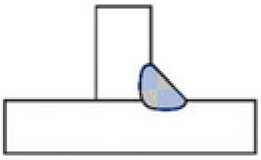


Fig. 1.3.2: Fillet weld

Groove Weld: A groove weld is used to join two plates or sections with a groove or gap between them. The groove can be prepared in various shapes, such as square, V-shaped, U-shaped, J-shaped, or bevelled, to accommodate the welding process and provide sufficient penetration and fusion. Groove welds are commonly used in structural applications and require precise preparation and control to ensure proper weld quality.

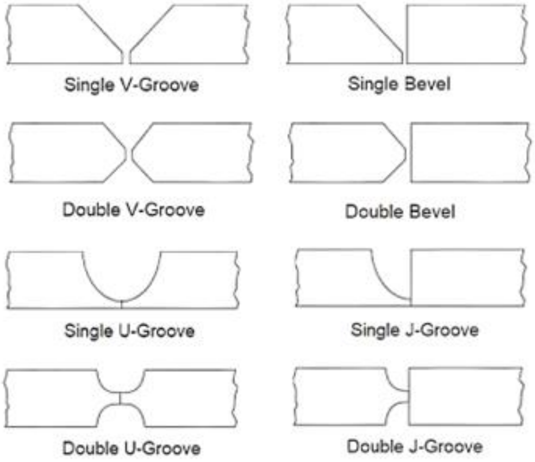


Fig. 1.3.3: Groove weld types

Butt Weld: Butt welding involves joining two adjacent edges of base material, creating a fusion in either full penetration, where the entire joint thickness melds, or partial penetration, where only part fuses. Butt weld profiles can vary from square, V-shaped, U-shaped, to bevelled, depending on joint configuration. In contrast, a butt joint joins two materials end-to-end without overlap, with options for square or bevelled configurations. The choice of welding method is dictated by joint thickness and requirements, and achieving a strong butt joint relies on meticulous fit-up, alignment, and precise welding parameters.

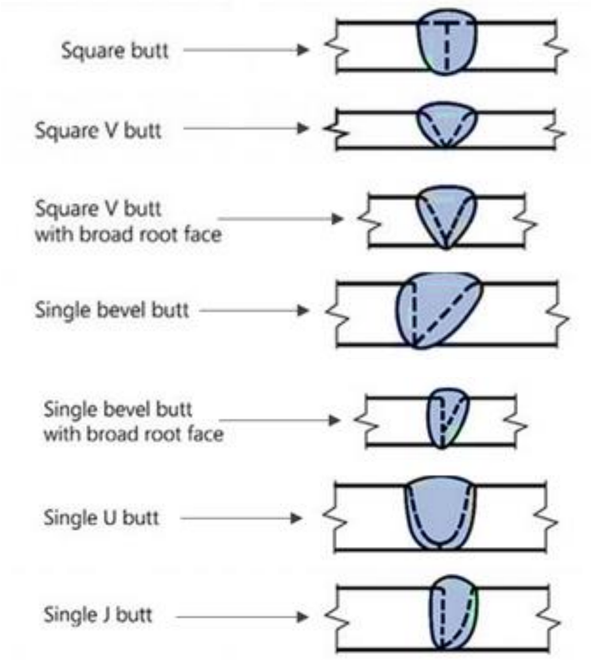


Fig. 1.3.4: Butt weld types

Fig. 1.3.5: Butt joint

Lap Joint: In a lap joint, two overlapping pieces of material are joined together by welding along the overlapping region. It is commonly used for joining thin plates or sheet metal components. The strength of a lap joint depends on the size of the overlap, the weld length, and the welding process used.

Fig. 1.3.6: Lap joint

T-Joint: A T-joint is formed when the end of one piece is welded to the surface of another piece, creating a T-shaped configuration. T-joints are widely used in structural applications and require careful consideration of welding parameters and reinforcement to ensure adequate strength and load transfer.

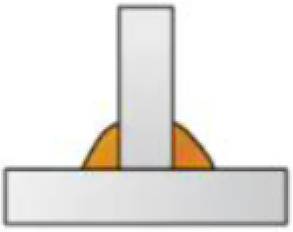


Fig. 1.3.7: T-joint

Corner Joint: A corner joint is created when two pieces of material meet at a 90-degree angle and are welded together. It is commonly used in box structures, frames, and piping systems. Proper weld preparation, penetration, and reinforcement are crucial in corner joints to ensure structural integrity and resistance to stress concentrations.

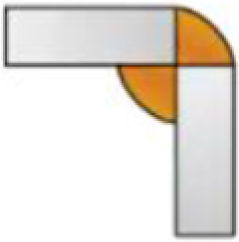


Fig. 1.3.8: Corner joint

Assessing the weld profiles and joint configurations is vital in welding technology as they directly impact the strength, load-bearing capacity, and serviceability of the welded joint. The examination of these features involves visually inspecting the weld cross-section, measuring dimensions, evaluating reinforcement, and verifying the conformity to the specified requirements. It is important to adhere to applicable standards, codes, and specifications when evaluating weld profiles and joint configurations. These standards provide guidelines for acceptable weld profiles, dimensions, reinforcement, and quality criteria based on the specific application and material being welded. By carefully examining weld profiles and joint configurations, welders, inspectors, and engineers can ensure that the weld meets the necessary strength and integrity requirements, minimizing the risk of failure and ensuring the longevity and performance of the welded structure or component.

Weld Size, Shape and Penetration

Weld size, shape, and penetration are crucial parameters in welding that directly affect the strength, integrity, and performance of the welded joint. Evaluating and understanding these aspects is essential for assessing the quality of the weld and ensuring its suitability for the intended application.

Weld Size: Weld size refers to the dimensions of the weld cross-section, including its length, width, and depth. The size of the weld is typically specified in terms of its leg length, throat thickness, or weld width. Proper weld size is important for achieving the required strength and load-carrying capacity of the joint. It is determined by factors such as the joint design, material thickness, welding process, and welding parameters.

Weld Shape: The shape of the weld cross-section can vary depending on the joint configuration, welding process, and welding technique used. Common weld shapes include convex, concave, and flat profiles. The desired shape is determined by the welding procedure and the requirements of the application. The shape of the weld can influence factors such as stress concentration, fatigue resistance, and the ability to withstand applied loads.

Weld Penetration: Weld penetration refers to the depth to which the weld metal extends into the base material. It is a measure of the fusion and bonding between the weld and the base metal. Proper weld penetration is essential for achieving a strong and reliable joint. Insufficient penetration may result in incomplete fusion, lack of joint strength, and reduced load-carrying capacity. Excessive penetration, on the other hand, can lead to excessive heat input, distortion, and potential defects.

The assessment of weld size, shape, and penetration involves visual inspection, measurement, and evaluation techniques. Visual inspection allows for the direct observation of the weld cross-section and identification of any deviations from the specified requirements. Measurement tools such as weld gauges, callipers, and microscope techniques can be used to accurately determine the dimensions and assess the weld quality.

It is important to consult relevant standards, codes, and specifications when evaluating weld size, shape, and penetration; they provide guidelines and acceptance criteria for the desired weld dimensions and penetration depth based on the specific application, material type, and welding process being used. Adhering to these standards ensures that the weld meets the necessary strength and integrity requirements. Proper communication and documentation of the weld size, shape, and penetration findings are essential for quality control and traceability. These records serve as a reference for future inspections, repairs, and maintenance activities. By closely examining and assessing weld size, shape, and penetration, welders, inspectors, and engineers can ensure that the weld joint meets the necessary strength and integrity requirements.

Reinforcement and Fillet Weld Profiles

Reinforcement and fillet weld profiles are important aspects of macroscopic examination in welding technology. They provide valuable information about the quality, strength, and structural integrity of the welded joint. Understanding and evaluating these features is crucial for ensuring the reliability and performance of the weld.

Reinforcement

Reinforcement, also known as weld reinforcement or weld crown, refers to the excess weld metal that extends beyond the original surface of the base material. It is typically observed on the upper side of a weld joint, commonly seen in fillet welds and butt welds. Reinforcement plays a significant role in enhancing the strength and load-carrying capacity of the joint by providing additional material for stress distribution. However, excessive reinforcement can lead to issues such as stress concentration, reduced fatigue life, and potential interference with adjacent components or structures.

Proper assessment of weld reinforcement involves measuring its height or depth, which can be done using visual inspection, weld gauges, or profilometers. The acceptable levels of reinforcement are usually specified in relevant standards, codes, or project specifications, taking into account factors such as the welding process, joint design, and material thickness. Evaluating reinforcement ensures compliance with these requirements and ensures the structural integrity of the welded joint.

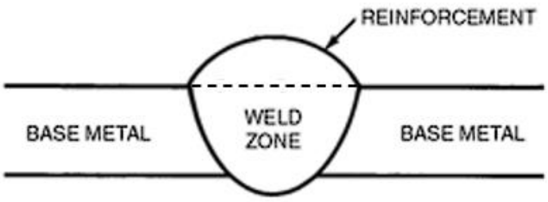


Fig. 1.3.9: Reinforcement in welding

Fillet Weld Profiles

Fillet welds are commonly used in various welding applications, especially for joining components with lap joints, T-joints, or corner joints. The fillet weld profile refers to the shape and dimensions of the weld cross-section in these joints. It plays a vital role in determining the strength, load-carrying capacity, and fatigue resistance of the joint.

The fillet weld profile is typically characterized by its leg length, throat thickness, and toe radius. The leg length refers to the distance from the joint root to the intersection of the fillet weld with the base material. The throat thickness represents the shortest distance between the root and the face of the weld. The toe radius refers to the smoothness and roundness of the transition between the weld and the base material.

Proper evaluation of fillet weld profiles involves visual inspection and accurate measurement using tools such as fillet weld gauges or profile gauges. The measured values are then compared against the specified requirements outlined in applicable standards, codes, or project specifications. Deviations from the required fillet weld profiles may indicate issues such as insufficient weld size, inadequate fusion, or improper welding techniques. Addressing these deviations is necessary to ensure the desired joint strength and integrity. The assessment of reinforcement and fillet weld profiles is essential for evaluating the quality and structural integrity of welded joints. Through visual inspection and accurate measurements, welders, inspectors, and engineers can verify the compliance of welds with relevant standards and specifications. By ensuring proper reinforcement and fillet weld profiles, the strength, reliability, and performance of the welded joint can be upheld, minimizing the risk of failure and promoting overall weld quality.

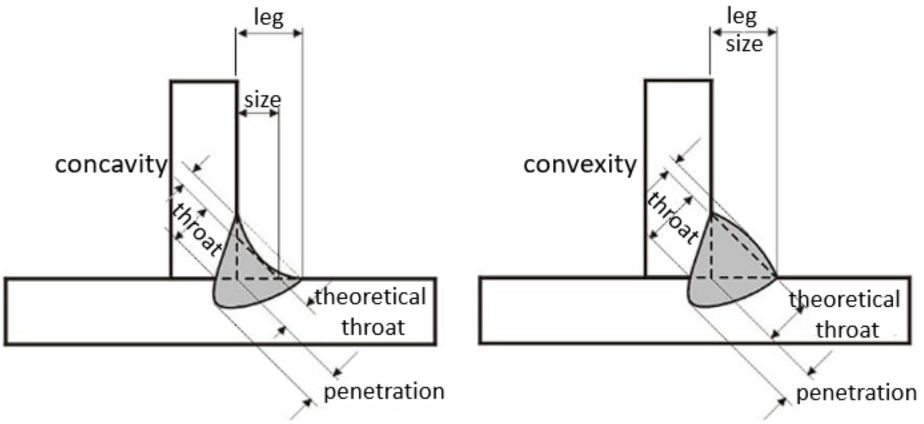


Fig. 1.3.10: Geometrical elements of fillet weld profiles [1.3.25]

Groove Weld Profiles and Bevel Angles

Groove weld profiles and bevel angles are important aspects of macroscopic examination in welding technology. They provide valuable information about the geometry, strength, and integrity of groove weld joints. Understanding and evaluating these features is crucial for ensuring the quality and performance of the weld.

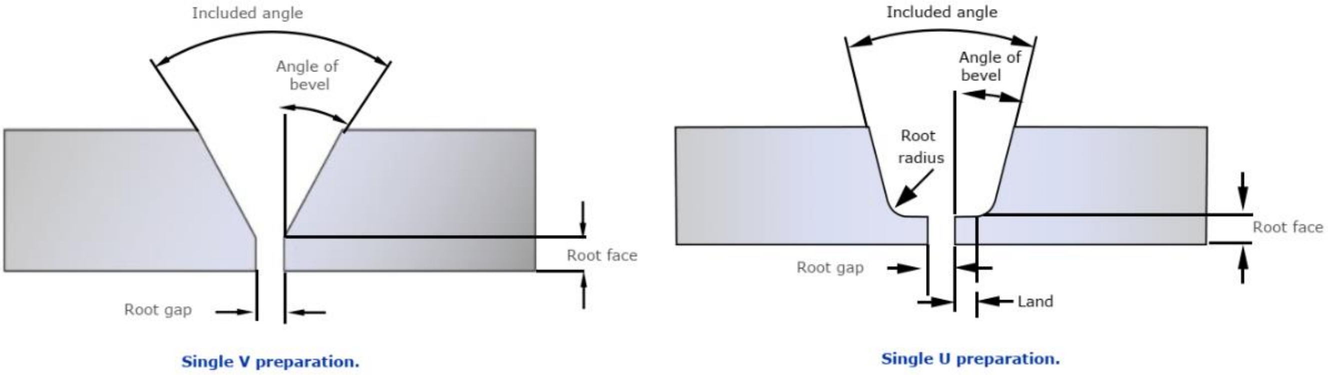


Fig. 1.3.11: Bevel angle representation for V- and U-groove welding profiles [1.3.26]

Groove Weld Profiles

Groove welds are commonly used in various welding applications where two or more components are joined by filling a groove between them. The groove weld profile refers to the shape and dimensions of the weld cross-section in the groove joint. It plays a vital role in determining the strength, load-carrying capacity, and fatigue resistance of the joint.

The groove weld profile is typically characterized by its depth, width, and the angle of the sidewalls. The depth represents the distance from the root of the weld to the surface of the base material. The width refers to the distance between the sidewalls of the groove. The angle of the sidewalls, also known as the included angle, is the angle formed between the sidewalls of the groove.

Proper evaluation of groove weld profiles involves visual inspection and accurate measurement using tools such as depth gauges, width gauges, or profile gauges. The measured values are then compared against the specified requirements outlined in applicable standards, codes, or project specifications. Deviations from the required groove weld profiles may indicate issues such as improper weld penetration, inadequate fusion, or incorrect groove preparation. Addressing these deviations is necessary to ensure the desired joint strength and integrity.

Bevel Angles

Bevel angles are an essential aspect of groove welds, particularly in joints with a V-groove or U-groove configuration. The bevel angle refers to the angle formed between the prepared surfaces of the base material before welding. It determines the shape and size of the groove and influences factors such as weld penetration, weld volume, and ease of welding.

Proper evaluation of bevel angles involves visual inspection and accurate measurement using tools such as protractors or angle gauges. The measured values are then compared against the specified requirements outlined in applicable standards, codes, or project specifications. Deviations from the required bevel angles may indicate issues such as improper groove preparation, incorrect welding parameters, or improper joint fit-up. Rectifying these deviations is necessary to ensure proper weld penetration, fusion, and joint strength.

The assessment of groove weld profiles and bevel angles is crucial for evaluating the quality and integrity of groove weld joints. By visually inspecting and accurately measuring these features, welders, inspectors, and engineers can verify the compliance of welds with relevant standards and specifications. By ensuring proper groove weld profiles and bevel angles, the strength, reliability, and performance of the welded joint can be maintained, minimizing the risk of failure and promoting overall weld quality.

1.4.3. Defects in Welded Joints

Welded joints are susceptible to various types of defects that can compromise their structural integrity and performance. In this subchapter, we will explore the different types of defects that can be identified through macroscopic examination. We will discuss defects such as porosity, lack of fusion, undercutting, excessive reinforcement, cracks, and surface irregularities. Understanding the causes, characteristics, and implications of these defects is essential for evaluating weld quality and ensuring the long-term reliability of welded structures.

Common Weld Defects and their Causes

Understanding the common weld defects and their causes is essential for preventing their occurrence and ensuring high-quality welds. In this technical text, we will explore some of the most common weld defects and the factors that contribute to their formation.

Porosity

Porosity is a prevalent weld defect characterized by the presence of small gas pockets or voids within the weld metal. It can weaken the weld and make it susceptible to cracking. The main causes of porosity include:

- Contaminants on the surface of the base metal or filler material, such as oil, moisture, or rust.
- Improper shielding gas coverage or gas flow during welding, leading to inadequate protection against atmospheric gases.
- Excessive welding parameters, such as high current or travel speed, which can result in the entrapment of gases.

Fig. 1.3.12.1: Porosity shown through 2-dimensional drawing

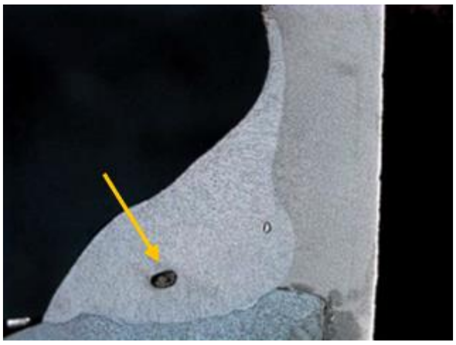


Fig. 1.3.12.2: A macrographic example of porosity [1.3.25]

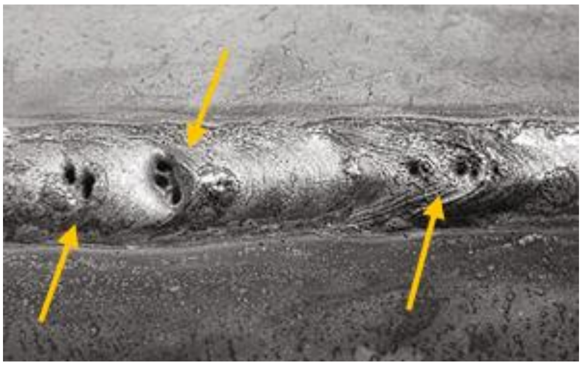


Fig. 1.3.12.3: An example of porosity [1.3.27]

Lack of Fusion

Lack of fusion occurs when there is incomplete bonding between the base metal and the filler material or between adjacent layers of the weld. It can lead to weak joints and reduced load-carrying capacity. Some common causes of lack of fusion include:

- Insufficient heat input, which prevents proper melting and bonding of the base metal and filler material.
- Inadequate penetration, where the weld does not reach the full depth of the joint, resulting in incomplete fusion.
- Poor joint fit-up, with gaps or misalignment preventing proper fusion between the metal surfaces.

Fig. 1.3.13.1: Lack of fusion shown through 2-dimensional drawing

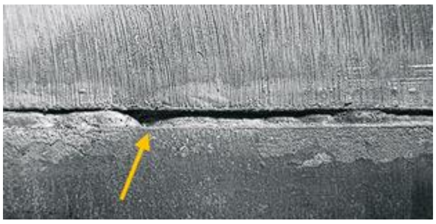


Fig. 1.3.13.2: An example of lack of fusion [1.3.28]

Cracks

Cracks in welded joints are defects that can occur in various forms, such as hot cracks, cold cracks, or stress cracks. Cracks can significantly compromise the structural integrity of the weld and may propagate under applied loads or service conditions. The causes of cracks include:

- High levels of residual stress, which can be caused by improper welding techniques, inadequate preheating, or rapid cooling.
- Inadequate joint design, leading to stress concentrations and localized areas of high stress.
- Incompatible welding consumables or mismatched base metal properties, resulting in metallurgical incompatibility and increased susceptibility to cracks.

Fig. 1.3.14.1: Crack shown through 2-dimensional drawing

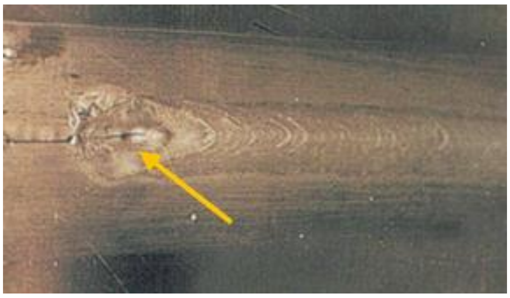


Fig. 1.3.14.2: An example of crack [1.3.25]

Undercuts

Undercuts are a groove-like depression along the weld toe or the sidewalls of the weld. It weakens the joint and can act as a stress concentration point. Undercuts can be caused by:

- Excessive heat input or high welding currents, leading to excessive melting and material removal at the weld edges.
- Incorrect welding technique, such as improper manipulation of the welding arc or excessive weaving.

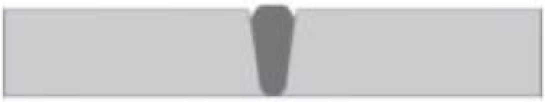


Fig. 1.3.15.1: Undercuts shown through 2-dimensional drawing

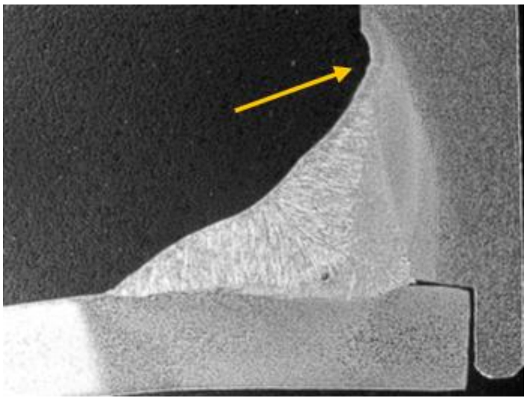


Fig. 1.3.15.2: A macrographic example of an undercut [1.3.25]

Spatter

Spatter refers to the ejection of small molten metal droplets during welding, which can deposit on nearby surfaces. While spatter does not directly affect the integrity of the weld, it can create cosmetic issues and may indicate improper welding conditions or electrode contamination.

Fig. 1.3.16.1: Spatter shown through 2-dimensional drawing

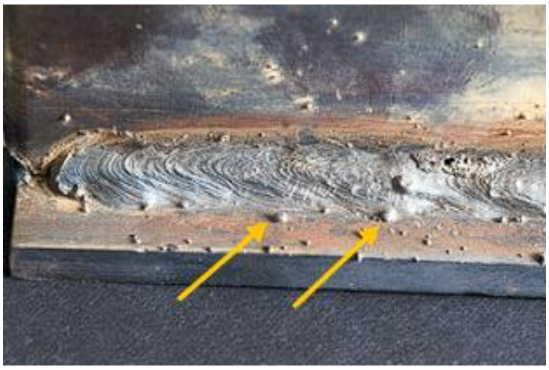


Fig. 1.3.16.2: An example of spatter [1.3.25]

Incomplete Penetration

Incomplete penetration occurs when the weld does not fully penetrate the joint thickness. It can weaken the joint and compromise its load-bearing capacity. Causes of incomplete penetration include:

- Insufficient heat input or welding parameters that do not provide adequate energy to melt and fuse the entire joint thickness.
- Improper joint preparation, such as a joint gap that is too narrow or joint surfaces that are not properly cleaned and prepared.
- Improper welding technique, such as incorrect electrode angle or improper manipulation of the welding arc.

Fig. 1.3.17.1: Incomplete penetration shown through 2-dimensional drawing

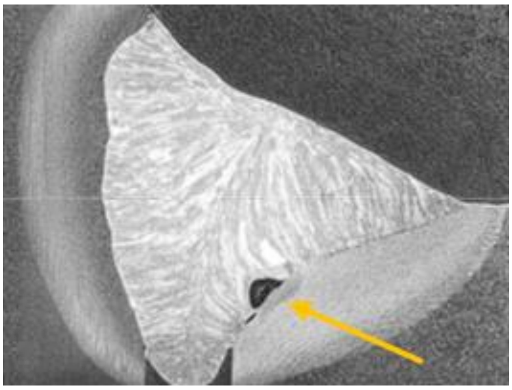


Fig. 1.3.17.2: A macrographic example of incomplete penetration [1.3.25]

Excessive Penetration

Excessive penetration refers to the weld metal penetrating too deeply into the base metal, resulting in excessive fusion. It can lead to distortion, loss of joint strength, and potential defects on the backside of the weld. Excessive penetration is often caused by:

- Excessive heat input or high welding currents, which cause excessive melting and penetration into the base metal.
- Improper joint design or fit-up, such as joints with insufficient root gap or incorrect bevel angle.

Fig. 1.3.18.1: Excessive penetration shown through 2-dimensional drawing

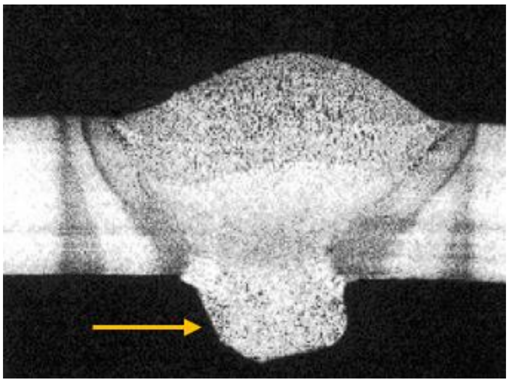


Fig. 1.3.18.2: A macrographic example of excessive penetration [1.3.29]

Inclusions

Inclusions are foreign materials, such as slag or non-metallic particles, that are trapped within the weld metal. They can weaken the weld and act as stress concentration points. Inclusions can be caused by:

- Contamination of the welding consumables, such as the filler material or flux, with dirt, rust, or moisture.
- Improper welding technique, such as excessive manipulation or movement of the electrode, causing slag inclusion.

Fig. 1.3.19.1: Inclusions shown through 2-dimensional drawing

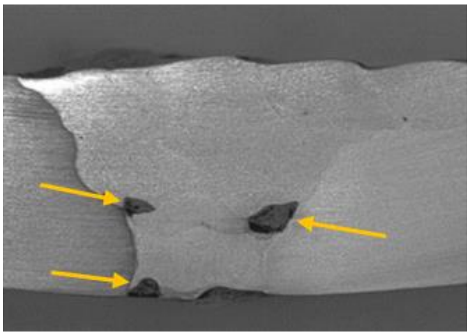


Fig. 1.3.19.2: A macrographic example of slag inclusion [1.3.25]

Distortion

Distortion refers to the deformation or misalignment of the base metal or the welded joint due to the welding process. It can result in dimensional inaccuracies, misfits with adjoining components, or stress concentrations. Distortion is influenced by various factors, including:

- Thermal expansion and contraction during welding, especially when welding thick sections or using high heat inputs.
- Improper joint design, such as inadequate provision for expansion and contraction or inadequate use of fixturing to control distortion.

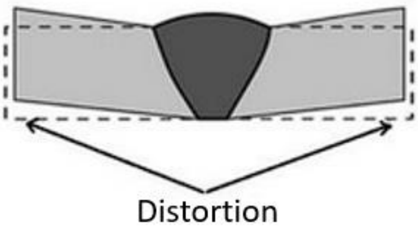


Fig. 1.3.20.1: Distortion shown through 2-dimensional drawing [1.3.30]

Fig. 1.3.20.2: An example of distortion [1.3.31]

Inadequate Reinforcement

Inadequate reinforcement refers to the insufficient build-up of weld metal in fillet welds or inadequate weld bead height in groove welds. It can impact the strength and load-carrying capacity of the joint. Causes of inadequate reinforcement include:

- Insufficient weld size or improper welding technique, resulting in inadequate deposition of weld metal.
- Improper selection of welding parameters or consumables, leading to insufficient fusion or incomplete weld bead formation.

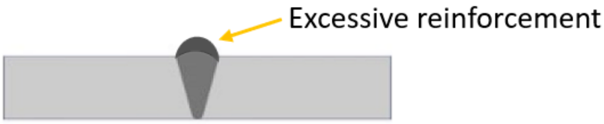


Fig. 1.3.21.1: Excessive reinforcement shown through 2-dimensional drawing

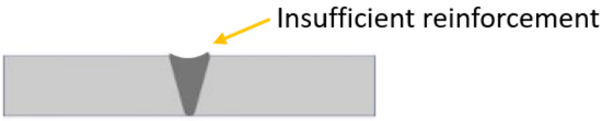


Fig. 1.3.21.2: Insufficient reinforcement shown through 2-dimensional drawing

Porosity and Gas Inclusions

Porosity and gas inclusions are common defects that can occur in welded joints and have a significant impact on their mechanical properties and performance. Understanding the causes, characteristics, and methods of detection of these defects is crucial for ensuring the quality and reliability of welds. In this section, we will delve into the topic of porosity and gas inclusions and their implications in the field of metallography.

Porosity refers to the presence of small voids or cavities within the weld metal or at the weld interface. These voids can vary in size, shape, and distribution. They are typically caused by the entrapment of gases, such as hydrogen, nitrogen, or oxygen, during the solidification of the weld metal. The sources of these gases can include moisture, contaminants on the surfaces being welded, or the decomposition of certain compounds present in the base metal or welding consumables.

Gas inclusions, on the other hand, are discrete gas-filled cavities that are typically larger than the pores associated with porosity. They can be spherical, elongated, or irregular in shape and are often found in the weld metal. Gas inclusions can result from the entrapment of gases during solidification or from the release of dissolved gases as the weld cools. The presence of gas inclusions can have detrimental effects on the mechanical properties of the weld, such as reducing its ductility, increasing its susceptibility to cracking, and compromising its structural integrity.

Causes of Porosity and Gas Inclusions

Moisture and Contaminants: Moisture or contaminants present on the surfaces being welded can lead to the release of gases during welding, resulting in porosity and gas inclusions.

Improper Shielding: Inadequate shielding gases or fluxes can fail to provide sufficient protection against atmospheric gases, leading to their incorporation into the weld metal.

Base Metal and Consumables: Certain compounds present in the base metal or welding consumables may decompose during welding, releasing gases that can cause porosity and gas inclusions.

Welding Parameters: Improper welding parameters, such as excessive heat input or improper arc control, can generate excessive gas levels in the weld pool, leading to porosity.

Characteristics of Porosity and Gas Inclusions

Size and Shape: Porosity can range from small, spherical voids to larger irregular-shaped cavities. Gas inclusions are often larger and can have various shapes, including spherical, elongated, or irregular.

Distribution: Porosity and gas inclusions can be randomly distributed throughout the weld or concentrated in specific areas, depending on the welding conditions and material properties.

Connectivity: Porosity can be interconnected, forming networks within the weld, while gas inclusions are typically discrete and isolated.

Effect on Mechanical Properties: Both porosity and gas inclusions can reduce the weld's mechanical properties, such as strength, ductility, and toughness, making it more prone to failure.

Methods of Detection

Visual Inspection: Surface examination can reveal the presence of visible porosity or gas inclusions, although it may not detect smaller or subsurface defects.

Metallographic Examination: Cross-sectional samples of the weld can be prepared and analysed using optical microscopy or electron microscopy. This allows for precise characterization of porosity and gas inclusions, including their size, shape, distribution, and connectivity.

Non-destructive Testing (NDT): Techniques like ultrasonic testing, radiographic testing, or eddy current testing can detect and locate internal defects, including porosity and gas inclusions, without the need for sample preparation or destruction.

Penetrant Testing: This method involves applying a liquid penetrant to the weld surface, which can seep into and highlight surface-breaking porosity or gas inclusions, aiding their detection.

Accurate detection and characterization of porosity and gas inclusions enable welders and inspectors to assess the quality of the weld and take appropriate corrective actions. By identifying the root causes of these defects, such as adjusting welding parameters, optimizing shielding practices, or improving material cleanliness, steps can be taken to minimize their occurrence and enhance the integrity and reliability of welded joints.

Lack of Fusion and Incomplete Penetration

Lack of fusion and incomplete penetration are common weld defects that can significantly impact the integrity and strength of welded joints. In metallography, the study of these defects plays a crucial role in understanding their causes, characteristics, and methods of detection.

Causes of Lack of Fusion and Incomplete Penetration

Inadequate Heat Input: Insufficient heat input during the welding process can prevent proper fusion between the base metal and the filler metal, leading to a lack of fusion or incomplete penetration.

Improper Welding Technique: Incorrect manipulation of the welding torch or electrode, such as improper travel speed or electrode angle, can result in inadequate fusion and penetration.

Joint Design and Fit-Up: Poor joint design, including excessive gap or poor fit-up between the base metals, can impede proper fusion and penetration.

Weld Pool Control: Inadequate control of the molten weld pool, such as excessive weaving or improper manipulation of the arc, can contribute to insufficient fusion and penetration.

Characteristics of Lack of Fusion and Incomplete Penetration

Lack of Fusion: Lack of fusion occurs when there is a failure to bond the filler metal or base metal adequately. It appears as an unfused area between the base metal and the weld metal.

Incomplete Penetration: Incomplete penetration refers to the failure of the weld metal to penetrate through the entire thickness of the joint. It is characterized by a partial fusion between the base metal and the weld metal, resulting in a lack of continuity in the joint.

Location and Extent: Lack of fusion and incomplete penetration can occur at various locations within the weld, such as the root, sidewalls, or surface. The extent of the defect can range from localized discontinuities to spanning a significant portion of the joint.

Effect on Joint Strength: Lack of fusion and incomplete penetration can significantly reduce the strength and load-bearing capacity of the welded joint. These defects create potential paths for crack initiation and propagation, leading to premature failure under applied stresses.

Methods of Detection

Visual Inspection: Visual examination of the weld surface can often reveal the presence of lack of fusion or incomplete penetration. However, it may not be sufficient for detecting internal defects or for precise characterization.

Metallographic Examination: Cross-sectional analysis of the weld using optical microscopy or electron microscopy allows for detailed inspection of the weld zone. This method helps identify the presence, extent, and location of lack of fusion and incomplete penetration.

Radiographic Testing: Non-destructive testing techniques like X-ray or gamma ray radiography can detect internal defects, including lack of fusion and incomplete penetration, by providing a two-dimensional image of the weld.

Ultrasonic Testing: Ultrasonic examination utilizes sound waves to detect and evaluate internal defects. It can effectively detect lack of fusion and incomplete penetration by analysing the echoes reflected from the defects.

Undercuts and Excessive Penetration

Undercuts and excessive penetration are common weld defects that can occur during the welding process. In the field of metallography, these defects are of great importance as they can affect the structural integrity and mechanical properties of welded joints. Let's explore these defects, including their causes, characteristics, and methods of detection.

Causes that lead to the occurrence of Undercuts

Undercuts are typically caused by excessive heat or improper welding techniques. Common causes for undercuts include:

- High welding current or voltage leading to excessive heat input.
- Inadequate control of welding parameters, such as travel speed and arc length.
- Incorrect torch angle or improper manipulation technique.
- Insufficient or improper filler metal selection.

Characteristics of Undercuts

Undercuts are characterized by a groove or cavity along the edges of the weld joint. The depth and width of the undercut can vary depending on the severity of the defect. Undercuts can weaken the joint by reducing the effective throat thickness, compromising the mechanical strength and fatigue resistance.

Methods of Detection for Undercuts

Metallography offers various methods for detecting and evaluating undercuts in welded joints:

Visual Inspection: Undercuts are often visible to the naked eye. Visual inspection can help identify the presence and extent of the defect.

Macroscopic Examination: By sectioning the weld joint and examining it under a microscope, the depth and extent of undercuts can be measured and evaluated.

Microstructural Analysis: Metallographic techniques such as etching can reveal the microstructural changes associated with undercuts, helping to determine its severity and impact on the weld joint.

Causes of Excessive Penetration

Excessive penetration refers to the weld metal extending beyond the desired depth into the base metal. It can occur due to factors such as:

- High welding current or voltage resulting in excessive heat input.
- Improper joint preparation or fit-up, leading to inadequate control of weld pool shape and size.
- Incorrect welding technique or improper manipulation of the welding torch.
- Inadequate shielding gas or flux coverage.

Characteristics of Excessive Penetration

Excessive penetration is characterized by excessive weld metal penetration into the base metal. This can lead to issues such as lack of fusion, distortion, and reduced joint strength. The excessive penetration can be visually observed by examining the cross-section of the weld joint.

Methods of Detection of Excessive Penetration

Metallography provides several techniques for detecting and evaluating excessive penetration:

Visual Inspection: Excessive penetration can be visually identified by examining the cross-section of the weld joint.

Macroscopic Examination: By preparing cross-sections of the weld joint and observing them under a microscope, the depth and extent of excessive penetration can be assessed.

Microstructural Analysis: Metallographic techniques, including etching, can reveal the penetration depth and assess its impact on the weld joint's microstructure and mechanical properties.

Understanding the causes, characteristics, and detection methods for undercuts and excessive penetration is crucial in the field of metallography. Through visual inspection, macroscopic examination, and microstructural analysis, metallographers can assess the severity of these defects and their impact on the integrity and performance of welded joints. This knowledge allows for effective quality control and helps ensure the production of sound welds in various industries.

Cracks in Welded Joints

Cracks in welded joints present significant challenges in the field of welding technology and represent significant defects that can occur in welded joints, compromising the structural integrity and mechanical properties of the welded components. Understanding the causes, characteristics, detection, and evaluation of these defects is essential for ensuring the reliability and safety of welded structures. By implementing appropriate preventive measures, conducting thorough inspections, and employing effective repair methods, engineers and practitioners can mitigate the risks associated with cracks, leading to improved weld quality, enhanced structural performance, and increased safety in various industries and applications.

Types and Characteristics of Cracks in Welded Joints

- Hot cracks: Liquation cracks, solidification cracks
- Cold cracks: Hydrogen-induced cracks, delayed cracks, inclusion cracks, etc.
- Stress corrosion cracks
- Fatigue cracks
- Brittle fractures
- Ductile fractures
- Micro-cracks and micro-voids

Factors that Influence the Formation of Cracks

- Welding parameters: Heat input, welding speed, preheating
- Material properties: Strength, toughness, susceptibility to hydrogen embrittlement
- Residual stresses

- Welding technique: Joint design, weld bead shape, filler metal selection
- Environmental conditions: Moisture, corrosive media, temperature

Detection and Evaluation of Cracks

Visual inspection: Surface examination, macroscopic assessment

Non-destructive testing (NDT) techniques: Ultrasonic testing, radiography, magnetic particle testing, dye penetrant testing

Microscopic examination: Metallography, optical microscopy, electron microscopy

Fracture surface analysis: Fractography, crack path analysis, determination of fracture modes

Mechanical testing: Tensile testing, Charpy impact testing, fracture toughness testing

Evaluation of the severity of the Crack extent regarding repair

- Crack sizing and measurement
- Determination of crack propagation potential
- Assessment of crack growth rates
- Suitability for service calculations and standards

Mitigation and Repair of Cracks

- Weld repair techniques: Weld overlay, re-welding, post-weld heat treatment
- Stress relief methods: Heat treatment, stress relieving annealing
- Weld modification and reinforcement
- Material selection and process optimization to prevent crack formation

Reporting and Documentation

- Detailed documentation of crack and fracture assessments
- Reporting formats and standards (e.g., ASTM, ISO)
- Communication of assessment findings to relevant stakeholders
- Recommendations for repair, replacement, or further investigation

Surface Irregularities and Spatter

Surface irregularities and spatter are common issues encountered in welding processes that can affect the appearance, functionality, and quality of welded joints. Surface irregularities refer to deviations from the desired smooth and uniform surface, while spatter refers to the expulsion of molten metal droplets during welding. The present technical text provides an in-depth overview of surface irregularities and spatter in welding, including their types, causes, detection, prevention, and mitigation techniques.

Types and Characteristics of Surface Irregularities

- Rough surfaces: Incomplete fusion, insufficient penetration, improper welding parameters

- Undercuts and underfill: Excessive heat input, improper welding technique, inadequate joint preparation
- Overlapping and excessive reinforcement: Incorrect welding technique, improper weld size control
- Surface cracks: Thermal stress, rapid cooling, improper heat treatment
- Surface porosity: Contaminants, improper shielding gas, inadequate welding technique
- Oxidation and temper colours: Insufficient shielding gas coverage, exposure to air, improper cleaning

Causes of Surface Irregularities and Spatter

- Improper welding parameters: Welding current, voltage, travel speed
- Incorrect electrode or filler wire selection
- Poor joint fit-up and preparation
- Contaminants on the workpiece or welding consumables
- Inadequate shielding gas coverage or gas flow rate
- Presence of moisture or oil on the workpiece surface
- Insufficient cleaning of the workpiece before welding
- Welding technique and operator skill

Detection and Evaluation of Surface Irregularities

Visual inspection: Surface examination for irregularities, spatter, and defects

Optical microscopy: Examination of surface features and morphology

Non-destructive testing (NDT) techniques: Ultrasonic testing, radiography, magnetic particle testing

Prevention and Control of Surface Irregularities and Spatter

- Proper welding parameters and technique optimization
- Adequate joint preparation and fit-up
- Effective cleaning and removal of contaminants
- Selection of appropriate shielding gas and flow rate
- Use of anti-spatter agents and coatings
- Maintenance and calibration of welding equipment
- Training and qualification of welders

Mitigation and Repair of Surface Irregularities

- Grinding and surface refinishing
- Weld profile modification and blending
- Removal of excess reinforcement and undercuts
- Use of appropriate filler materials for repair

Reporting and Documentation

- Detailed documentation of surface irregularities and spatter
- Reporting formats and standards (e.g., ISO, AWS)
- Communication of assessment findings to relevant stakeholders
- Recommendations for prevention, mitigation, and repair

Surface irregularities and spatter in welding can significantly impact the quality and performance of welded joints. Implementing appropriate welding parameters, optimizing joint preparation, maintaining cleanliness, and utilizing proper shielding gas and anti-spatter measures are key in achieving smooth, defect-free surfaces. Through continuous improvement, monitoring, and adherence to established standards, professionals can ensure high-quality welds with superior aesthetics, functionality, and reliability.

1.4.4. Weld Quality Assessment

Assessing the quality of a weld is a critical aspect of welding technology. In this subchapter, we will discuss how macroscopic examination is used to evaluate the overall quality of a weld. We will explore the acceptance criteria, including the size, location, and severity of defects, as well as the visual appearance and alignment of the weld. By following established guidelines and standards, we can make objective assessments and determine the acceptance or rejection of welded joints, for the intended applications, based on macroscopic examination findings.

Acceptance Criteria for Weld Quality

Acceptance criteria for weld quality serve as guidelines and standards to assess the suitability and integrity of welded joints. These criteria define the allowable limits for various weld characteristics and ensure that the weld meets the specified requirements and performance expectations. Establishing clear and consistent acceptance criteria is crucial for quality control, compliance with industry standards, and ensuring the safety and reliability of welded structures. This section provides an overview of the key factors and parameters considered in determining acceptance criteria for weld quality.

Welding Codes and Standards

- International standards: ISO 3834, ISO 15614, ISO 9606, etc.
- National standards: AWS D1.1, ASME Section IX, EN ISO 15614, etc.
- Industry-specific standards: API 1104, ASME B31.3, etc.

Weld Attributes and Acceptance Levels

- Weld dimensions: Size, shape, reinforcement, penetration, and fillet weld profiles
- Discontinuities: Porosity, cracks, lack of fusion, incomplete penetration, undercuts, etc.
- Visual appearance: Surface irregularities, spatter, discoloration, and contamination
- Mechanical properties: Tensile strength, impact toughness, hardness, etc.

Non-Destructive Testing (NDT) Techniques

- Ultrasonic testing (UT): Detection of internal flaws, discontinuities, and bonding defects
- Radiographic testing (RT): Evaluation of internal and surface defects using X-rays or gamma rays
- Magnetic particle testing (MT): Detection of surface and near-surface defects in ferromagnetic materials
- Liquid penetrant testing (PT): Identification of surface-breaking defects and cracks
- Visual inspection: Examination of weld appearance, dimensions, and surface conditions

Acceptance Levels and Criteria

- Acceptance levels: Acceptable, repairable, or rejectable based on the severity and impact of defects
- Acceptance criteria: Specific limits and thresholds for each weld attribute or parameter
- Defect classification: Categorization of defects based on size, location, and type

Evaluation and Interpretation

- Qualified personnel: Certified inspectors or NDT technicians for accurate evaluation
- Documentation and record-keeping: Proper documentation of inspection results, procedures, and acceptance decisions
- Compliance with standards: Verification of weld quality against applicable codes and specifications

Weld Repair and Post-Weld Examination

- Repair procedures: Defined methods and requirements for addressing non-conformities
- Post-repair examination: Validation of repaired welds to ensure compliance with acceptance criteria

In the European Union (EU), several standards are applicable to the detection and evaluation of lack of fusion and incomplete penetration in welded joints. These standards provide guidelines and requirements for testing methods, acceptance criteria, and quality control procedures.

EN ISO 5817 - Welding - Fusion-welded joints in steel, nickel, titanium, and their alloys (Quality levels for imperfections): This standard specifies quality levels for lack of fusion and incomplete penetration, among other imperfections, in fusion-welded joints. It provides acceptance criteria based on visual inspection and radiographic examination.

EN ISO 17636-2 - Non-destructive testing of welds - Radiographic testing - Part 2: X- and gamma-ray techniques with film: This standard specifies the requirements for radiographic testing of welds using X-ray and gamma-ray techniques with film. It covers the detection and evaluation of lack of fusion and incomplete penetration, as well as other weld defects.

EN ISO 17636-3 - Non-destructive testing of welds - Radiographic testing - Part 3: Digital techniques: This standard provides requirements for radiographic testing of welds using digital techniques. It includes the detection and evaluation of lack of fusion and incomplete penetration, along with other weld defects.

EN ISO 16810 - Non-destructive testing - Ultrasonic testing - Time-of-flight diffraction technique as a method for detection and sizing of discontinuities: This standard specifies the time-of-flight diffraction (TOFD) technique for the detection and sizing of weld discontinuities, including lack of fusion and incomplete penetration.

EN ISO 23278 - Non-destructive testing of welds - Magnetic particle testing: This standard outlines the requirements for using magnetic particle testing to detect surface and near-surface defects in welds. It provides criteria for evaluating the indications and determining their acceptability based on size, shape, and location.

EN ISO 3452 - Non-destructive testing - Penetrant testing: This standard defines the requirements for conducting liquid penetrant testing to detect surface-breaking defects in welds. It specifies the acceptance criteria for evaluating the indications based on their size, shape, and visibility.

EN ISO 10042 - Welding - Arc-welded joints in aluminium and its alloys - Quality levels for imperfections: This standard specifically addresses the quality levels for imperfections in arc-welded joints in aluminium and its alloys. It defines the acceptable limits and sizes of imperfections to ensure the weld integrity.

EN ISO 15614-1 - Specification and qualification of welding procedures for metallic materials - Welding procedure test - Part 1: Arc and gas welding of steels and arc welding of nickel and nickel alloys: This standard focuses on the qualification of welding procedures for metallic materials. It provides guidelines for conducting welding procedure tests, including the acceptance criteria for weld quality. **Part 2:** Arc welding of aluminium and its alloys: This standard specifies how a preliminary welding procedure specification is qualified by welding procedure tests.

EN ISO 14731 - Welding coordination - Tasks and responsibilities: This standard defines the tasks and responsibilities of welding coordination personnel involved in ensuring weld quality. It outlines the requirements for managing welding processes, including inspection and acceptance criteria.

EN ISO 3834 - Quality requirements for fusion welding of metallic materials: This standard specifies quality requirements for the fusion welding of metallic materials. It covers aspects such as welding procedures, personnel qualifications, inspection, and acceptance criteria to ensure the overall quality of welded joints.

Visual Inspection and Acceptance Standards

Visual inspection plays a crucial role in assessing the quality of welds, and there are several European standards that provide guidelines for visual inspection and acceptance criteria.

EU standards related to visual inspection and acceptance standards:

EN ISO 17637 - Non-destructive testing of welds - Visual testing of fusion-welded joints

This standard specifies the requirements for visual testing of fusion-welded joints. It outlines the procedures, equipment, and acceptance criteria for evaluating the surface conditions, dimensions, and overall appearance of welds using visual inspection techniques.

EN ISO 5817 - Welding - Fusion-welded joints in steel, nickel, titanium, and their alloys (Quality levels for imperfections)

As mentioned earlier, this standard provides quality levels for imperfections in fusion-welded joints. It includes visual acceptance criteria for various types of imperfections such as porosity, undercut, lack of fusion, etc.

EN ISO 13920 - Welding - General tolerances for welded constructions - Dimensions for lengths and angles - Shape and position

This standard specifies general tolerances for welded constructions and provides guidelines for dimensional and geometrical deviations in welded structures. It helps define acceptance criteria based on allowable deviations from specified dimensions and angles.

EN 1290 - Welding - Determination of Ferrite Number (FN) in austenitic and duplex ferritic-austenitic Cr-Ni stainless steel weld metals

Although this standard is specific to ferrite determination in stainless steel welds, it highlights the visual acceptance criteria for determining the ferrite content in welds using various methods such as macroscopic examination and etching techniques.

EN ISO 17635 - Non-destructive testing of welds - General rules for metallic materials

This standard establishes general rules for non-destructive testing of welds in metallic materials. It includes provisions for visual inspection, as well as other testing methods such as radiographic testing, ultrasonic testing, and magnetic particle testing.

EN ISO 10275 - Welding - Joint preparation - Part 3: Metal inert gas welding and tungsten inert gas welding of copper and its alloys

While specific to copper and its alloys, this standard provides guidelines for joint preparation in metal inert gas (MIG) welding and tungsten inert gas (TIG) welding. It includes visual acceptance criteria for weld quality, including surface conditions and penetration.

EN ISO 10042 - Welding - Arc-welded joints in aluminium and its alloys - Quality levels for imperfections

This standard specifies quality levels for imperfections in arc-welded joints in aluminium and its alloys. It defines visual acceptance criteria for various types of imperfections such as porosity, lack of fusion, cracks, and undercut.

Dimensional Requirements and Alignment

Weld quality assessment for dimensional requirements and alignment involves evaluating the conformity of welded joints to specified dimensional tolerances and alignment criteria. The assessment aims to ensure that the welded components meet the intended design dimensions, fitment, and alignment, thereby ensuring the structural integrity and functionality of the final product.

In the field of metallography, several techniques and methods can be employed to assess weld quality concerning dimensional requirements and alignment. These include:

Visual Inspection: Visual inspection is the most basic and commonly used method to assess dimensional requirements and alignment in welded joints. It involves visually examining the weld joint for any deviations from the specified dimensions, such as excessive weld reinforcement, underfill, or excessive root penetration. Visual inspection also helps in detecting misalignments, angular deviations, or distortions that may affect the overall fit and function of the welded component.

Measurement and Metrology: Precision measurement tools, such as callipers, micrometres, height gauges, and laser alignment systems, can be employed to measure critical dimensions and alignments of welded joints. These measurements are compared against the specified dimensional tolerances provided in engineering drawings or applicable standards to assess the weld quality. Measurement techniques should ensure accuracy and repeatability to obtain reliable results.

Coordinate Measurement Machines (CMM): CMMs are advanced metrology devices that enable precise and automated measurements of complex geometries. CMMs use probes to capture the three-dimensional coordinates of specific features or reference points on the welded joint. The acquired data is compared against the CAD model or specified dimensional requirements to evaluate the weld quality in terms of dimensional conformity and alignment.

Non-Destructive Testing (NDT) Techniques: NDT methods, such as ultrasonic testing, magnetic particle testing, or laser scanning, can also be utilized to assess dimensional requirements and alignment in welded joints. These techniques can provide detailed information about internal and external features, identify any deviations from the specified dimensions, and evaluate alignment characteristics without causing damage to the weld.

Statistical Analysis: Statistical analysis techniques, such as process capability studies, can be employed to assess the overall weld quality concerning dimensional requirements and alignment. These analyses involve collecting data from a representative sample of welded joints, analysing the data statistically, and determining the process capability to meet the specified dimensional tolerances and alignment criteria.

European standards, such as EN ISO 13920, EN 1090-2, and EN ISO 5817, provide acceptance criteria and guidelines for assessing weld quality in terms of dimensional requirements and alignment. These standards define tolerance limits, inspection methods, and measurement techniques to ensure compliance with the specified dimensional requirements. Weld quality assessment in terms of dimensional requirements and alignment is essential to guarantee the functionality, performance, and structural integrity of welded components and to meet industry standards and customer expectations.

Evaluating Defect Severity and Classification

Weld quality assessment for evaluating defect severity and classification involves determining the significance and impact of defects on the structural integrity, performance, and serviceability of welded joints. This assessment aims to categorize defects based on their severity levels, prioritize corrective actions, and ensure compliance with applicable standards and specifications.

To assess the severity and classification of weld defects, various techniques and methods can be employed in the field of metallography. These include:

Visual Inspection: Visual inspection is the initial step in evaluating defect severity and classification. It involves visually examining the weld joint for the presence of defects such as cracks, porosity, lack of fusion, undercutting, and spatter. The inspector assesses the size, shape, location, and distribution of the defects to determine their severity and classification.

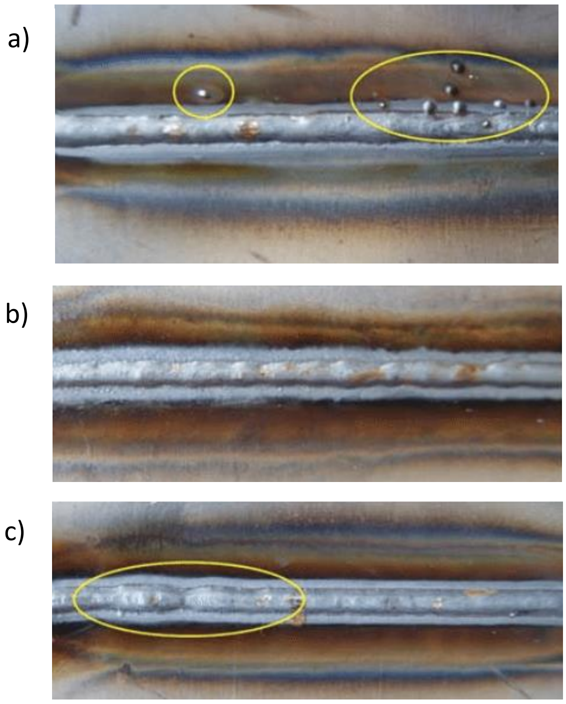


Fig 1.3.22: Imperfections found during visual inspections: local spatters (a); uneven heat-affected zone (b); local risers and incompletely filled groove of weld face (c) [1.3.32]

Non-Destructive Testing (NDT) Techniques: NDT methods, such as ultrasonic testing, radiographic testing, magnetic particle testing, and dye penetrant testing, are commonly used to assess defect severity and classification. These techniques provide detailed information about the size, shape, location, and extent of defects within the welded joint. NDT methods can help identify critical defects that may compromise the integrity of the weld and classify them based on their severity levels.

Table 1.3.1: Several techniques used in non-destructive testing:

Internal defect	RT: Radiographic Testing
	UT: Ultrasonic Testing
	VI: Visual Inspection
Surface defect ET	MT: Magnetic Particle Testing
Surface opening defect	PT: Penetrant Testing

Macroscopic Examination: Macroscopic examination involves the use of macrographs and cross-sectioning of the weld joint to assess defect severity and classification. Macrographs provide a detailed visual representation of the defects, allowing for better evaluation of their dimensions, distribution, and relationship to the surrounding weld structure. Cross-sectioning involves cutting the weld joint to expose the internal features and defects, enabling a thorough examination of their nature, size, and position.

Acceptance Criteria and Standards: Weld quality assessment for defect severity and classification relies on established acceptance criteria and standards. European standards, such as EN ISO 5817 and EN ISO 10042, provide guidelines and specifications for evaluating the severity levels of various weld defects. These standards define acceptance levels and provide criteria for classifying defects based on their size, length, depth, and other parameters.

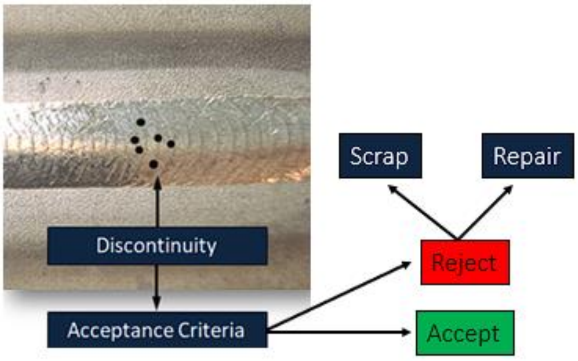


Fig. 1.3.23: Critical Weld Assessment: Applying Acceptance Criteria for Quality Evaluation [1.3.33]

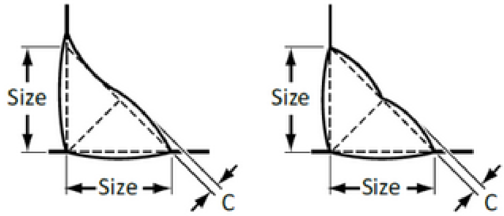


Fig. 1.3.24.1: Acceptable fillet weld profiles [1.3.33]

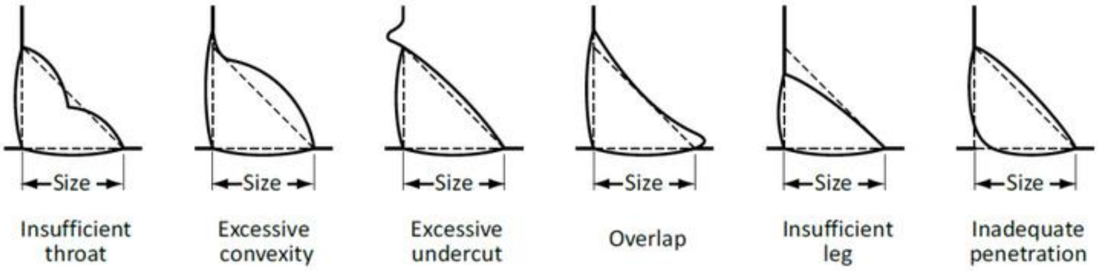


Fig. 1.3.24.2: Unacceptable fillet weld profiles [1.3.33]

Expert Judgment: In some cases, the expertise and experience of qualified personnel play a crucial role in assessing defect severity and classification. Skilled metallurgists, welding inspectors, and engineers with a deep understanding of welding processes and defect characteristics can provide valuable insights and judgments in determining the severity of defects and their impact on weld quality.

During the weld quality assessment, defects are typically classified into different categories, such as critical, major, and minor defects, based on predefined acceptance criteria. The classification helps prioritize corrective actions, determine the need for repair or rework, and ensure compliance with relevant standards and specifications. It is important to note that the severity and classification of defects may vary depending on the specific application, weld type, material, and industry requirements. Therefore, weld quality assessment should consider the specific context and criteria defined by applicable standards and specifications. By evaluating defect severity and classification, weld quality assessment helps in identifying critical defects that may compromise the integrity of the welded joint and ensures that appropriate corrective measures are implemented. This assessment plays a crucial role in maintaining the structural integrity, performance, and safety of welded components in various industries and applications.

Non-Destructive Testing for Weld Quality Assessment

Weld quality assessment through non-destructive testing (NDT) techniques is a crucial aspect of ensuring the integrity and reliability of welded joints. NDT methods are employed to evaluate the quality of welds without causing any damage to the welded components, allowing for comprehensive inspection and assessment of weld defects.

Several NDT techniques are commonly used in weld quality assessment, including:

Ultrasonic Testing (UT): UT utilizes high-frequency sound waves to detect and evaluate internal and surface defects in welded joints. Ultrasonic waves are transmitted into the weld, and the reflected waves are analyzed to identify indications such as lack of fusion, porosity, cracks, and other discontinuities. The size, location, and nature of these indications are assessed to determine their impact on weld quality.

Fig. 1.3.25: Ultrasonic testing of weld [1.3.34]

Radiographic Testing (RT): RT involves the use of X-rays or gamma rays to create an image of the weld and detect internal defects. Radiographic images provide a detailed view of weld discontinuities, including cracks, lack of fusion, porosity, and inclusions. The size, shape, and density of these indications are examined to assess their severity and impact on weld quality.

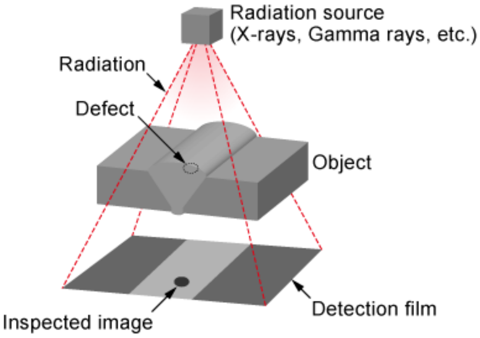


Fig. 1.3.26: Radiographic Testing [1.3.35]

Magnetic Particle Testing (MT): MT is primarily used to detect surface-breaking defects in ferromagnetic materials. It involves magnetizing the weld area and applying magnetic particles, which will gather at areas with magnetic flux leakage caused by surface discontinuities such as cracks and lack of fusion. The indications are examined visually to determine their severity and influence on weld quality.

Fig. 1.3.27: Magnetic Testing [1.3.36]

Liquid Penetrant Testing (PT): PT is employed to detect surface-breaking defects in non-porous materials. A liquid penetrant is applied to the surface of the weld, and after a specified dwell time, excess penetrant is removed, and a developer is applied. The penetrant that seeps into surface defects becomes visible under proper lighting conditions, allowing for the identification and assessment of defects such as cracks and porosity.

Fig. 1.3.28: Liquid Penetrant Testing [1.3.37]

Visual Inspection (VI): Although considered a separate inspection method, visual inspection is an essential component of weld quality assessment. It involves a thorough visual examination of the weld joint to identify surface defects, such as cracks, undercutting, spatter, and incomplete fusion. Visual inspection complements other NDT techniques and provides a qualitative assessment of weld quality.

Fig. 1.3.29: Visual Inspection [1.3.38]

During NDT for weld quality assessment, indications and defects are evaluated based on predefined acceptance criteria provided by relevant standards, such as EN ISO 17636 and EN ISO 9712. These standards define the allowable sizes, lengths, locations, and types of indications that are acceptable in welds. The findings from NDT are recorded and documented, allowing for further analysis and decision-making regarding the acceptance or rejection of the weld. NDT techniques provide valuable insights into the internal and surface conditions of welded joints, allowing for the detection, evaluation, and characterization of weld defects. By utilizing these techniques, weld quality assessment can identify critical defects, assess their severity, and determine the appropriate actions needed to maintain the integrity and reliability of the welds. It is important to note that NDT techniques should be performed by qualified personnel who have received proper training and certification in accordance with relevant standards, such as EN ISO 9712. This ensures the accuracy and reliability of the assessment and enhances the overall quality control of welded components in various industries.

1.4.5. Weld Repair and Post-Weld Examination

Weld repairs are often necessary to rectify defects or improve the integrity of a joint. This subchapter will focus on the role of macroscopic examination in assessing the effectiveness of weld repairs. We will discuss the techniques and procedures used to evaluate the repaired joint, ensuring that it meets the required standards and specifications. Additionally, we will explore the importance of post-weld examination to verify the integrity and reliability of the repaired joint.

Macroscopic examination is a fundamental technique in welding technology that allows for the comprehensive evaluation of welded joints. By examining macroscopic features, identifying defects, assessing weld quality, and ensuring the effectiveness of repairs, we can enhance the performance, safety, and reliability of welded structures and components. So, let's dive into the fascinating world of macroscopic examination and its application in welding technology!

Macroscopic examination specifications for welding technology may vary depending on the industry and application. However, some common specifications include:

1. Magnification: The level of magnification used during macroscopic examination should be appropriate for the size of the defect being examined. Typically, a magnification of 5x to 10x is used for general weld examination, while a magnification of up to 50x may be used for more detailed examination. Additionally, it is important to note that the magnification should not be so high that the field of view becomes too small, making it difficult to identify the location or extent of the defect. The use of higher magnifications may also increase the likelihood of false positives, where surface irregularities that are not defects are identified as such. Therefore, the appropriate level of magnification should be determined based on the size and type of defects being examined, as well as the surface finish and texture of the material being inspected. This can help to ensure that defects are accurately identified, and that the inspection process is efficient and effective

2. Lighting: Adequate lighting should be used during macroscopic examination to ensure that all surface defects are visible. The lighting should be directed at the surface being examined at an angle that provides optimal visibility. The lighting used during macroscopic examination should be bright enough to illuminate all surface defects and minimize shadows or glare that may obscure the view. The direction of the lighting should also be carefully considered to ensure that the surface being examined is illuminated at an angle that provides optimal visibility. This may involve using multiple light sources or adjusting the angle of the lighting to minimize glare and shadows.

In addition, the colour temperature of the lighting should also be considered. Different types of lighting, such as fluorescent or LED, may produce different colour temperatures, which can affect the appearance of surface defects. The appropriate colour temperature should be selected based on the material being inspected and the type of defects being examined.

Overall, adequate lighting is essential for macroscopic examination to ensure that all surface defects are visible and accurately identified. The lighting should be carefully selected and positioned to provide optimal visibility and minimize any glare or shadows that may obscure the view

3. Surface preparation: The surface being examined should be cleaned and prepared according to industry standards. Any surface contaminants, such as oil, grease, or rust, should be removed to ensure that all surface defects are visible. Sampling of samples

For welded joints, it is necessary to perform examinations on all characteristic areas (BM, HAZ, WELD), which is why the metallographic samples include the entire cross-section of the joint.

Depending on the needs of the research, the samples are taken differently. Especially for damage analyses, the sampling is carried out taking into account the working hypotheses that must be verified and the condition and nature of the material under investigation, in many cases reference samples from unaffected areas or from similar material not subject to operating conditions are examined in parallel.

When checking the welding technologies, the basic materials and the assimilation of the welding materials, the samples are taken in such a way that they are representative of the entire investigated batch. Failure to ensure this condition leads to wrong interpretations with serious consequence.

The sampling and preparation of metallographic samples is carried out according to STAS 4203.

Obtaining the flat surface is achieved by filing, milling or polishing. Grinding is carried out with the help of metallographic papers that show an increasing fineness of the abrasive particles formed by silicon carbide. According to the grain of the metallographic papers used, grinding can be:

- coarse when using metallographic paper with coarse grain (80, 100, 180, 240, 320);
- fine when using metallographic paper with fine grain (400, 500, 600, 800);
- very fine when using very fine metallographic papers (1000, 1200, 1500, 2000, 2500).

Grinding can be performed manually or mechanically. In manual sanding (recommended for large, welded samples) the paper is placed on a glass plate mounted on a wooden support. The metallographic sample being lightly pressed always moves in one direction, performing a back-and-forth movement. At each change of paper, it is necessary to rotate the sample by 90° so that the new wrinkles form a right angle with the previous ones. Sanding begins with coarse-grained paper (300...400) and ends with fine-grained (800...900) and extra-fine (1000).

In the case of mechanical grinding, horizontal or vertical grinding machines are used where the metallographic paper is fixed on rotating discs. During the grinding operation, the following rules must be observed:

• do not switch to another metallographic paper until after it has been ascertained that all the scratches from the previous sanding have disappeared;

• the metallographic paper is cleaned of metal or abrasive dust every time it is used (if dry sanding is done);

• during grinding, the part must not heat up;

• the same papers will not be used for ferrous and non-ferrous alloys;

• at the end of the grinding operation, the sample is washed to remove all inclusions that adhered during grinding.

Polishing of the welded samples is performed mechanically with the polishing machine equipped with a rotating disk on which a merino wool felt is fixed. During polishing, a suspension of Al oxide, Mg oxide, and diamond dust of different granulations is deposited on the felt. After polishing, the surface of the sample must have a mirror appearance (without wrinkles). The polished sample is washed with water, degreased with alcohol and then dried by dabbing with cotton wool or filter paper, drying in a stream of warm air is recommended.

Metallographic attack the purpose of the operation is to highlight the crystalline structure. The polished surface is attacked with appropriate reagents that selectively dissolve or colour the constituents present, making them distinguishable from one another. The attack is carried out either by immersion or by dabbing the surface of the sample with a piece of cotton soaked in reagents. If the sample is not sufficiently attacked, the operation is repeated. In general, to obtain strong counters, reagents will be used as diluted as possible, so the attack will be made with longer holding times. Also, for research at a larger size of the microscope, a weaker attack will be performed, for the observation in better conditions of the details of the structure, and vice versa in the case of research at a small size. Practically, the sample is considered attacked when the prepared surface has lost its metallic lustre in all the characteristic areas, becoming matte and the areas can be observed. Too intense an attack distorts the structure.

After the metallographic attack, the sample is washed with water, then with alcohol and dried by pressing on filter paper or under a stream of warm air. The choice of the reagent for the metallographic attack is made according to the standard SR CEN ISO/TR 16060.

4. Acceptance criteria: Acceptance criteria for surface defects should be defined based on industry standards and the requirements of the application. The criteria should specify the type, size, and location of defects that are acceptable and those that are not. Acceptance criteria for surface defects should be established based on industry standards and the requirements of the application. This involves specifying the type, size, and location of defects that are acceptable and those that are not. The acceptance criteria may vary depending on the material being inspected, the intended use of the product, and the importance of the welded joint in the overall structure.

For example, in the aerospace industry, the acceptance criteria for surface defects in welded joints may be much stricter than in other industries due to the critical role that aerospace components play in ensuring safety. In this case, the acceptance criteria may specify that any surface defects, regardless of size or location, are not acceptable.

Similarly, in the automotive industry, the acceptance criteria for surface defects may be less strict, but still require that defects be within certain size and location limits to ensure that the welded joint meets the required strength and durability standards.

Establishing clear acceptance criteria is important for ensuring that welded products meet the required quality standards and are safe for their intended use. This can help to prevent defects and failures, reduce the risk of accidents, and ensure that products meet customer expectations. Acceptance levels for imperfections in welded joints are carried out according to SR EN ISO 5817. The classification of geometric imperfections in welded joints of metallic materials is carried out according to SR EN ISO 6520-1.

5. Reporting: A detailed report should be provided that includes the results of the macroscopic examination, including any defects found and their location. The report should also include recommendations for corrective action, if necessary. After macroscopic examination is performed, a detailed report should be generated that includes the results of the examination, any defects found, and their location on the welded joint. The report should also include recommendations for corrective action, if necessary, based on the acceptance criteria and industry standards.

The report should be clear and concise, providing a summary of the inspection results, including any defects found, their size and location. It should also include photographs or diagrams that illustrate the location and extent of any defects.

If any defects are identified that do not meet the acceptance criteria, the report should include recommendations for corrective action, such as repair or replacement of the welded joint. The report should also provide a clear timeline for corrective action and any necessary follow-up inspections.

Overall, a detailed report is essential for documenting the inspection results and ensuring that any defects are identified and addressed in a timely and effective manner. This can help to improve the quality and safety of welded products and ensure that they meet the required industry standards and customer expectations.

Importance of Weld Repair

Weld repair plays a significant role in maintaining the integrity and functionality of welded components. When defects or discontinuities are detected in welded joints during inspection or testing, weld repair becomes necessary to rectify the issues and restore the desired quality and performance of the weld. The importance of weld repair can be highlighted in the following aspects:

Structural Integrity: Welds are often critical components in structures, machinery, and equipment. Any defects or flaws in the weld can compromise the structural integrity, leading to potential failures or accidents. Weld repair helps address these issues by eliminating or mitigating the defects, ensuring that the welded joint regains its intended strength and load-bearing capacity.

Functional Performance: Welded components are subjected to various operating conditions, including mechanical stress, temperature fluctuations, and corrosive environments. Defective welds may hinder the functionality of the component, affecting its performance and reliability. Weld repair ensures that the weld meets the required specifications and standards, allowing the component to perform its intended function effectively.

Cost-Effectiveness: Weld repair is often a more cost-effective solution compared to the replacement of entire components or structures. Repairing a defective weld eliminates the need for complete rework or replacement, saving time, resources, and expenses. It also minimizes production downtime and associated costs, making it a practical choice in many scenarios.

Compliance with Standards and Regulations: Welded components in various industries are governed by specific standards and regulations to ensure safety, reliability, and quality. Weld repair helps bring the welded joints back into compliance with these standards, ensuring that the repaired weld meets the required specifications and is suitable for its intended application.

Environmental Considerations: Repairing a defective weld minimizes the generation of scrap material and waste, contributing to sustainable practices. By salvaging and repairing welded components, the need for raw materials and energy-intensive manufacturing processes is reduced, resulting in environmental benefits.

To ensure the effectiveness and reliability of weld repair, it is crucial to follow established procedures and guidelines outlined in relevant standards, such as EN ISO 5817 and EN ISO 15614. These standards provide specifications for weld repair techniques, materials, and qualification requirements, ensuring that the repaired weld meets the necessary quality and performance criteria. Additionally, weld repair should be carried out by qualified personnel with expertise in welding techniques and repair procedures. Proper inspection and testing should be conducted post-repair to verify the quality and integrity of the repaired weld. This ensures that the repaired weld meets the required standards and can safely perform its intended function.

The importance of weld repair lies in its ability to restore the integrity, functionality, and compliance of welded components. By addressing defects and discontinuities, weld repair ensures structural soundness, functional performance, and cost-effectiveness. It enables the reuse and preservation of components, reduces waste, and contributes to overall safety, reliability, and sustainability in welding practices.

Assessing the Need for Weld Repair

Assessing the need for weld repair is an essential step in maintaining the quality and integrity of welded components. It involves evaluating the severity and impact of defects or discontinuities present in the welds and determining whether repair is necessary. The following factors are considered during the assessment process:

Inspection Results: Welded joints undergo various inspection methods, such as visual inspection, non-destructive testing (NDT), and macroscopic examination, to identify any defects or discontinuities. The inspection results provide valuable information about the type, size, location, and extent of the defects. Based on these findings, the need for repair can be assessed.

Applicable Standards and Codes: Welded components are subject to specific standards and codes that outline the acceptance criteria for weld quality. These standards specify the maximum permissible defect sizes and types. By comparing the inspection results with the relevant standards, it is possible to determine whether the defects exceed the acceptable limits and require repair.

Structural and Functional Considerations: The significance of the welded joint in the overall structure or component is evaluated. If the defect or discontinuity compromises the structural integrity or functional performance, repair becomes necessary. Factors such as load-bearing capacity, stress concentration, fatigue life, and service conditions are considered to assess the impact of the defects on the weld's performance.

Safety and Regulatory Compliance: Welded components are often used in safety-critical applications, such as in the aerospace, automotive, and oil and gas industries. Assessing the need for repair involves considering the safety implications of the defects. Welds that pose a risk to personnel safety or fail to comply with industry regulations must be repaired to ensure compliance and prevent potential hazards.

Cost and Time Considerations: The cost and time required for repair are evaluated in relation to the significance of the defects and the value of the welded component. Factors such as the availability of repair resources, the impact of repair on production schedules, and the cost-effectiveness of repair versus replacement are taken into consideration.

Based on the assessment, a decision is made whether to proceed with weld repair. If the defects are deemed significant and impact the weld's integrity or performance, repair is typically recommended. The repair process involves preparing the weld surface, removing the defective portion, applying suitable repair techniques (such as welding or other approved methods), and conducting post-repair inspection and testing to verify the quality and integrity of the repaired weld. It is important to note that the assessment of the need for weld repair should be carried out by qualified personnel with expertise in welding technology and relevant standards. These individuals should have a thorough understanding of weld defects, their implications, and the appropriate repair methods to ensure that the repaired weld meets the required quality and performance criteria. Assessing the need for weld repair involves evaluating inspection results, considering applicable standards and codes, assessing structural and functional considerations, ensuring safety and regulatory compliance, and considering cost and time factors. By conducting a comprehensive assessment, informed decisions can be made regarding the necessity and appropriateness of weld repair, ensuring the continued integrity and performance of welded components.

Methods and Techniques for Weld Repair

When it is determined that a weld requires repair due to defects or discontinuities, various methods and techniques can be employed to restore its integrity and functionality. The choice of repair method depends on factors such as the type and severity of the defects, the material being welded, and the specific requirements of the application. Here are some common methods and techniques used for weld repair:

Welding Repair: Welding is the most common method for repairing weld defects. It involves adding additional weld metal to the defective area to fill gaps, correct insufficient penetration, or fuse discontinuities. The welding process used for repair depends on factors such as the base material, defect type, and available equipment. Common welding processes include shielded metal arc welding (SMAW), gas tungsten arc welding (GTAW), gas metal arc welding (GMAW), and flux-cored arc welding (FCAW). Proper welding techniques and parameters must be employed to ensure sound and high-quality repairs.

Weld Overlay: Weld overlay, also known as cladding, is a technique where a layer of weld metal is applied to the surface of the base material to improve its properties or protect it from corrosion. It is often used to repair or enhance worn or damaged surfaces. Weld overlay can be performed using processes such as GTAW, GMAW, or plasma transferred arc (PTA) welding. The choice of overlay material depends on factors such as the desired properties, compatibility with the base material, and environmental conditions.

Mechanical Repair: In some cases, mechanical methods are used for weld repair. These methods involve removing the defective portion of the weld or the entire weld and replacing it with a new weld or a mechanical fastening method. Mechanical repair techniques include grinding, machining, milling, or drilling to remove the defective weld and preparing the area for re-welding or joining. Mechanical repairs are often employed for minor defects or when welding is not feasible or practical.

Brazing and Soldering: For certain types of defects or materials, brazing or soldering techniques can be used for repair. These processes involve melting a filler metal with a lower melting point than the base material and using capillary action to join the parts. Brazing and soldering are commonly used for repairs in non-ferrous metals, electrical components, and delicate assemblies.

Heat Treatment: In some cases, heat treatment techniques are employed as a part of the weld repair process. Heat treatment can help relieve residual stresses, improve mechanical properties, or reduce the risk of cracks. Processes such as post-weld heat treatment (PWHT) or stress relieving may be applied after the repair welding to ensure the desired metallurgical and mechanical properties are achieved.

Regardless of the repair method employed, it is crucial to follow industry standards, specifications, and best practices. Qualified personnel with expertise in welding and repair techniques should carry out the repairs to ensure proper execution. Post-repair inspection and testing are also necessary to verify the quality and integrity of the repaired weld. The selection of the appropriate method depends on factors such as the type and severity of defects, base material, and application requirements. It is essential to follow established procedures and guidelines to ensure the effectiveness and long-term reliability of the repaired weld.

Evaluating the Effectiveness of Weld Repairs

Evaluating the effectiveness of weld repairs is a crucial step in the welding process to ensure that the repaired weld meets the required quality standards and can withstand the intended service conditions. The evaluation involves assessing various aspects of the repair, including its structural integrity, mechanical properties, and dimensional accuracy.

Key factors to consider when evaluating the effectiveness of weld repairs:

Visual Inspection: Visual inspection is the initial step in evaluating the effectiveness of a weld repair. The repaired area should be visually examined to ensure that the defects have been adequately addressed and that the weld profile, reinforcement, and overall appearance meet the specified requirements. Any signs of incomplete fusion, cracks, porosity, or other defects should be carefully examined to ensure they have been properly repaired.

Non-Destructive Testing (NDT): Non-destructive testing methods play a vital role in assessing the quality of weld repairs. Techniques such as ultrasonic testing, magnetic particle testing, dye penetrant testing, radiographic testing, or eddy current testing can be employed to detect subsurface defects, discontinuities, or other hidden flaws that may affect the integrity of the repaired weld. NDT helps verify the soundness and reliability of the repair and ensures that any residual defects are identified and addressed.

Mechanical Testing: Mechanical testing is often performed to evaluate the strength, hardness, ductility, and other mechanical properties of the repaired weld. Tensile testing, hardness testing, impact testing, or bend testing can be conducted to assess the weld's mechanical performance and compare it against the specified requirements. These tests provide quantitative data on the weld's strength and integrity, helping determine whether the repair has restored the weld's mechanical properties to an acceptable level.

Dimensional Inspection: Dimensional inspection is essential to ensure that the repaired weld conforms to the specified dimensions, including length, width, height, and throat thickness. Precision measuring tools such as callipers, micrometres, gauges, or optical measurement systems can be used to assess the dimensional accuracy of the repair. Proper dimensional alignment and fit-up are crucial for ensuring the structural integrity and functionality of the repaired weld.

Post-Repair Documentation: It is important to maintain comprehensive documentation of the weld repair process, including repair procedures, inspection reports, test results, and any other relevant information. This documentation serves as a reference for future inspections, audits, or quality assessments and provides a record of the repair's effectiveness and compliance with applicable standards and specifications.

Compliance with Standards and Specifications: The effectiveness of a weld repair should be evaluated based on the applicable industry standards, codes, and specifications. These documents outline the acceptance criteria, quality requirements, and inspection procedures that must be followed. The repaired weld should meet or exceed the specified standards to ensure its fitness for service and long-term reliability.

By conducting a thorough evaluation that includes visual inspection, non-destructive testing, mechanical testing, dimensional inspection, and compliance with standards, the effectiveness of weld repairs can be accurately assessed. This evaluation ensures that the repaired weld meets the necessary quality standards and provides confidence in its structural integrity and performance under operating conditions.

Post-Weld Examination for Quality Verification

Post-weld examination is a critical step in the welding process that involves verifying the quality and integrity of the welded joint after the completion of welding operations. This examination aims to ensure that the weld meets the required standards, specifications, and performance criteria. It helps identify any potential defects, discontinuities, or imperfections that may have occurred during welding and assesses the overall quality of the weld.

Key aspects of post-weld examination for quality verification:

Visual Inspection: Visual inspection is the most basic and commonly used method for post-weld examination. It involves a thorough visual examination of the welded joint to identify any visible defects or imperfections. The inspector assesses the weld for issues such as incomplete fusion, lack of penetration, undercutting, porosity, cracks, spatter, or any other irregularities. Visual inspection also verifies the overall weld profile, reinforcement, and appearance, ensuring they conform to the specified requirements.

Non-Destructive Testing (NDT): Non-destructive testing techniques are employed to evaluate the internal and surface integrity of the weld without causing damage. Various NDT methods such as ultrasonic testing, radiographic testing, magnetic particle testing, liquid penetrant testing, or eddy current testing can be used to detect subsurface defects, discontinuities, or other hidden flaws. NDT helps ensure that the weld is free from internal defects, such as porosity, cracks, lack of fusion, or inclusions, which could compromise its strength and performance.

Mechanical Testing: Mechanical testing is performed to assess the mechanical properties and strength of the welded joint. Tensile testing, hardness testing, impact testing, or bend testing can be conducted on test specimens extracted from the weld to evaluate its strength, ductility, toughness, and other mechanical characteristics. Mechanical testing provides quantitative data on the weld's performance, allowing for comparison against specified requirements and standards.

Dimensional Inspection: Dimensional inspection verifies the dimensional accuracy and conformity of the weld to the specified requirements. It involves measuring key dimensions of the weld, such as length, width, height, and throat thickness, using precision measuring tools. Dimensional inspection ensures that the weld conforms to the required dimensions, fit-up tolerances, and geometric parameters necessary for its intended application.

Documentation and Record-Keeping: Proper documentation and record-keeping are essential in post-weld examination. Detailed records should be maintained, including inspection reports, test results, welder qualifications, welding procedure specifications, and any other relevant documents. This documentation provides evidence of compliance, allows for traceability, and serves as a reference for future inspections, audits, or quality assessments.

Compliance with Standards and Specifications: Post-weld examination should be conducted in accordance with applicable standards, codes, and specifications. These documents define the acceptance criteria, quality requirements, and inspection procedures that must be followed. Compliance with standards ensures that the weld meets the necessary quality standards and provides assurance of its performance and reliability in service.

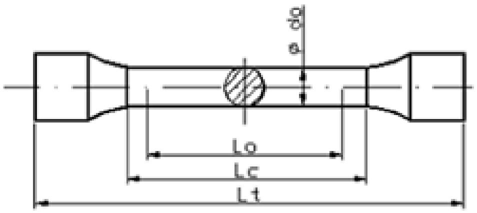
Post-weld examination plays a vital role in verifying the quality and integrity of welded joints. By conducting thorough visual inspection, employing appropriate non-destructive testing techniques, performing mechanical and dimensional testing, and maintaining proper documentation, the quality of the weld can be effectively verified. This examination process ensures that the weld meets the required standards, enhances its structural integrity, and contributes to the overall success and reliability of the welded component or structure.

1.4.6. Methods for Mechanical Testing of Metallic Materials

Mechanical tests refer to all determinations regarding the behaviour of metallic materials under specific mechanically imposed conditions, conventionally established. Mechanical tests vary based on the type of macroscopic deformation generated (tension, compression, bending, torsion, hardness, etc.), the mode of load application (static, dynamic, variable in magnitude and direction), the temperature at which the test is conducted (hot, cold, at room temperature), etc. The most common approaches involve static tensile tests, hardness tests, and dynamic impact bending tests.

a) Static Tensile Tests

These tests are conducted on specimens in the form of bars, wires, or strips with standardized dimensions. The most used shape and primary dimensions of the tensile specimen are presented in Figure 1.3.30.



L_t = total length

L_c = calibrated length

L_o = initial length between markers

d_o = initial diameter

Fig. 1.3.30: Tensile static test specimen

The initial length L_0 and initial diameter d_0 are chosen in such a way that the dimensional ratio, denoted as $n = L_0/d_0$, has a value of either n = 5 or n = 10. The processing of specimens is described in STAS 7324 - 75, while the shape, dimensions, and conditions for specimen preparation are specified in STAS 200 - 87.

The machines used for tensile tests are equipped with a recorder that plots the relationship between the axial force F and the specimen's elongation Δl. Figure 1.3.31 depicts a tensile stress-strain curve for a low-carbon steel.

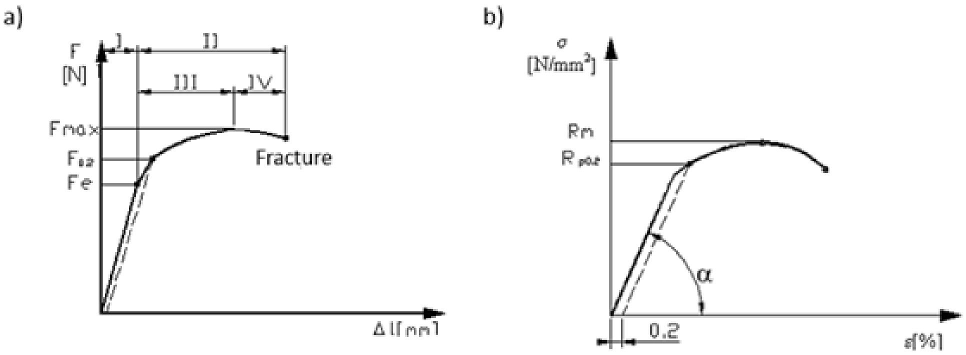


Fig. 1.3.31: a) Tensile curve of a low-carbon steel; b) Conventional tensile curve of a low-carbon steel.

We distinguish:

I - The domain of elastic deformation.

II - The domain of plastic deformation.

III - Uniform deformation.

IV - Non-uniform deformation (necking).

In domain I, elastic deformation of the material occurs, following Hooke's law: $\varepsilon = \sigma/E$.

The conventional tensile curve (Figure 1.3.32) represents the relationship between the mechanical stress σ, defined as the ratio of force to cross-sectional area ($\sigma = F/A$ [N/mm²]), and the relative elongation ε, defined as the ratio of the specimen's elongation Δl to its initial length L_o.

$$\varepsilon = \frac{\Delta l}{L_0} \cdot 100 \ [\%]$$

From this curve, the following characteristics can be determined:

- Tensile strength, Rm, defined as the ratio of the maximum load to the initial cross-sectional area of the specimen:

$$R_m = \frac{F_{max}}{S_o} \left[\frac{N}{mm^2} \right]$$

- Conventional yield strength, $R_{p0.2}$, obtained as the ratio of the load that produces a plastic deformation of 0.2% to the initial cross-sectional area of the specimen:

$$R_{p0,2} = \frac{F_{0,2}}{S_0}\left[\frac{N}{mm^2}\right]$$

- Longitudinal elastic modulus, E, representing the slope of the linear portion of the conventional tensile curve: αtgE = Δσ/Δε;
- Elongation at break, A_n, defined as the ratio of the specimen's elongation to its initial length:

$$A_n = \frac{L_f - L_0}{L_0} \cdot 100[\%]$$

where L_f is the length of the specimen after rupture, and n is the dimensional factor.

- Necking at break, Z, expressed as the difference between the initial and final cross-sectional areas of the specimen relative to the initial cross-sectional area:

$$Z = \frac{S_0 - S_f}{S_0} \cdot 100[\%]$$

where S_0 is the initial cross-sectional area of the specimen, and S_f is the final cross-sectional area in the rupture zone.

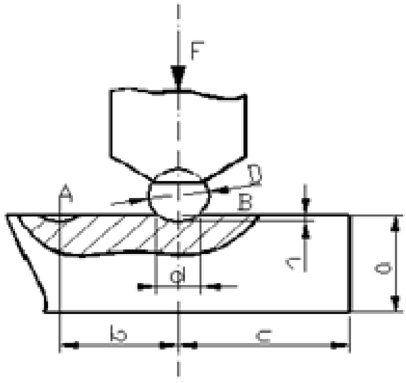
b) Hardness Tests

Hardness is defined as the property of a material to resist plastic deformation caused by localized static or dynamic contact loads on its surface. It is one of the most used mechanical properties for characterizing metallic materials and depends on the chemical composition and structural state of the material.

Several methods exist for determining hardness based on the size of the impression produced by a penetrator pressed with a specific force onto the material's surface. The choice of the most suitable hardness testing method considers:

- The nature of the material being tested.
- The precision of the test.
- The dimensions of the sample.
- The cost-effectiveness of the determination.

The Brinell method involves indenting the material's surface with a steel ball of diameter D, perpendicular to the surface, as shown in the scheme in Figure 1.3.32.



D = ball diameter
h = depth of the trace
d = trace diameter
a = thickness of the part
b = distance between two traces
c = distance between the trace and the edge of the part

Fig. 1.3.32: Determination of Brinell hardness

Hardness is expressed as the ratio of the applied force F to the spherical cap area S of the impression left by the penetrator on the surface being tested:

$$HB = \frac{2 \cdot F}{\pi \cdot D \cdot \left(D - \sqrt{D^2 - d^2}\right)} \ [\mathrm{daN/mm^2}]$$

The duration of the load, the minimum distance between two traces (b), and the distance between the trace and the edge of the sample (c) depend on the hardness of the material.

The minimum thickness of the samples subjected to the test (a) should be 8 times the depth of the impression to avoid the influence of the apparatus support on the hardness.

In practice, the hardness value is derived from tables based on the diameter of the trace, for a specific load force and ball diameter.

This method is used for soft alloys (non-ferrous metals, and annealed steels).

Hardness is denoted by the measured value followed by the symbol HB and a group of three numbers indicating the ball diameter in mm, the load force in N, and the duration in seconds.

Example: 270HB5/700/5

The Rockwell method allows the evaluation of hardness through the depth of the residual impression of the penetrator. The penetrator can be a diamond cone with a 120° apex angle or a hardened steel ball.

This method applies to both hard and very hard materials (hardened steel, white cast iron) as well as soft materials. Testing with a diamond cone (HRC) or a ball (HRB) is done according to the scheme in Figure 1.3.33, the penetrator is initially pressed onto the measurement surface with an initial force F0. The measurement depth device is set to zero, and then the force F1 is applied. After an interval of 15, 30, or 60 seconds, the over force F1 is removed, and the HRC hardness is determined by subtracting the actual penetration depth e from the Rockwell unit size of the conventional constant E.

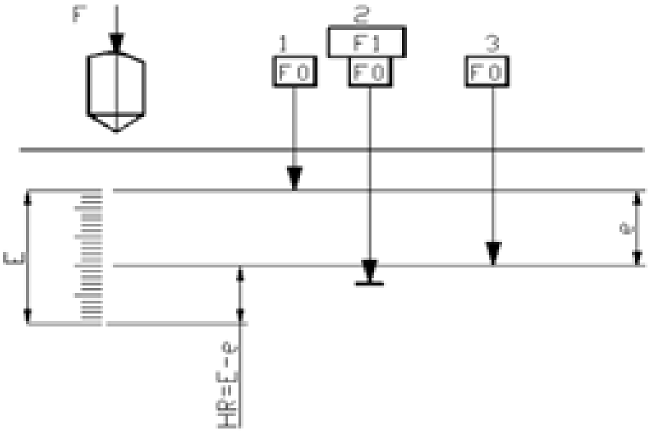


Fig. 1.3.33: Determination of Rockwell Hardness

Notation of hardness using this method is done using the symbol HRC (or HRB) followed by the hardness value.

Example: 45 HRC

The Vickers method (Figure 1.3.34) is similar in principle to the Brinell method, involving the application of a penetrator with the shape of a right pyramid with a square base and a vertex angle of 136° on the test surface. Vickers hardness is denoted as HV and is determined as the ratio of the applied force F to the lateral area of the impression left by the penetrator. In practice, the area is determined based on the average value of the diagonal of the trace, according to the formula:

$$HV = 1{,}8544 \cdot \frac{F}{d^2} \ [\text{daN/mm}^2]$$

This method is applicable to both soft and hard materials. It can also be used for measuring microhardness by coupling a testing apparatus with an optical metallographic microscope. In this way, the hardness of structural constituents and very fine structures can be determined. In this case, HV hardness is determined using the formula:

$$HV = 1{,}8544 \cdot \frac{F}{(d+1{,}5)^2}$$

Where d is the diagonal of the trace, and 0.25 is a correction factor.

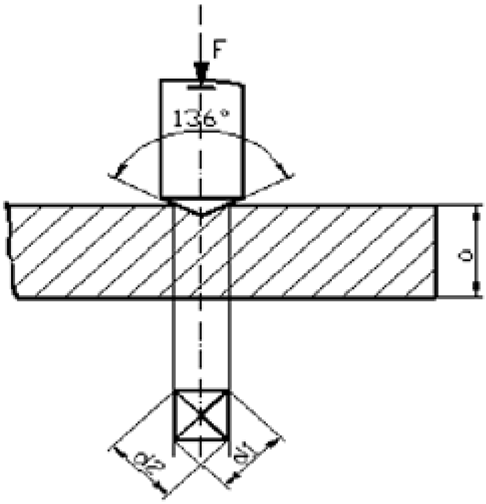


Fig. 1.3.34: Determination of Vickers Hardness

Hardness notation is done using the symbol HV, followed by the hardness value and followed by an indicator representing the load in daN (if it differs from 30 daN), followed by the duration of load retention in seconds (if it differs from 10...15s).

c) Dynamic Impact Bending Test (Resilience Test)

It constitutes a method for assessing the toughness of materials.

Toughness represents the material's resistance to abrupt crack propagation.

The most used apparatus is the Charpy pendulum hammer, equipped with a weight G that oscillates around the center O (Figure 1.3.35).

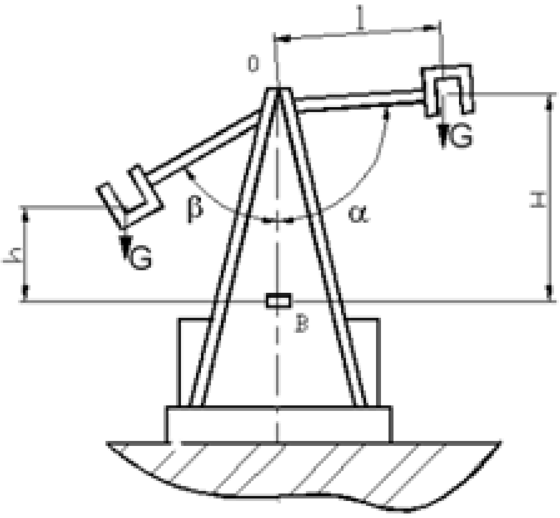


Fig. 1.3.35: Charpy pendulum hammer.

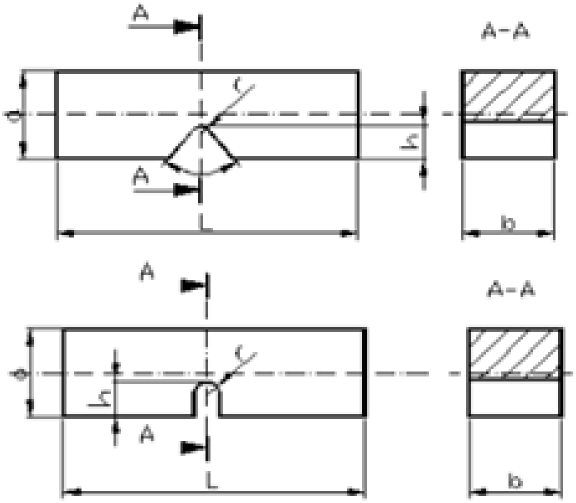


Fig. 1.3.36: Specimens for impact bending test

The specimens used are typically prismatic, with dimensions of 10 x 10 x 55 mm and a "U" or "V" notch (Figure 1.3.36).

To break the specimen placed on two supports on the frame, the hammer falls from the initially set height H, and after the specimen breaks, it is raised to height h.

The energy consumed for breaking the specimen is given by the equation:

$$W = W_0 - W_f = G \cdot (H - h)$$

where:

W_0 = potential energy of the hammer in the initial position.

W_f = potential energy in the final position.

In practice, the apparatus allows the determination of the angle α that the hammer's rod of length l makes with the vertical when in the initial position, and the angle β that the hammer's rod makes with the vertical after the specimen has broken. Expressing heights H and h in terms of angles α and β and length l, the energy consumed in the process of breaking the specimen can be written as:

$$W = G \cdot l(\cos\beta - \cos\alpha) \ [\mathrm{J}]$$

In the case of testing specimens with a "U" notch, resilience is defined as the ratio of the energy consumed to break the specimen to the cross-sectional area at the notch. It is symbolized as KCU and is expressed in J/cm². Thus, the notation KCU 150/2/5 = 100 J/cm² signifies a resilience value of 100 J/cm², determined with a pendulum hammer having a potential energy W_0 = 150 J, on "U" notched specimens with a depth of h = 2 mm and width of b = 5 mm.

In the test with "V" notched specimens, resilience represents the energy consumed for breaking. In this case, the symbol is KV, and it is expressed in J. The notation KV 300/5 = 70 J represents the value of the breaking energy (70 J) determined with a hammer having potential energy W0 = 300 J on a "V" notched specimen with a width of b = 5 mm.

Resilience testing is more representative when conducted at different temperatures to determine the transition temperature from ductile to brittle fracture, as materials behave differently at various working temperatures.

It can be observed that the energy absorbed during fracture decreases with decreasing temperature (Figure 1.3.37).

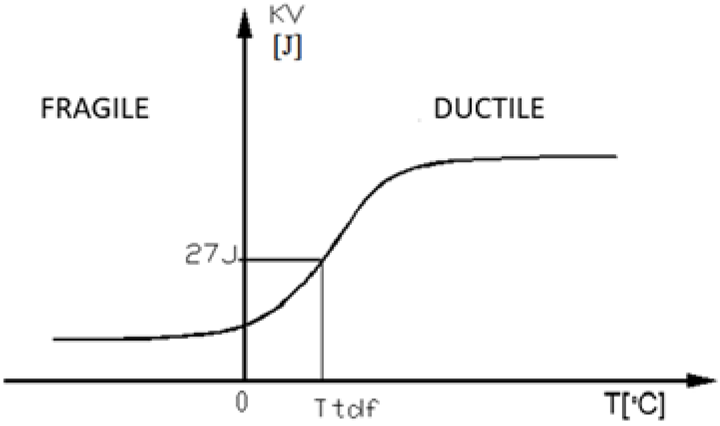


Fig. 1.3.37: Resilience variation with temperature

In general, it is estimated that the temperature at which the ductile-to-brittle transition (TDBT) occurs is the temperature at which the value of the breaking energy is KV = 27J. On resilience specimens, the ductility of the material can be assessed by the proportion of the crystalline surface to the total fracture surface.

Conclusions

In conclusion, macroscopic examination is an indispensable tool in the field of welding technology. It provides valuable insights into the characteristics, defects, and quality of welded joints, allowing for effective evaluation and assessment. Throughout this e-course, we have explored the various aspects of macroscopic examination, including its definition, purpose, advantages, and limitations.

We have learned that macroscopic examination serves as a fundamental method for assessing weld quality, identifying defects, and ensuring the integrity of welded joints. It allows for visual inspection of weld profiles, dimensions, and surface irregularities, providing essential information about joint configuration, penetration, reinforcement, and overall appearance. By analysing macroscopic features, such as weld size, shape, and reinforcement, we can gain valuable insights into the welding process and the integrity of the joint.

Moreover, macroscopic examination complements other inspection techniques, including non-destructive testing and microscopic examination. It helps in the detection and characterization of defects such as porosity, cracks, lack of fusion, and incomplete penetration. By combining visual inspection with other testing methods, we can obtain a comprehensive understanding of the weld's quality and performance.

Throughout this course, we have emphasized the importance of adhering to established standards and specifications. European standards provide clear guidelines for macroscopic examination and weld quality assessment, ensuring consistency and reliability in the inspection process. These standards outline acceptance criteria, defect classifications, and evaluation methods, enabling effective quality control and verification.

Additionally, we have explored the significance of post-weld examination, including weld repair and post-repair verification. Post-weld examination allows for the assessment of the effectiveness of repair procedures, ensuring that the repaired joint meets the required standards and specifications.

Overall, macroscopic examination plays a crucial role in weld quality control, defect identification, and ensuring the reliability of welded joints. It enables us to make informed decisions regarding the acceptance or rejection of welds, evaluate the performance and integrity of joints, and implement appropriate corrective measures. By understanding the principles and techniques of macroscopic examination, we can enhance the quality, safety, and durability of welded structures and components.

As professionals in the field of metallography and welding technology, it is essential to continually improve our knowledge and skills in macroscopic examination. This course has provided a solid foundation in understanding the significance, methodologies, and applications of macroscopic examination. By applying the knowledge gained in this course, we can contribute to the advancement of welding technology, promote quality assurance, and ensure the successful implementation of welded structures in various industries.

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1.5. Microscopic examination and its application in welding technology

The optical (light) microscope remains the most important tool for the study of microstructure, despite the evolution of sophisticated electron metallographic instruments. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are valuable tools as well; however, all examinations of microstructure should begin with the use of an optical microscope, starting at low magnification, such as 25×, followed by progressively higher magnifications to assess the basic characteristics of the microstructure efficiently.

Most microstructures can be observed with the optical microscope and identified based on their characteristics. Identification of questionable or unknown constituents may be aided by observation of their hardness relative to their matrix, by their natural colour, by their response to polarized light and by their response to selective etchants. These observations are compared to known details about the physical metallurgy of the material being examined. If doubt still remains or if the structure is too fine to observe, more sophisticated techniques must be implemented used in conjunction with optical microscopy, rather than as a substitute.

The scanning electron microscope is one of the most versatile instruments for investigating the microstructure of metallic materials. Compared to the optical (light) microscope, it expands the resolution range by more than one order of magnitude to approximately 10 nm (100) in routine instruments, with ultimate values below 3 nm (30). Useful magnification thus extends beyond 10,000× up to 150,000×, closing the gap between the optical and the transmission electron microscope. Compared to optical microscopy, the depth of focus, ranging from 1 m at 10,000× to 2 mm (0.08 in.) at 10×, is larger by more than two orders of magnitude, due to the very small beam aperture.

Scanning electron microscopy (SEM) offers possibilities for image formation that are usually easy to interpret and will reveal clear pictures of as polished and etched cross sections as well as rough surfaces. Energy-dispersive x-ray analysis using equipment routinely attached to the scanning electron microscope features semiquantitative and, in favourable cases, quantitative analysis of composition from a small volume with good lateral resolution. SEM has found a wide range of applications in materials research, materials development, failure analysis, and quality control.

Using microscopic examination, different aspects of a weld can be seen and analysed. Aspects such as imperfections or defects (Fig. 1.4.1.), microstructures (Fig. 1.4.2.) and the thermal regime of the welding process.

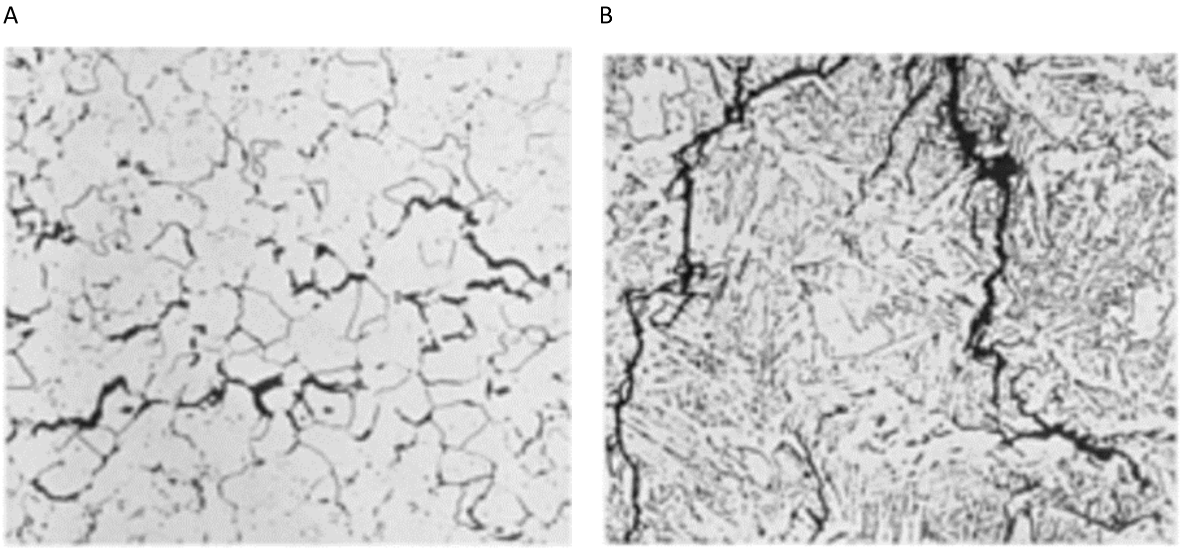


Fig. 1.4.1. A. Cracks in weld metal in ASTM A285, Grade C, steel plate. The cracks, which resulted from caustic embrittlement, are trans granular and inter-granular. Etch: Nital. 275×. B. Fissures, in ASTM A285 Grade C steel plate that had been in service one year at 480°C in a refinery vessel, caused by hydrogen penetration. Ferrite with only a few carbide particles--a result of hydrogen decarburization in service. Nital. 275x (Source: ASM Handbook Volume 9. Metallography and Microstructures).

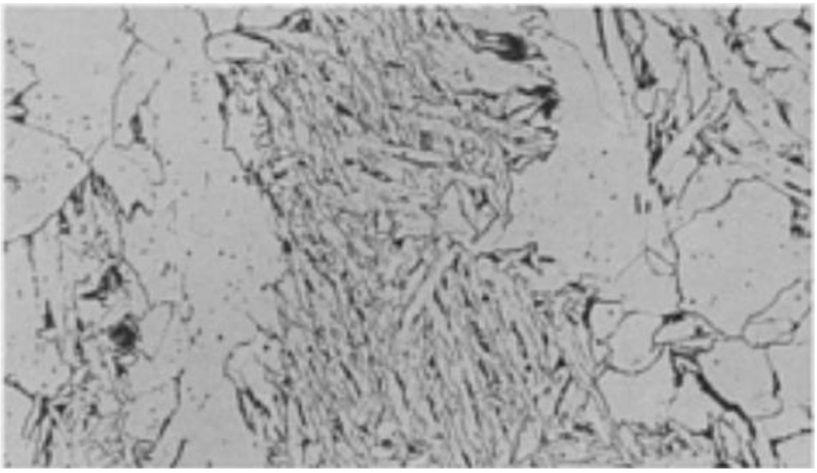


Fig. 1.4.2. 16 mm A-36 steel plate, multiple-pass shielded metal arc single-V butt weld. Heat input: 1.3 kJ/mm. Weld wire: AWS E7018. Fusion zone microstructure containing bainite and ferrite-carbide aggregate in coarse grain boundary ferrite. 2% nital. 500× (Source: ASM Handbook Volume 9. Metallography and Microstructures).

1.5.1. Equilibrium phases

The phase represents a homogeneous part of a system that has the same chemical composition and the same state of aggregation in its entire volume, being separated from other parts of the system by phase interfaces, as for example grain boundaries. The structure of the phase depends on the shape, size and the reciprocal arrangement of atoms.

The microstructural constituents can be made up of two or more phases which simultaneously separate from the liquid at the lowest solidification temperature of the system (eutectic transformation) or that appear as a result of phase transformations in the solid state (eutectoid transformation). It is identified using optical or electronic microscopy and may contain: pure metal, solid solution, defined compound or mechanical mixture.

During the heating or cooling processes, or when changing the state of aggregation from liquid to solid, metallic alloys undergo phase transformations in the solid state. The main types of transformations are: homogeneous transformations, which take place simultaneously in the whole volume of the piece, without germination and growth processes and heterogeneous transformations, which occur through germination and growth (with diffusion, without diffusion).

The main equilibrium phases that can be found in non-alloyed steel or low alloyed steel are: **ferrite, pearlite and cementite.**

Alpha Ferrite is the stable form of Iron at room temperature. It is soft and ferromagnetic at temperatures below 727 °C and paramagnetic between 727 and 912 °C. Alpha ferrite is a large component of mild steel but is present in all Iron carbon compounds that have been cooled to room temperature, co-existing with other forms such as Cementite.

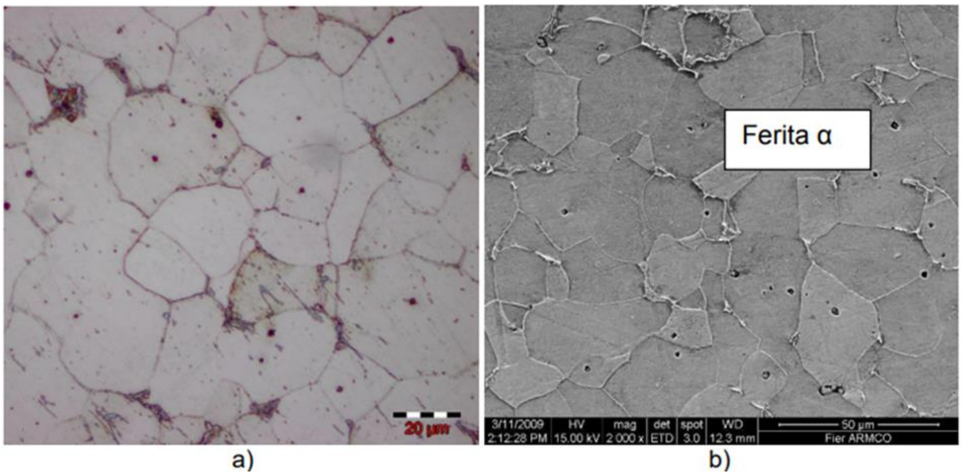


Fig. 1.4.4. ALPHA FERRITE: a) Optical microscopy(200x); b) SEM(2000x).

Austenite, also known as gamma-phase iron (γ-Fe), is a metallic, non-magnetic allotrope of iron or a solid solution of iron with an alloying element. In plain-carbon steel, austenite exists above the critical eutectoid temperature of 727 °C. It exists at room temperature in some stainless steels due to the presence of nickel stabilizing the austenite at lower temperatures.

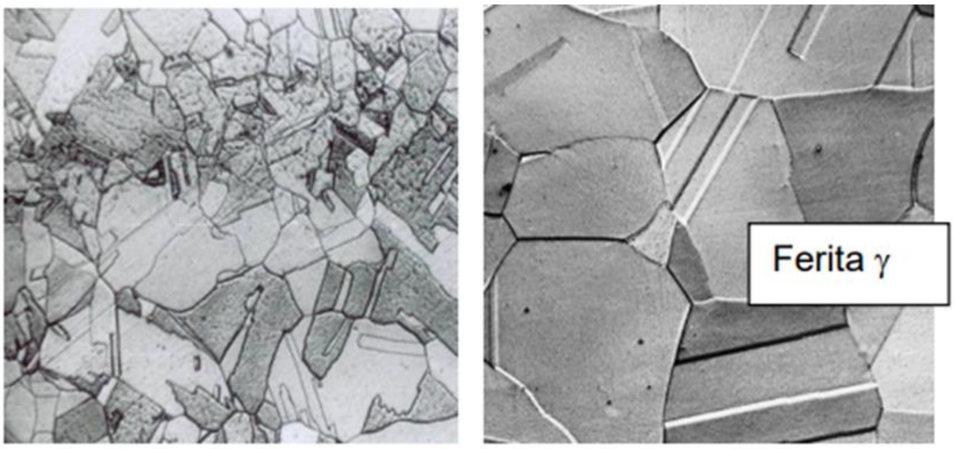


Fig. 1.4.5. AUSTENITE

Delta ferrite is a standing phase only at very high temperatures, close to the melting point; it becomes stable when allied with ferrite stabilizers such as: Cr, Mo, Si, Nb. Thus, delta ferrite forms during the cooling phase of manufacturing stainless steel. It starts as an austenite grain boundary phase, which then turns into a delta ferrite phase as it cools down.

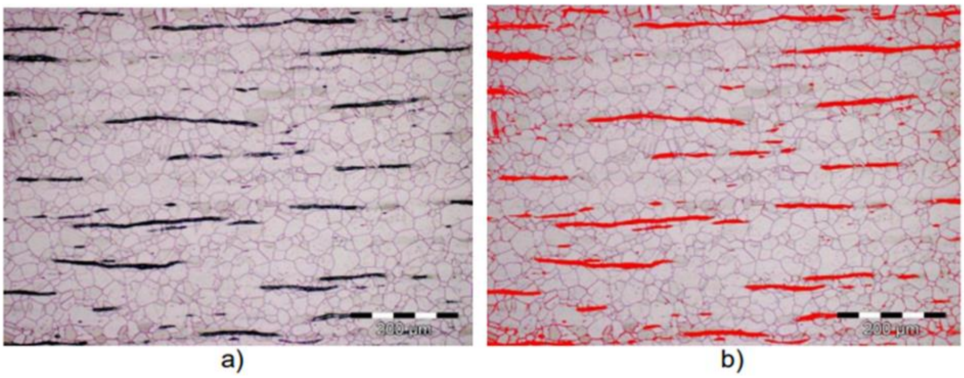


Fig. 1.4.6. Delta ferrite in stainless steel(16% Cr, 10% Ni) disposed in between austenite grains. a) Real micro-structure; b) Processed image using an Image Analysis program.

Pearlite is a two-phased, lamellar (layered or plate-like) structure composed of alternating layers of alpha-ferrite and cementite that occurs in some steels and cast irons. Pearlite only forms under special conditions which must be controlled to create this alloy phase. It is formed by eutectoid decomposition of austenite upon cooling by diffusion of C atoms, when ferrite and cementite grow contiguously, C precipitating as Fe_3C between laths of ferrite at the advancing interface, leaving parallel laths of Fe and Fe_3C which is pearlite.

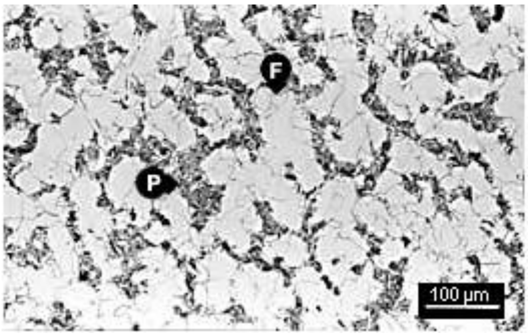
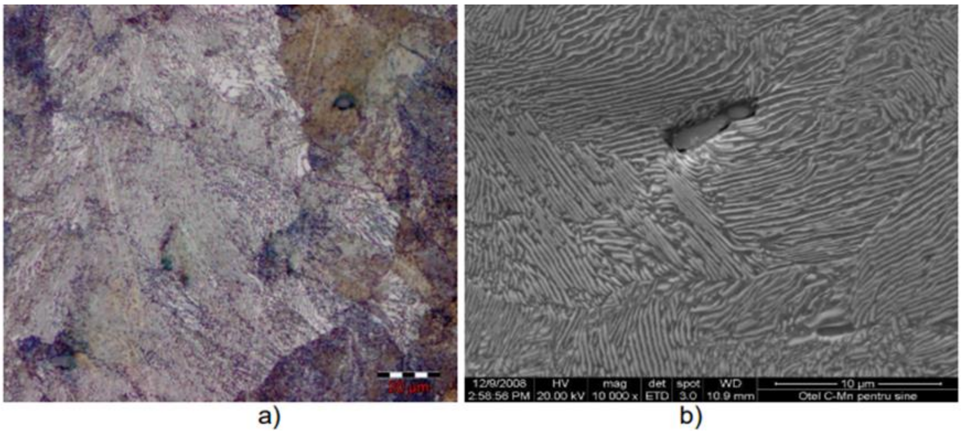


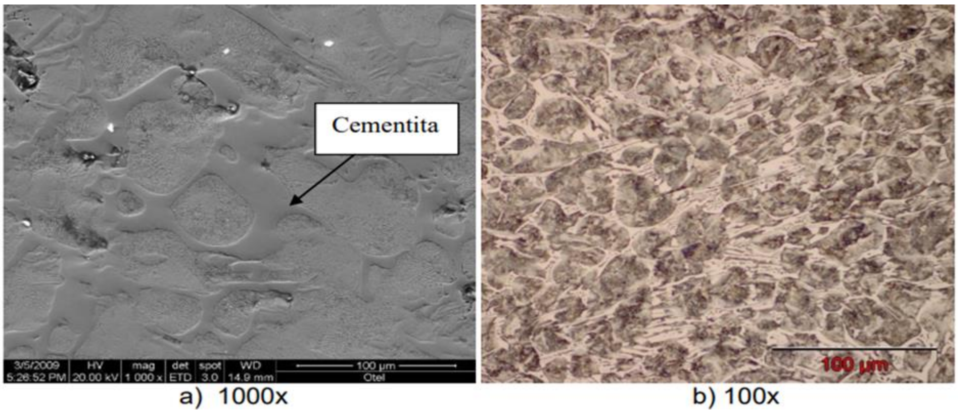
Fig. Alpha-ferrite(F) and Peralite (P). Source: TWI ()



a) b)

Fig. 1.4.76. Lamellar pearlite. Etch: Nital 2%: a) Optical Microscopy(1000x); b) SEM (10000x)

Cementite (or iron carbide) is a compound of iron and carbon, more precisely an intermediate transition metal carbide with the formula Fe_3C. By weight, it is 6.67% carbon and 93.3% iron. It is a hard and brittle structure, having a Brinell hardness of 700-750HB usually. In hypereutectoid steel can be found surrounding the pearlite grains while in cast iron has a needle-shaped structure or polyhedral crystals.



a) 1000x b) 100x

Fig. 1.4.87. Hypereutectoid steel. a) Pearlite and cementite(SEM); b) Optical microscopy

1.5.2. Heat treatments

Heat treatment is the process of heating and cooling metals, using specific predetermined methods to obtain desired properties. In simple terms, heat treatment is the process of heating the metal, holding it at that temperature, and then cooling it back. During the process, the metal part will undergo changes in its mechanical properties. This is because the high temperature alters the microstructure of the metal. And microstructure plays an important role in the mechanical properties of a material.

The final outcome depends on many different factors. These include the time of heating, time of keeping the metal part at a certain temperature, rate of cooling, surrounding conditions, etc. The parameters depend on the heat treatment method, type of metal and part size. Hardness tests can be performed in order to check the success of the heat treatment performed.

Normalization is a heat treatment whose main effect is the recrystallization of the phase in order to reduce grain size, while it also has the positive effects of dehydrogenation and stress relief.

The normalization can be complete, if the holding temperature is chosen at values slightly above the value of point AC_3: T_H = AC3 + (30÷50) °C, which corresponds to a value of about 950÷1050°C. The choice of the upper temperature value must be chosen very carefully in order to avoid overheating. Cooling is carried out in ventilated air or in oil, the cooling speed being higher than the equilibrium according to the Fe-C diagram but lower than the martensitic tempering speed.

The grain-refining effect is obtained by the double transformation of phase when heating up (Pearlite ® Austenite) and when cooling down (Austenite ® Ferrite + Pearlite). With this process, from an austenite grain can be obtained 3 grains of ferrite.

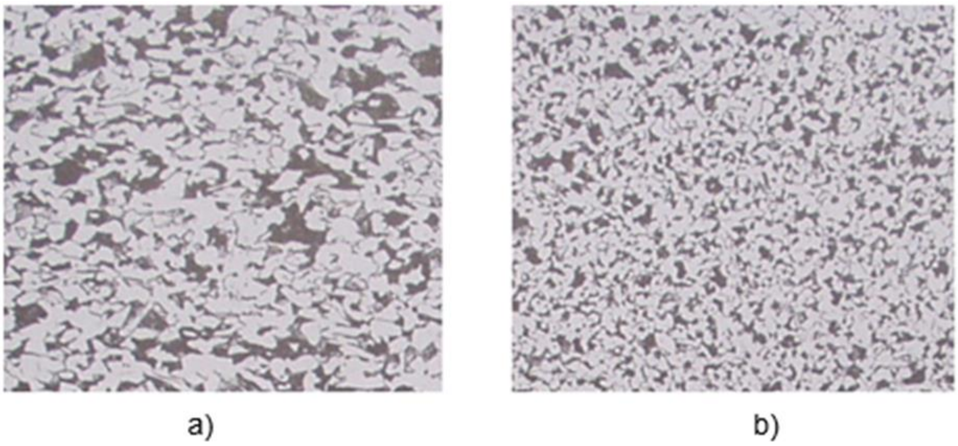


Fig. 1.4.98. Grain-refining through normalization of carbon steel type SA 515 Gr 70 thickness = 30mm: a) before normalization; b) after normalization at 900°C/1 hour.

Quenching is a heat treatment that aims to bring the material to a state very far from equilibrium, by heating it at a high temperature (Over AC_3) and then cooling it at sufficiently high speeds to prevent totally or partially the microstructural transformation that would have taken place in equilibrium conditions. Many times, the quenched material does not satisfy to the desired extent only some characteristics, in particular those of mechanical resistance and hardness, affecting on the other hand those of plasticity and tenacity.

To achieve an optimal effect regarding the technological behaviour in working conditions, the hardened parts are subjected to a subsequent heat treatment (tempering).

Tempering is a heat treatment applied after quenching in order to improve the characteristics that have been weakened due to quenching. The parts are heated up to a temperature lower than AC_1 and then air cooled.

In the Fig. 1.4.10a and 1.4.10b is possible to observe the C45 steel in commercial condition. In this condition, the microstructure exhibit ferrite and pearlite like principal structures. The Fig 1.4.10a was obtained with optical objective 20X and the figure 1.4.10b was obtained with 100X magnification. The figures 1.4.10c and 1.4.10d were obtained with 50X and 100X magnification respectively, material condition: quenched in water. The figures show a long structure in needle form. After cooling the resulting structure is martensite. The figures 1.4.10e and 1.4.10f were obtained with 50X and 100X magnification, material condition: quenched and tempered (Q-T) and the structure observed was tempered martensite.

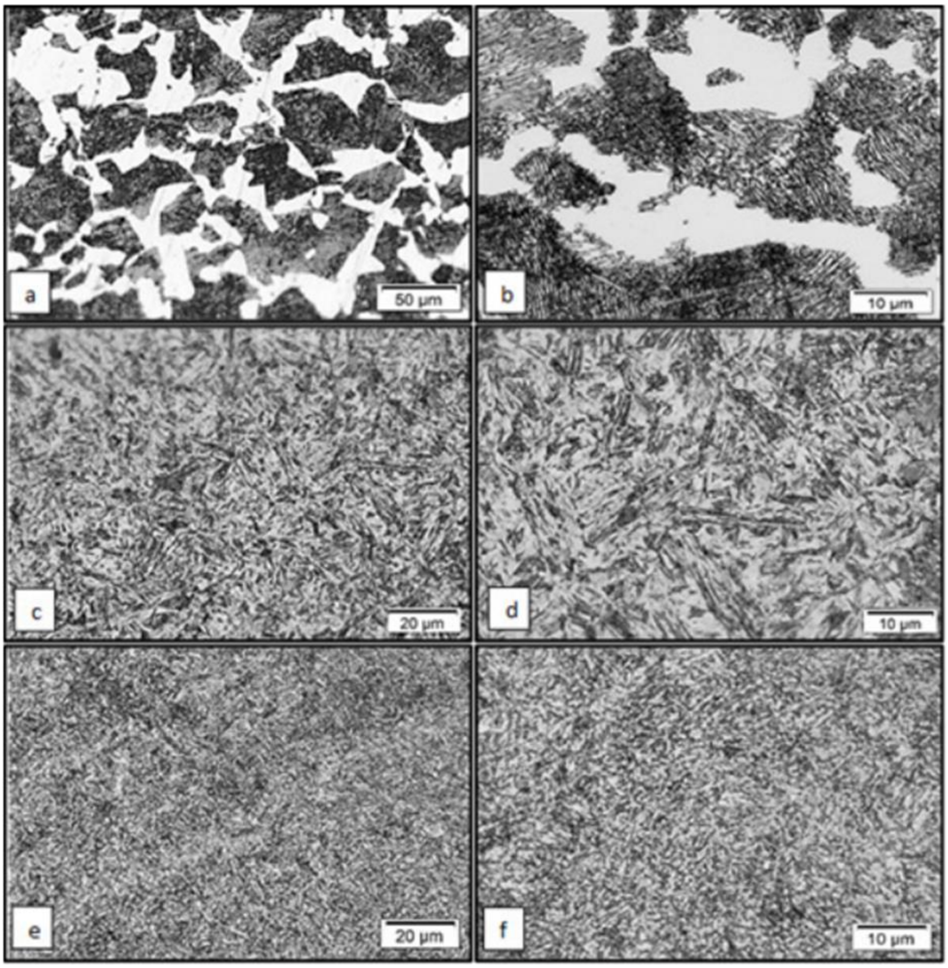
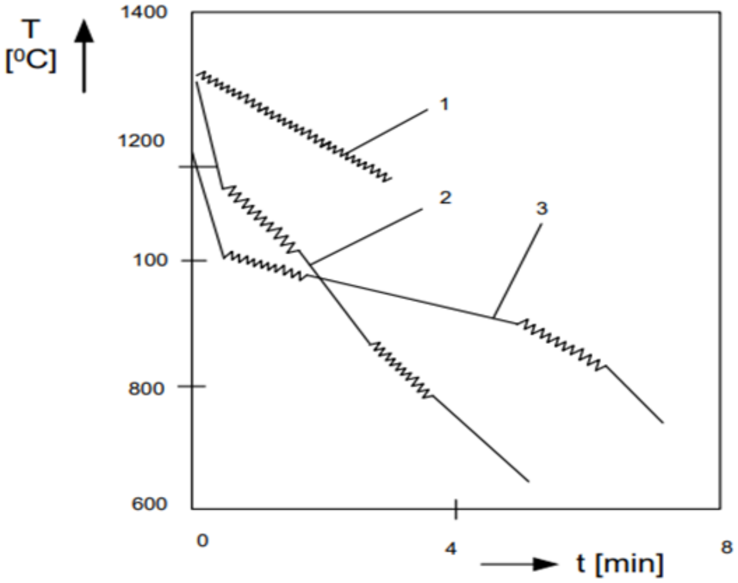


Fig. 1.4.9 Optical microstructures obtained for C45 steel quenched in water. a) Commercial condition at 20x. b) Commercial condition at 100x. c) water quenched at 50x. d) water quenched at 100x. e) Quenched and Tem-

pered at 50x. f) Quenched and tempered at 100x. (Source: http://www.iaeme.com/IJMET/index.asp 146 editor@iaeme.com International Journal of Mechanical Engineering and Technology (IJMET) Volume 9, Issue 7, July 2018, pp. 146–154, Article ID: IJMET_09_07_017)

Thermo-mechanically controlled processed (TMCP) steels are obtained when combining plastic deformation through rolling with heat treatment (thermomechanical rolling process). It is a unique procedure because it ensures the best combination of resistance and tenacity on low carbon contents, which cannot be obtained through any other type of heat treatment.



1 – classical hot rolling deformation, high temperatures throughout the whole process; 2 – hot rolling using controller temperature; 3- hot rolling combined with thermo-mechanical treatment, after an intermediate cooling that is being done at lower temperature.

By combining the effects of microalloying with hot rolling deformation at lower and lower temperatures, a decrease of approximately 8 times of the ferritic grain size is obtained, which causes an important increase in the yield strength and, above all, an improvement essential of the tenacity characteristics.

Fig. 1.4.11. Microstructure of TMCP steel

1.5.3. Microstructural aspects of a weld

In the following set of images obtained using optical microscopy different areas of a carbon steel welded joint and their specific structures can be seen, such as:

a) Base material: polygonal-ferrite (white colour) and pearlite (lamellar disposal, brown-light blue colour);

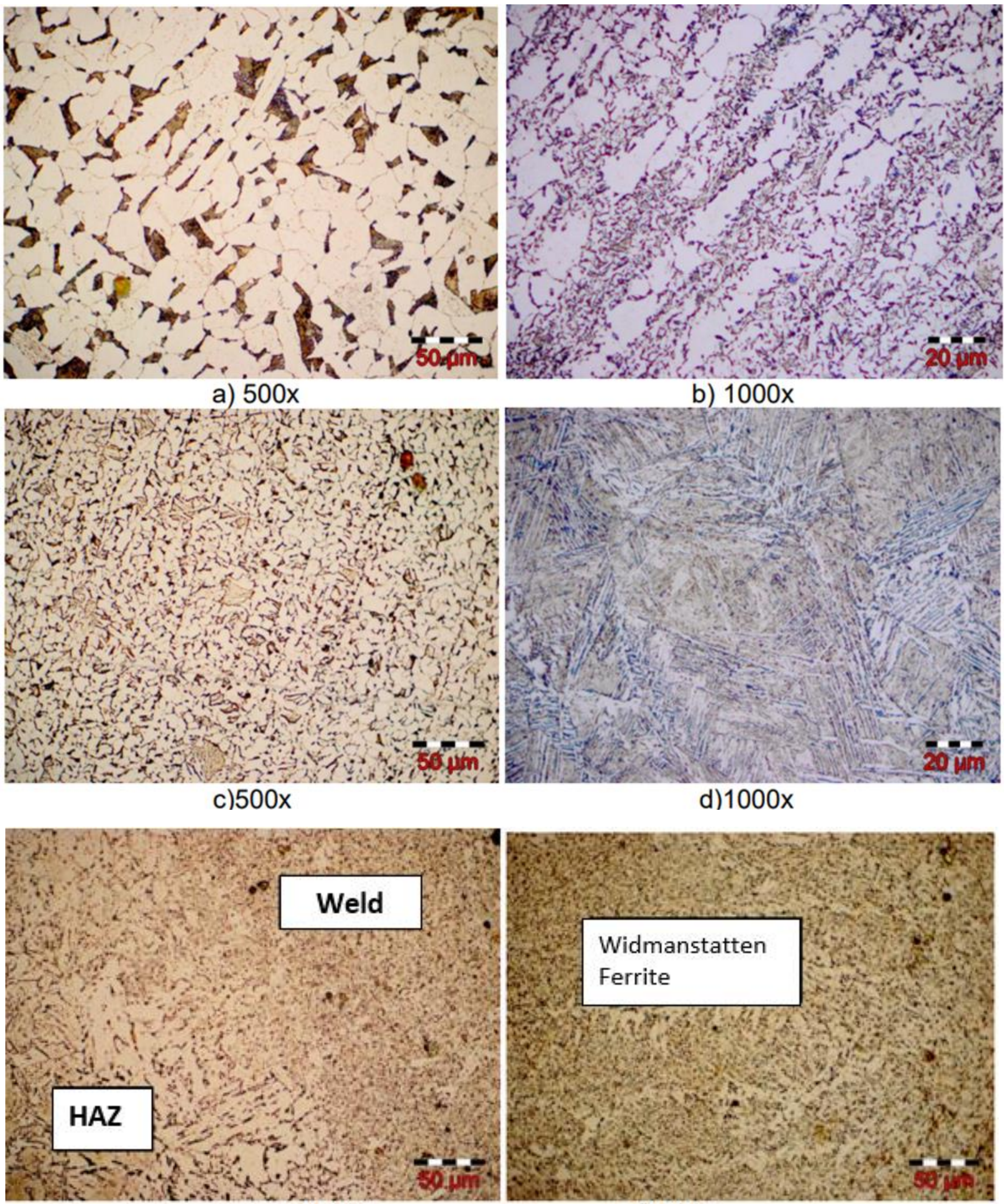
b) Recrystallization micro area of the HAZ: slightly modified ferrite and pearlite;

c) Normalizing micro area: fine ferrite and pearlite that has been recrystallised;

d) Overheated micro area: pearlite and Widmanstatten ferrite;

e) Fusion line: where the grain size difference of the HAZ can be seen;

f) Welded seam: fine ferrite, Widmanstatten ferrite and carbides precipitates.



a) 500x b) 1000x

c)500x d)1000x

e) 500x f) 500x

When cooling is carried out immediately below AC_3, ferrite starts to nucleate on the boundaries of the austenite grains and the formed grains increase in the form of massive blocks forming the so called limits of allotriomorphic grains.

At increased values of cooling, the growth tendency of ferrite starts to manifest itself in the form of massive lamellar structures, called the Widmanstätten structure, which then become refined as the cooling rate increases.

References:

[1.4.1] Voiculescu I, Geantă V, Vasile I M, 2016, Aliaje feroase pentru structuri sudate (Ferrous alloys for welded structures).

[1.4.2]International Journal of Mechanical Engineering and Technology (IJMET). Volume 9, Issue 7, July 2018, pp. 146–154, Article ID: IJMET_09_07_017.

[1.4.3]ASM Handbook Volume 4. Heat Treating.

[1.4.4]ASM Handbook Volume 6. Welding, Brazing and Soldering.

[1.4.5]ASM Handbook Volume 9. Metallography and Microstructures.

2. CU2: Specific Methods for Sample Preparation for Macro and Microscopic Examinations

2.1. Electrolytic polishing, chemical polishing, coloured etching – purpose and application

In this chapter, we will discuss three essential methods used in the field of metallography: electrolytic polishing, chemical polishing, and coloured etching. These techniques play a vital role in the examination and analysis of metallic materials, offering unique insights into their microstructural features and surface characteristics.

2.1.1. Introduction to Electrolytic Polishing Definition, Purpose and How It Works

Also known as electrochemical polishing, anodic polishing or electrolytic polishing, electropolishing is especially useful for polishing and deburring parts that are brittle or have complex geometries. Electropolishing improves surface finish by reducing surface roughness by up to 50%. The Electropolishing principle is presented in the figure 2.1.1.

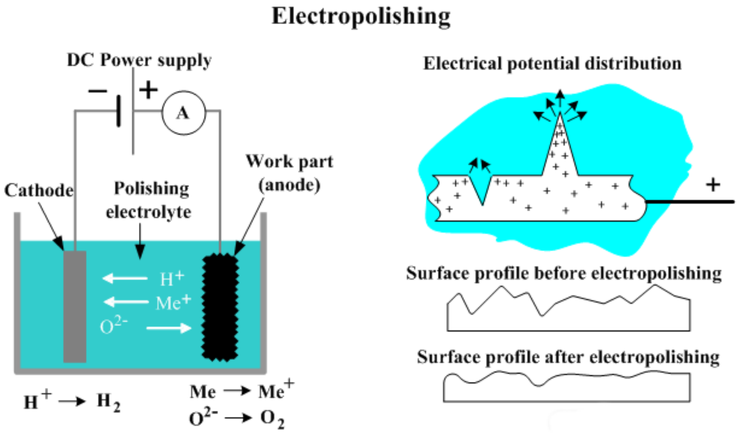
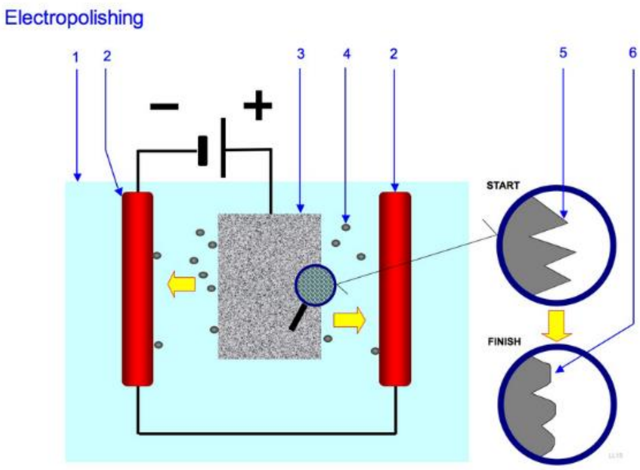


Figure 2.1.1 Electrolytic Polishing Principle [2.1.1]

The purpose of electrolytic polishing is twofold: surface refinement and surface preparation. By subjecting the specimen to an electric current in the presence of an electrolyte, this technique eliminates surface irregularities, such as scratches, deformation, and surface contaminants. The result is a smooth and uniform surface that facilitates accurate microstructural analysis and observations, as seen in figure 2.1.2. The primary objective of electrolytic polishing is to create a surface that accurately reflects the true microstructure of the material.



1. Electrolyte
2. Cathode
3. Workpiece to polish (Anode)
4. Particle moving from the workpiece toward the cathode
5. Surface before polishing
6. Surface after polishing

Figure 2.1.2. How Electropolishing Works [2.1.2]

In electropolishing, the metal part or workpiece serves as the positively charged anode. The workpiece is connected to the positive terminal of a DC power rectifier. The negatively charged cathode, generally made of stainless steel or zirconium, is connected to the negative terminal of the DC power rectifier.

Both anode and cathode are immersed in a temperature-controlled bath of electrolyte solution. The electropolishing chemicals for stainless steel typically consist of a high-viscosity mixture of sulfuric acid and phosphoric acid.

Electrical current from the rectifier is conducted from the anode to the cathode through the electrolyte. The electrical current causes metal ions on the surface of the part to oxidize and dissolve into the electrolyte. This process can dissolve extremely small, tightly controlled amounts of metal, resulting in micron-level thickness of surface removal as we can see in video from figure 2.1.3.

Fig. 2.1.3 Electrolytic Sample Preparation Polishing [2.1.3]

Important parameters for a successful electropolishing process

The amount of metal surface removal is controlled by the following factors:

- Electrolyte chemical composition;
- Temperature of electrolyte (typically 76 °C – 82 °C);
- Length of time of exposure to electrical current;
- Density of electrical current (varies by electrolyte; typical range is 140 – 250 amps/m^2);
- Composition of metal alloy undergoing electropolishing.

In the process of electropolishing, burrs and other peaks of surface roughness attract greater electrical current density and are the first to erode, in a phenomenon known as anodic levelling. The preferential removal of protruding portions of the surface structure leads to a smoother metal surface.

Electrolyte Flow and Agitation

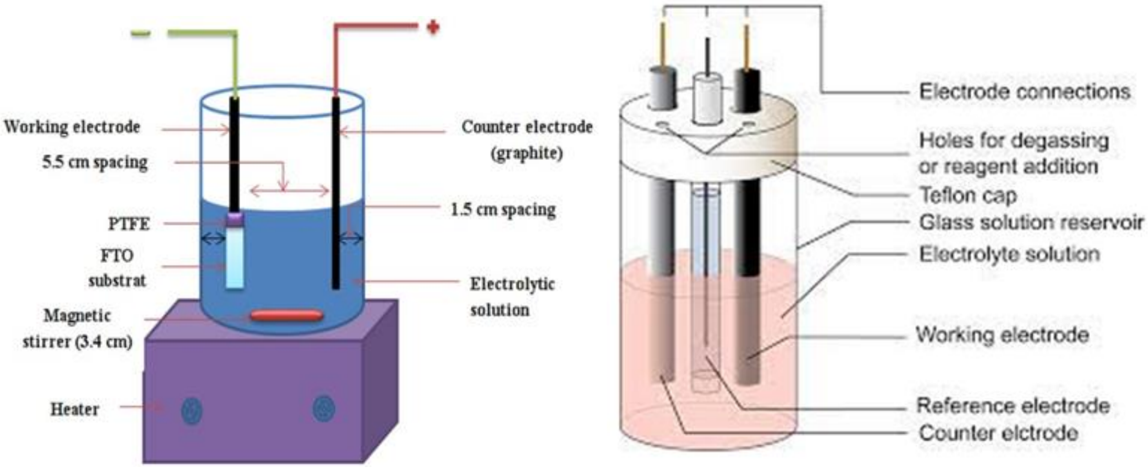
Maintaining a consistent and controlled electrolyte flow is essential for effective electrolytic polishing. The flow of the electrolyte helps to remove debris and prevent the accumulation of dissolved material on the sample's surface, which could hinder the polishing process. It also promotes uniformity in material removal, ensuring a consistent surface finish.

Mixing techniques, such as mechanical stirring or ultrasonic mixing, can further enhance the electrolyte flow and improve polishing results. Mixing helps to dislodge air/gas bubbles, promote uniform distribution of the electrolyte, and enhance mass transfer at the sample surface. This ensures that the entire surface is exposed to the electrolyte, preventing localized effects and achieving a more uniform removal of material.

Electrode Configuration

The configuration and arrangement of electrodes in electrolytic polishing influence the distribution of electrical current and material removal across the sample's surface. The electrodes serve as conductors for the electrical current, and their placement and geometry can be adjusted to control the polishing process.

Typically, two types of electrode configurations are used: single electrode and dual electrode. In a single electrode configuration, the specimen itself acts as one electrode, while a separate counter electrode is used. In a dual electrode configuration, both the specimen and a separate auxiliary electrode are immersed in the electrolyte solution. The choice of electrode configuration depends on the specific requirements of the sample and the desired polishing results. In the figures below, unit etching with two and three electrodes are presented.



a) b)

Fig. 2.1.4. Electrode configuration with: a) 2 electrodes, b) three electrodes [2.1.4].

Current Density and Voltage Parameters

The current density and voltage parameters are critical variables that govern the material removal rate and surface finish during electrolytic polishing. The current density refers to the amount of electrical current applied per unit area of the sample's surface. It determines the rate of material removal, with higher current densities resulting in faster removal and potentially rougher surfaces. The current density is controlled by adjusting the applied voltage and the surface area of the electrode.

The applied voltage also influences the electrolytic polishing process. It determines the driving force for the electrochemical reactions and material removal. By adjusting the voltage, researchers can control the material removal rate, surface roughness, and the selection of the polishing process. Optimizing the current density and voltage parameters requires careful consideration of the sample material, electrolyte composition, and desired surface characteristics. It often involves iterative testing and adjustment to achieve the desired results while avoiding excessive material removal or surface damage.

How Much Material Does Electropolishing Remove?

The amount of material that is removed by electropolish systems is directly proportional to the run time / cycle time and the amp-minutes that pass through the surface area of the part. All of these variables can be controlled to create a consistent, reliably controlled surface finish.

The thickness of the removed material layers is commonly within 0.0008 mm to 0.0018 mm for most deburring and polishing applications after machining. With precise monitoring, electropolishing can remove as little as 0.00025 mm. For an electropolishing process with longer cycle time and amp-minutes, as much as 0.008 mm to 0.0125 mm of material may be removed. The longer the electrical current is applied, the more material is removed but the less uniform is the resulting surface finish. Thus, the process is best applied to micro burrs, or those that protrude less than about 0.0038 mm from the part surface. Examples of material removal specifications by application are shown in the table 2.1.1.

Table 2.1.1. Material removal specifications by application

Metal alloy	**Material removal control in the process**	**Minimum/ cleaning**	**Microfinish**	**Microdeburring**	**Corrosion resistance**
Titanium	0.0254 mm per surface	0.0254 mm per surface	0.0635 mm per surface	0.127 per surface	N/A
Nitinol	0.0254 mm per surface	0.0254 mm per surface	0.0635 mm per surface	0.127 per surface	N/A
Brass	0.0635 mm per surface	0.0635 mm per surface	0.127 - 0.254 mm per surface	0.127 - 0.254 mm per surface	N/A
Copper	0.0635 mm per surface	0.0635 mm per surface	0.127 - 0.254 mm per surface	0.127 - 0.254 mm per surface	N/A
Steel	0.0635 mm per surface	0.0635 mm per surface	0.127 - 0.254 per surface	0.127 - 0.254 mm per surface	N/A
Stainless Steel	0.0254 mm per surface	0.0254 mm per surface	0.127 per surface	0.127 - 0.254 mm per surface	0.127 mm per surface
Aluminium	0.0635 mm per surface	0.0635mm per surface	0.127 - 0.254 mm per surface	0.127 - 0.254 mm per surface	N/A

Electropolishing is a controlled electrochemical process that selectively removes surface material, typically a few micrometres to tens of micrometres in depth. The rate of material removal in electropolishing is influenced by factors such as the composition and concentration of the electrolyte, temperature, current density, and duration of the process. These parameters can be adjusted to achieve the desired surface finish and to control the amount of material removal. It's important to note that electropolishing is primarily used to improve surface finish, enhance cleanliness, and reduce surface defects rather than to remove significant amounts of material. The process is typically more focused on levelling and smoothing the surface, removing imperfections, and improving surface aesthetics. To determine the precise amount of material removed during electropolishing for a specific application, it is recommended to consult the technical specifications provided by the equipment manufacturer or conduct experimental testing under controlled conditions.

Electrolytic preparation

Electrolytic preparation uses an electrolytic reaction cell containing a liquid electrolyte with two electrodes: an anode and cathode. The sample to be polished / etched forms the anode. Current is applied which forces the metal of the anode to dissolve, to move and deposit itself on the cathode as a coating. The electrodes are connected to an external power supply and voltage is applied to cause reaction within the cell.

Shown below in figure 2.1.5 is the characteristic curve for an electrolytic cell. This curve is dependent on the electrolyte used and will vary for different electrolytes. Control of the voltage and current density at the anode, plus electrolyte composition, temperature and mixing are all critical in achieving the desired polishing/etching characteristics. Establishing adequate control of these parameters can be difficult and further, many of the electrolytes are hazardous or even explosive. In the case of the latter, temperature control is critical. Electrolytic polishing or etching without the necessary experience and safety measures in place shall not be attempted.

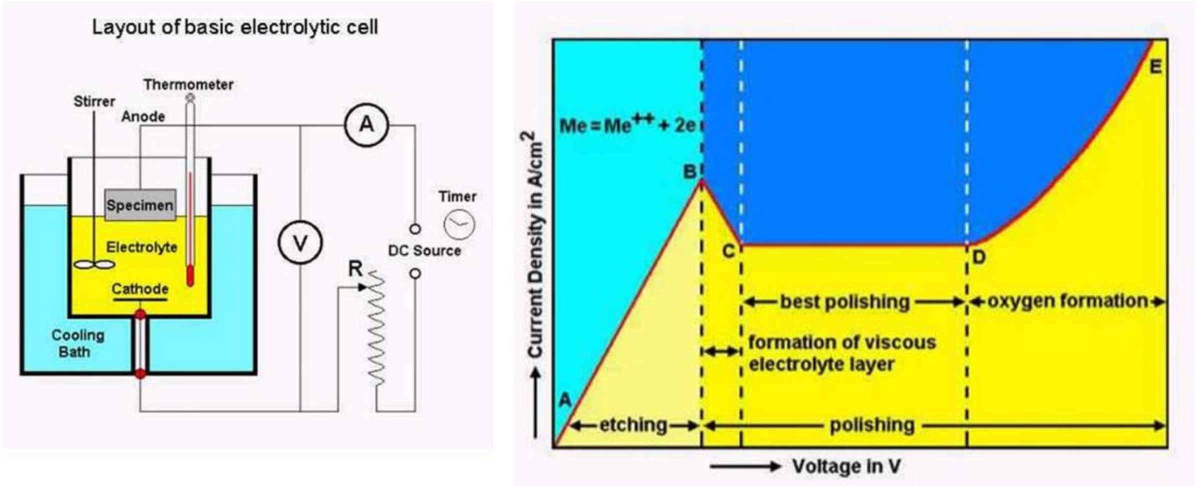


Fig. 2.1.5 Characteristic curve for an electrolytic cell [2.1.5]

Factors controlling etching/polishing characteristics include:

- Electrolyte composition;
- Electrolyte temperature;
- Electrolyte stirring;
- Area to be polished/etched (current density);
- Voltage.

Benefits of Electropolishing

Metal pipes are used mostly to transport gases and liquids. There are domestic, low cost and less sensitive applications like water supply lines to high cost, commercial, high sensitive applications such as medical equipment. The different applications would require different standards and qualities. In addition to the mechanical and chemical qualities sometimes the dimensions must be accurate.

The pipes are made to be seamless through drawing procedures to overcome this issue. The seamless pipes are dimensionally accurate and can have less absolute roughness. But when the application is extremely sensitive and needs the inside of the pipe to be as clean and as smooth as possible, electropolishing is applied. The procedure is carried out by a process called electrolysis where the product loses atom by atom on the surface and comes to a polished finish.

Table 2.1.2 Benefits of Electropolishing

Benefit	**Description**
Decorative appearance	The bright and shiny finish, often highly reflective, is aesthetically pleasing and uniform, without fine lines that occur with abrasive polishing.
Microscopic smoothness	Levels peaks and valleys and provides up to 50% improvement in surface roughness (Ra). Surface roughness refers to the texture or appearance of an external surface, capturing its irregularities and microscopic variations in height or depth. It plays a crucial role in determining how surfaces interact with each other, affecting friction, wear, and sealing properties. Conversely, internal roughness refers to the condition of a surface within a material or component, typically associated with imperfections or irregularities that are not visible externally. Internal roughness, such as microstructural flaws or inconsistencies, can impact the material's structural integrity and mechanical properties but is often more challenging to detect or evaluate compared to surface roughness, often requiring specialized techniques for assessment. Unlike mechanical finishing, electropolishing does not smear, bend, stress or fracture the crystalline metal surface.
Deburring	On a microscopic level, electropolishing removes small pieces of displaced surface material that can snag and break.
Ultra-clean finish	Creates a clean surface that is easier to sterilize. Removes embedded contaminants that can lead to corrosion.
Removes surface defects	Increases operational life by removing cracks from the surface, thus eliminating sites where corrosion can take hold. Helps parts that need to flex and bend to last longer.

Reduces friction	Prolongs life of parts in constant motion. Reduces galling on threaded parts, improving sealing.
Eliminates oxide scale	Removes weld discoloration from heat tint and oxide scale.
Good for fragile parts	Works on delicate parts that might be damaged in a tumbling process. Can replace some mechanical finishing techniques such as grinding, buffing, milling and blasting.
Good for complex parts	Can polish areas that are inaccessible by other polishing methods without causing damage. Effectively treats tiny surfaces on complex components.
Size control	Can be used to slightly reduce size of parts if necessary.

However, the process has its downfalls as well. Even with this method, the absolute roughness persists to some extent. The time taken for the process could be tolerated but it is costly, and some of the acids and other chemicals used would have an impact on the finished product. While removing the surface layers, the process removes impurities as well, resulting in a higher corrosion resistance.

Etching

Directly polished surfaces can be inspected using EBSD (electron backscatter diffraction), but in many cases the pattern quality is improved by etching, as it is shown in figure 2.1.6. Additionally, etching delineates the grain structure, which is of obvious benefit. However, etching may attack a second phase preferentially, or attack grain boundaries excessively. Caution should be exercised when choosing and using etchants. Inspect the sample surface using a light microscope before and after etching to assess the effect. Materials that are difficult to polish may benefit from repeated etching and re-polishing. This method can expose an undamaged surface suitable for EBSD when conventional polishing and etching fails to achieve an adequate surface. Using special acid or alkali resistant cloths, it is also possible to add dilute etchants to the polishing cloth during polishing. Although this can be effective, it can be difficult to control.

Any etchant that is used must dissolve the specimen surface in an even manner, and not leave behind oxides or reaction product layers. Such layers can completely suppress diffraction.

Many etchants listed in metallographic textbooks are 'contrast etches' which rely on the formation of different thickness oxide layers to generate colours visible using a light microscope. Therefore, such etchants are generally not suitable for EBSD.

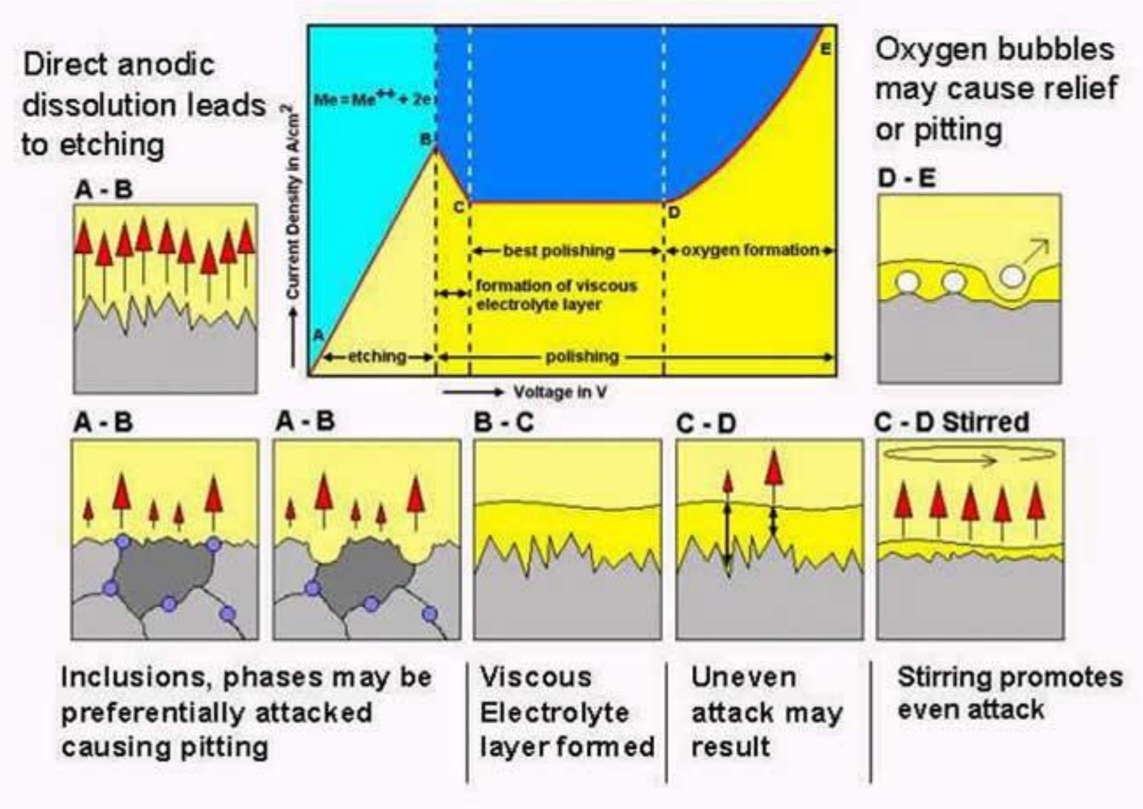


Fig. 2.1.6 Sample surface function to voltage and current density [2.1.5]

Common Applications for Electropolishing

Since the 1950s, scientists have commercially used the combination of electricity and chemicals to improve the surface finish of metals. It is a common finishing procedure across a wide variety of industries, including the following:

- Surgeons and dentists use electropolishing to keep their tools clean and functioning well.
- Food, beverage and pharmaceutical processors benefit from the sterilizing features of electropolishing.
- Appliance manufacturers use electropolishing in appliances like refrigerators and washing machines to make parts last longer and look better.
- Aerospace manufacturers use electropolishing on flight-critical parts to reduce friction and ensure top performance.
- Semiconductor manufacturers use electropolishing with fragile, irregularly shaped products.
- Automakers and the racing industry electropolish gears and fuel lines to reduce friction, boost performance and ensure long life of parts.

Examples of metal parts commonly electropolished:

- Piping and tubing
- Fasteners

- Springs
- Wire racks
- Blades

Electrolytic polishing finds wide application in industries such as aerospace, medical, food, automotive and materials research, helping to assess grain boundaries, surface defects and phase distribution.

Which Metals Work with Electropolishing?

Electropolishing Stainless Steel: is the most common use of electropolishing. As it is presented in figure 2.1.7. Electropolished stainless steel has a smooth, shiny, ultra-clean finish that resists corrosion. Although nearly any metal will work, the most commonly electropolished metals are 300- and 400-series stainless steel.

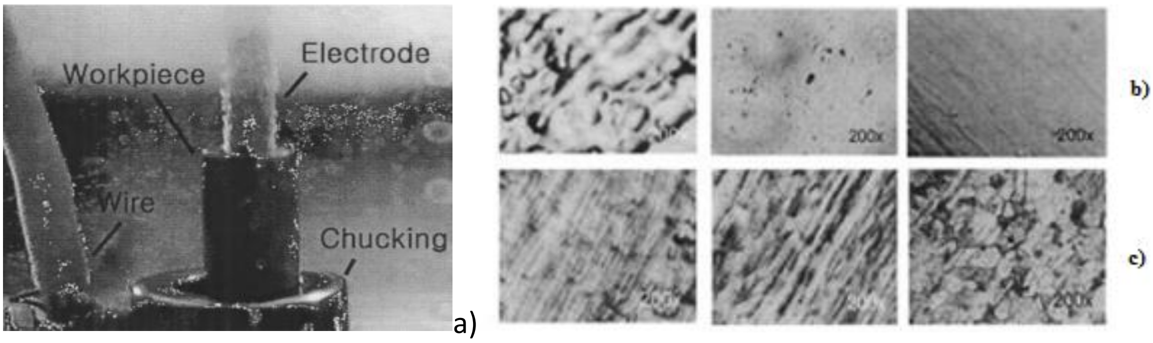


Fig.2.1.7 Electropolishing for Stainless Steel (STS316L) pipe: a) The experimental set-up, b) optical micrographics, c) metallographic micrographs [2.1.6]

Parts made from 400-series stainless steel (SS) have the advantage of increased hardness and strength at the cost of increased susceptibility to corrosion. By electropolishing stainless steel, engineers can use 400-series SS and still provide protection from corrosion. If using 400-series stainless steel, making the move to electropolish stainless steel parts can give you the best of both worlds – corrosion resistance, hardness and strength.

Electropolishing of Stainless Steel after Welding can remove the tough oxide scale and heat tint that can occur in stainless steel after welding. Flash electropolishing is a technique to remove scale or oxidation from the surface of a heat-treated metal. For example, some surgical instruments made of stainless steel (SS) 17-4 will appear brown after welding and/or heat treatment. To remove the brown appearance, parts are flash electropolished. The “flash” refers to how quickly the polishing takes place: only 10 seconds of electropolishing compared to a minimum of 2 minutes for most applications.

Flash electropolishing stainless steel is the only way to remove scale without a highly labour-intensive process, and it has the added benefit of achieving consistent results. In contrast, using blasting to remove scale on an SS 17-4 instrument will take 4 times as long and often yields inconsistent, wavy results in the surface finish after cleaning. Note that flash electropolishing is distinct from “flash attack,” a defect that can occur in a passivation process.

In addition to stainless steel, other metals compatible with electropolishing include:

- Aluminium;
- Brass;
- Carbon steels;
- Cobalt chrome;
- Copper alloys (figure 2.1.8);
- Nickel alloys;
- Titanium;
- Nitinol.

Only limited success can be achieved with cast metals, such as alloys containing significant amounts of silicon, sulphur or carbon. Electropolishing aluminium and zinc die castings does not work well, but most other alloys of aluminium yield good results. Titanium and nitinol are also compatible with electropolishing; however, the electrolyte solution for these metals is highly flammable and requires cooling systems to prevent the electrolyte from combusting at room temperature.

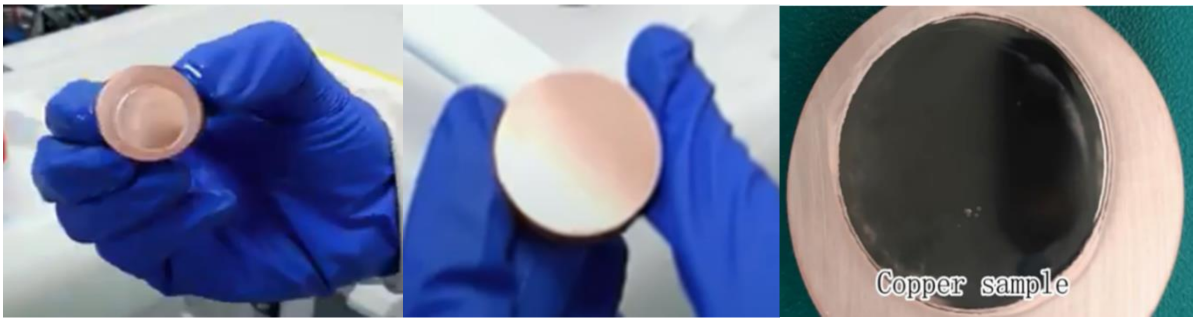


Fig. 2.1.8. Electropolishing sample of Copper [2.1.7]

In the table 2.1.3 are presented major parameters involved in the process of Electropolishing for stainless steel and in table 2.1.4 for copper.

Table 2.1.3 Common parameters of electropolishing process on stainless steel workpiece:

Electrolyte	**Stainless steel type**	**Temperature**	**Current density range**	**Voltage range**
Phosphoric acid, sulphuric acid and glycerol mixture	304	30 to 700 °C	0.5 to 1A/cm^2	Not specified

Phosphoric acid, sulphuric acid and glycerol mixture	304	5 to 800 °C	0.5 to 1A/cm^2	Not specified
Phosphoric acid, sulphuric acid and glycerol mixture	304	30 to 700 °C	0.5 to 1A/cm^2	Not specified
Phosphoric acid, sulphuric acid and water	Fe13Cr	25 to 700 °C	0.01 to 0.225 A/cm^2	-0.81 to 1.45 V
Four electrolytes (mixtures of sulphuric acid, phosphoric acid and glycerol)	316L	50 to 800 °C	Not specified	Voltage scanning rate 5 mV/s
Mixtures of sulphuric acid, phosphoric acid and water with/without glycerine	316L	50 to 950 °C	0.5 to 2,5 A/cm^2	Not specified
Mixtures of sulphuric acid, phosphoric acid and water glycerol	316L	65 to 700 °C	Not specified	2.5 to 10V

Table 2.1.4 Common parameters of electropolishing process on copper workpiece:

Electrolyte	Temperature	Current density range	Voltage range
Anhydrous orthophosphoric acid	250°C	Not specified	Not specified
Orthophosphoric acid solution (non-agitated solution)	-5,60°C to 530°C	0.2 to 1.5 A/dm^2	0,7 to 0,9 V
Orthophosphoric acid/water solution (agitated solution)	-5,60°C to 530°C	0.2 to 1.5 A/dm^2	Not specified
Orthophosphoric acid/water solution	250°C	0.0625 A/cm^2	0.8 to 1.2 V
Phosphoric acid/water solution with/without glycerol, ethylene glycol	170 to 250 °C	2 to 4.2 mA/cm^2	0.25 to 1.5 V
Phosphoric acid/ copper phosphate/water solution	250°C	50 to 1000 mA/cm^2	0.2 to 0.8 V

Orthophosphoric acid/sulphuric acid/water solution	250°C	0.1 A/cm^2	1.8 to 2.2 V

Concluding remarks

Electropolishing is a distinctive metallic surface treatment used to remove rough irregularities from metal surfaces. Although the theory is straightforward, multiple parameters influence the workpiece's surface current density and electropolishing results, such as electrolyte type, temperature, concentration, voltage, anode/cathode distance, etc. Precise tuning of these parameters is essential to achieve the desired surface finishes for metal workpieces of different shapes and sizes, with particular attention given to the electropolishing electrolyte.

Over time, the metals requiring electropolishing have evolved, with stainless steels now comprising 95% of the current market compared to copper and copper alloys in the past. Empirical observations initially shaped the technique, but several theories have been developed to understand the underlying mechanisms.

For optimal electropolishing results, workpiece surfaces often undergo both pre-treatment and post-treatment processes. Evaluation of surface finishes is done through various microscopic techniques, depending on the required surface roughness and scale of observation.

The advantages of electropolishing include enhancing metal surface smoothness, appearance, corrosion resistance, removal of contaminants, and refinement of the surface oxidation layer.

Beyond decorative purposes, electropolishing has found broader applications in various industries, including food, beverage, pharmaceutical, biomedicine, electronics, semiconductors, automotive, aerospace, and more. As time progresses, electropolishing will continue to play a crucial role in processing diverse metal surface finishes.

2.1.2. The Principle of Chemical Polishing

Chemical polishing, also known as chemical etching, is a surface treatment process that utilizes chemical reactions to remove material from a metal surface selectively. The purpose of chemical polishing is to produce a mirror-like finish, eliminate surface defects, and enhance the visibility of microstructural features.

The chemical processing is highly efficient to remove a thin layer of material from the entire immersed surface of a part, resulting in a smooth, flawless surface, revealing microstructural details for further analysis, and provides a safe and cost-effective solution by simply immersing parts in special tanks. This process effectively removes particles, pores, debris, contaminants and structural damage present in the surface layer. In particular, it achieves higher removal rates along the tops and edges, effectively removing burrs down to a size of about 0.5 mm. The removal rate can be adjusted according to the concentration of chemicals in the tank, the temperature of the solution and the current on the part surface. By controlling these parameters, repeatability of processing can be guaranteed.

Figure 2.1.9 illustrates the process of chemical polishing. When a metal surface is immersed in a chemical polishing solution, it dissolves into metal ions, and these ions diffuse within the polishing solution. In situations where the metal dissolves at a faster rate than ion diffusion, the metal ions accumulate at the concave regions. As depicted in Figure 2.1.9 (a), the density of cations, primarily hydrogen ions (H+), in the polishing solution decreases at the concave areas. This results in a smooth surface since the etching rate at the convex portions is higher than at the concave parts, as shown in Figure 2.1.9 (b) and (c).

Moreover, chemical polishing can effectively remove substances that were not eliminated by precision cleaning, as indicated in Figure 2.1.9 (a) and (b). These persistent substances may include adherent impurities like chemically altered surface layers like oxidation scales formed during welding, as represented in Figure 2.1.9 (a) – α, β, and γ.

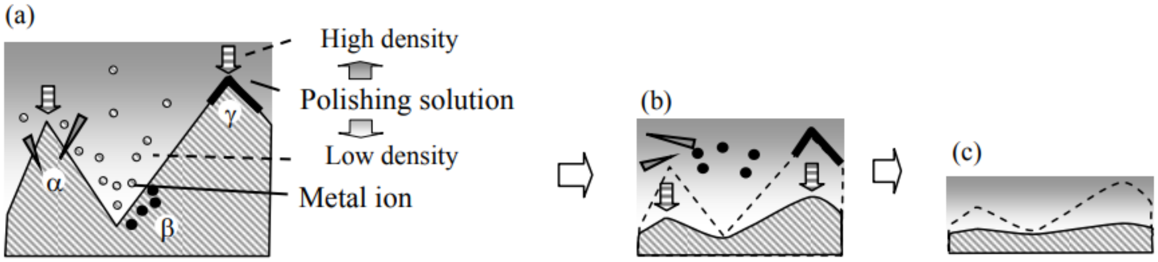


Figure 2.1.9 Representation of the Chemical Polishing principle [2.1.8]

Chemical polishing is commonly used as a quick method to achieve satisfactory results rather than aiming for a flawless surface. However, in cases where preparing a deformation-free surface using other techniques proves challenging, especially when dealing with soft metals or when encountering various difficulties, chemical polishing may offer the most suitable approach for preliminary or final preparation.

For refractory metals, chemical polishing is often conducted after mechanical polishing to enhance polarised light response (e.g., for Zr, Hf) or to eliminate minor deformations (e.g., Nb, Ta, V). The technique is designed to produce a smooth and flat surface by selectively dissolving or etching specific microstructural constituents while leaving others mostly unaffected.

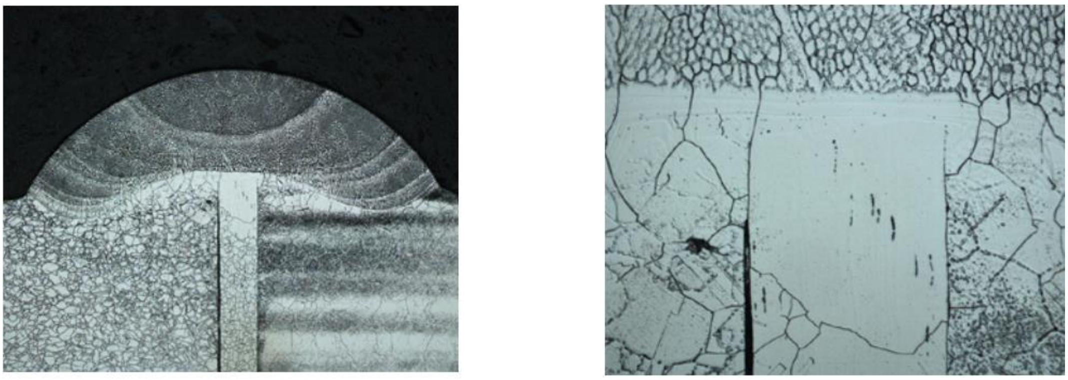
Achieving the desired material removal rate, selectivity, and surface quality relies on adjusting the specific etchant composition and process parameters. Chemical polishing represents a valuable method for refining surfaces in challenging situations, ensuring optimal results for various applications. In figure 2.1.10 an electrical system for chemical polishing is presented.

Fig. 2.1.10 Chemical Polishing System [2.1.9]

Chemical deburring and polishing processes are used in the following applications: individual components in immersion tanks using baskets or suspension equipment, pipes and long drilled holes by pumping the solution through the components, small parts, mostly loose as bulk, provided they cannot damage each other; wires and strips by continuous processing on reel-to-reel equipment or in a loose bundle, large containers by means of spraying or sprinkling.

Acid-Based Etchants

Acid-based etchants are commonly used in chemical polishing due to their high reactivity and ability to dissolve a wide range of metallic materials as in figure 2.1.11. Examples of acid-based etchants include nitric acid (HNO3), hydrochloric acid (HCl), and sulfuric acid (H2SO4). These etchants are effective in removing oxides, scale, and surface impurities. Acid-based etchants are particularly useful for materials such as stainless steels, aluminium alloys, and titanium alloys. The composition and concentration of the acid-based etchants can be adjusted to achieve different levels of selectivity and surface finishes.



Mechanically polished and electrolytically etched section through micropulse Tungsten Inert Gas weld in Inconel 625 alloy. Etched in 10% oxalic acid; 10 Volts for 10 secs. Bright field, 2.5x.

Same material. Microstructure in the weld consists of primary solid solution and fine irresolvable secondary phase. Section shows pronounced grain coarsening of base material in proximity of fusion face. Bright field, 10x.

Fig. 2.1.11 Inconel 625 alloy before and after the chemical attack [2.1.10]

Alkaline-Based Etchants

Alkaline-based etchants are often employed for chemical polishing of metals and alloys. Sodium hydroxide (NaOH) and potassium hydroxide (KOH) are common alkaline-based etchants used in metallography. Alkaline etchants are effective in removing oxide layers and can provide smooth and shiny surfaces. Alkaline-based etchants are suitable for materials like stainless steel like in figure 2.1.12, copper, brass, and certain aluminium alloys. The concentration and temperature of the alkaline etchants can be adjusted to control the etching rate and selectivity.

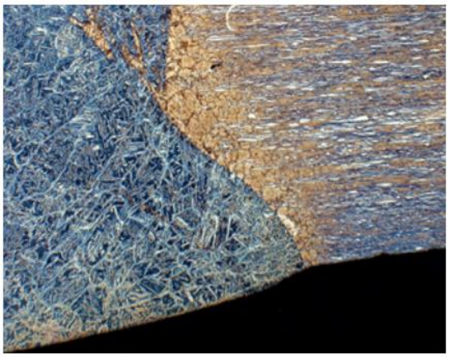
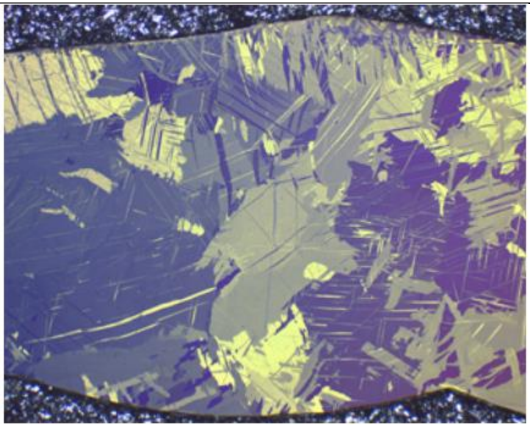
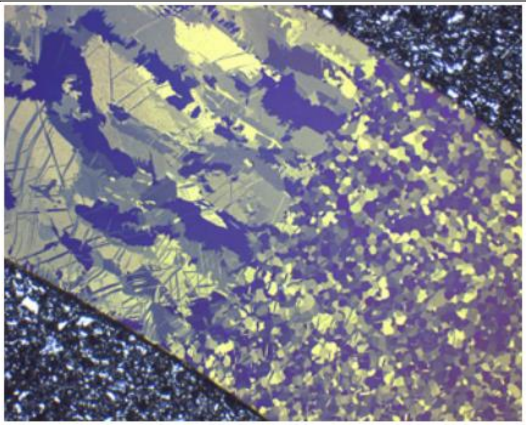
	
Weld-base metal interface on Duplex stainless steel. Mechanically prepared; electrolytically etched in 40% aqueous sodium hydroxide solution. Bright Field, 25x.	Same material. Weld microstructure consists mainly of delta ferrite and austenite. Bright Field, 200x.

Fig. 2.1.12 Duplex stainless steel before and after the chemical attack [2.1.10]

Mixed Etchants

Mixed etchants consist of a combination of chemical compounds, offering a broader range of etching capabilities compared to individual acid or alkaline etchants. Mixed etchants can provide improved selectivity and controlled material removal rates for specific microstructural constituents, as presented in figure 2.1.13. One example is the Kroll's reagent, which is a mixture of nitric acid and hydrofluoric acid. It is commonly used for the chemical polishing of titanium and titanium alloys, providing a bright and reflective surface finish.

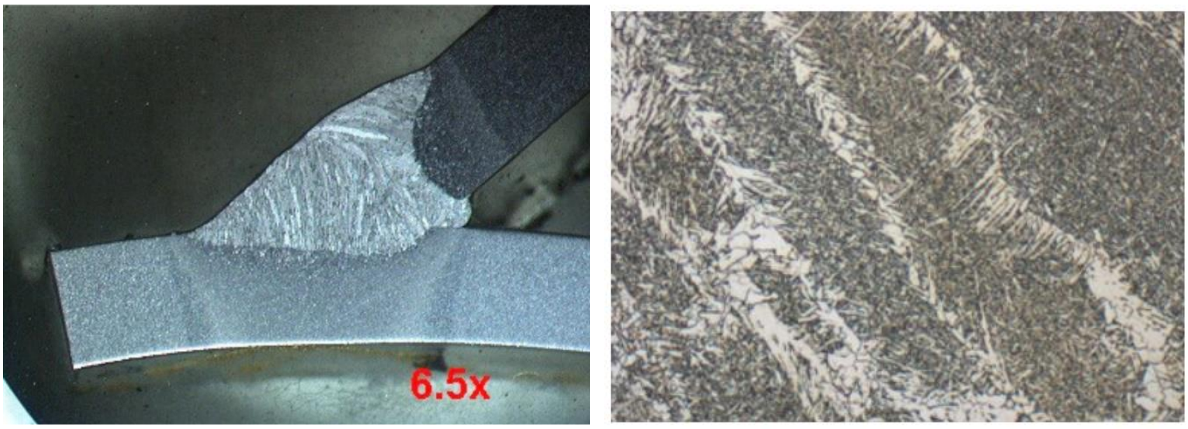
	

Weld area. Mechanically polished weld section of full penetration weld in grade 1 titanium. The weld microstructure consists of Widmanstätten α phase. Polarised light with a sensitive tint plate (λ1/4-plate), 50x.	Same material. Parent-heat affected zone interface. Polarised light with a sensitive tint plate (λ1/4-plate), 50x.

Fig. 2.1.13 Grade 1 titanium before and after the chemical attack [2.1.10]

Special Etchants for Specific Alloys

Certain alloys may require specialty etchants tailored to their unique microstructures and compositions. These specialty etchants are designed to selectively reveal specific microstructural features or phases. Examples include Nital (figure 2.1.14), which is a mixture of nitric acid and ethanol used for revealing grain boundaries in steels, and Beraha reagent, used for revealing grain boundaries in copper and copper alloys.



Ground and etched macro section through a MIG weld in carbon steel, etched with 4% Nital. Bright field, 6.5x

Polished and etched micro section through MAG welded carbon steel. Microstructure consists of acicular and primary ferrite. Etched with 2% Nital. Bright field, 200x

Fig. 2.1.14 Carbon steel before and after the chemical attack [2.1.10]

Temperature and time play important roles in chemical polishing. The etching rate is highly dependent on the temperature of the etchant. Higher temperatures generally result in faster material removal rates, although excessively high temperatures may lead to uncontrolled and non-uniform etching. The etching time is another critical parameter that affects the extent of material removal and surface finish. Longer etching times can result in increased material removal and smoother surfaces. However, the etching time should be carefully controlled to avoid over-etching and excessive removal of the desired microstructural features. Optimizing the temperature and etching time parameters is crucial to achieve the desired surface finish, selectivity, and preservation of microstructural details.

In general, a ground specimen is immersed in the polishing agent, or swabbed with the solution, until a polish is obtained, and it is then etched or washed and dried, as appropriate. Reagents are listed in Table 2.1.5 [2.1.11].

Table 2.1.5: Chemical polishing reagents

Metal	Reagent*	Quantity	Time	Temp. (0C)	Remarks
Aluminium and alloys	Sulphuric acid (1.84)	25 ml	30 s-2 min	85	Very useful for studying alloys containing intermetallic compounds, e.g., Al-Cu, Al-Fe, Al-Si alloys
	Orthophosphoric acid	70 ml			
	Nitric acid	5 ml			
Beryllium	Sulphuric acid (1.84)	1 ml	Several minutes	49-50	Rate of metal removal is approx. 1 µm/min 1. Passive film formed may be removed by immersion for 15-30 s in 10 % sulphuric acid
	Orthophosphoric acid (1.75)	14 ml			
	Chromic acid	20 g			
	Water	100 ml			
Cadmium	Nitric acid (1.4)	75 ml	5-10 s	20	Cycles of dipping for a few seconds, followed immediately by washing in a rapid stream of water are used until a bright surface is obtained
	Water	25 ml			
Copper	Nitric acid	33 ml	1-2 min	60-70	Finish is better when copper oxide is absent
	Orthophosphoric acid	33 ml			
	Glacial acetic acid	33 ml			
Copper alloys	Nitric acid	30 ml	1-2 min	70-80	Specimen should be mixed
	Hydrochloric acid	10 ml			

	Orthophosphoric acid	10 ml			
	Glacial acetic acid	50 ml			
Copper-zinc alloys	Nitric acid (1.40)	80 ml	5 s	40	Use periods of 5 s immersion followed immediately by washing in a rapid stream of water. Slight variations in composition are needed for α-β and β-γ brasses to prevent differential attack. With β-γ alloys, a dull film forms and this can be removed by immersion in a saturated solution of chromic acid in fuming nitric acid for a few seconds followed by washing.
	Water	20 ml			
Germanium	Hydrofluoric acid +	15 ml	5-10 s	20	-
	Nitric acid	25 ml			
	Glacial acetic acid	15 ml			
Hafnium	Nitric acid	45 ml	5-10 s	20	As for zirconium
	Water	45 ml			
	Hydrofluoric acid +	8-10 ml			
Iron	Nitric acid	3 ml	2-3 min	60-70	Dense brown viscous layer forms on surface; layer is soluble in solution. Low carbon steels can also be polished, but the cementite is attacked preferentially.
	Hydrofluoric acid (40%)+	7 ml			
	Water	30 ml			
Irons and steels	Distilled water	80 ml	15 min	35	The solution must be prepared freshly before use.
	Oxalic acid (100 gl-1)	28 ml			

	Hydrogen peroxide (30%)	4 ml			Careful washing is necessary before treatment. A microstructure is obtained similar to that produced by mechanical polishing, followed by etching with nital.
Lead	Hydrogen peroxide (30%)	80 ml	Periods of 5-10 s	20	Use Russel´s reagent to check that any flowed layer has been removed before final polishing in this reagent.
	Glacial acetic acid				
Magnesium	Fuming nitric acid	75 ml	Periods of 3 s	20	The reaction reaches almost explosive violence after about a minute, but if allowed to continue it ceases after several minutes, leaving a polished surface ready for examination.
	Water	25 ml			Specimen should be washed immediately after removal from solution.
Nickel	Nitric acid (1.40)	30 ml	1/2 – 1 min	85-95	This solution provides very good polish
	Sulphuric acid (1.84)	10 ml			
	Orthophosphoric acid (1.70)	10 ml			
	Glacial acetic acid	50 ml			
Silicon	Nitric acid (1.40)	20 ml	5-10 s	20	1:1 mixture also used
	Hydrofluoric acid (40%)+	5 ml			
Tantalum	Sulphuric acid (1.84)	50 ml	5-10 s	20	Solution is useful for preparing surfaces prior to anodising
	Nitric acid (1.40)	20 ml			

	Hydrofluoric acid (40%)+	20 ml			
Titanium	Hydrofluoric acid (40%)+	10 ml	30-60 s	-	Swab until satisfactory
	Hydrogen peroxide (30%)	60 ml			
	Water	30 ml			
	Hydrofluoric acid (40%)+	10 ml			Few seconds to several minutes function to alloy
	Nitric acid (1.40)	10 ml			
	Lactic acid (90%)	30 ml			
Zinc	Fuming nitric acid	75 ml	5-10 s	20	As for cadmium
	Water	10 ml			
	Chromium trioxide	20 g	3-30 min	20	Solution must be replaced frequently
	Sodium sulphate	1.5 g			
	Nitric acid (1.40)	5 ml			
	Water	100 ml			
Zirconium (also Hafnium)	Acid ammonium fluoride	10 g	1/2 -1 min	30-40	Rate of dissolution varies markedly with temperature and is about 20-60 µm min-1 in the given range
	Nitric acid (1.40)	40 ml			
	Flousilicic acid	20 ml			
	Water	100 ml			
	Nitric acid (1.40)	40-45	5-10 ml	-	Reaction is vigorous at air/solution interface, and specimen is therefore held near surface of liquid. Hydrogen peroxide (30%) can be used in place of water.
	Water	40-45 ml	Repeated		
	Hydrofluoric acid (40%)+	10-15 ml			

* Acids are concentrated, unless otherwise indicated.

Hydrofluoric acid produces irreversible bone damage and presents a range of other hazards. Even dilute hydrofluoric acid solutions should be handled with great care. Note that hydrofluoric acid attacks laboratory glassware.

The etchant's chemical composition determines the type of reactions that occur during chemical polishing. For example, acid-based etchants typically involve proton donation and the formation of soluble metal salts. Alkaline-based etchants, on the other hand, rely on hydroxide ion reactions to dissolve the surface. Surface passivation and inhibition mechanisms can also influence the etching process. These phenomena involve the formation of protective oxide layers or the presence of inhibitors that slow down or halt the etching reaction in certain regions. Controlling these mechanisms is crucial for achieving uniform material removal and desired surface finish.

Mechanisms of Chemical Polishing

The mechanisms of chemical polishing involve the selective dissolution or etching of different microstructural constituents based on their reactivity with the etchant. This selectivity depends on factors such as crystallographic orientation, chemical composition, and surface defects.

In some cases, the etchant preferentially attacks grain boundaries or other crystallographic planes, leading to the removal of material from these regions. Other etchants may target specific phases or constituents, resulting in their preferential dissolution. The selectivity is influenced by the etchant composition, temperature, and process parameters.

a. Microstructure Examination: Chemical polishing is widely used for microstructure examination. By providing a smooth and polished surface, it allows for clear observation and analysis of microstructural features, including grain boundaries, phases, inclusions, and precipitates. Microstructure examination facilitated by chemical polishing aids in understanding the material's composition, grain size, grain morphology, and distribution of phases. It is crucial for assessing the effects of processing, heat treatment, and alloying on the microstructure and subsequent mechanical properties.

b. Grain Size Determination: Chemical polishing plays an important role in determining grain size in metallic materials. By revealing the grain boundaries clearly, it enables accurate measurement and analysis of grain size using optical or electron microscopy. Grain size determination is essential for evaluating the material's mechanical properties, such as strength, hardness, and fatigue resistance.

c. Surface Relief Analysis: Chemical polishing is employed for surface relief analysis, particularly in materials with complex microstructures. By selectively removing material and enhancing the visibility of surface features, it allows for the examination of surface relief variations, such as surface undulations, deformation patterns, and texture. Surface relief analysis facilitated by chemical polishing aids in understanding surface deformation mechanisms, surface treatments' effectiveness, and the impact of mechanical and thermal processes on the surface morphology.

d. Second-Phase Particle Detection: Chemical polishing is valuable for the detection and analysis of second-phase particles within metallic materials. By preferentially dissolving the matrix phase, it enhances the visibility and contrast of the second-phase particles, enabling their identification, measurement, and characterization. Second-phase particle detection facilitated by chemical polishing provides understanding regarding distribution, size, morphology, and volume fraction of the second-phase particles. It provides valuable insights into the material's strength, toughness, wear resistance, and other mechanical properties.

Chemical polishing is a versatile technique with various applications in metallography. It enables microstructure examination, grain size determination, surface relief analysis, and second-phase particle detection. By selectively removing material and revealing microstructural features, chemical polishing contributes to the understanding and characterization of metallic materials.

2.1.3. Coloured etching. Definition and purpose

Introduction to Coloured Etching

Coloured etching, also known as tint etching or tinting, refers to the process of staining or colouring specific microstructural constituents within a metal sample using specialized etchants. The purpose of coloured etching is to enhance the visibility and contrast of different phases, inclusions, grain boundaries, and other microstructural features. By selectively colouring these constituents, the technique enables their identification, characterization, and analysis.

Coloured etching is a technique used in metallography to selectively stain or colour different microstructural constituents within a metallic material. By employing specific etchants, the technique enhances the visibility and contrast of various phases, inclusions, and grain boundaries, as shown in figure 2.1.15.

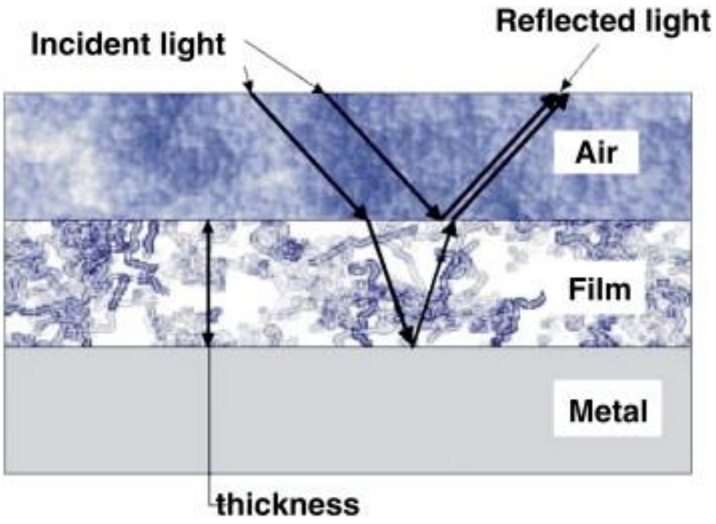


Fig. 2.1.15 Colour Etching: Etching solutions are chemically manipulated to achieve precise colouring by forming a stable, typically 0.04–0.5 µm thick layer on the specimen's surface. These thin films consist of oxides, sulphides, or complex molybdate compounds and exhibit a range of colours attributed to interference effects [2.1.12]

Coloured etching plays a significant role in the analysis and characterization of materials, providing valuable information about their microstructural features.

Mechanisms of Coloured Etching

Interaction of Etchants with Microstructural Constituents

The mechanisms of coloured etching involve the selective interaction of etchants with specific microstructural constituents, as shown in figure 2.1.16. Different etchants exhibit varying affinities towards different phases, inclusions, or grain boundaries, resulting in distinct colour formation.

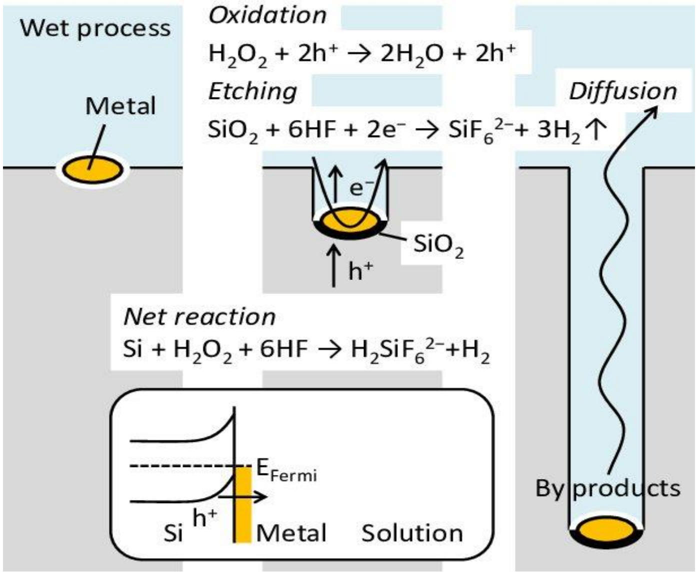


Fig. 2.1.16 Simplified mechanism for metal-assisted chemical etching (MaCE) of silicon in HF and H_2O_2 mixed solution [2.1.13].

The etchants may react differently with the constituents due to factors such as chemical composition, crystallographic orientation, and surface condition. These interactions can lead to the formation of coloured compounds or complexes on the surface of the microstructural features, making them more visible and distinguishable.

Principles of Colour Formation

Colour arises due to the absorption, reflection, or interference of specific wavelengths of light by the coloured compounds or complexes formed on the surface as shown in figures 2.1.17 (left) and 2.1.18 (right).

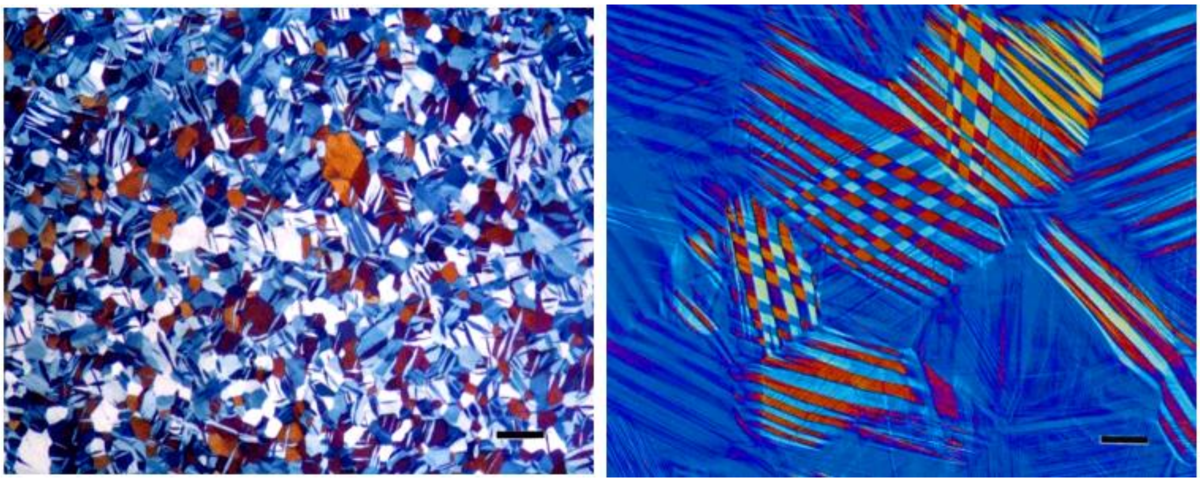


Fig. 2.1.17 (left) and 2.1.18 (right): Grain structure on high-purity Zr (left) that was hot worked and cold drawn (note mechanical twins) and viewed in polarized light and of Spangold (Au – 19Cu-5Al) that was polished and cycled through the shape-memory effect to produce martensite and Nomarski differential interference illumination was used to image the surface upheaval due to the shear reaction at the free surface. The magnification bars are 100 and 50 µm, respectively [2.1.14].

The colours produced by colour (tint) etchants are visible under bright-field illumination, and in many cases further enhancement is attained using polarized light. Colours are developed by interference in the same manner as with heat tinting or vacuum deposition. As noted, colour is determined by the thickness of the film, usually in the sequence of yellow, red, violet, blue, and green when viewed using white light. With anodic systems, the film forms only over the anodic phase, but its thickness can vary with the crystallographic orientation of the phase. For cathodic systems, because the film thickness over the cathodic phase is generally consistent, only one colour is produced, which will vary as the film grows during etching. Therefore, to obtain the same colour each time, the etching duration must be constant. This can be accomplished by timing the etch and observing the macroscopic colour of the specimen during staining.

Factors Affecting Colour Development

Several factors can influence the colour development in coloured etching. These include the composition and concentration of the etchant, temperature, etching time, and surface preparation of the sample, like in figures 2.1.19 and 2.1.20. Additionally, the presence of alloying elements, impurities, or specific crystallographic orientations can affect the colour intensity and contrast.

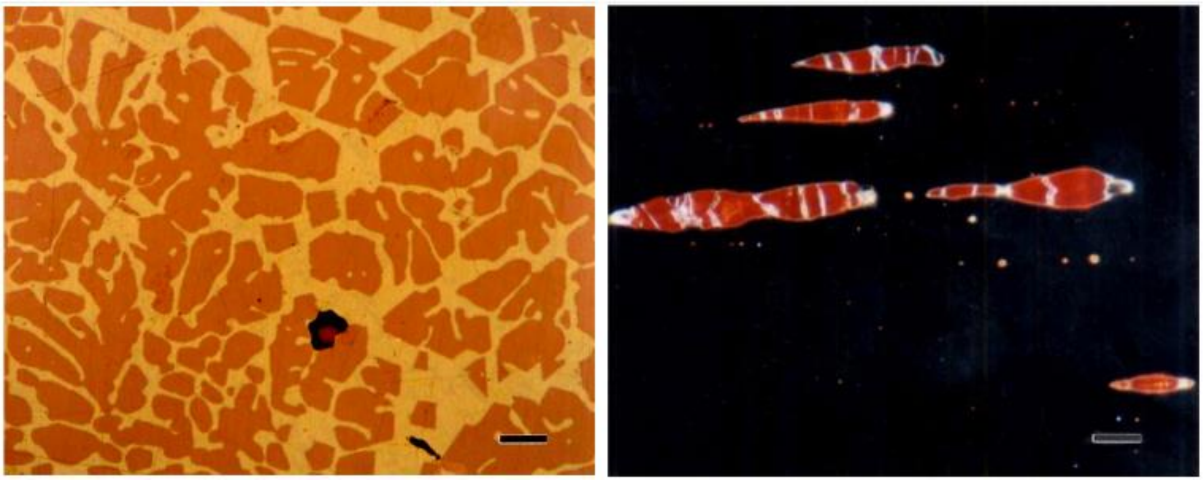


Fig. 2.1.19 (left) and 2.1.20 (right) showing natural reddish-purple colour of the $AuAl_2$ intermetallic (left) in bright field and cuprous oxide's characteristic ruby red colour in dark field illumination (tough-pitch arsenical copper specimen). The magnification bars are 50 and 10 µm, respectively [2.1.14].

Understanding these factors is crucial for achieving optimal colour development and contrast in coloured etching. Careful control and optimization of the etching parameters are necessary to obtain reliable and reproducible results.

Coloured etching is particularly useful when the microstructural features of interest are not clearly distinguishable in the un-etched sample. It allows for improved observation and examination under optical or electron microscopy, facilitating a better understanding of the material's composition, structure, and properties.

Many metals etched with standard reagents to reveal the grain boundaries often yield only a high percentage of the boundaries, rather than all of the boundaries. Colour etchants, however, reveal the grain structure completely. In the case of metals with annealing twins, it can be very difficult to rate the grain size when a standard etchant reveals a portion of the grain and twin boundaries. In fact, it can be quite difficult to make a precise measurement of the grain size, even manually, with such a specimen, as distinguishing between grain and twin boundaries (the latter must be ignored in the measurement) is not simple. However, with a colour etched microstructure it is relatively easy to separate grain from twin boundaries, at least manually. Further, the films grow as a function of crystal orientation. Therefore, one can detect any preferred crystallographic orientation by the narrowness of the colour range present. A wide range of colours is present in a random pattern in figure 2.1.21.

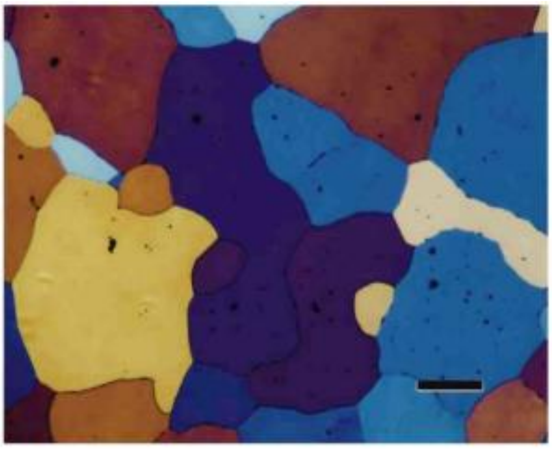


Fig. 2.1.21 Super-pure aluminium anodized crystal orientation is highlighted with the Barker reagent (30 Vdc, 2 minutes). The magnification bar is 200 µm long [2.1.14]

Super-pure aluminium anodized crystal orientation is random. If a narrow range of colours is present in the grains, then a preferred orientation is highlighted. Tint etch compositions are given at the end of the chapter. Specimen preparation must be better when using colour methods compared with black and white methods because the epitaxially grown films are sensitive to residual preparation-induced damage that was not removed. This level of preparation is required in image analysis work and can be easily obtained by a knowledgeable metallographer with the proper equipment. Electrolytic polishing is not required to get damage-free surfaces. The most common tint etchants are those that deposit a sulphide-based interference film on the specimen, as shown in Figures 2.1.22 and 2.1.23. These are the best-known tint etches and usually the easiest to use.

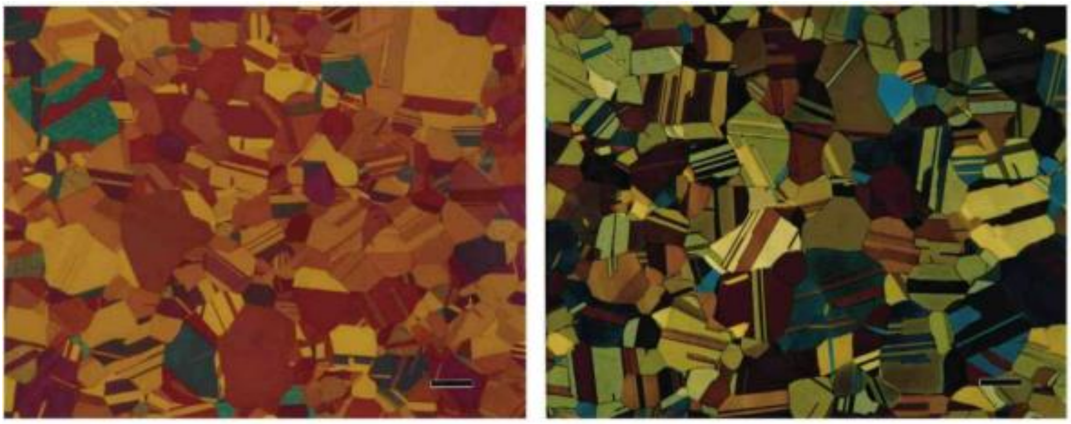


Fig. 2.1.22 and 2.1.23: FCC twinned grain structure of cartridge brass, Cu - 30% Zn, after cold reduction and full annealing, tint etched with Klemm's I (left) and Klemm's III (right) reagents and viewed with polarized light plus sensitive tint. Magnification bars are 200 µm long [2.1.14]

Klemm and Beraha have developed the most widely used sulphide-based tint etchants using sodium thiosulfate, $Na_2S_2O_3$, and potassium metabisulfite, K2S2O5. Klemm's I, II, III (Figures 2.1.22 and 2.1.23) and one of Berahas reagents utilize both ingredients (Figure 2.1.24), while Beraha recommends a range of HCl concentrations used with potassium metabisulfite (Figure 2.1.25) for etching a variety of iron-based alloys.

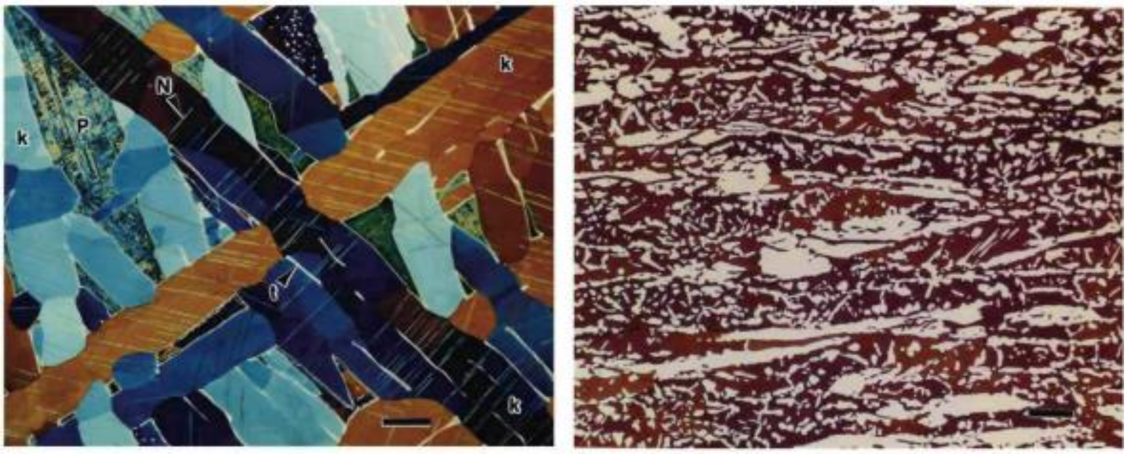


Fig. 2.1.24 and 2.1.25: Fine octahedrite grain structure of the Gibeon meteorite (left) revealed with Berahas reagent (100 mL water, 10g $Na_2S_2O_3$ and 3g $K_2S_2O_5$) and ferrite in 7 Mo PLUS duplex stainless steel plate revealed using Berahas reagent (85 mL water, 15 mL HCl, 1 g $K_2S_2O_5$). The magnification bars are 500 and 50 µm long, respectively [2.1.14].

These etchants can be used to colour ferrite and martensite in cast iron, carbon and low-alloy steels. The HCl-based reagents vary widely in concentration and can be used to colour the grain structures of stainless steels (Figure 2.1.26), Ni-based and Co-based alloys. Sodium metabisulfite has been used in a number of concentrations, from about 1 to 20 g per 100 mL water, and is a safe, reliable, useful colour etch for irons and steels (Figure 2.1.27).

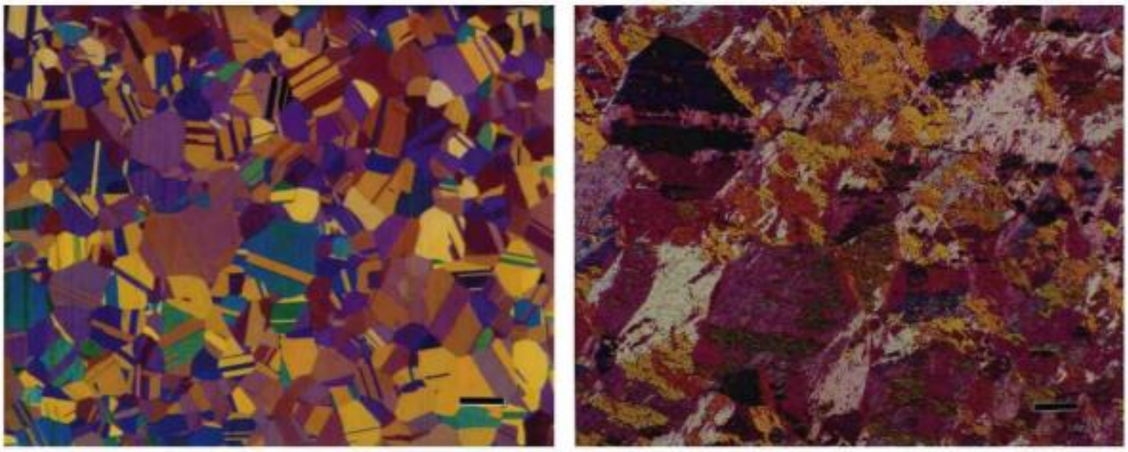


Fig. 2.1.26 and 2.1.27: FCC twinned grain structure of heading quality Custom Flo 302 stainless steel revealed using Berahas BI reagent and lath martensite grain structure of over-austenitized (1093 °C) AerMet 100 ultra-high strength steel revealed using 10% sodium metabisulfite. Both viewed with polarized light plus sensitive tint. The magnification bars are 100 µm long [2.1.14]

Beraha also developed etchants based upon sulfamic acid, a weak organic acid, which has not been used much, although they are quite useful, reliable and easy to employ. The sulfamic acid-based reagents are applicable to cast iron, low-carbon and alloy steels, tool steels, and martensitic stainless steels (Figure 2.1.27).

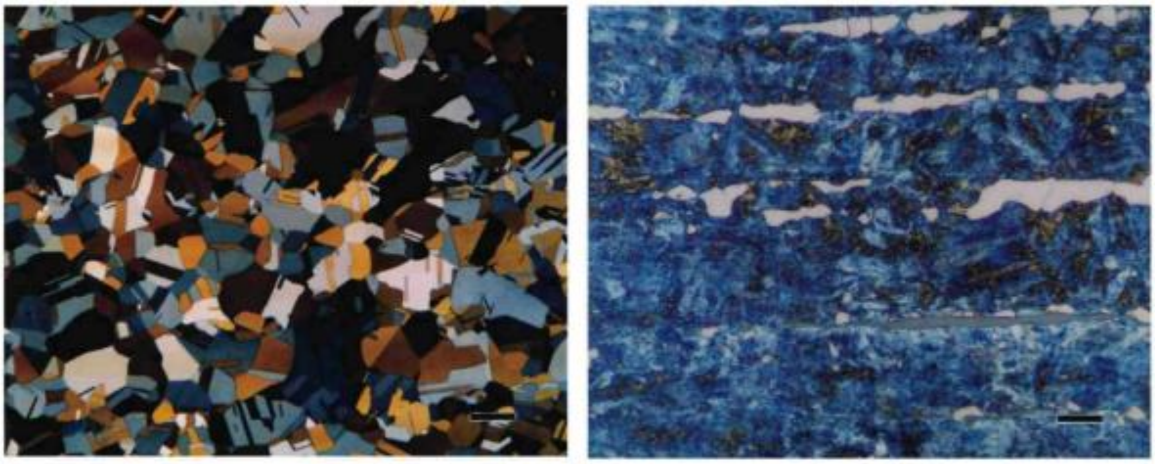


Fig. 2.1.27 and 2.1.28: Twinned FCC grain structure in Fe-39% Ni revealed by Beraha's sulfamic acid reagent (left) and tempered martensite grain structure of Carpenter Project 70 type 416 martensitic stainless steel revealed with Beraha's CdS reagent (right). The white grains are delta ferrite, and the gray inclusions are sulphides. Viewed with polarized light plus sensitive tint. The magnification bars are 100 and 200 µm long, respectively. [2.1.14]

Beraha also developed two rather specialized tint etches that deposit cadmium sulphide (Figure 2.1.28) or lead sulphide (Figure 2.1.29) films on the surfaces of steels and 4 copper-based alloys. These two etchants are quite useful, although tedious to make.

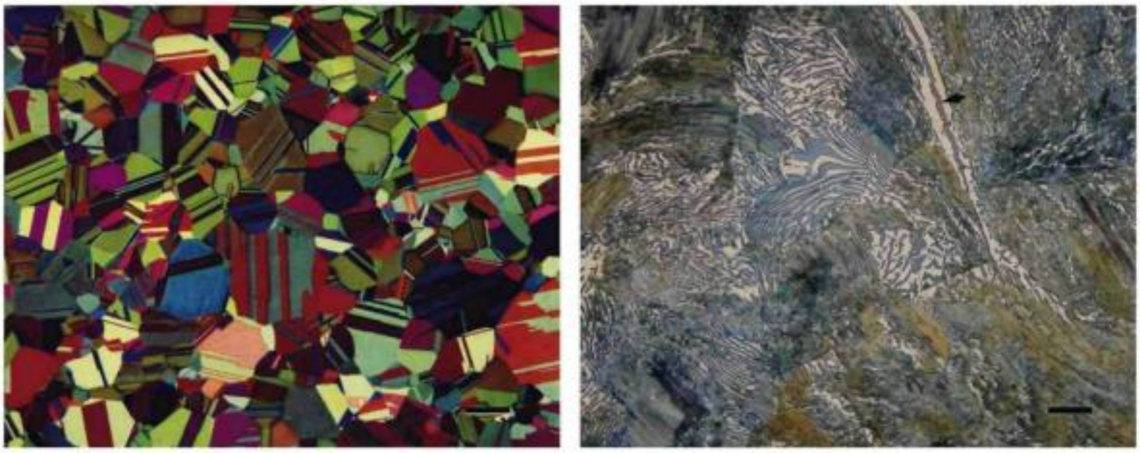


Fig. 2.1.29 and 2.1.30: Cartridge brass micrograph shown in Figures 2.1.22 and 2.1.23 tint etched with Beraha's PbS reagent (left) and cementite in a hot rolled Fe-1% C binary alloy coloured with Beraha's sodium molybdate reagent (right). Magnification bars are 200 and 20 µm long, respectively [2.1.14].

His CdS reagent is useful for carbon and alloy steels, tool steels, and ferritic, martensitic and precipitation hardenable stainless steels, while the PbS reagent does an excellent job on copper-based alloys and can be used to colour sulphides in steels white (the specimen is pre-etched with nital and the etch colours the darkened matrix, so that the white sulphides are visible). Beraha also developed two tint etchants that utilize molybdate ions in nitric acid. They colour cementite in steels (Figure 2.1.30).

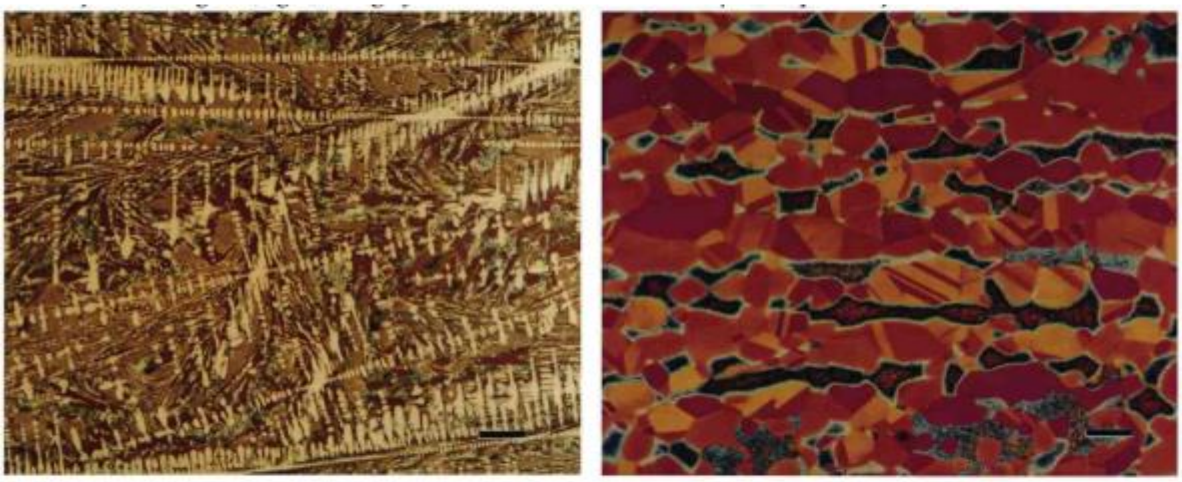


Fig. 2.1.31 and 2.1.32: Cementite in the chill cast surface of gray iron etched with Beraha's selenic acid reagent for cast iron (left) and twinned FCC alpha phase and beta phase (mottled and outlined) in Cu-40% Zn revealed using Beraha's selenic acid reagent for copper alloys. Magnification bars are 50 and 20 µm long, respectively [2.1.14].

He also developed tint etchants that deposit elemental selenium on the surface of steels (Figure 2.1.31), nickel-based alloys and copper-based alloys (Figure 2.1.32). There are a number of other tint etchants that have been developed by a variety of metallographers. Lichtenegger and Bloch, for example, developed an unusual reagent that will colour austenite (Figure 2.1.33) in duplex stainless steels, rather than ferrite (as nearly all others do).

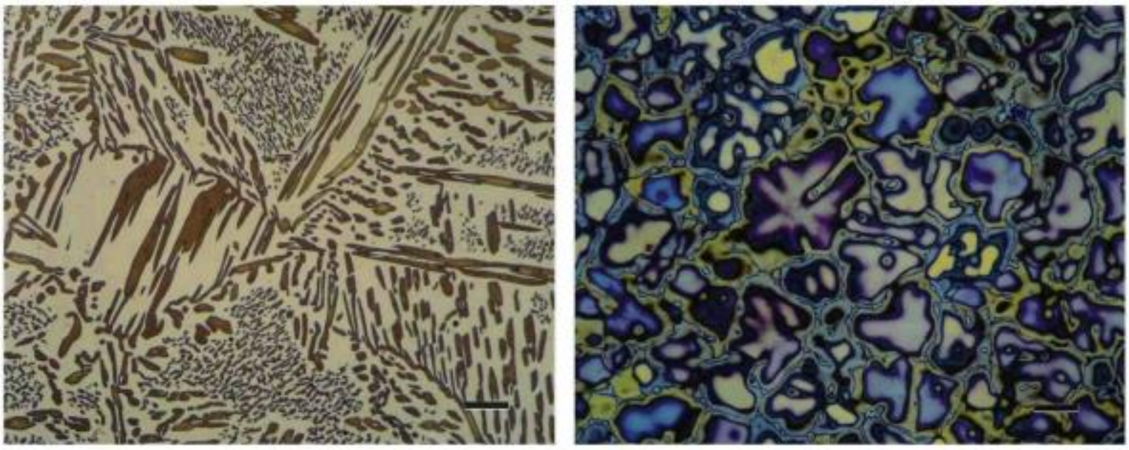


Fig. 2.1.33 and 2.1.34: Austenite coloured in ASTMA890 Grade 5A cast duplex stainless steel with the LBl reagent (left) and the cast grain structure of 206 aluminium revealed using Week's reagent for Al alloys (right). The magnification bars are 100 and 50 µm long, respectively [2.1.14].

Week developed a number of tint etchants, while utilizing many of them in the research. Several were developed to colour aluminium (Figure 2.1.34) or titanium alloys (Figure 2.1.35).

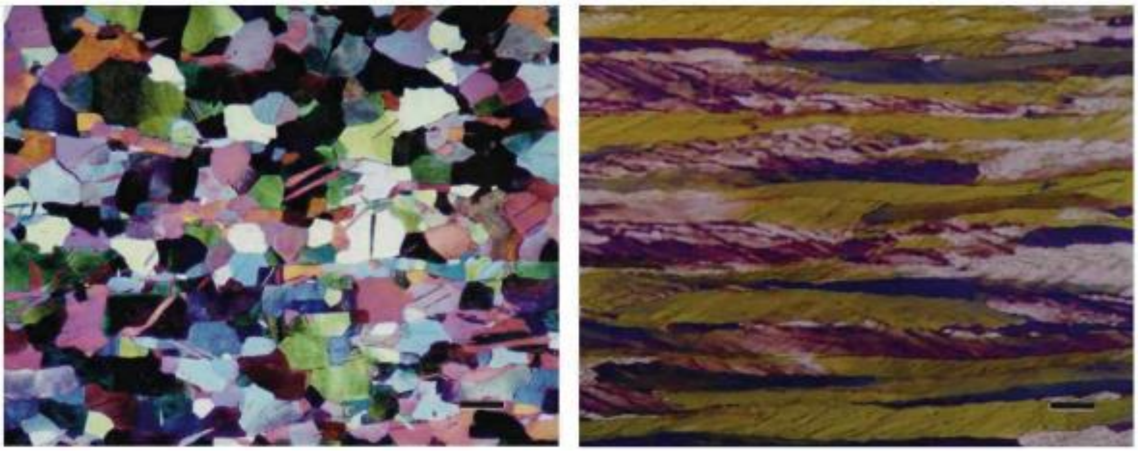


Fig. 2.1.35 and 2.1.36: Grain structure of as-rolled CP Ti (ASTM F67, Grade 2) containing mechanical twins (left) etched with modified Week's reagent and cold rolled pure molybdenum (right) coloured with the reagent developed by Oak Ridge National Laboratory. Magnification bars are 100 and 20 µm long, respectively [2.1.14]

In each case, it is easier to develop good colour with the cast alloys than with the wrought alloys. Two etchants have been found useful for colouring theta phase, AlCu2, in Al-Cu alloys; Lienard developed one of the easiest to use. Several colour etchants have been developed for molybdenum (Figure 2.1.36) and for tungsten [2.1.15].

Applications of Coloured Etching

In the context of metallurgy and welded joints, coloured etching is a valuable technique that can be used for specific applications:

Weld Defect Analysis: Coloured etching can be employed to reveal and highlight defects in welded joints, such as porosity, cracks, inclusions, and lack of fusion. By using specific etchants, different weld features can be emphasized, aiding in the assessment of weld quality and integrity.

Microstructural Examination: Coloured etching allows for the examination of the microstructure of the weld and heat-affected zone (HAZ). Different microstructural constituents may react differently to various etchants, enabling researchers and inspectors to distinguish between phases and identify potential issues related to grain structure and distribution.

Phase Identification: In certain cases, welds can consist of multiple phases or alloys. Coloured etching can help differentiate between these phases by selectively revealing their unique microstructural features. This information is crucial for understanding the composition and properties of the welded joint.

Intermetallic Compound Detection: Welding can lead to the formation of intermetallic compounds, which can have significant effects on the weld's mechanical properties. Coloured etching can aid in identifying and characterizing these compounds, helping to assess their potential impact on the weld's performance.

Residual Stress Analysis: Coloured etching combined with specific techniques, such as layer removal methods, can be utilized to analyse and visualize the distribution of residual stresses in welded joints. This information is vital for evaluating the structural integrity and potential for stress-related failure.

Grain Boundary Examination: Coloured etching can reveal grain boundaries in the weld metal and HAZ. Grain boundary characterization is essential for assessing the susceptibility to intergranular corrosion and for determining the weld's overall mechanical properties.

It's worth noting that the selection of appropriate etchants and preparation methods is critical in coloured etching for metallurgical applications. Different alloys and welding processes may require specific etching solutions and conditions to achieve accurate and meaningful results. Additionally, in modern metallurgy, other non-destructive and quantitative techniques, such as microscopy, electron microscopy, and X-ray diffraction, are often used in conjunction with coloured etching for a comprehensive analysis of welded joints.

Conclusions

The examples shown have demonstrated the great value of colour and tint etching for examining microstructures of metals. Solutions exist to develop colour with most commercial alloy systems. The examples clearly demonstrate the value of these reagents in revealing the grain structure fully, even for the most difficult to etch specimens. Further, they are selective in nature that can be quite useful for quantitative metallographic studies. Tint etchants reveal segregation very clearly and either EDS or WDS can be performed on a tint-etched surface without any problems from the interference surface layer.

Etch Compositions

Table 2.1.6 and 2.1.7 presents recipes for common coloured reagents [2.1.15].

Table 2.1.6

Coloured reagents	Recipes reagents
Klemm's I	50 mL stock solution, 1 g $K_2S_2O_5$ (stock solution is water saturated with $Na_2S_2O_3$)
Klemm's III	5 mL stock solution, 45 mL water, 20 g $K_2S_2O_5$ (stock solution as for Klemm's I)
Beraha's 10/3 reagent	10g $Na_2S_2O_3$, 3g $K_2S_2O_5$ and 100 mL water
Beraha's BI	100 mL stock solution (1000 mL water, 200 mL HCl, 24 g NH_4FHF) plus 0.1 – 0.2 g $K_2S_2O_5$ for martensitic stainless steel and 0.3 – 0.6 g $K_2S_2O_5$ for austenitic and ferritic stainless steels.

Beraha’s sulfamic acid reagent No. III	100 mL water, 3 g $K_2S_2O_5$, 2 g NH_2SO_3H (two other similar compositions were published) for carbon and alloy steels.
Beraha’s sulfamic acid reagent No. IV	100 mL water, 3 g $K_2S_2O_5$, 1 g NH_2SO_3H, 0.5 – 1 g NH_4FHF for high Cr tool steels and martensitic stainless steels.
Beraha’s CdS and PbS reagents	CdS stock solution: 1000 mL water, 240 g $Na_2S_2O_3 \bullet 5H_2O$, 20-25 g cadmium chloride (or cadmium acetate), 30 g citric acid; PbS stock solution: 1000 mL water, 240 g $Na_2S_2O_3 \bullet 5H_2O$, 30 g citric acid, 24 g lead acetate.

*Mix each solution in the precise order that is given.

Table 2.1.7

Coloured reagents	Recipes reagents
Beraha’s sodium molybdate reagent	Stock Solution: 1000 mL water, 10 g $Na_2MoO_4 \bullet 2H_2O$. Pour off about 100 mL of the stock solution and add HNO_3 to bring the pH to 2.5 – 3.0. For steels, add small amounts of NH4FHF to control coloration (none for cast iron). Colours cementite.
Beraha’s selenic acid reagent for cast iron	100 mL ethanol, 2 mL HCl, 1 mL selenic acid
Beraha’s selenic acid reagent for Cu alloys	300 mL ethanol, 2 mL HCl, 0.5-1 mL selenic acid
Lichtenegger and Blöch LB1 reagent	20 g of ammonium bifluoride, NH_4FHF, and 0.5 g potassium metabisulfite, $K_2S_2O_5$, dissolved in 100 mL water (use hot water). Etch at 25-30 ºC.
Weck’s reagent for Al	100 mL water, 4 g $KMnO_4$ and 1 g NaOH
Modified Weck’s reagent for Ti	100 mL water, 25 mL ethanol and 2 g ammonium bifluoride. The original formula specified 50 mL of ethanol, but that produces etch artefacts.

2.1.4. Differences between electrolytic polishing, chemical polishing, coloured etching

Electrolytic Polishing, Chemical Polishing, and Coloured Etching are three different surface treatment techniques used for metals, each with distinct principles and applications, namely:

Surface Defect Detection and Evaluation

Surface defect detection and evaluation are crucial in material analysis to assess the quality and integrity of the surface. Each of the surface treatment techniques—electrolytic polishing, chemical polishing, and coloured etching—offers specific advantages and considerations for surface defect detection and evaluation:

a. Surface Defect Detection and Evaluation after Electrolytic Polishing: Electrolytic polishing generally produces smooth and defect-free surfaces due to the removal of surface irregularities during the process. Surface defects such as scratches, pits, and cracks are less likely to be introduced during electrolytic polishing, making it suitable for defect-sensitive analyses.

After electrolytic polishing as shown in the figure 2.1.37, the surface can be visually inspected or observed under microscopy to detect any remaining defects. For more detailed defect evaluation, advanced techniques like scanning electron microscopy (SEM) can be used to examine the polished surface at higher magnifications.

Fig. 2.1.37 Surface defect detection before and after Electrolytic Polishing for Copper 101 [2.1.16]

b. Surface Defect Detection and Evaluation after Chemical Polishing: Chemical polishing may introduce surface defects, especially if the process is not carefully controlled. Surface defects like etch pits, surface roughness, and other irregularities can be introduced during chemical polishing, potentially impacting defect evaluation. Special attention should be given to avoid misinterpreting defects arising from the chemical polishing process itself rather than inherent material flaws as illustrated in figure 2.1.38. Advanced techniques such as optical profilometry or atomic force microscopy (AFM) can be used to quantitatively assess surface roughness and detect defects introduced during chemical polishing.

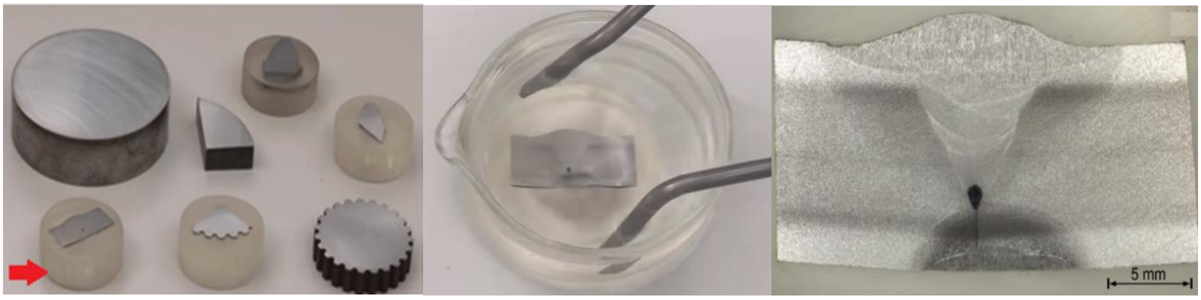


Fig. 2.1.38 Surface defect detection before and after Chemical Polishing for Stainless Steel [2.1.17]

c. Surface Defect Detection and Evaluation after Coloured Etching: Coloured etching can help highlight and reveal defects on the metal surface due to the contrasting colours or shades produced by specific etchants, as presented in figure 2.1.39. Defects such as cracks, inclusions, and porosity may be more easily identified and evaluated after coloured etching. The technique is particularly useful for defect-sensitive analysis of welded joints and other critical components. However, it is essential to ensure that the coloured etchant does not mask or interfere with the detection of certain types of defects.

In all cases, the choice of surface treatment technique depends on the specific requirements of defect detection and evaluation. It's important to consider the potential impact of the surface treatment itself on defect introduction and ensure appropriate controls are in place during the sample preparation process. Additionally, complementary techniques, such as non-destructive testing (NDT) methods like ultrasonic testing, dye penetrant testing, and X-ray inspection, may be used in conjunction with surface treatment to provide a comprehensive assessment of surface defects and material quality.

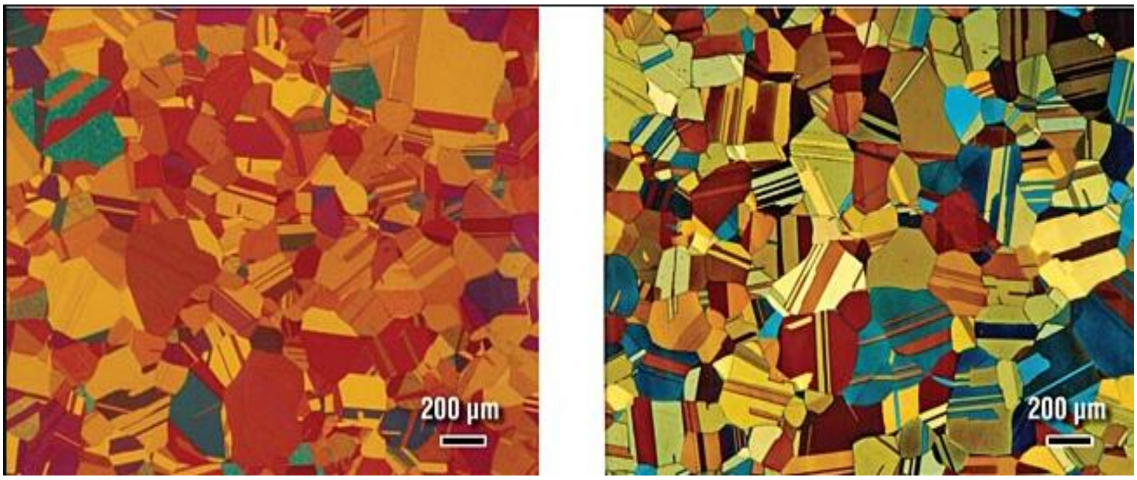


Fig. 2.1.39 Observe the microstructure of the specimen, etching with Berahal solution (3 g $K_2S_2O_5$, 10 g $Na_2S_2O_3$ in 100 mL distilled water) were performed after a traditional specimen preparation technique [2.1.14]

Grain Boundary Assessment

Grain boundary assessment is an important aspect of microstructural analysis, especially in metallurgy and materials science. Each of the surface treatment techniques—Electrolytic polishing, Chemical polishing, and Coloured etching—have their advantages and considerations for grain boundary assessment:

a. Grain Boundary Assessment after Electrolytic Polishing: Grain boundaries are generally well-preserved and clearly visible after electrolytic polishing, making it suitable for grain boundary analysis, as presented in figure 2.1.40.

High-quality optical microscopy can be used to observe and analyse grain boundaries, enabling the identification of grain size, shape, and distribution. Electron backscatter diffraction (EBSD) is often employed to obtain detailed information about crystallographic orientation and grain boundary character after electrolytic polishing.

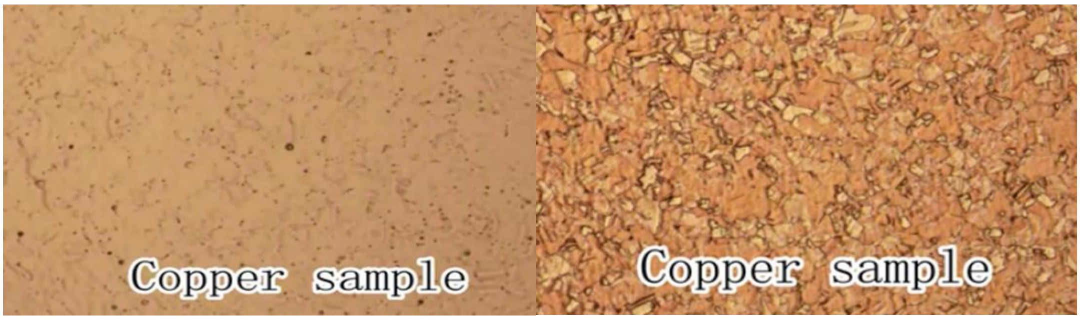


Fig. 2.1.40 Grain Boundary Assessment after Electrolytic Polishing for Copper 101 [2.1.7]

b. Grain Boundary Assessment after Chemical Polishing: Attention must be taken to ensure that the chemical polishing process does not alter the grain boundaries significantly. High-resolution microscopy techniques, such as SEM and TEM, may be necessary to evaluate grain boundaries after chemical polishing, as shown in Figure 2.1.41. Grain boundary analysis can be challenging in certain materials due to the potential for preferential etching along grain boundaries.

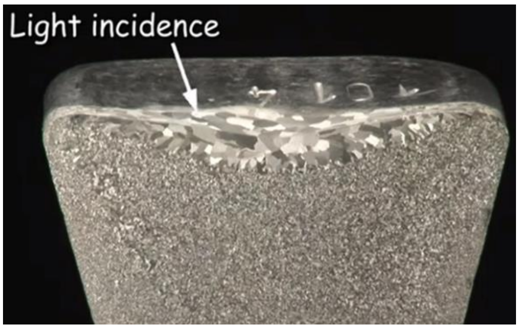


Fig. 2.1.41 Grain Boundary Assessment after Chemical Polishing for Stainless Steel [2.1.17]

c. Grain Boundary Assessment after Coloured Etching: Coloured etching can be advantageous for grain boundary assessment as it highlights grain boundaries with contrasting colours or shades.

The technique enables the visualization and identification of grain boundaries, facilitating grain size and distribution analysis. Coloured etching can be particularly useful for evaluating grain boundary behaviour in welds and other multi-phase materials. However, care must be taken to ensure that the coloured etchant does not obscure or distort the grain boundaries, as shown in figure 2.1.42.

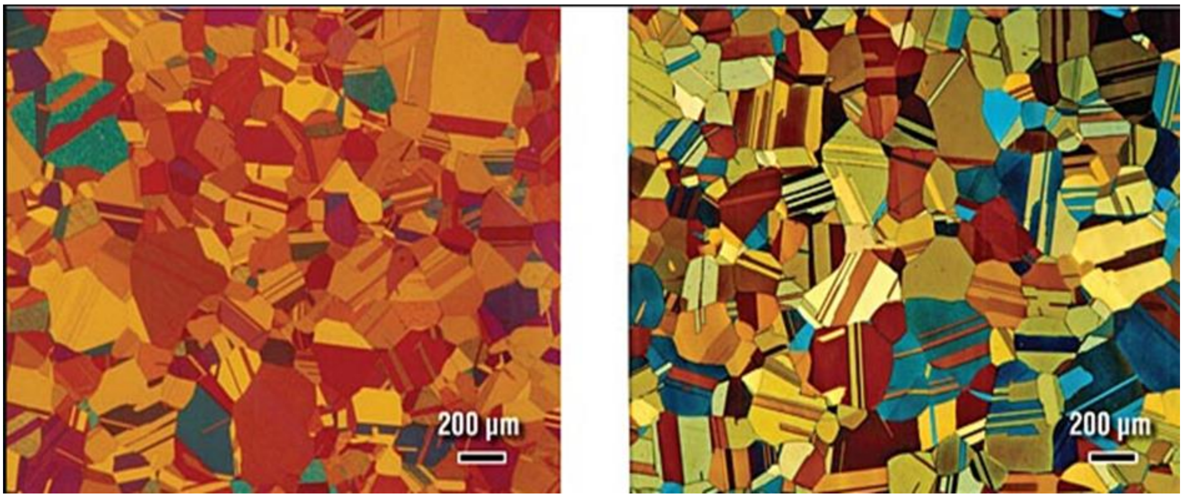
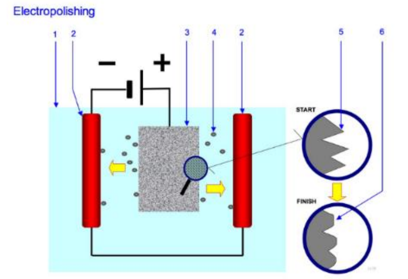
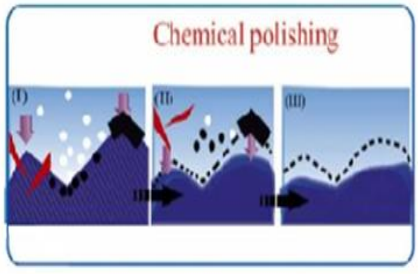
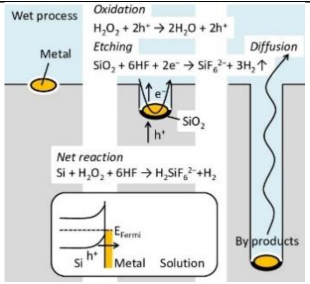


Fig. 2.1.42 FCC twinned grain structure of cartridge brass, Cu-30% Zn, after cold reduction by 50% and full annealing, tint etched with Klemm's I (left) and Klemm's III (right) reagents and viewed with polarized light plus sensitive tint. Magnification bars are 200 µm long. [2.1.14].

In general, all three surface treatment techniques can be used for grain boundary assessment, but careful consideration is required to choose the most appropriate method depending on the material type, analysis objectives, and the potential impact of the surface treatment itself. Advanced microscopy techniques like SEM, TEM, and EBSD are often employed to obtain detailed information about grain boundaries, crystallographic orientation, and grain boundary character after surface treatment. Additionally, it's crucial to use standardized procedures and validated techniques to ensure reliable and accurate grain boundary analysis.

Differences between processes

Furthermore, we will present a comparison between electrolytic polishing, chemical polishing, and coloured etching processes:

		
ELECTROLYTIC POLISHING	CHEMICAL POLISHING	COLORED ETCHING

Principle

Electrolytic Polishing: Electrochemical process utilizing an electric current to smooth the metal surface by controlled dissolution of surface irregularities.

Chemical Polishing: Chemical process involving the selective dissolution of a thin metal layer through a chemical reaction to achieve a polished and shiny surface.

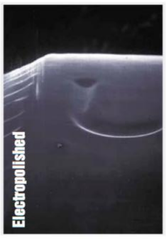
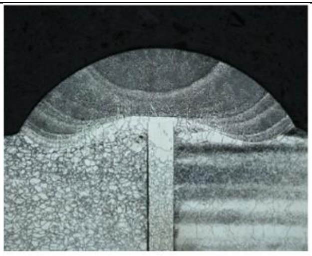
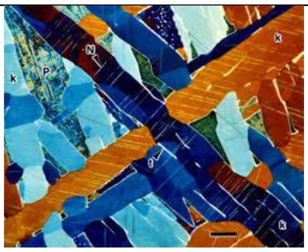
Coloured Etching: Metallographic technique using specific etchants to reveal and highlight different microstructural features with contrasting colours or shades.

Surface Finish

Electrolytic Polishing: Produces a smooth, mirror-like surface with minimal mechanical deformation.

Chemical Polishing: Creates a polished and shiny appearance, but the surface may be affected by introduced artefacts or irregularities.

Coloured Etching: Provides a surface with contrasting colours or shades, highlighting microstructural features without significantly altering the surface finish.

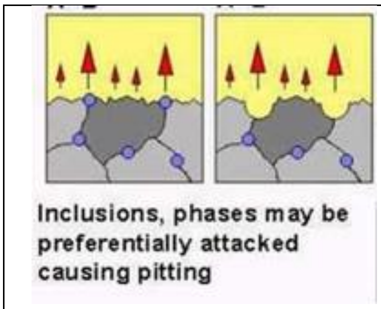
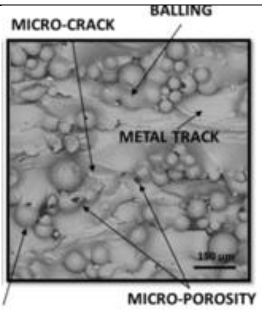
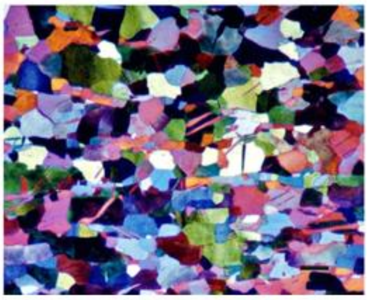
		
ELECTROLYTIC POLISHING	CHEMICAL POLISHING	COLORED ETCHING

Defect Introduction

Electrolytic Polishing: Minimizes the introduction of surface defects due to its non-mechanical nature.

Chemical Polishing: May introduce surface defects or etch patterns depending on the control of the chemical reaction.

Coloured Etching: Typically, does not introduce defects, but care must be taken to ensure the coloured etchant does not obscure certain defects.

		
ELECTROLYTIC POLISHING	CHEMICAL POLISHING	COLORED ETCHING

Grain Boundary Assessment

Electrolytic Polishing: Suitable for grain boundary analysis, providing well-preserved and visible grain boundaries as in figure 2.1.43.

Chemical Polishing: Challenging for grain boundary assessment due to potential artefacts introduced during the chemical etching process.

Coloured Etching: Useful for highlighting grain boundaries with contrasting colours, aiding grain boundary evaluation.

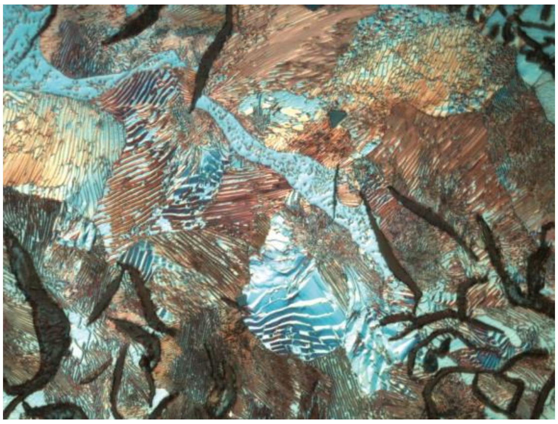


Fig. 2.1.43 Interlamellar spacing of pearlite in a gray cast iron [2.1.18]

Surface Defect Detection

Electrolytic Polishing: Allows for effective defect detection on smooth surfaces with minimal artefacts.

Chemical Polishing: May introduce surface defects, making defect evaluation more complex.

Coloured Etching: Facilitates the identification of defects by contrasting colours, as can be seen in the figure 2.1.44, particularly useful for detecting defects in critical components.

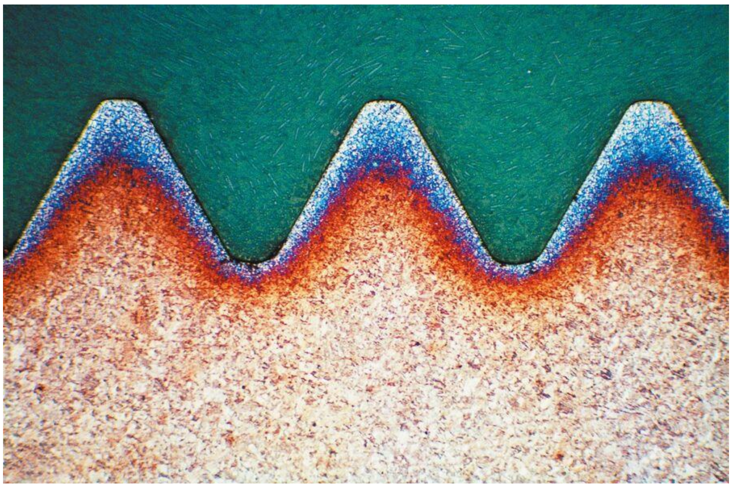


Fig. 2.1.44 Surface hardening of steels [2.1.18]

Microstructure Analysis

Electrolytic Polishing: Enables accurate microstructural examination with minimal interference.

Chemical Polishing: Requires careful control to avoid misinterpreting defects introduced during the process.

Coloured Etching: Valuable for microstructural analysis, particularly in welds and multi-phase materials, as can be seen in the figure 2.1.45, by highlighting microstructural constituents.

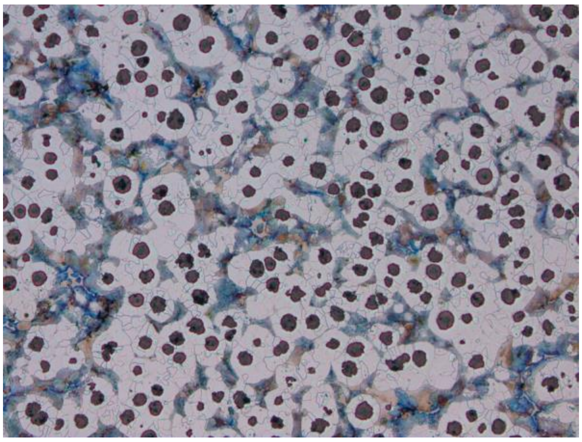


Fig. 2.1.45 Ductile iron with spheroidal graphite (HC PL Fluotar 10x objective, brightfield). [2.1.18]

Applications

Electrolytic Polishing: Primarily used in metallography for preparing samples for microscopy and microstructural analysis.

Chemical Polishing: Applied in electronics, optics, jewellery, and aerospace industries for achieving high-quality surface finishes.

Coloured Etching: Extensively used in metallurgy and materials science for microstructural examination of various metal components, as shown in figure 2.1.46.

Fig. 2.1.46 Electropolishing of surfaces: theory and applications [2.1.19]

In summary, each process has its strengths and weaknesses, and the choice of technique depends on the specific requirements of the analysis, the material type, and the desired level of surface finish and accuracy.

Final remarks

This chapter provided an in-depth exploration of electrolytic polishing, chemical polishing, and coloured etching, focusing on their definitions, purposes, mechanisms, and applications in materials science and engineering. Electrolytic polishing's controlled electrochemical reactions offer valuable insights into microstructure analysis, surface defect detection, grain boundary assessment, and surface coating preparation, supporting the development of advanced metallic materials.

Chemical polishing has proven effective in microstructure characterization, surface finish improvement, phase identification, and materials evaluation, with its ability to selectively remove material enhancing the visibility of microstructural features and defect characteristics.

Coloured etching, through interaction with microstructural constituents, is a valuable technique for phase identification, inclusion detection, and grain size analysis, providing a means to visualize and differentiate various microstructural features based on colour.

These techniques offer complementary approaches to studying metallic materials, with applications ranging from materials research to failure analysis and process optimization.

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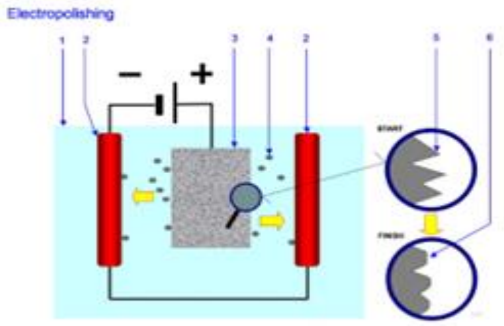
2.2. Demonstration of Electrolytic Polishing and Colored Etching

2.2.1. Electrolytic polishing – purpose, application, and demonstration

Electrolytic polishing is a specialized technique used to remove material from a metal surface through controlled electrochemical reactions. This process is commonly used for preparing samples for microscopy, metallography, and other applications where a high-quality surface finish is required. It is used to enhance the surface quality of metallic specimens for microscopic examination. It involves the controlled application of electrical current and an electrolyte solution to selectively remove material from the sample's surface, resulting in a polished and clean surface finish.



1. Electrolyte
2. Cathode
3. Workpiece to polish (Anode)
4. Particle moving from the workpiece toward the cathode
5. Surface before polishing
6. Surface after polishing

Figure 2.2.1. How Electropolishing Works (source: Best Technology)

In electropolishing, the metal part or workpiece serves as the positively charged anode. The workpiece is connected to the positive terminal of a DC power rectifier. The negatively charged cathode, generally made of stainless steel or zirconium, is connected to the negative terminal of the DC power rectifier.

Both anode and cathode are immersed in a temperature-controlled bath of electrolyte solution. Electrical current from the rectifier is conducted from the anode to the cathode through the electrolyte. The electrical current causes metal ions on the surface of the part to oxidize and dissolve into the electrolyte. This process can dissolve extremely small, tightly controlled amounts of metal, resulting in micron-level thickness of surface removal.

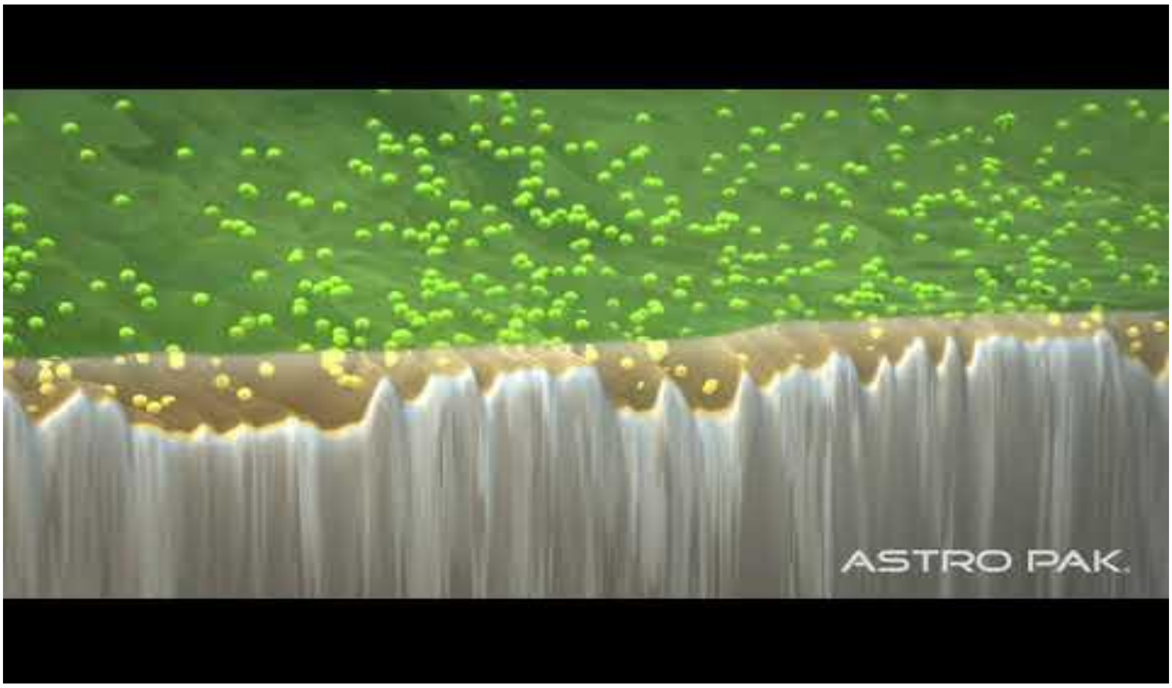


Figure 2.2.2. What is Electropolishing? | How Does Electropolishing Work? (source: AstroPaK)

The amount of metal surface removal is controlled by the following factors: electrolyte chemical composition, temperature of electrolyte (typically 76 °C – 82 °C), length of time of exposure to electrical current, density of electrical current (varies by electrolyte; composition of metal alloy undergoing electropolishing. Supported metal alloys for electropolishing are aluminium, carbon steel, stainless steel, copper alloys, nitinol, titanium, tool steel, specialty/exotic alloys. The electropolishing chemicals for stainless steel typically consist of a high-viscosity mixture of sulfuric acid and phosphoric acid.

2.2.2. Hands-on practice with electrolytic polishing

It is typically conducted in laboratory or industrial settings for the purpose of improving the surface finish of metal samples. Electrolytic polishing is a specialized technique used to remove material from a metal surface through controlled electrochemical reactions. This process is commonly used for preparing samples for microscopy, metallography, and other applications where a high-quality surface finish is required.

Before we begin the process, make sure to follow all safety precautions. This includes wearing appropriate personal protective equipment, working in a well-ventilated area, and adhering to any safety guidelines provided by your institution or workplace.

Materials and Equipment's used in electrolytic polishing

- Metal samples to be polished

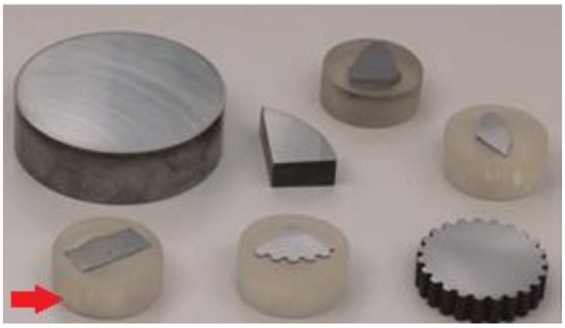


Figure 2.2.3. Samples ready for electropolishing (source: Kemet International Ltd)

- Electrolytic polishing equipment, including a power supply, electrolyte solution, and electrodes

Figure 2.2.4. Equipment for electrolytic polisher with the electrolytic cell uncover (source: American Buehler Instrument Co., Ltd.)

Figure 2.2.5. Equipment for electrolytic polisher with the electrolytic cell over (source: Kemet International Ltd)

- Safety goggles, gloves, and lab coat
- Glass containers for electrolyte solution

Figure 2.2.6. Electrolyte solutions (source: Micro Lit)

- Polishing cloth or pads
- Distilled water
- Voltage and amperage meters

Figure 2.2.7. Voltage and amperage meters (source: Pulsivo USA)

- Polishing equipment (e.g., grinder or sandpaper) for initial sample preparation

Figure 2.2.8. Polishing equipment (source: AzOm)

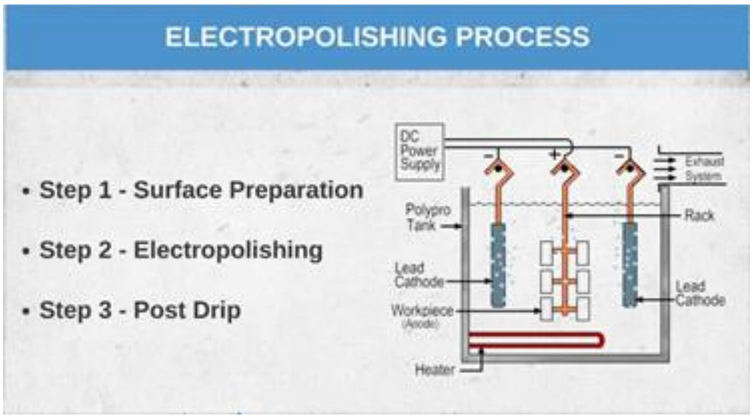
- Scrubbing brush

Electrolytic polishing work procedure

Sample Preparation:

1. The operator should start with a metal sample that requires polishing and ensure that it is clean and free of any contaminants.
2. If the sample exhibits significant surface imperfections, the operator may need to perform initial mechanical polishing using a grinder or sandpaper to eliminate major defects.
3. Subsequently, the sample should be rinsed thoroughly with distilled water to eliminate any debris and contaminants.

The most commonly used in Electropolishing industry are ASTM B912 and ASTM E1558-09. The standard provides guidance on how to electropolish stainless steel.



ELECTROPOLISHING PROCESS

- Step 1 - Surface Preparation
- Step 2 - Electropolishing
- Step 3 - Post Drip

Figure 2.2.9. Electropolish Process Steps (source: Best Technology Ltd.)

In this preparation phase, the operator should remove contaminants such as oil and grease that interfere with uniformity when electric current is applied. After cleaning, be careful that the stainless-steel parts avoid contact with hands or equipment. Improper cleaning is a common cause of part rejection.

Electrolyte Solution Preparation

1. An appropriate electrolyte solution should be prepared for the specific metal and application. The composition of the electrolyte will vary based on the material being polished.
2. The operator should pour the electrolyte solution into a glass container.

Table 2.2.1 Types of electrolytic solution (source: Quora)

Stainless Steel	10g Oxalic Acid, 100ml Water
	5 ml Sulfuric Acid, 100 ml Water
For austenitic-ferritic steels (duplex)	20-40 % aqueous sodium hydroxide solution
For automatic grain size measurements of austenitic steels	60 % distilled water 40 % nitric acid

For example, the actual steps for electropolishing of stainless steel is performed in this phase to smooth, brighten and deburr the metal.

- Electropolish stainless steel parts – Treatment times can vary from 10 seconds to 20 minutes.

- ➢ Boil off/Spray rinse, or Dead rinse – This tank captures the used electrolyte solution.

(a)

(b)

Figure 2.2.10. (a) Different types of electrolytes suitable for specific SC applications and (b) impact of the electrolyte on the performance of electrochemical SCs. (source: Tiwari, Santosh & Thakur, Anukul & De Adhikari, Amrita & Zhu, Yanqiu & Wang, Nannan. (2020). Current Research of Graphene-Based Nanocomposites and Their Application for Supercapacitors. 10. 2046. 10.3390/nano10102046.)

Electrolytic Cell Setup

1. The positive electrode (anode) and the negative electrode (cathode) need to be connected to the power supply.
2. The operator needs to submerge the metal sample in the electrolyte solution and connect it to the cathode.
3. The anode should be made of a material that does not dissolve in the electrolyte. Common choices include platinum or graphite.

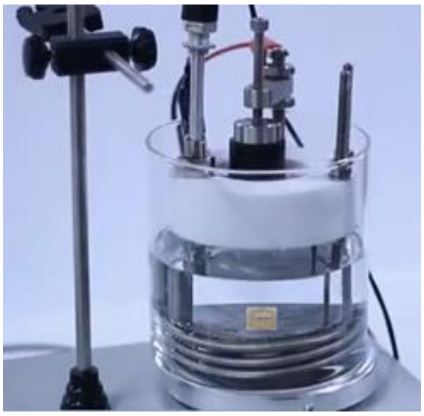


Figure 2.2.11. Special unit cell for electropolishing (source: American Buehler Instrument Co., Ltd.)

Electrolytic Polishing

1. The operator needs to turn on the power supply and set the voltage and amperage to the desired values for your application. The specific parameters will depend on the metal and the extent of polishing required.
2. The operator will observe the material removal from the metal sample. The metal dissolves at the anode and is deposited at the cathode. The process should be carefully monitored to achieve the desired finish.
3. The operator should keep the voltage and amperage within the recommended range to prevent overheating or damaging the sample.

Figure 2.2.12. DPF-2 Electrolytic Polishing and Etching Machine (source: Micro Lit)

Rinsing and Final Cleaning

1. Once the desired polishing is achieved, the operator needs to turn off the power supply.
2. The operator needs to carefully remove the sample from the electrolyte solution.
3. The operator needs to rinse the sample with distilled water to remove any residual electrolyte.
4. The operator needs to use a polishing cloth or pad to give the sample a final polish and remove any remaining imperfections.

Final Inspection

The operator needs to examine the polished sample under a microscope or for other quality control purposes to ensure the desired finish has been achieved.

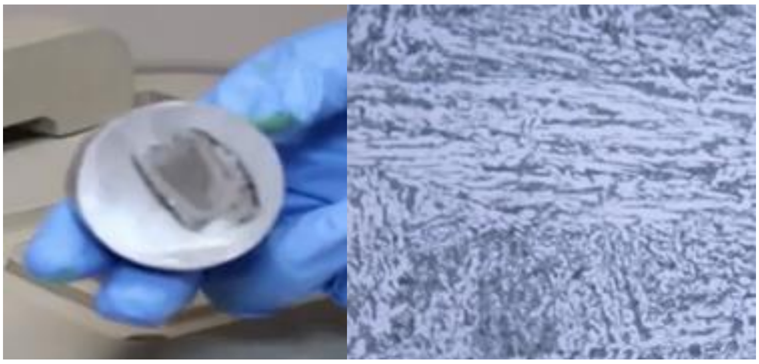


Figure 2.2.13. The microscope structure of stainless-steel sample after been electropolish (source: Kemet International Ltd)

2.2.3. Common issues and solution when using the electropolishing technique for metallographic sample preparation

Electrolytic polishing is a precise electrochemical process used to achieve a high-quality surface finish on metal samples, however, it can encounter several common issues that may affect the quality of the results, in the following we will present the most common:

Uneven Polishing

Issue: The metal sample may undergo uneven polishing, resulting in an uneven surface finish.

Figure 2.2.14. Uneven polishing on stainless steel sheet (source: AzOm)

Solution: The operator must ensure that the sample is properly secured and positioned in the electrolyte solution, stir the solution to maintain uniformity and adjust the electrical parameters (voltage and current) to provide consistent polishing across the entire sample.

Over-Polishing

Issue: Over-polishing occurs when the sample is polished for too long, leading to excessive material removal and potential damage to the sample.

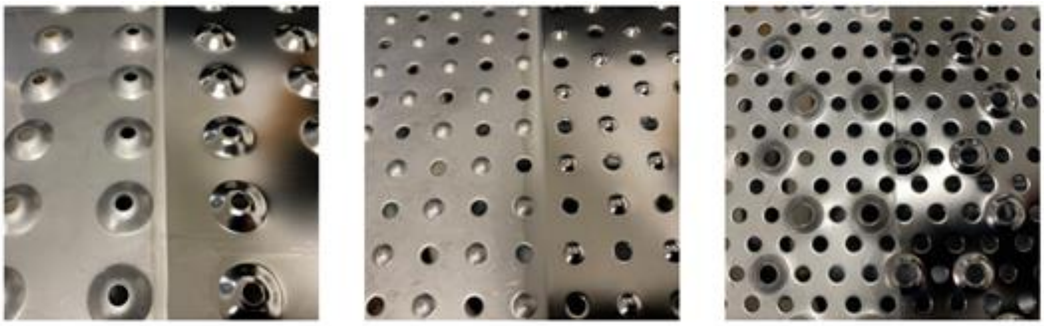


Figure 2.2.15. Stainless Steel Graepels/Graepels (source: Perforators & Weavers Ltd/Graepel Perforators Ltd)

Solution: The operator must monitor the polishing process closely and adhere to recommended polishing times and adjust the voltage and current settings to control the rate of material removal. A timer should be implemented to prevent over-polishing.

Pitting and Dimpling

Issue: Pitting and dimpling result from localized corrosion during electrolytic polishing, causing irregular depressions on the sample surface.

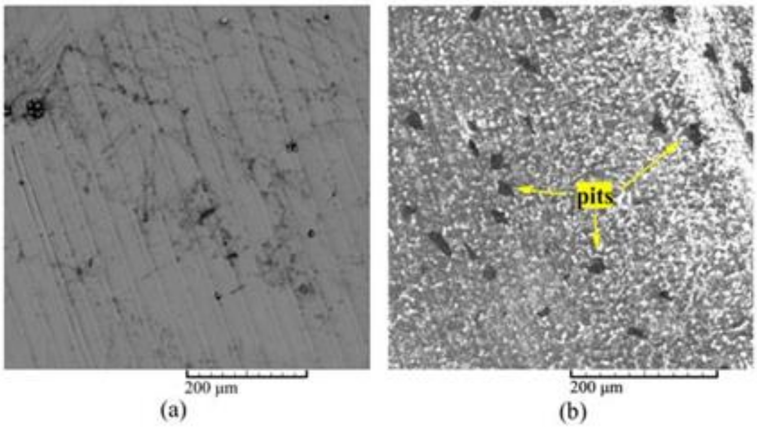


Figure 2.2.16. SEM image of (a) a ground steel disc surface and (b) an electropolished steel disc surface. (source: Mašović, R.; Miler, D.; Čular, I.; Jakovljević, S.; Šercer, M.; Žeželj, D. The Effect of Steel Electropolishing on the Tribological Behavior of a Steel–Bronze Pair in the Mixed and Boundary Lubrication Regimes. *Lubricants* **2023**, *11*, 325. https://doi.org/10.3390/lubricants11080325)

Solution: The operator must check the composition and temperature of the electrolyte solution, as well as the anode-cathode distance and ensure that the electrolyte is well-mixed. Properly clean and prepare the sample before polishing to remove any contaminants.

Rough or Scratched Surface

Issue: If the sample's surface appears rough or scratched after electrolytic polishing, it may be due to inadequate mechanical preparation or dirty polishing equipment.

Solution: The operator must ensure that the sample is properly prepped with initial mechanical polishing and use clean and well-maintained polishing equipment and check the quality of the polishing cloth or pad for any abrasive particles that may scratch the surface.

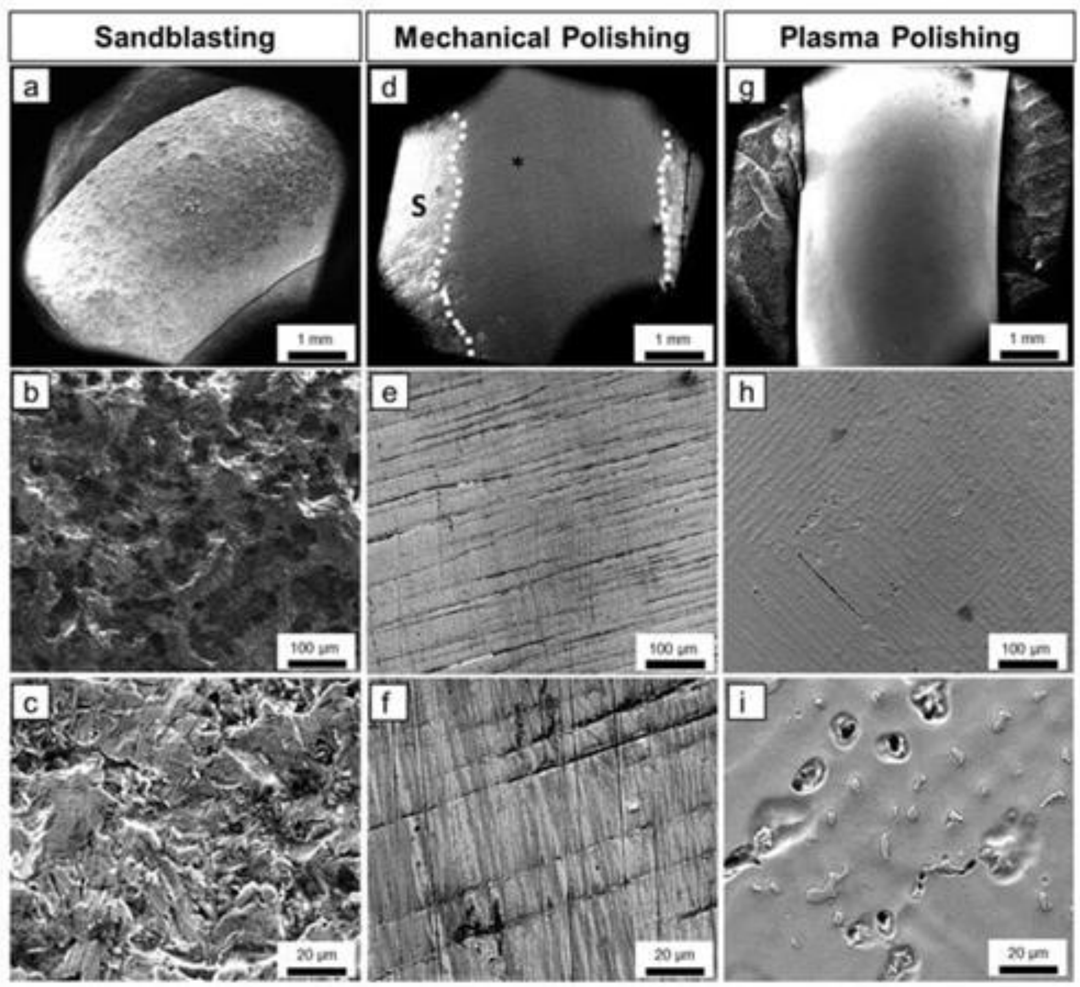


Figure 2.2.17. SEM micrographs revealing the comparison of sandblasted, mechanically polished and plasma electrolytically polished Co-Cr alloys. (a) Overview of sandblasted depicting a rough surface morphology. (b) Close-up view of sandblasted showing a rough and fissured surface. (c) High magnification reveals an irregular surface with ridges, edges and deeper porosities. (d) Overview of mechanically polished sample depicting a clear border between mechanically polished (*) and sandblasted area (S). (e,f) High magnification of mechanically polished sample reveals a smooth surface with numerous polishing marks. (g) Overview of plasma electrolytically polished sample showing a comprehensive smooth surface. (h) Close-up of plasma electrolytically polished sample showing a smooth surface with numerous inhomogeneities. (i) High magnification of plasma electrolytically polished sample reveals a smooth surface with interspersed insular structures. (source: Witzke, Katharina &

Kensbock, Renko & Willsch, Caroline & Fricke, Katja & Bekeschus, Sander & Metelmann, Hans-Robert. (2023). Mechanical and Plasma Electrolytic Polishing of Dental Alloys. Materials. 16. 6222. 10.3390/ma16186222.)

Gas Bubbles and Dendritic Growth

Issue: The formation of gas bubbles or dendritic growth on the sample surface can occur during electrolytic polishing.

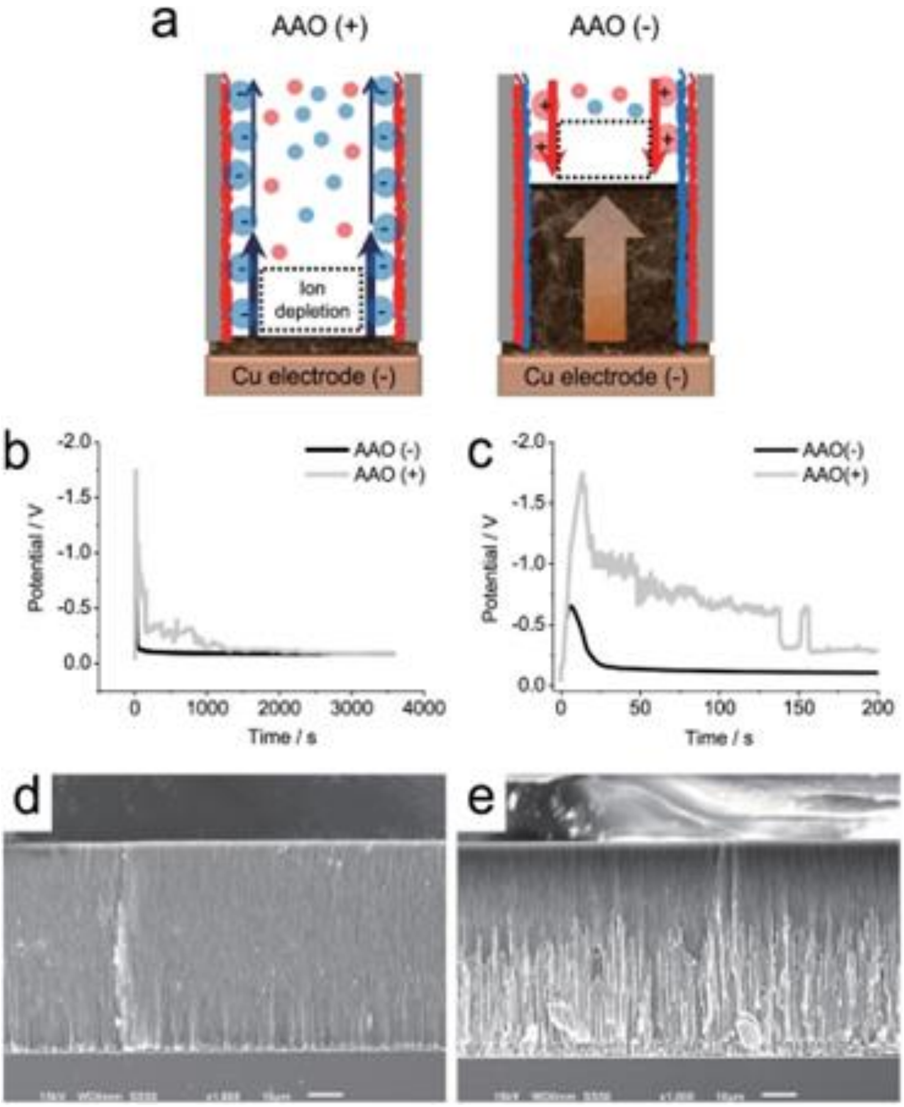


Figure 2.2.18. (a) Effect of SC on electrodeposition in charged nanopores during OLC. (b) V-t curves of AAO(+) and AAO(−) for an applied current of −6 mA. (c) Magnification of data of (b) for first 200 s. SEM images of electrodeposited Cu nanowires in (d) AAO(+) and (e) AAO(−). (source: Han, Ji-Hyung & Khoo, Edwin & Bai, Peng & Bazant, Martin. (2014). Over-limiting Current and Control of Dendritic Growth by Surface Conduction in Nanopores. Scientific reports. 4. 10.1038/srep07056.)

Solution: The operator must make sure that the sample is free from trapped air or gas bubbles before starting the polishing process then adjust the agitation and temperature of the electrolyte to prevent the formation of bubbles.

Etching Patterns or Streaks

Issue: Unwanted etching patterns or streaks may appear on the polished surface, often due to inconsistent polishing conditions.

Figure 2.2.19. Streaks on machined SS304 after electropolishing (source: Global Spec)

Solution: The operator must maintain steady electrical parameters and control the flow of electrolyte to ensure even distribution. Properly clean and prepare the sample to avoid variations in surface composition.

Inadequate Luster or Brightness

Issue: If the polished surface lacks the desired luster or brightness, it may result from suboptimal electrolyte composition or conditions.

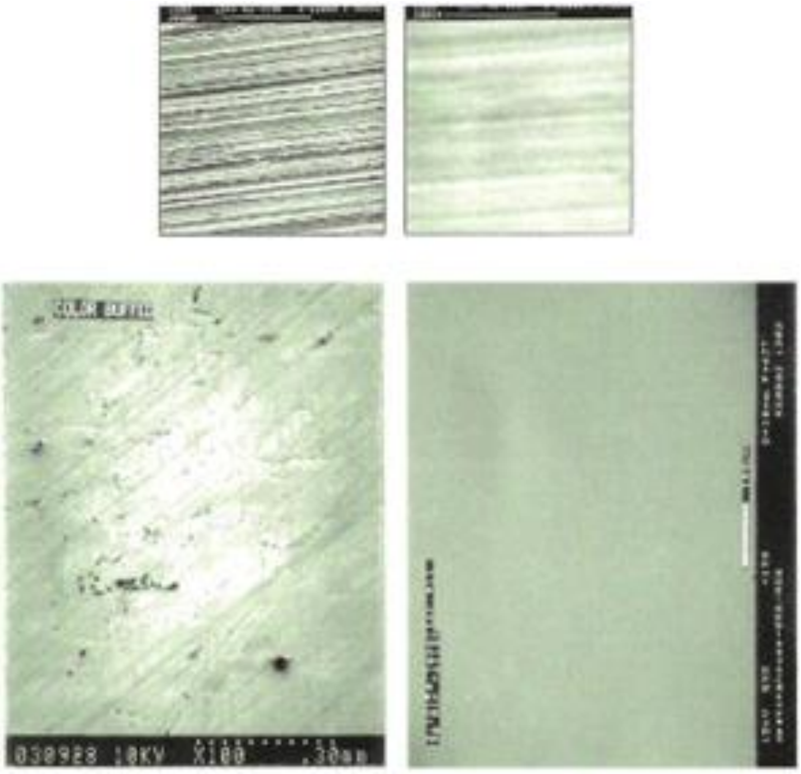


Figure 2.2.20. Optical (top) and SEM (bottom) photomicrographs showing transformation of mechanically polished rough surface on the left to smooth finish after electropolishing (source: Global Spec)

Solution: The operator must adjust the composition of the electrolyte solution to enhance the polishing result. Experiment with different parameters, such as temperature, voltage, and current, to achieve the desired finish.

Safety Concerns

Issue: Safety issues can arise if proper safety precautions are not followed when working with chemicals and electrical equipment.

Figure 2.2.21. Protective equipment (source: Micro Lit)

Solution: The operator must always follow safety guidelines and wear the appropriate personal protective equipment, work in a well-ventilated area, and be trained on the proper handling of chemicals and equipment.

2.2.4. Handling and disposal of electrolytes from electrolytic polishing

An operator or metallographic specialist must follow specific procedures for the safe handling and disposal of electrolytes used in electrolytic polishing.


Electrolyte LF For Cleaning, 1 Litre
Electrolyte For Dark Marking, 100 Ml
Electrolyte For Light Marking, 100 Ml
Electrolyte C For Polishing, 1 Litre
Electrolyte For Cleaning, 1 Litre

Figure 2.2.22. Electrolyte solutions (source: Micro Lit)

Handling Electrolytes from Electrolytic Polishing

The operator or specialist must perform electrolyte handling in a well-ventilated area, such as a fume hood or a space with good room ventilation, to minimize exposure to fumes or vapours. Containers holding electrolytes should be clearly labelled with their names, compositions, hazards, and any necessary handling precautions. Specific labels should be used, and records should be maintained. It's important to store electrolytes in chemical-resistant containers designed for this purpose. Glass or plastic containers are common choices.

Figure 2.2.23. Electrolyte C+ (source: Micro Lit)

Operators or specialists should avoid direct skin contact with the electrolyte. If contact occurs, immediate rinsing of the affected area with plenty of water is essential. When handling electrolytes the operator should refrain from eating, drinking, or smoking to prevent accidental ingestion. When transferring or mixing electrolytes, care should be taken to avoid splashes or accidents. It's advisable to add acids to water, not the other way around. The operator or specialist should ensure that materials used for handling electrolytes (containers, stirrers, etc.) are compatible with the specific electrolyte and won't react with it.

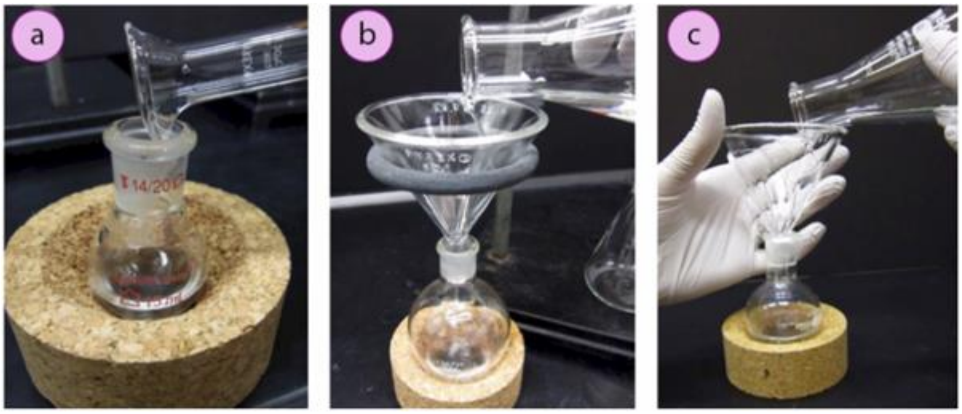


Figure 2.2.24. Pouring liquid, b) Pouring into a funnel held with ring clamp, c) Pouring into a funnel held by hand. (source: Micro Lit)

All personnel working with electrolytes from electrolytic polishing should be educated on the risks and safety procedures. Ongoing training and awareness are crucial.

Regulatory Compliance with the disposal of electrolytes from electrolytic polishing

Operators or metallographic specialists must adhere to local, state, and federal regulations governing the disposal of hazardous waste. These regulations can vary by location. Different electrolytes should be kept separate, and mixing during disposal should be avoided. In cases where neutralization is necessary, the individual should follow chemical compatibility guidelines for safe neutralization. If there is uncertainty about the proper disposal methods, operators or specialists should consult with a qualified chemist, waste disposal expert, or their organization's environmental health and safety department. Waste containers should be clearly labelled with their contents and any hazards to ensure safe handling during disposal.

It's crucial to ensure that waste containers are tightly sealed to prevent leaks or spills. Operators or specialists should arrange for the pickup and disposal of hazardous waste through licensed and authorized waste disposal services or facilities. Attempting to dispose of hazardous waste independently is not recommended.

Maintaining records of the disposal process, including dates, disposal methods, and relevant safety data sheets (SDS), is essential for reference and compliance. Operators or specialists should be prepared for emergencies and have spill response kits, safety showers, and eyewash stations available in the area where electrolytes from electrolytic polishing are handled.

2.2.5. Troubleshooting and tips in electrolytic polishing metallographic samples

Electrolytic polishing of metallographic samples is a crucial step in preparing materials for microscopic examination. However, issues may arise during the process (please see previous section), thus in this section we will mention sample contamination, a common issue that occurs in metallographic laboratories. The issue is the contaminants on the sample that can interfere with the polishing process. To achieve a successful process, the operator must ensure the sample is free of contaminants, clean all equipment, and use clean electrolyte solutions. Other surface defects, such as scratches, cracks, and pits, can significantly affect the performance and reliability of metallic materials. Electrolytic polishing plays a vital role in surface defect detection by providing a clean and smooth surface that facilitates their visibility and characterization.

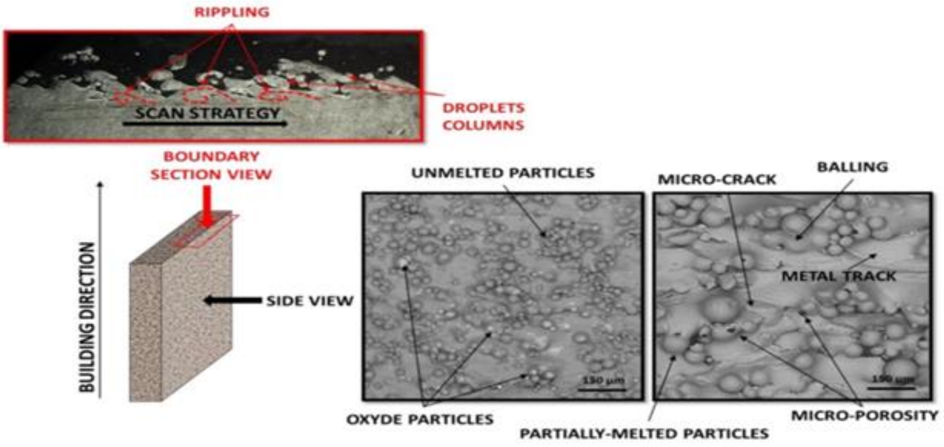


Figure 2.2.25. Representation of surface defects of additively manufactured objects (source: Acquesta, Annalisa & Monetta, Tullio. (2023). Green Approach for Electropolishing Surface Treatments of Additive Manufactured Parts: A Comprehensive Review. Metals. 13. 874. 10.3390/met13050874.)

By removing surface irregularities and contaminants, electrolytic polishing enhances the contrast between the defects and the surrounding material. This enables precise identification, measurement, and evaluation of surface flaws.

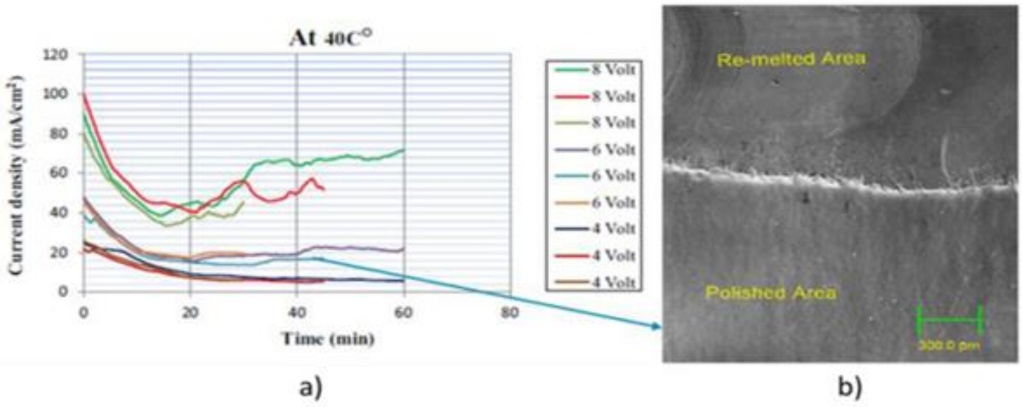


Figure 2.2.26. Chrono – ampero-metric curves obtained performing the EP treatment at 6 V at 40 °C for a duration time between 30 and 60 min. (**b**) SEM picture of the electropolished sample applying a voltage of 6 V at a

temperature of 40 °C for a duration of 45 min. (source: Acquesta, A.; Monetta, T. Green Approach for Electropolishing Surface Treatments of Additive Manufactured Parts: A Comprehensive Review. *Metals* 2023, *13*, 874. https://doi.org/10.3390/met13050874)

The ability to detect and characterize surface defects is crucial for assessing the material's quality, durability, and suitability for specific applications.

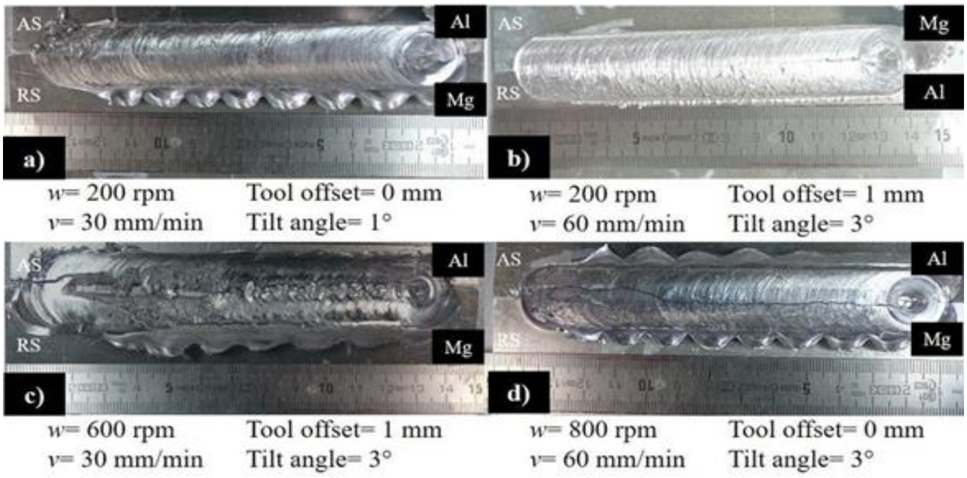


Figure 2.2.27. Defects of the welds, a) excessive flashing, b) crack, c) surface lack of fill and excessive flashing and d) Crack and excessive flashing. (Advancing side: AS and retreating side: RS) (source: Hernández-García, D. & Saldaña Garcés, Rocio & Vázquez, F.J. & Gutiérrez-Castañeda, E.J. & Deaquino-Lara, R. & Verdera, David. (2017). Friction Stir Welding of Dissimilar AA7075-T6 to AZ31B-H24 Alloys. MRS Advances. 2. 1-9. 10.1557/adv.2017.609.)

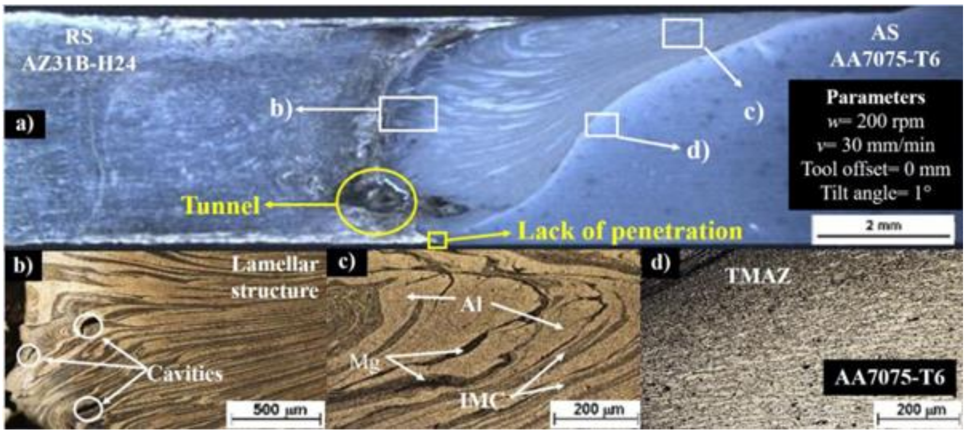


Figure 2.2.28. Micrographs of the cross-section of dissimilar AA7075-T6 to AZ31B-H24 joint by FSW, with internal defects such as tunnelling, lack of penetration (a) and cavities (b), in (c) it shows the dispersion between the Mg and Al in the SZ; and in (d) it is possible to observe a decrease of approximately 70% in the grain size of the thermomechanical affected zone (TMAZ) (source: Hernández-García, D. & Saldaña Garcés, Rocio & Vázquez, F.J. & Gutiérrez-Castañeda, E.J. & Deaquino-Lara, R. & Verdera, David. (2017). Friction Stir Welding of Dissimilar AA7075-T6 to AZ31B-H24 Alloys. MRS Advances. 2. 1-9. 10.1557/adv.2017.609.)

General Tips

- ✓ The operator should properly prepare the sample by removing any surface contaminants and performing initial mechanical polishing to eliminate major defects.
- ✓ The operator should tailor the electrolyte composition to the specific metal being polished, different metals may require different electrolytes.
- ✓ The operator should ensure uniform electrolyte distribution by agitating it to prevent stagnation or the formation of gas bubbles.
- ✓ The operator should continuously monitor the polishing process to achieve the desired finish and use timers if necessary to prevent over-polishing.
- ✓ The operator should adhere to safety guidelines, wear appropriate personal protective equipment, and work in a well-ventilated area when handling chemicals and electrical equipment.
- ✓ The operator should experiment with voltage, current, and temperature settings to find the ideal conditions for your specific metal and finish requirements.
- ✓ The operator should maintain detailed records of the polishing process, including parameters, electrolyte composition, and any issues encountered, to aid in troubleshooting and process improvement.
- ✓ Maintenance should regularly maintain and clean the polishing equipment, including the electrodes and the power supply, to ensure consistent performance.

2.2.6. Real-world examples of electrolytic polishing and coloured etching

Electrolytic polishing is commonly used in various real-world applications to achieve a high-quality surface finish on metal samples. In metallurgical laboratories, electrolytic polishing is used to prepare metallographic samples for microscopic examination. This technique allows metallurgists to study the microstructure of metals, such as examining the grain boundaries, phases, and inclusions, which is essential for quality control and material analysis. The aerospace and automotive industries use electrolytic polishing to prepare metal samples for non-destructive testing (NDT). This includes inspecting the integrity of materials, welds, and components through techniques like dye penetrant testing and magnetic particle inspection. Electrolytic polishing is employed in semiconductor manufacturing to prepare thin metal films or wafers for inspection, electron microscopy, and electron diffraction. It ensures the semiconductor components have a smooth and defect-free surface.

Figure 2.2.29. Electrolytic Polishing & Etching System (source: Metkton USA)

Electrolytic polishing is employed in the electronics industry to create a smooth and defect-free surface on metal components, including connectors and PCBs. This is important for ensuring reliable electrical connections and reducing signal interference. In the production of solar panels and wind turbine components, electrolytic polishing is used to prepare metal surfaces, such as aluminium or copper, for improved energy efficiency and corrosion resistance.

Electrolytic polishing is used to prepare metal components for optical systems and astronomical instruments. Smooth and reflective surfaces are vital for telescopes, mirrors, and lenses to achieve accurate observations and images.

Figure 2.2.30. ElectroMet 4 Polishing & Etching Demo (source: BuehlerMaterials)

Manufacturers of medical implants and devices use electrolytic polishing to improve the surface finish of metal components. This is crucial for reducing the risk of bacterial adhesion and ensuring the biocompatibility of the materials. In the field of dentistry, electrolytic polishing is employed to refine the surface of dental prosthetics like crowns, bridges, and dental implants. A smooth and polished surface helps reduce the risk of plaque buildup and enhances patient comfort.

2.2.7. Colored Etching – purpose, application and demonstration

Coloured etching is a technique used in metallography to selectively stain or colour different microstructural constituents within a metallic material. By employing specific etchants, the technique enhances the visibility and contrast of various phases, inclusions, and grain boundaries.

Figure 2.2.30. Macroscopic Techniques (source: MaterialScience2000)

Coloured etching plays a crucial role in metallography by providing a means to differentiate and highlight various microstructural constituents. It aids in the identification of phases, inclusions, grain boundaries, and other features that may be difficult to distinguish in unattached samples. This enhanced visibility allows for a more comprehensive analysis and understanding of the material's structure and properties. In addition to phase identification, coloured etching enables the characterization of grain size, grain boundaries, second-phase particles, and other microstructural parameters. It facilitates the assessment of microstructural homogeneity, the detection of segregation or intermetallic phases, and the analysis of material defects or anomalies. It allows for improved observation and examination under optical or electron microscopy, facilitating a better understanding of the material's composition, structure, and properties.

Principles of Colour Formation

The etchants may react differently with the constituents due to factors such as chemical composition, crystallographic orientation, and surface condition. These interactions can lead to the formation of coloured compounds or complexes on the surface of the microstructural features, making them more visible and distinguishable.

The principles of colour formation in coloured etching are based on the interaction between the etchant and the microstructural constituents. Colour arises due to the absorption, reflection, or interference of specific wavelengths of light by the coloured compounds or complexes formed on the surface.

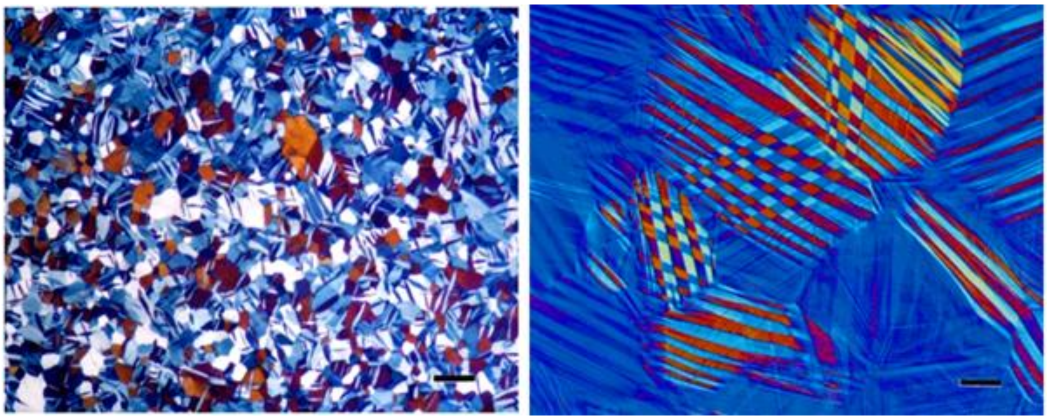


Figure 2.2.31. (left) and Figure 2.2.32. (right): Grain structure on high-purity Zr (left) that was hot worked and cold drawn (note mechanical twins) and viewed in polarized light and of Au – 19Cu-5Al that was polished and cycled through the shape-memory effect to produce martensite and Nomarski differential interference illumination was used to image the surface upheaval due to the shear reaction at the free surface. The magnification bars are 100 and 50 µm, respectively (source: Vac Aero Coloured Metallography)

The colours produced by colour (tint) etchants are visible under bright-field illumination, and in many cases further enhancement is attained using polarized light. Colours are developed by interference in the same manner as with heat tinting or vacuum deposition. As noted, colour is determined by the thickness of the film, usually in the sequence of yellow, red, violet, blue, and green when viewed using white light. With anodic systems, the film forms only over the anodic phase, but its thickness can vary with the crystallographic orientation of the phase. For cathodic systems, because the film thickness over the cathodic phase is generally consistent, only one colour is produced, which will vary as the film grows during etching. Therefore, to obtain the same colour each time, the etching duration must be constant. This can be accomplished by timing the etch and observing the macroscopic colour of the specimen during staining.

Coloured etching Sample Preparation

Metallographic preparation steps are the same as in the previous process followed by metallographic etching.

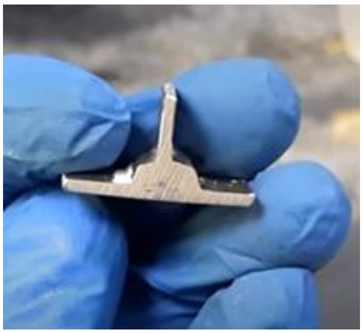


Figure 2.2.33.Preparation of the sample for colour etching (source: Vac Aero Coloured Metallography)

Metallographic etching is carried out on the polished surface by etching with reagents appropriate to the materials of the welded sample which selectively dissolve or colour the constituents present, making them distinguishable from each other. Etching is carried out by dabbing the surface of the test piece with a piece of cotton wool soaked in the reagents. The purpose of the operation is to reveal the crystal structure.

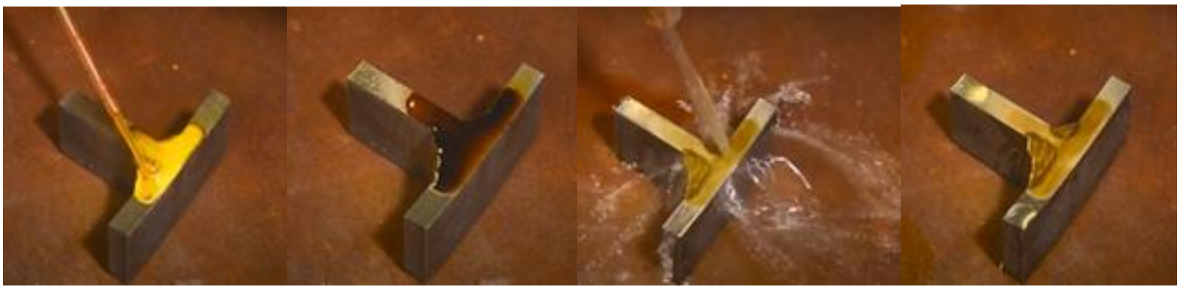


Figure 2.2.34. Weld colour Etching (source: Vac Aero Coloured Metallography)

2.2.8. Hands-on practice with Colored Etching

Materials and Equipment used when colour etching metallographic samples:

- ✓ metallographic samples (typically prepared, mounted, and polished metal samples);

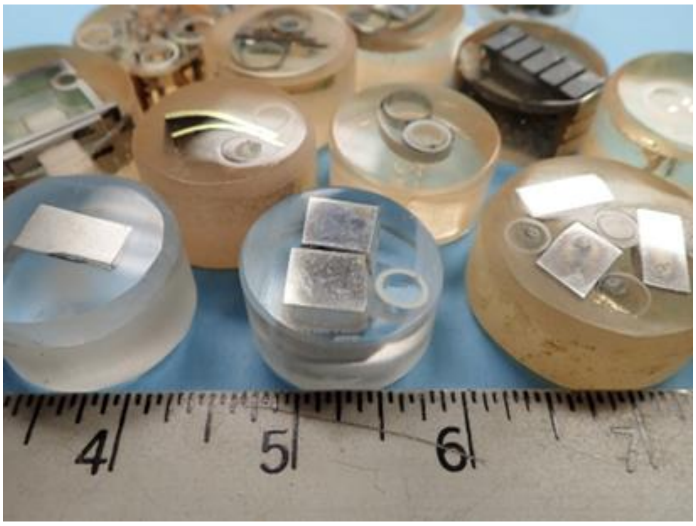
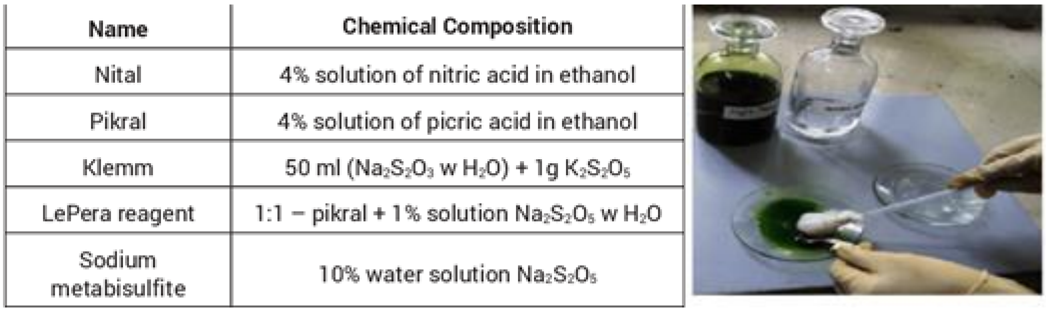


Figure 2.2.35. Metallographic samples (source: Metallurgical Engineering Services, Inc.)

- ✓ chemical reagents specifically formulated to react with different phases or constituents in the metal to produce colours;



Name	Chemical Composition
Nital	4% solution of nitric acid in ethanol
Pikral	4% solution of picric acid in ethanol
Klemm	50 ml ($Na_2S_2O_3$ w H_2O) + 1g $K_2S_2O_5$
LePera reagent	1:1 – pikral + 1% solution $Na_2S_2O_5$ w H_2O
Sodium metabisulfite	10% water solution $Na_2S_2O_5$

Figure 2.2.36. Chemical reagents (source: Micro Lit)

✓ grinding and polishing machines or equipment, abrasive papers, and polishing cloths or pads,

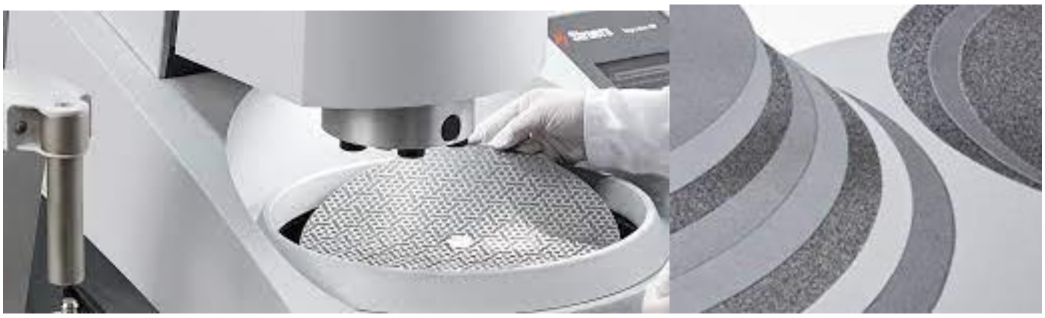


Figure 2.2.37. Grinding and polishing machines or equipment, abrasive papers (source: Kemet International Ltd)

✓ personal protective equipment (PPE) such as safety goggles, gloves, and a lab coat,

✓ microscope to examine the etched samples,

Figure 2.2.38. Metallographic microscope (source: Olympus)

✓ laboratory glassware for storing and handling etchants,

Figure 2.2.39. Laboratory glassware (source: Kemet International Ltd)

- ✓ distilled water for rinsing samples,

Figure 2.2.40. Distilled water (source: Micro Lit)

- ✓ timer or clock to ensure accurate etching times,
- ✓ chemical-resistant materials, such as polyethylene or polypropylene containers for etchant handling,

Figure 2.2.41. Acid Etch PET Plastic Bottles 13g Plastic Storage Jar With PP Screw Cap (source: Micro Lit)

- ✓ ventilated workspace to minimize exposure to fumes and ensure proper chemical disposal,

Procedure for coloured etching

Sample Preparation:

a. The specialist should start with well-prepared metallographic samples that have been mounted and polished to a high level of finish.

b. The specialist must ensure that the samples are clean and free from any contaminants or residues.

Figure 2.2.42. **Sample preparation and sample example** (source: Kemet International Ltd)

Selection of coloured Etchant:

a. The specialist should choose the appropriate coloured etchant based on the specific metal and the features you want to reveal.

b. The specialist should refer to the manufacturer's guidelines or published resources for information on suitable etchants for your application.

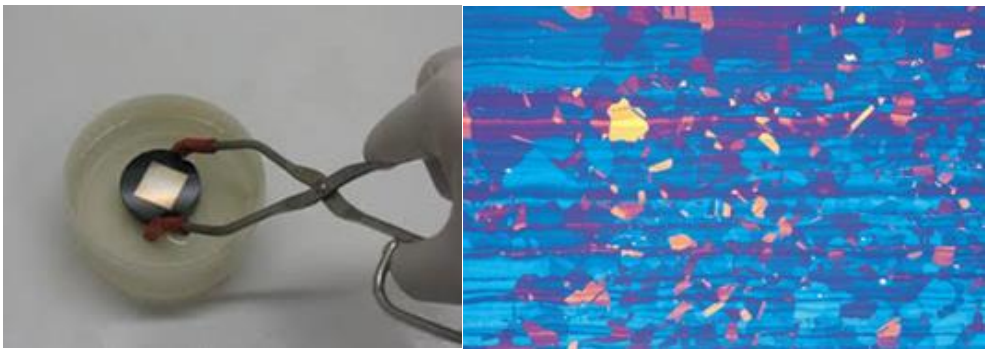


Figure 2.2.43. Sample etching (right) and high alloy steel (left) etched with colour etchants (Beraha, Lichtenegger), with V2A-lye or Adler-etchant containing hydrochloric acid (source: Struers)

Etchant Preparation:

a. The specialist should carefully follow the manufacturer's instructions for preparing the coloured etchant.

b. Some etchants may need to be mixed with a specific solvent or prepared at a particular concentration.

Common Types of Coloured Etchants (please see appendix)

Many metals etched with standard reagents to reveal the grain boundaries often yield only a high percentage of the boundaries, rather than all of the boundaries. Colour etchants, however, reveal the grain structure completely. In the case of metals with annealing twins, it can be very difficult to rate the grain size when a standard etchant reveals a portion of the grain and twin boundaries. In fact, it can be quite difficult to make a precise measurement of the grain size, even manually, with such a specimen, as distinguishing between grain and twin boundaries (the latter must be ignored in the measurement). However, with a colour etched microstructure it is relatively easy to separate grain from twin boundaries, at least manually.

Further, the films grow as a function of crystal orientation. Therefore, one can detect any preferred crystallographic orientation by the narrowness of the colour range present. If a wide range of colours is present in a random pattern, the crystal orientation is random. If a narrow range of colours is present in the grains, then a preferred orientation is present.

Specimen preparation must be better when using colour methods than for black and white methods because the epitaxial grown films are sensitive to residual preparation-induced damage that was not removed. This level of preparation is required in image analysis work and can be easily obtained by a knowledgeable metallographic expert with the proper equipment. Electrolytic polishing is not required to get damage-free surfaces.

The most common tint etchants are those that deposit a sulphide-based interference film on the specimen. These are the best-known tint etches and usually the easiest to use. Klemm and Beraha have developed the most widely used sulphide-based tint etchants using sodium thiosulfate, $Na_2S_2O_3$, and potassium metabisulfite, $K_2S_2O_5$.

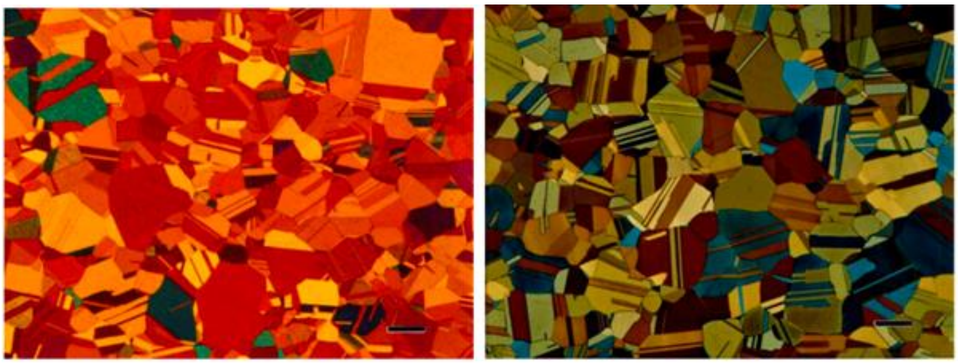


Figure 2.2.44. and Figure 2.2.45. FCC twinned grain structure of cartridge brass, Cu – 30% Zn, after cold reduction by 50% and full annealing, tint etched with Klemm's I (left) and Klemm's III (right) reagents and viewed with polarized light plus sensitive tint. Magnification bars are 200-µm long, respectively (source: Vac Aero Coloured Metallography)

Klemm's I, II, III (Figures 2.2.12 and 2.2.13) and one of Beraha's reagents utilize both ingredients (Figure 2.2.13) while Beraha recommends a range of HCl concentrations used with potassium metabisulfite (Figure 2.2.14) for etching a variety of iron-based alloys.

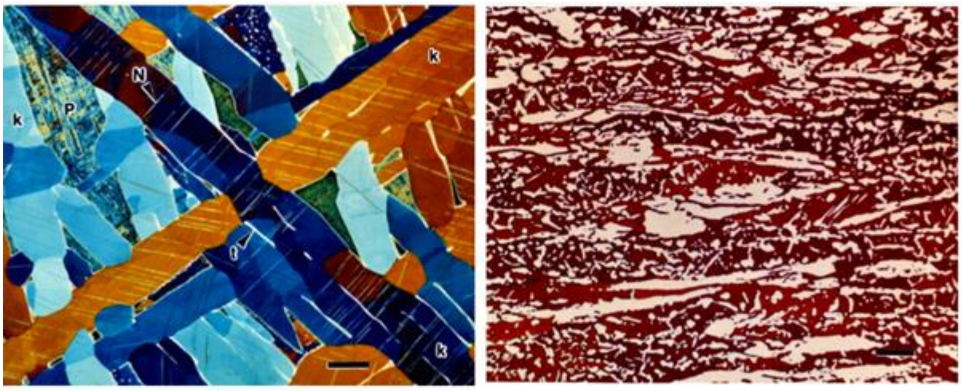


Figure 2.2.46. and Figure 2.2.47. : Fine octahedrite grain structure of the Gibeon meteorite (left) revealed with Beraha's reagent (100 mL water, 10 g $Na_2S_2O_3$ and 3 g $K_2S_2O_5$) and ferrite in 7 Mo PLUS duplex stainless steel plate revealed using Beraha's reagent (85 mL water, 15 mL HCl, 1 g $K_2S_2O_5$). The magnification bars are 500 and 50 µm long, respectively [2.2.10].

These etchants can be used to colour ferrite and martensite in cast iron, carbon and low-alloy steels. The HCl-based reagents vary widely in concentration and can be used to colour the grain structures of stainless steels (2.2.15), Ni-based and Co-based alloys. Sodium metabisulfite has been used in a number of concentrations, from about 1 to 20 g per 100 mL water, and is a safe, reliable, useful colour etch for irons and steels. (source: Vac Aero Coloured Metallography).

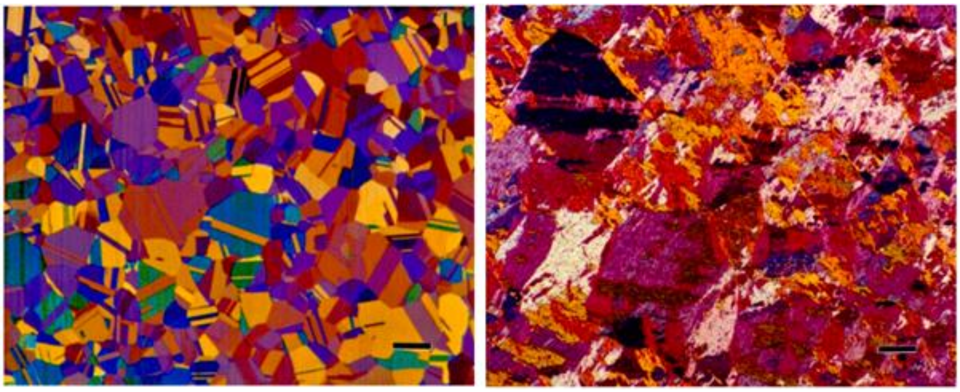


Figure 2.2.48. and 2.2.49: FCC twinned grain structure of heading quality Custom Flo 302 stainless steel revealed using Beraha's B1 reagent and lath martensite grain structure of over-austenitized (1093 ºC) AerMet 100 ultra-high strength steel revealed using 10% sodium metabisulfite. Both viewed with polarized light plus sensitive tint. The magnification bars are 100 µm long (source: Vac Aero Coloured Metallography)

Beraha also developed etchants based upon sulfamic acid, a low concentration organic acid, that have not been used much, although they are quite useful, reliable and easy to use. The sulfamic acid-based reagents are applicable to cast iron, low-carbon and alloy steels, tool steels, and martensitic stainless steels (Figure 2.2.17). Beraha also developed two rather specialized tint etches that deposit cadmium sulphide (Figure 2.2.18) or lead sulphide (Figure 2.2.19) films on the surfaces of steels and copper-based alloys. These two etchants are quite useful, although toxic to work with. His CdS reagent is useful for carbon and alloy steels, tool steels, and ferritic, martensitic and precipitation hardenable stainless steels while the PbS reagent does an excellent job on copper-based alloys and can be used to colour sulphides in steels, white (the specimen is pre-etched with nital and the etch colours the darkened matrix, so that the white sulphides are visible).

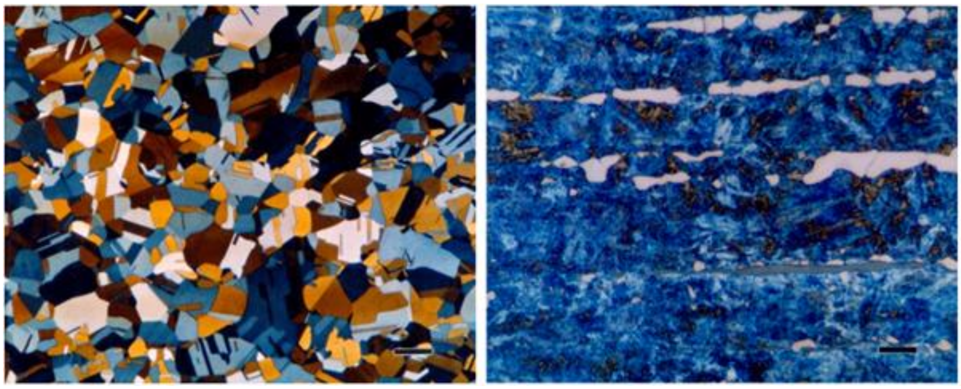


Figure 2.2.50. and 2.2.51: Twinned FCC grain structure in Fe-39% Ni revealed by Beraha's sulfamic acid reagent (left) and tempered martensite grain structure of 416 martensitic stainless steel revealed with Beraha's CdS

reagent (right). The white grains are delta ferrite and the gray inclusions are sulphides. Viewed with polarized light plus sensitive tint. The magnification bars are 100 and 200 µm long, respectively (source: Struers)

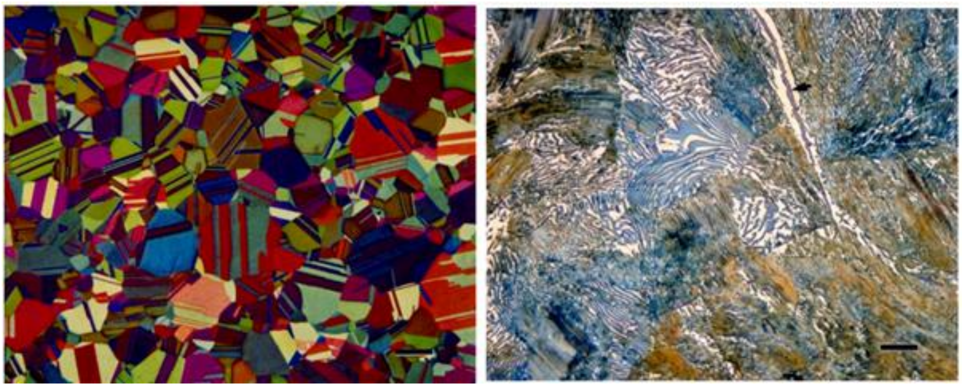


Figure 2.2.52.and 2.2.53: Cartridge brass micrograph shown in Figures 2.2.11 and 2.2.12 tint etched with Beraha's PbS reagent (left) and cementite in a hot rolled Fe-1% C binary alloy coloured with Beraha's sodium molybdate reagent (right). Magnification bars are 200 and respectively 20 µm, (source: Vac Aero Coloured Metallography)

Beraha also developed two tint etchants that utilize molybdate ions in nitric acid. They colour cementite in steels (Figure 2.2.52). He also developed tint etchants that deposit elemental selenium on the surface of steels (Figure 2.2.53), nickel-based alloys and copper-based alloys (Figure 2.2.54).

There are a number of other tint etchants that have been developed by a variety of metallography's. Lichtenegger and Blöch, for example, developed an unusual reagent that will colour austenite (Figure 2.2.56) in duplex stainless steels, rather than ferrite (as nearly all others do).

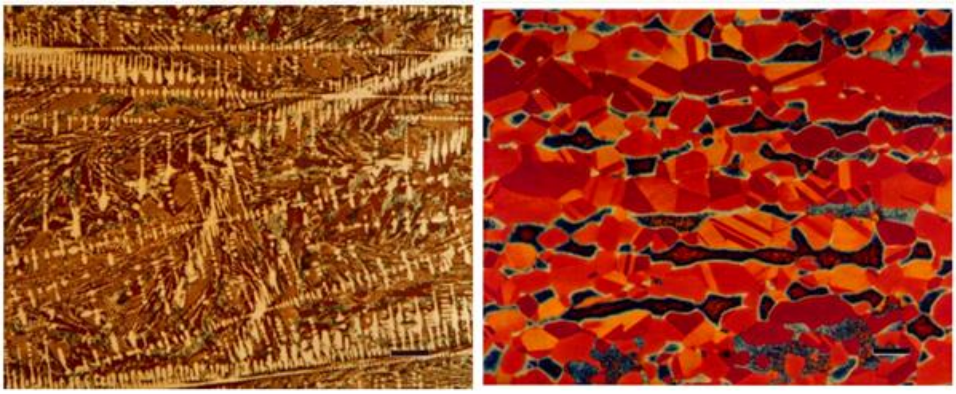


Figure 2.2.54. and 2.2.55: Cementite in the chill cast surface of gray iron etched with Beraha's selenic acid reagent for cast iron (left) and twinned FCC alpha phase and beta phase (mottled and outlined) in Cu-40% Zn revealed using Beraha's selenic acid reagent for copper alloys. Magnification bars are 50 and 20 µm long, respectively (source: Struers)

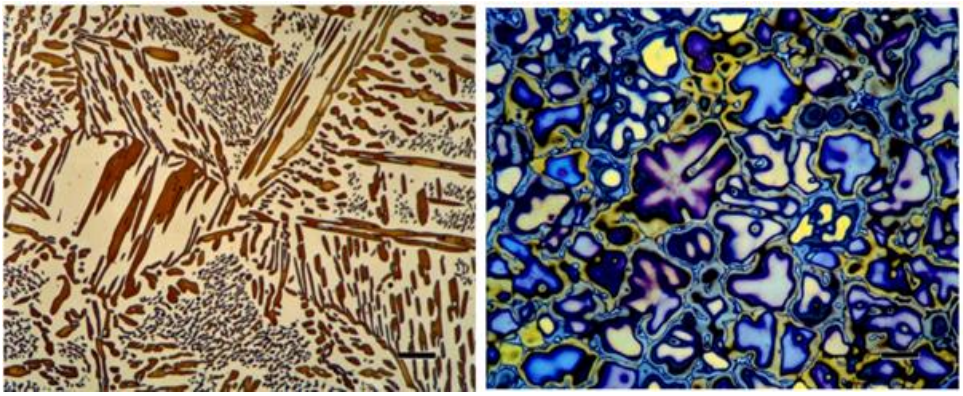


Figure 2.2.56. and 2.2.57: Austenite coloured in ASTM A890 Grade 5A cast duplex stainless steel with the LB1 reagent (left) and the cast grain structure of 206 aluminium revealed using Weck's reagent for Al alloys (right). The magnification bars are 100 and respectively 50 µm long, (source: Vac Aero Coloured Metallography)

Weck developed a number of tint etchants, while utilizing many of them in her research. Several were developed to colour aluminium (Figure 2.2.57) or titanium alloys (Figure 2.2.58). In each case, it is easier to develop good colour with the cast alloys than with the wrought alloys. Two etchants have been found useful for colouring theta phase, AlCu2, in Al-Cu alloys; Lienard developed one of the easiest to use. Several colour etchants have been developed for molybdenum (Figure 2.2.59) and for tungsten.

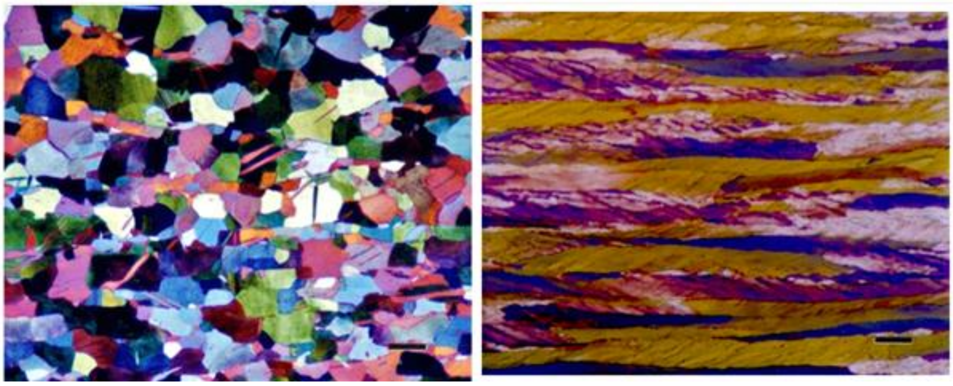


Figure 2.2.58. and 2.2.59: Grain structure of as-rolled CP Ti (ASTM F67, Grade 2) containing mechanical twins (left) etched with modified Weck's reagent and cold rolled pure molybdenum (right) coloured with the reagent developed by Oak Ridge National Laboratory. Magnification bars are 100 and 20 µm long, respectively (source: Vac Aero Coloured Metallography)

Etching Process:

a) The specialist should immerse the polished metallographic sample into the coloured etchant solution.
b) The specialist should monitor the etching process carefully, the time needed for etching varies based on the metal and the specific etchant used.
c) The specialist should use a timer to ensure that the etching time is precise.

d) The specialist should remove the sample from the etchant at the end of the predetermined etching time.

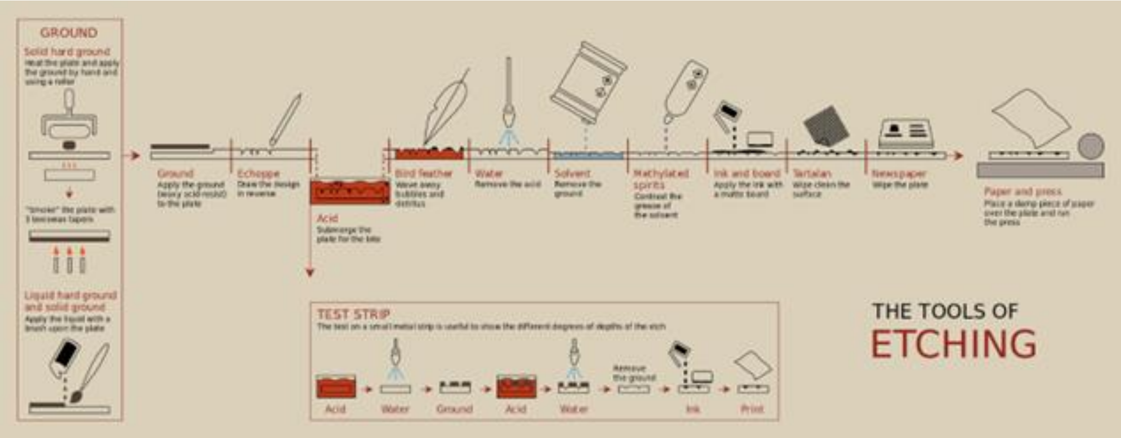


Figure 2.2.60. The tools of the etching's modern technique (source: Wikipedia)

Rinsing and Cleaning:

a) The specialist should rinse the etched sample thoroughly with distilled water to remove any residual etchant.

b) The specialist should use a soft brush or scrubbing pad to gently clean the surface if necessary.

Figure 2.2.61. **Distilled water for sample rinsing** (source: Mirco Lit)

Microscopic Examination:

a) The specialist should allow the sample to dry or use compressed air to remove excess moisture.

b) The specialist should examine the etched sample under a microscope to observe the coloured microstructural features.

c) The specialist should document and photograph the results for analysis and record-keeping.

Figure 2.2.62. Metallurgical Microscopes (source: Nikon)

Repeatability and Optimization:

If needed, the specialist should repeat the process with variations in etching time or etchant concentration to optimize the results and enhance the contrast.

Proper sample preparation and adherence to safety precautions are essential to obtain accurate and meaningful results.

2.2.9. Common issues and solution when using the coloured etching for metallographic sample preparation

When using coloured etching for metallographic sample preparation, it is essential to carefully control the etching conditions, understand the chemistry of the etchant, and maintain meticulous sample preparation to achieve the desired results and accurate microstructural analysis. Experimentation and practice are often necessary to optimize the etching process for specific materials and microstructures.

Common Issues

Inadequate Contrast or Colour Development

Issue: The etched sample does not exhibit the expected contrast or colours.

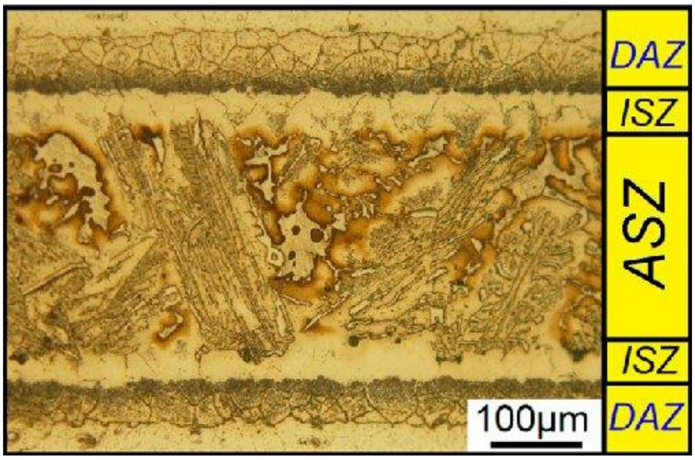


Figure 2.2.63. Optical micrograph showing the microstructure gradient in the HX/Ni-13Cr-4.5Si-4.2Fe-2.8B/HX brazed joint. (source: Ghasemi, Ali & Pouranvari, Majid. (2018). Intermetallic phase formation during brazing of a nickel alloy using a Ni–Cr–Si–Fe–B quinary filler alloy. Science and Technology of Welding and Joining. 24. 1-10. 10.1080/13621718.2018.1553280.)

Solution: The specialist verifies the compatibility of the etchant with the specific metal and microstructure, he/she needs to ensure the etchant concentration and etching time are appropriate for the sample. Consider alternative etchants that may provide better results.

Over-Etching

Issue: Over-etching occurs when the sample exhibits excessive colour development, making it challenging to discern microstructural features.

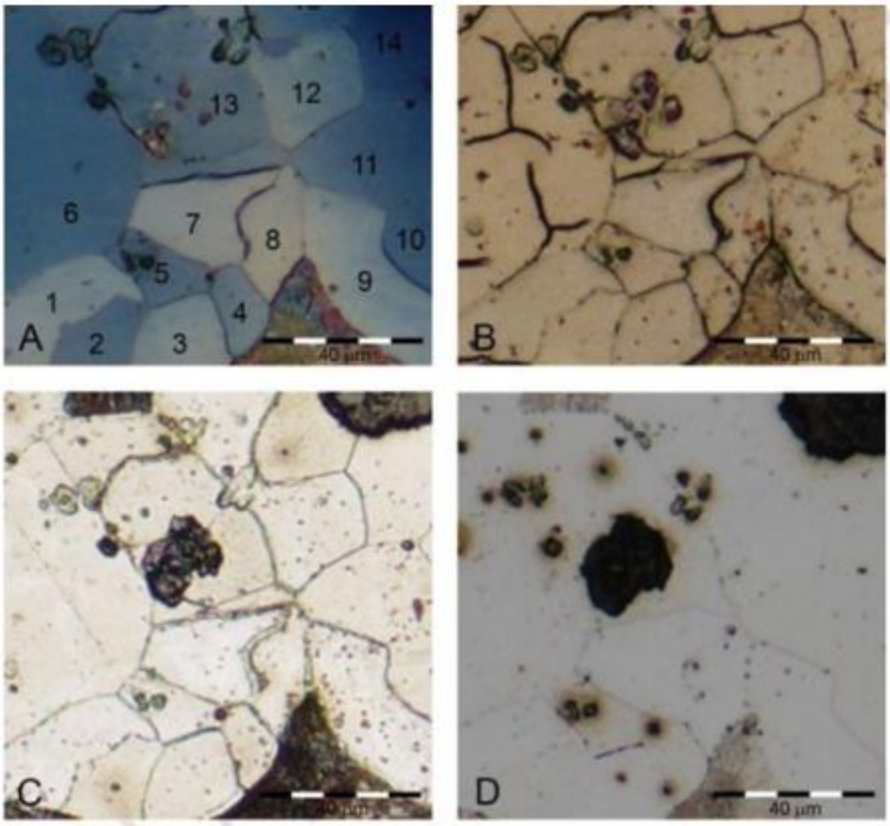


Figure 2.2.64. Optical micrographs of specimen (A) after colour etching with the Beraha-I solution, (B) after over etching with the Beraha-I solution, (C) after removing the interfering layer by 1% sulfuric acid, and (D) after etching by Nital. (source: Ghasemi, Ali & Pouranvari, Majid. (2018). Intermetallic phase formation during brazing of a nickel alloy using a Ni–Cr–Si–Fe–B quinary filler alloy. Science and Technology of Welding and Joining. 24. 1-10. 10.1080/13621718.2018.1553280.)

Solution: The specialist should reduce the etching time or concentration to prevent over-etching and monitor the sample closely during etching to avoid excessive colour development.

Under-Etching

Issue: The sample does not show the desired level of colour or contrast.

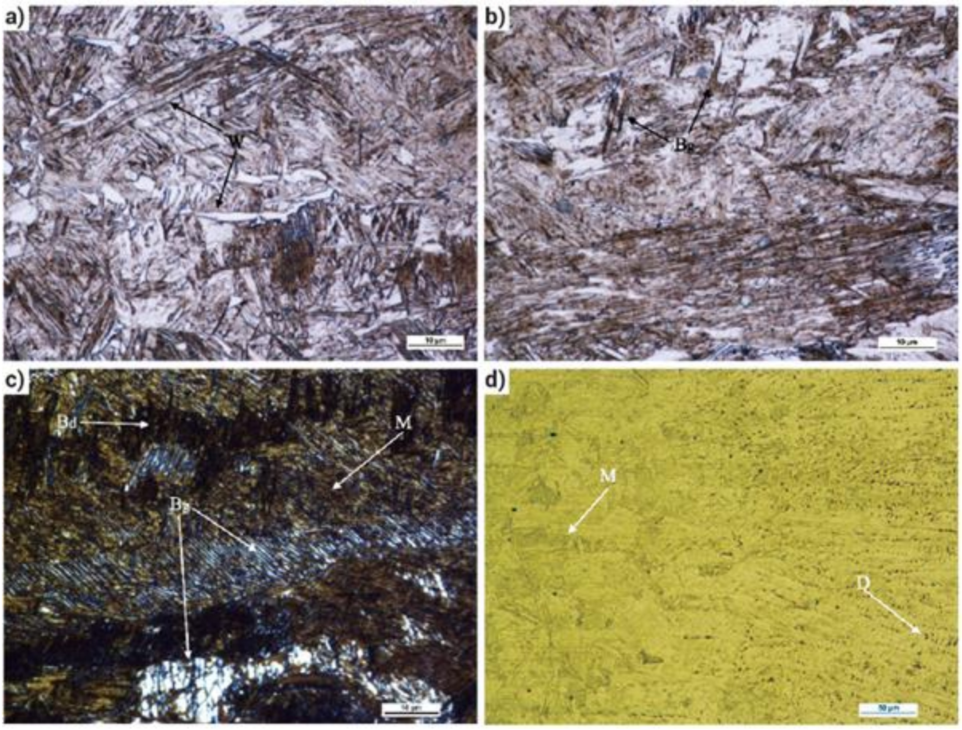


Figure 2.2.65. TRIP steel microstructure, LOM: a) HAZ, Nital etched, b) Fusion zone, Nital etched, c) Fusion zone, Nital + Klemm reagent etched, d) Fusion zone and HAZ, Pikral etched (source: Dobras, Daniel & Rutkowska-Gorczyca, Małgorzata. (2018). Application of color etching to study the microstructure of TRIP steel after laser remelting. Welding Technology Review. 90. 10.26628/wtr.v90i12.984.)

Solution: The specialist should increase the etching time or concentration to achieve the desired contrast. He/she needs to ensure the sample surface is properly cleaned and free of contaminants before etching.

Uneven Colour Distribution

Issue: Colours are not evenly distributed on the sample, resulting in variations in contrast.

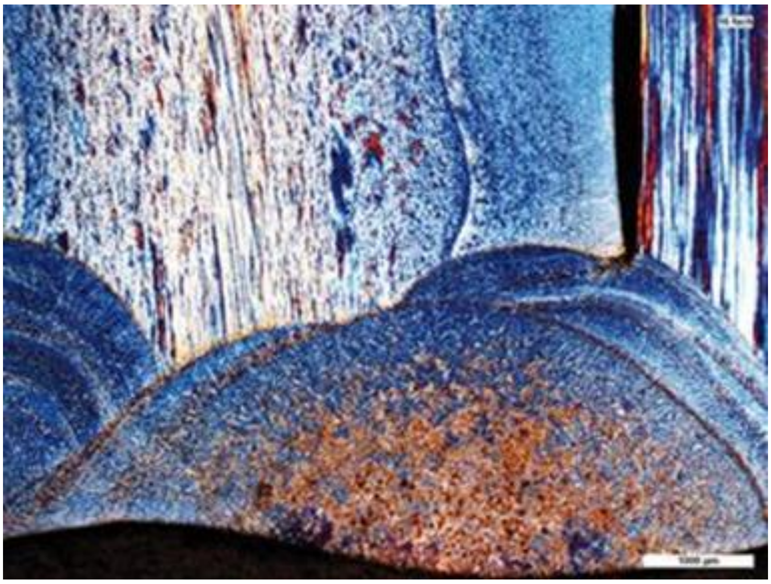


Figure 2.2.66. Colour etching of various grain or mixed-crystal areas and sulphate layers of different thicknesses - Laser welding connection of various austenitic steel wires (B) (source: Leica Microsystems)

Solution: The specialist should improve the sample preparation, ensuring a consistent finish before etching and agitate the etchant solution during the process to maintain even colour distribution.

Staining or Discoloration

Issue: The sample may exhibit unwanted staining or discoloration during the etching process.

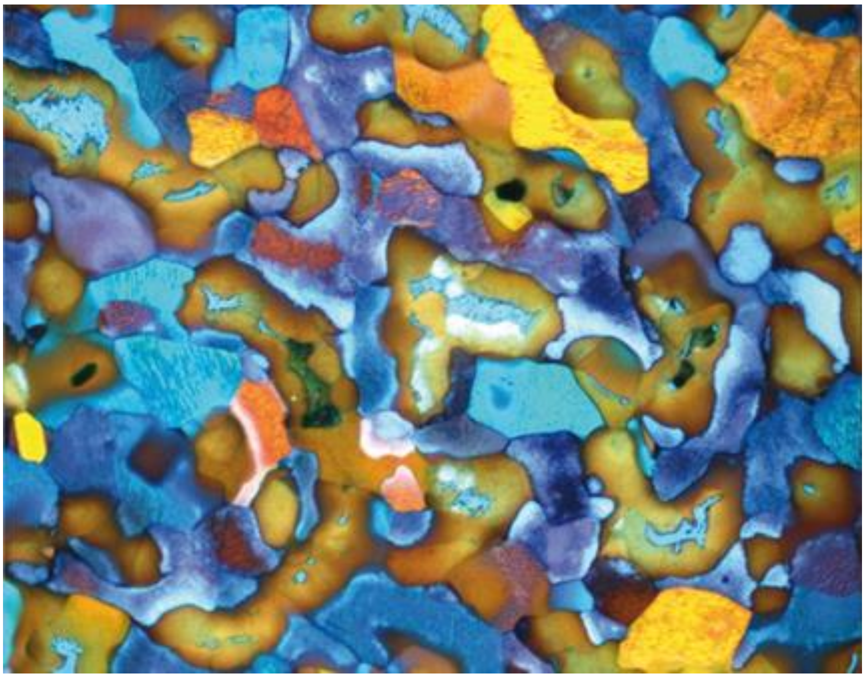


Figure 2.2.67. Concentration differences in a bronze wire (K) (source: Leica Microsystems)

Solution: The specialist should review the compatibility of the etchant with the metal and microstructure and ensure that the sample is properly cleaned before etching to avoid contaminants that can cause staining.

Contamination

Issue: Contaminants on the sample surface can interfere with the etching process.

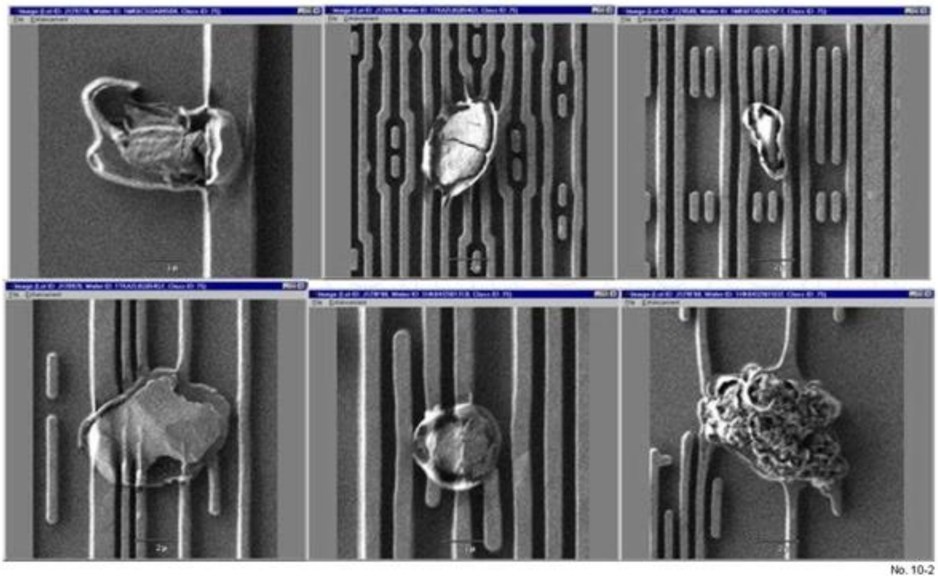


Figure 2.2.68. Killer defects generated in aluminium metal etch processes. (source: Steel Data)

Solution: The specialist should thoroughly clean and degrease the sample before etching to remove contaminants. Use clean laboratory equipment, such as containers and brushes, to minimize the risk of contamination.

Safety Precautions

Issue: Inadequate safety precautions may lead to personal exposure to hazardous etchants.

Figure 2.2.69. Personal Protective Equipment (PPE) (source: MicroLit)

Solution: The technician should adhere to proper safety protocols and use personal protective equipment (PPE), including safety goggles, gloves, and a lab coat and work in a well-ventilated area or under a fume hood to reduce exposure to fumes.

Inconsistent Results

Issue: When obtaining consistent results across different samples can be challenging.

Solution: The technician should maintain precise control of etchant concentration, etching time, and sample preparation techniques for repeatability and document and standardize the etching process to achieve consistent outcomes.

Inadequate Record-Keeping

Issue: Poor documentation makes it difficult to analyse and replicate results.

Solution: The technician should maintain detailed records of the etching process, including etchant composition, concentration, etching time, and sample characteristics, to facilitate future analysis and troubleshooting.

2.2.10. Handling and disposal of reagents from Colored Etching

Handling and disposal of reagents used in coloured etching are critical aspects of safe and environmentally responsible laboratory practices. coloured etching reagents often contain chemicals that can be hazardous, so it's essential to follow proper procedures for both handling and disposing of these materials. Proper handling and disposal of reagents used in coloured etching are essential for maintaining a safe and environmentally responsible laboratory. Always prioritize safety and compliance with local regulations when working with hazardous materials.

Handling Reagents for coloured Etching

Personal Protective Equipment (PPE): The specialist needs to wear appropriate personal protective equipment, including safety goggles, gloves, a lab coat, or protective clothing, and possibly a face shield to protect against splashes or contact with reagents.

Ventilation: The specialist needs to perform reagent handling in a well-ventilated area, such as a fume hood or a space with good room ventilation, to minimize exposure to fumes or vapours.

Labelling: The specialist needs to ensure clear labelled containers that hold reagents, with their names, compositions, hazards, and any necessary handling precautions. The use of specific labels and record keeping is mandatory.

Use Appropriate Containers: The specialist needs to store reagents in chemical-resistant containers designed for this purpose; glass or plastic containers are common choices.

Minimize Skin Contact: The specialist should avoid direct skin contact with the reagent; if contact occurs, rinse the affected area immediately with plenty of water.

No Eating or Drinking: The specialist needs to ensure not to eat, drink, or smoke while handling reagents to prevent accidental ingestion.

Handle with Care: The specialist needs to be cautious when transferring or mixing reagents to avoid splashes or accidents.

Check Compatibility: The specialist needs to ensure that materials used for handling reagents (containers, stirrers, etc.) are compatible with the specific reagent and won't react with it.

Education and Training: All personnel working with reagents used in coloured etching need to be educated on the risks and safety procedures; ongoing training and awareness are crucial.

Disposal of Reagents from coloured Etching

Regulatory Compliance: The laboratory needs to adhere to local, state, and federal regulations governing the disposal of hazardous waste, these regulations can vary by location. The specialist needs to keep different reagents separate, and do not mix them during disposal. In some cases, the specialist may need to neutralize the reagent before disposal, he/she should follow chemical compatibility guidelines for safe neutralization.

Consult with Experts: If the specialist is uncertain about the proper disposal methods, consult with a qualified chemist, waste disposal expert, or your organization's environmental health and safety department.

Label Waste Containers: The specialist needs to clearly label waste containers with their contents and any hazards to ensure safe handling during disposal, he/she should ensure that waste containers are tightly sealed to prevent leaks or spills.

Waste Disposal Services: The laboratory needs to arrange for the pickup and disposal of hazardous waste through licensed and authorized waste disposal services or facilities, they should not attempt to dispose of hazardous waste on your own. The laboratory needs to maintain records of the disposal process, including dates, disposal methods, and any relevant safety data sheets (SDS) for reference. Laboratories need to be prepared for emergencies and have spill response kits, safety showers, and eyewash stations available in the area where reagents from coloured etching are handled.

2.2.11. Troubleshooting and tips in coloured etched metallographic samples

Several factors can influence the colour development in coloured etching. These include the composition and concentration of the etchant, temperature, etching time, and surface preparation of the sample. Additionally, the presence of alloying elements, impurities, or specific crystallographic orientations can affect the colour intensity and contrast.

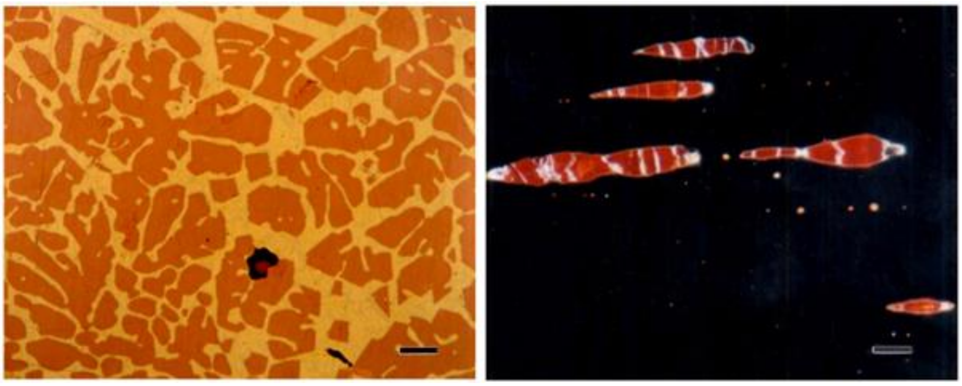


Figure 2.2.70. (left) and 2.2.71 (right) showing natural reddish-purple colour of the $AuAl_2$ intermetallic (left) in bright field and cuprous oxide's characteristic ruby red colour in dark field illumination (electrolytic copper specimen). The magnification bars are 50 and respectively 10 µm (source: Vac Aero Coloured Metallography)

Understanding these factors is crucial for achieving optimal colour development and contrast in coloured etching. Careful control and optimization of the etching parameters are necessary to obtain reliable and reproducible results.

2.2.12. Real-world examples of coloured etching

Coloured etching is a technique used in the field of metallography to reveal and highlight specific microstructural features in metallic materials. In the automotive and aerospace industries, coloured etching is often used to inspect welds in materials like aluminium and steel. This technique helps reveal the integrity of the weld, the presence of defects, and the quality of the weld bead and heat-affected zone. Coloured etching is an important tool in forensic engineering and failure analysis. It aids in identifying the causes of component failures, such as the presence of cracks, voids, or inclusions within a material. In the construction and civil engineering sectors, coloured etching is employed to inspect structural steel components. It helps assess the quality of steel and the presence of any inclusions, segregations, or defects that may compromise structural integrity. coloured etching is employed to examine engine components, such as pistons and crankshafts. It helps assess the quality of the material and detect any defects or irregularities that may affect performance.

Aircraft manufacturers and maintenance facilities use coloured etching to inspect critical components like turbine blades and landing gear. This technique assists in identifying microstructural issues that could lead to component failure. In the manufacturing of medical implants and devices, coloured etching is used to assess the quality and integrity of materials like stainless steel, titanium, and cobalt-chromium alloys. It helps ensure that medical devices meet stringent safety and performance standards.

In the semiconductor industry, coloured etching is used to inspect the quality of silicon wafers. The technique can reveal crystallographic defects, grain boundaries, and other features that can affect the performance of semiconductor devices. coloured etching is used in non-destructive testing of materials used in the oil and gas sector. It helps identify flaws or weaknesses in components like pipelines, pressure vessels, and welds, ensuring their reliability and safety.

Researchers and engineers use coloured etching to study the microstructure of various materials, including metals and alloys. It is employed to identify grain boundaries, phases, and inclusions, which are essential for understanding material properties and behaviour. In metallurgy and materials science use coloured etching to investigate the microstructure and phase composition of various metals and alloys. This information is crucial for designing and improving materials for specific applications.

Appendix

Etch Compositions

Klemm's I	50 mL stock solution, 1 g K2S2O5 (stock solution is water saturated with Na2S2O3
Klemm's III	5 mL stock solution, 45 mL water, 20 g K2S2O5 (stock solution as for Klemm's I)
Beraha's 10/3 reagent	10g Na2S2O3, 3g K2S2O5 and 100 mL water
Beraha's BI	100 mL stock solution (1000 mL water, 200 mL HCl, 24 g NH4FHF) plus 0.1 – 0.2 g K2S2O5 for martensitic stainless steel
Beraha's BI	100 mL stock solution (1000 mL water, 200 mL HCl, 24 g NH4FHF) plus 0.3 – 0.6 g K2S2O5 for austenitic and ferritic stainless steels.
Beraha's sulfamic acid reagent No. III	100 mL water, 3 g K2S2O5, 2 g NH2SO3H (two other similar compositions were published) for carbon and alloy steels.
Beraha's sulfamic acid reagent No. IV	100 mL water, 3 g K2S2O5, 1 g NH2SO3H, 0.5 – 1 g NH4FHF for high-Cr tool steels and martensitic stainless steels.
Beraha's CdS and PbS reagents	CdS stock solution: 1000 mL water, 240 g Na2S2O3 • 5H2O, 20-25 g cadmium chloride (or cadmium acetate), 30 g citric acid; PbS stock solution: 1000 mL water, 240 g Na2S2O3 • 5H2O, 30 g citric acid, 24 g lead acetate
Beraha's selenic acid reagent for cast iron	100 ethanol, 2 mL HCl, 1 mL selenic acid

2.2.13. Comprehend and describe the differences in terms of their effects on surface finish and microstructure of the sample

Microstructure analysis and examination of samples prepared through coloured etching technique

Microstructure analysis and examination are essential steps in the study of materials to understand their internal structure, grain boundaries, phases, defects, and other microstructural features.

Each of the surface treatment techniques—electrolytic polishing, coloured etching—offers unique advantages and considerations for microstructure analysis:

Microstructure Analysis after Electrolytic Polishing:	**Microstructure Analysis after Coloured Etching:**

Electrolytic polishing produces smooth and mirror-like surfaces on metal samples without introducing mechanical deformation. Microstructures revealed after electrolytic polishing are less likely to be altered by surface artefacts compared to other techniques. After electrolytic polishing, the metal sample can be directly observed under an optical or electron microscope for microstructural analysis. Grain boundaries, phase boundaries, and inclusions can be clearly identified and analysed.	Coloured etching uses specific etchants to reveal and highlight different microstructural features on metal surfaces, providing a contrast in colours or shades. Coloured etching is particularly useful for distinguishing different phases, grain boundaries, and intermetallic compounds in the microstructure. The contrast provided by coloured etching makes it easier to identify and characterize microstructural constituents. Care must be taken to use appropriate etchants and standardized procedures to ensure reliable and accurate results.

Overall, the two techniques—electrolytic polishing, coloured etching—play important roles in microstructure analysis. The choice of technique depends on the specific requirements of the analysis, the material being studied, and the desired level of surface finish and accuracy. In some cases, multiple techniques may be combined to gain a comprehensive understanding of the material's microstructure. Additionally, caution should be exercised to avoid introducing artefacts that could mislead the microstructural analysis. Advanced microscopy techniques, such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM), are often employed to achieve high-resolution microstructure examination after surface treatment.

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2.3. Image analysis as a tool in metallographic examination

Metallurgist have relied, in general, on qualitative descriptions of microstructures, for some sample the accuracy of such rating, although limited, is adequate for intended purpose, but in many cases, it is very important to obtain quantitative information; today, modern image analysis software connected to light microscopes allow for the formation of a quantitative output, such as linear or area measurements or phases or porosity percents.

In metallography, image analysis most often refers to digital quantitative evaluation of a microstructure image. Modern image analysis systems have become an important tool for quantitative measurements of microstructural aspects; quantitative measurements are typically length, width, and area, they are used for the evaluation of metallographic characteristics such as grain size, inclusions, layers, phases or other constituents.

In the past, the measurements had to be performed on image projectors, with magnification equal to the objectives, or photomicrographs, for which the concept of real magnification was important. Today, modern programs allow the association of the displayed image to the objective and to be able to carry out direct measurements on the monitor screen. This allows to carry out immediate quantitative analyses avoiding the use of correction parameters.

In general, the following precautions must be followed to obtain reliable and repeatable results.

2.3.1. Sampling and samples preparation

The samples must be selected and prepared properly. Random sampling is frequently employed to obtain statically significant data, because this implies that all regions and orientations of a metal item have an equal opportunity to be cut and tested. However, often it isn’t possible, and the sampling must be carried out on evaluation based on the investigated component (e.g., Welding joint).

It is difficult to establish rigid rules concerning the number and location of test samples.

After the samples are cut and identified, they must be prepared for the examination. Sample preparation (polishing and etching) must be performed carefully in order to avoid artifacts as distortions, engravings, rounded edges, etc.

Automatic grinding and polishing devices are recommended to obtain flatness and repeatability of results.

2.3.2. Field Selection

For many kinds of measurement, the technician/operator must decide how many fields are to be measured, how to space the fields, and the best magnification, for other kinds it is reported in standard methods or product specification (e.g., ASTM E562).

In general, as the magnification is raised, the field area is decreased; if the magnification is raised from 100X to 200X, four times as many fields must be measured to cover the same area.

The measurement area needed to obtain a certain measurements accuracy depends on the homogeneity of the structure. The number of the fields observed influences the accuracy of the measurements; in general, to reduce the observed accuracy to one-half the value obtained after a number x of measurements, four times x measurements must be performed in addition.

2.3.3. 2D measurements

The main quantitative outputs that may be requested in metallographic analysis are linear or area measurements. Usually, all image acquisition programs allow to perform direct measurements in real time on the monitor, associating the correct magnification on the basis of the object used.

Typically, linear measurements such as length, thickness, or area measurements by closed lines or image elaboration (by contrast) are required.

These measurements must be considered subject to a statistical error that can be calculated based on periodic check procedures with certified micrometres and data provided by the microscope manufacturer.

The main cases in which it may be required are listed below.

- Characteristic measurements of Welding joints (weld throat a, weld sides Z, imperfections length)
- Depth of decarburization.
- Depth of surface heat treatments (surface hardening, carburizing, nitriding, cyaniding, etc).
- Coating thickness (metallic coating, organic coating, ceramic coating, thermal spray).
- Depth of corrosion (after service or corrosion test).

A number of measurements should be made at spaced intervals in order to obtain a statistical estimate of the real depth.

2.3.3.1. Decarburization

Usually, the depth of decarburization is measured by optical microscope in order to increase the accuracy of the measurement, also microhardness or carbon analysis can be performed.

Often the decarburization layer isn’t uniform in thickness, for example, corners of rectangular sections exhibit greater decarburization depth than the faces, than several samples are selected from different test location.

Before polishing, the sample should be mounted by Bakelite or other hard resin to avoid rounding of the edges.

The microstructure is revealed by using “Nital” or “Picral” or other reagents as required.

Depth of decarburization is measured using the microscope acquisition software, after properly verifying that the magnification on the screen is correct.

The magnification is chosen based on the structure observed and the technician/operator should do at least five measurements, in random locations along the surface of each sample, then can calculate the average and standard deviation.

If a free-ferrite layer is present, thickness measurement is easier because there is a good contrast between this zone and adjacent partially decarburized layer.

In a hypoeutectoid ferrite pearlite steel sample, the operator looks for the depth where the amount of pearlite and ferrite appears to become constant.

While the evaluation is more difficult in a sample which the partially decarburized layer consists of martensite and/or bainite, where no clear difference is observed.

2.3.3.2. Depth of surface heat treatments

Carburizing, nitriding, cyaniding, and flame or induction hardening treatments can be measured with the same method as decarburization. The accuracy of the microscopic method depends on the surface and core microstructures. For example, carburization is easier to evaluate in the unhardened steel, while the nitriding can be difficult to estimate.

Often, it is preferable to determine the depth by microhardness measurements or chemical analyses.

2.3.3.3. Coating thickness

There are several types of coating, we can have metallic coatings, such as zinc plating, chrome plating, nickel plating, organic coatings, ceramic coatings, and obtained with different processes such as thermal spray.

In general, the coating efficacy is determined by its thickness and the measurement must be performed in several fields, with particular attention to the edges and corners.

2.3.3.4. Depth of corrosion

The corrosion behaviour of a metal alloy is often estimated in penetration per unit of time (mm/year). For this reason, the measurement of the corrosion depth is often required, above the service or corrosion tests aimed at predicting the behaviour of a specific alloy.

If we want to estimate general corrosion it is necessary to know the starting thickness. The measurement of a not uniform corrosion or localized corrosion is easier than a general corrosion.

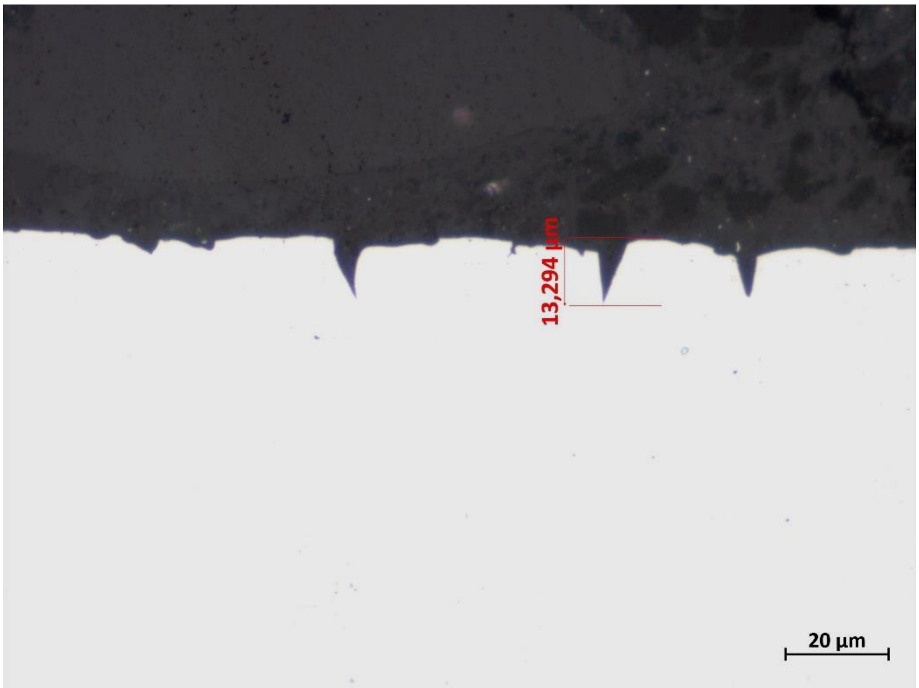


Fig. 2.3.1– measurement of localized corrosion depth on a specimen after laboratory corrosion tests. Source: IIS Laboratory

2.3.4. Determination of volume fraction

The determination of the volume fraction of a phases, constituent, and porosity in a microstructure is the most important and most common use of the image analysis.

Typically, this estimation can be carried out by two main methods:

- Determination of volume fraction by Point count method (ASTM E562).
- Determination of volume fraction by image analysis (ASTM E1245).

The determination is an estimate of the fraction of area, and it is subject to error.

2.3.4.1. Point counting

The point counting method for determination of volume fraction is described in the standard ASTM E562. It was proposed by three different scientists independently (Thomson in 1933, Glasgolev in 1933, and Chalkley in 1943).

A two-dimensional (or one dimensional) points grid is placed above the image, and then counting how many grid points are in the phase to be determined. Currently, the grid is applied using the image acquisition programs.

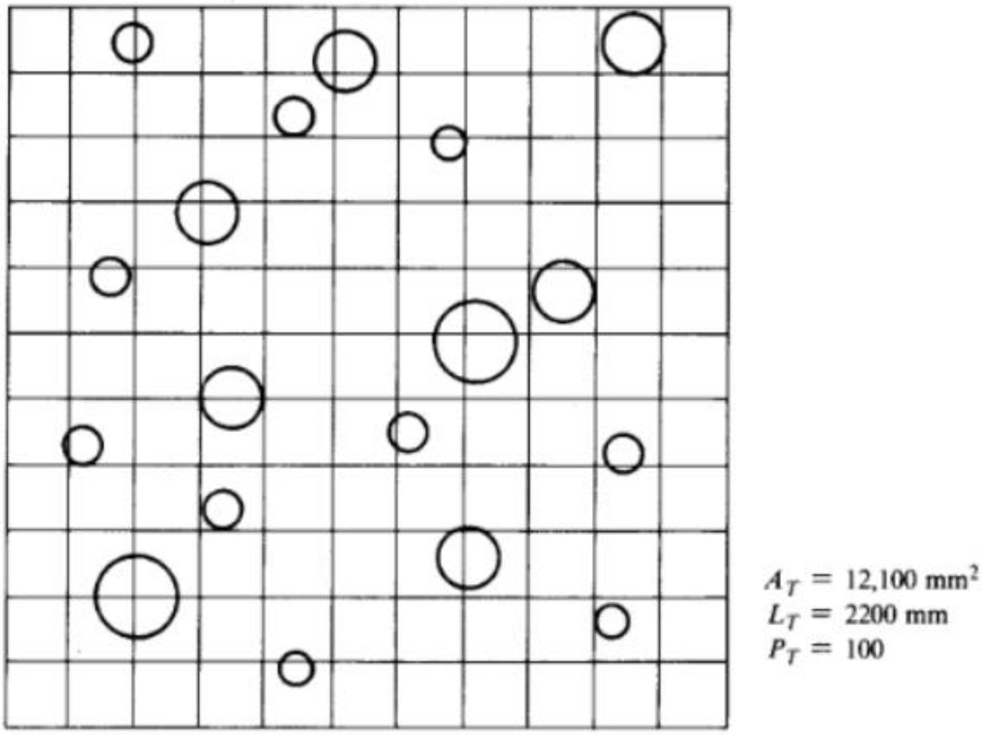
The magnification should be high enough so that the location of the test points with respect to the elements can be clearly discerned; it is important to choose a compromise between resolution and field size.

The point grid should be applied to a field selected at random; the points lying in the phase of interest P_a (for example delta ferrite) are counted, points lying in the particle or phase boundary are counted as one-half. The point fraction is calculated as shown below.

$$P_P = \frac{\Sigma P_\alpha}{P_T} = \frac{\Sigma P_\alpha}{nP_0}$$

Where n is the number of fields, P_0 is the number of the grid points.

Points grids of 16, 25, 49, 64, or 100 points are usually used; in order to measure a phase equal to about 50%, a grid of 16 point can be used. If the phase fraction decreases, greater points grid must be used (see tab 5.1).



Areal analysis

$$V_{V\alpha} = \frac{\Sigma A_\alpha}{A_T} = \frac{\text{areas of circles}}{\text{frame area}} = \frac{884.75}{12{,}100} = 0.073$$

Lineal analysis

$$L_{L\alpha} = \frac{\Sigma L_\alpha}{L_T} = \frac{\Sigma\ \text{intercept lengths}}{\text{total line length}} = \frac{152.3}{2200} = 0.069$$

Point count

$$P_{P\alpha} = \frac{\Sigma P_\alpha}{P_T} = \frac{\Sigma\text{points in } \alpha}{\text{total points}} = \frac{5 + 2\left(\frac{1}{2}\right)^{*}}{100} = 0.06$$

[*] Tangents to particles counted as $\frac{1}{2}$

Fig. 2.3.2– example illustrating three methods for estimating VV using an idealized microstructure of spherical intersected by a sectioning plane. Source: [2.3.3] ASTM E562:2011 – Standard Test Methods for Determining Volume Fraction by Sistematic Manual Point Count

Visual Area Fraction Estimate Expressed as a Percentage	***Grid Size (Number of Points, P_T)***
2 to 5 %	100
5 to 10 %	49
10 to 20 %	25
>20 %	16

Tab. 2.3.1 – guideline for grid size selection. Source: [2.3.3] ASTM E562:2011 – Standard Test Methods for Determining Volume Fraction by Sistematic Manual Point Count.

This method can be used to determine the fraction of a metallic phase, a precipitate, non-metallic inclusions, or porosity.

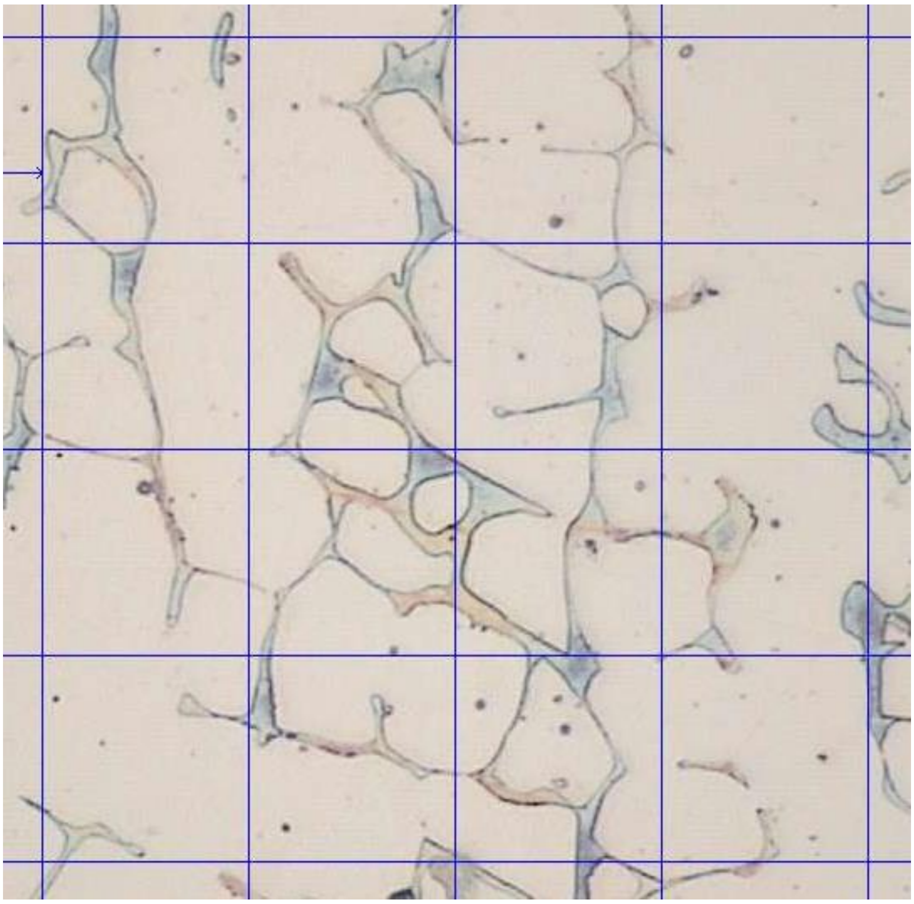


Fig. 2.3.3– example of determination of volume fraction of delta ferrite by a 25 points grid. Source: IIS laboratory.

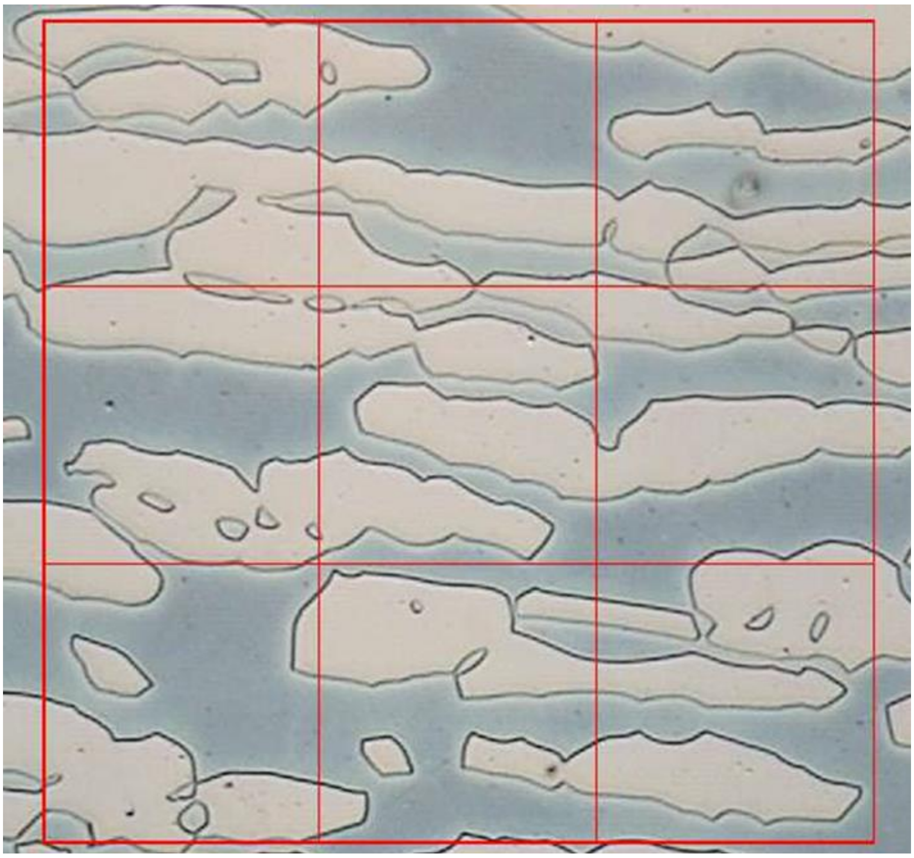


Fig. 2.3.4– example of determination of volume fraction of delta ferrite by a 16 points grid. Source: IIS laboratory.

2.3.4.2. *Determination of volume fraction by image analysis*

As an alternative to point counting method, an automatic image analyser can be used; this method is described in ASTM E1245.

The image is detected using a television type scanner tube and displayed on high resolution video monitor. Inclusions, phases, and porosity are detected and highlighted based on their grey-level intensity differences compared to each other and unetched matrix.

Measurements are made based on the nature of the discriminated picture point elements in the image. These measurements are made on each field of view selected. Statistical evaluation or feature-to-feature variability of the measurements.

This procedure is not suitable for assessing the exogenous inclusions in steels and other metals. Because of the sporadic, unpredictable nature of the distribution of exogenous inclusions, other methods involving complete inspection, for example, ultrasonic, must be used to locate their presence.

In general, test specimen orientation should be parallel to the hot-working axis and, most commonly, taken at the quarter-thickness location. Other test locations may also be sampled, for example, subsurface and centre location, as desired or required.

The surface to be polished should be large enough to allow the measurement of at least 100 fields at the necessary magnification. A minimum surface area of 160 mm^2 is preferred.

Metallography specimen preparation must be carefully controlled to produce acceptable quality surface for image analysis. The polishing procedure must not alter the true appearance of the constituents on the plane of polish by producing excessive relief, pitting, cracking or pullout. Minor fine scratches, such as from a 1 µm diamond abrasive, do not usually interfere with inclusion detection but heavier scratches are to be avoided. Proper cleaning of the specimen is necessary. Use an automatic grinding and polishing device is recommended.

The microscope light source should be checked for correct alignment and the illumination intensity should be adjusted to the level required by the monitor.

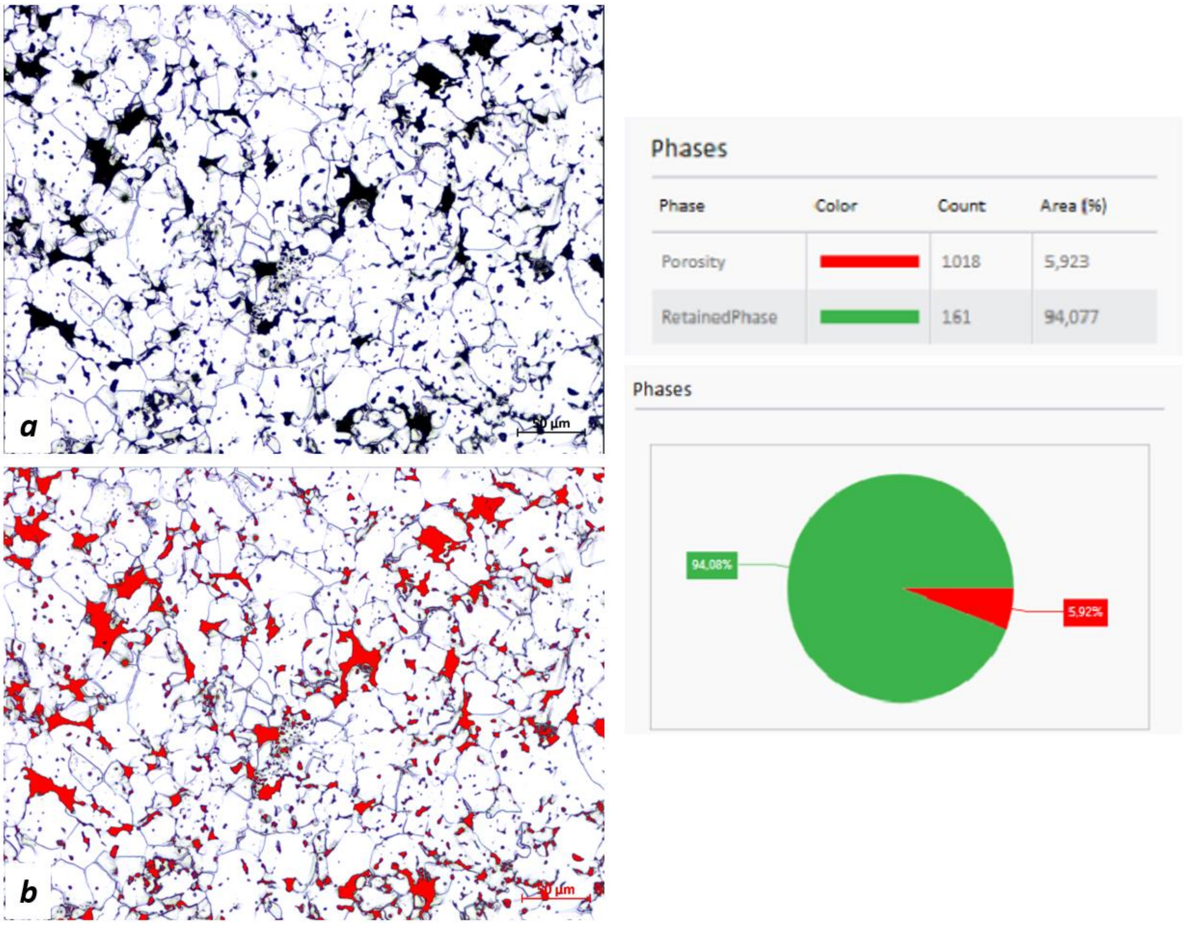
Adjustment of the magnification system to provide adequate resolution of the constituents/phases with the largest possible field size. Choice of the optimum magnification is a compromise between resolution and field-to-field measurement variability. Higher magnification objectives have higher numerical aperture rating and provide improved resolution. However, as magnification increases, the field-to-field measurements variability increases, which increases the standard deviation of the measurement. Also, as magnification increases, the field area decreases. For example, if the magnification is double, four times as many fields must be measured to cover the same test area.

Avoid use of lower magnification objectives that will not allow detection of the smaller constituents.

Gray level threshold settings are selected to allow independent detection of a specific discrete secondary phase, using a “flicker method” on switching back and forth between the inclusion image and thresholder image. The threshold setting may require a minor compromise between detection of the smallest constituents and over detection of the largest. The chosen threshold settings should be tried on constituents in several fields before beginning the analysis. For example, the threshold range for oxides is close to the black end of the reflectance scale while the range for sulphides is somewhat higher. An alternate approach to establish the threshold settings is to develop a grey-level reflectance histogram of the inclusion or constituent types that are present, as well as the matrix (usually in the as-polished condition). The histogram is used to identify the start (darkest grey) and end (lightest grey) of the range for each inclusion type, at the intersection grey level for two inclusions or constituent types with overlapping grey level ranges. Verify these settings by using the flicker method for several randomly selected fields.

The number of fields measured should be based on the relative accuracy of the measurements as defined in the following chapter or by agreement between manufacturer and end-user.

The computer program developed for data input image analyser control, measurement, and data analysis is read into the central processing unit to initiate the analysis. Input data regarding the specimen identify, data requester, calibration constant, date, number of fields, field spacing, and so forth, are entered.



Phases

Phase	Color	Count	Area (%)
Porosity	🟥	1018	5,923
RetainedPhase	🟩	161	94,077

Phases

Fig. 2.3.5– determination of the percentage of porosity in a sintered steel by image analysis. Detail of the microstructure before (a) and after (b) the image elaboration. Source: IIS laboratory

2.3.4.3. *Statistical analysis*

Analysis errors can be introduced from numerous sources; some of these are reported in the following:

- The sample selected could not be representative of the entire microstructure.
- Improper sample preparation.
- Operator competences and skills (e.g., inadequate ability to identify microstructures).
- The sample homogeneity, magnification, and number of measured fields affect data scatter and repeatability.

Microstructural variability is not a measurement of the errors, but it does influence test results, as the variability increases, the effort required to obtain a reliable statistical estimate to the structural parameters increases.

The calculation of the volume percentage estimate and percent relative accuracy are reported below. The value of the multiplier *t* can be found in the following table extracted from the ASTM E562.

No. of Fields n	***t***	***No. of Fields n***	***t***
5	2,776	19	2,101
6	2,571	20	2,093
7	2,447	21	2,086
8	2,365	22	2,080
9	2,306	23	2,074
10	2,262	24	2,069
11	2,228	25	2,064
12	2,201	26	2,060
13	2,179	27	2,056
14	2,160	28	2,052
15	2,145	29	2,048
16	2,131	30	2,020
17	2,120	40	2,000
18	2,110	60	1,960

Tab. 2.3.2 – 95% confidence interval multipliers. [3] *ASTM E562:2011 – Standard Test Methods for Determining Volume Fraction by Sistematic Manual Point Count. Source: [2.3.3]* ASTM E562:2011 – Standard Test Methods for Determining Volume Fraction by Sistematic Manual Point Count

The average $\overline{P_P}$, the standard deviation estimator S, the 95% confidence interval 95%CI, should be calculated and recorded for each set of fields; the equations used to calculate these values are shown below.

$$\bar{P}_p = \frac{1}{n}\sum_{i=1}^{n} P_p(i)$$

$$s = \left[\frac{1}{n-1}\sum_{i=1}^{n}\left[P_p(i)-\bar{P}_p\right]^2\right]^{1/2}$$

$$95\%\ \mathrm{CI} = t \times \frac{s}{\sqrt{n}}$$

The volume percentage estimate is given as:

$$V_v = \bar{P}_p \pm 95\,\%\ \text{CI}$$

An estimate of the percent relative accuracy associated with the estimate can be obtained as:

$$\%\ \text{RA} = \frac{95\,\%\ \text{CI}}{\bar{P}_p} \times 100$$

Estimates for the number of fields required to obtain a percent relative accuracy of 10, 20, or 33 % with different volume percentage and grid size are provided in the following table taken from ASTM E562. These values were calculated under the assumption that the features have a random distribution upon the metallographic section.

	33 % Relative Accuracy				***20 % Relative Accuracy***				***10 % Relative Accuracy***			
	Number of fields n for a grid of P_T				***Number of fields n for a grid of P_T***				***Number of fields n for a grid of P_T***			
Amount of volume fraction V_v in percent	***16 points***	***25 points***	***49 points***	***100 points***	***16 points***	***25 points***	***49 points***	***100 points***	***16 points***	***25 points***	***49 points***	***100 points***
2	110	75	35	(20)	310	200	105	50	1250	800	410	200
5	50	30	(15)	(8)	125	80	40	(20)	500	320	165	80
10	(25)	(15)	(10)	(4)	65	40	(20)	(10)	160	160	85	40
20	(15)	(5)	(5)	(4)	30	(20)	(10)	(5)	80	80	40	(20)

Tab. 2.3.3 – prediction of the number of fields (n) to be observed as a function of the desired relative accuracy and the estimated magnitude of the volume fraction of the constituent. Source: [2.3.3] ASTM E562:2011 – Standard Test Methods for Determining Volume Fraction by Sistematic Manual Point Count

The percent relative accuracy reported should always be calculated from the sample data and should not be taken from the previous table.

2.3.5. Grain Size

The properties and behaviour of a metal alloys are directly influenced by the grain size. In fact, the measurement of the grain size in a polycrystalline metal is one of the most important factors to determine, especially during production and quality control.

Usually, with the same chemical analysis, a fine grain steel has higher tensile characteristics and impact strength than a coarse grain steel. In contrast, a coarse grain steel could have a better behaviour in the creep regime than a fine grain steel.

The dimensions of grains observed on a cross section through the structure is used to determine the planar grain size. Several different measurements can be used to express the grain size:

- Average diameter
- Average area
- Number of grains per unit area
- Average intercept length number of grains per unit volume
- Average diameter based on average grain volume

To determine the grain size, the sections must be prepared by careful grinding and polishing to reveal the grains boundary by metallographic etching. Examples of procedures are described below:

- The ferritic grains shall be revealed by etching with “Nital”, or with an appropriate reagent.
- For steels with a single phase or two-phase austenitic structure (delta ferrite grains in an austenitic matrix) at room temperature, the grain shall be revealed with glyceregia, “Kalling,s reagent”, “Marble’s reagent”, or electrolytic aqueous 10% oxalic acid
- For steels with a martensitic or bainitic structure, sometimes the prior-austenitic grain can be detected. It shall be revealed with the following method reported in the UNI EN ISO 643:
    - “Bechet-Beaujard” method by etching with acqueous saturated picric acid solution.
    - “Kohn” method by controlled oxidation.
    - “McQuaid-Ehn” method by carburization.

The description of methods mentioned in the previous point are not covered by this chapter. Further information is provided in standard UNI EN ISO 643.

Both standards UNI EN ISO 643 and ASTM E112 show three methods to determine the average grain size. They are reported in the following points and described below:

- Comparison procedure.
- Planimetric (or Jeffries’) procedure.
- Intercept procedure – linear intercept procedure (Heyn) and circular intercept procedure.

ASTM E112 defines the grain size by an index G, known as the ASTM grain size, as shown in table 5.4; ASTM grain size index G is also used by the European standard UNI EN ISO 643.

	Grains/ Unit Area		***Average Grain Area***		***Average Diameter***		***Mean Intercept***		***N_L***
Grain size No. G	***No/In^2 at 100x***	***No./mm2 at 1x***	***mm^2***	***μm^2***	***mm***	***µm***	***mm***	***µm***	***No./mm***
00	0.25	3.88	0.2581	258064	0.5080	508.0	0.4525	454.5	2.21
0	0.50	7.75	0.1290	129032	0.3592	359.2	0.3200	320.0	3.12
0.5	0.71	10.96	0.0912	91239	0.3021	302.1	0.2691	269.1	3.71
1.0	1.00	15.50	0.0645	64516	0.2540	254.0	0.2263	226.3	4.42
1.5	1.41	21.92	0.0456	45620	0.2136	213.6	0.1903	190.3	5.26
2.0	2.00	31.00	0.0323	32258	0.1796	179.6	0.1600	160.0	6.25
2.5	2.83	43.84	0.0228	22810	0.1510	151.0	0.1345	134.5	7.43
3.0	4.00	62.00	0.0161	16129	0.1270	127.0	0.1131	113.1	8.84
3.5	5.66	87.68	0.0114	11405	0.1068	106.8	0.0951	95.1	10.51
4.0	8.00	124.00	0.00806	8065	0.0898	89,8	0.0800	80.0	12.50
4.5	11.31	175.36	0.00570	5703	0.0755	75.5	0.0673	67.3	14.87
5.0	16.00	248.00	0.00403	4032	0.0635	63.5	0.0566	56.6	17.68
5.5	22.63	350.73	0.00285	2851	0.0534	53.4	0.0476	47.6	21.02
6.0	32.00	496.00	0.00202	2016	0.0449	44.9	0.0400	40.0	25.0
6.5	45.25	701.45	0.00143	1426	0.0378	37.8	0.0336	33.6	29.73
7.0	64.00	99.00	0.00101	1008	0.0318	31.8	0.0283	28.3	35.36
7.5	90.51	1402.9	0.00071	713	0.0267	26.7	0.0238	23.8	42.04
8.0	128.00	1884.0	0.00050	504	0.0225	22.5	0.0200	20.0	50.00
8.5	181.02	2805.8	0.00036	356	0.0189	18.9	0.0168	16.8	59.46
9.0	256.00	3968.0	0.00025	252	0.0159	15.9	0.0141	14.1	70.71
9.5	364.04	5611.6	0.00018	178	0.0133	13.3	0.0119	11.9	84.09
10.0	512.00	7936.0	0.00013	126	0.0112	11.2	0.0100	10.0	100.0
10.5	724.08	11.223.2	0.000089	89.1	0.0094	9.4	0.0084	8.4	118.9
11.0	1024.00	15872.0	0.000063	63.0	0.0079	7.9	0.0071	7.1	141.4
11.5	1448.15	22446.4	0.000045	44.6	0.0067	6.7	0.0060	5.9	168.2
12.0	2048.00	31744.1	0.000032	31.5	0.0056	5.6	0.0050	5.0	200.0
12.5	2896.31	44892.9	0.000022	22.3	0.0047	4.7	0.0042	4.2	237.8
13.0	4096.00	63488.1	0.000016	15.8	0.0040	4.0	0.0035	3.5	282.8
13.5	5792.62	89785.8	0.000011	11.1	0.0033	3.3	0.0030	3.0	336.4
14.0	8192	126976.3	0.000008	7.9	0.0028	2.8	0.0025	2.5	400.0

Tab. 2.3.4 – Grain Size relationship Computed for uniform, randomly oriented, equiaxed grains. Source: [2.3.6] ASTM E112 – Standard Test methods for Determining Average Grain size

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2.3.5.1. *Comparison procedure*

The image examined on the screen is compared with a series of standard charts. The standard charts, at a magnification of 100 x, are numbered from - 07 to 17 so that it is equal to the index G. The comparison procedure shall be applied to completely recrystallized or cast materials with equiaxed grains.

When grain size estimations are made by the most convenient comparison method, repeated checks by specialists and/or interlaboratory tests, have shown that unless the appearance of the standard reflects that of the sample an error may occur. The ASTM E 112 shows four types of comparison charts that minimize such errors.

It is very important to verify that the magnification we are comparing is the same. Current software contains applications with loaded charts that allow for direct comparison.

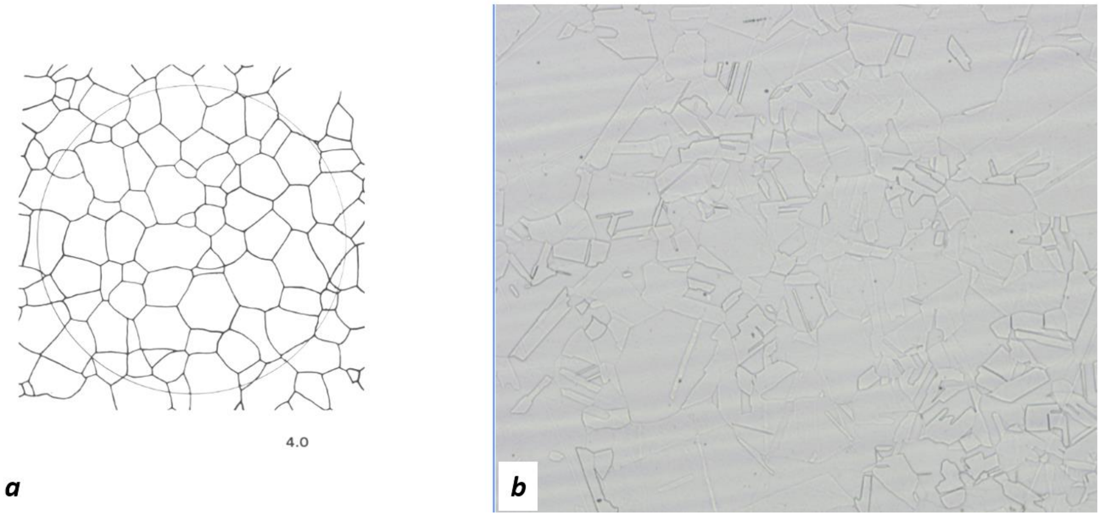


Fig. 2.3.6 – example of the comparison method. Image of a comparison chart (a) and detail of an austenitic steel obtained by OM. Source: IIS laboratory

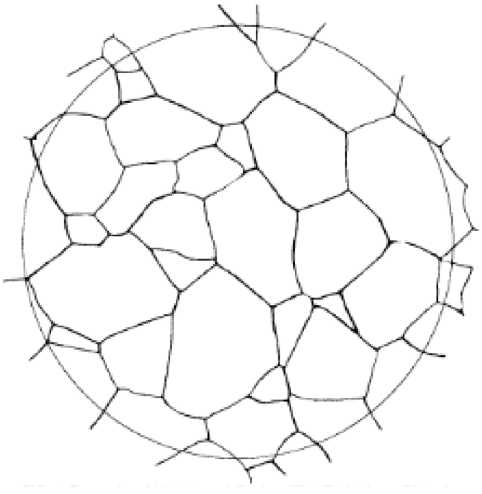


Fig. 2.3.7– example of untwinned grains (Flat Etchs) from plate I – Grain size No. 3 at 100X – image taken from ASTM E112. Source: [2.3.6] ASTM E112 – Standard Test methods for Determining Average Grain size

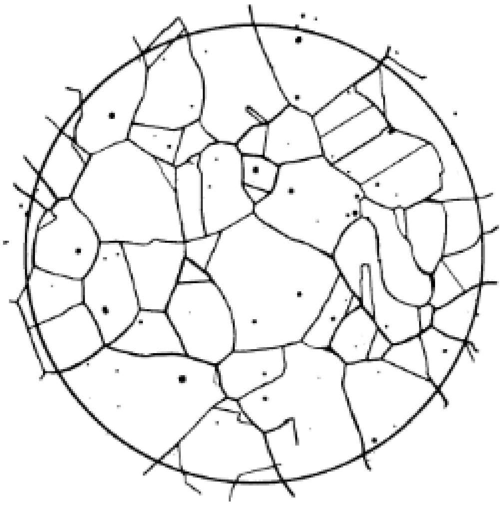


Fig. 2.3.8– example of twin grains (Flat Etchs) from plate II – Grain size No. 3 at 100X – image taken from ASTM E112. Source: [2.3.6] ASTM E112 – Standard Test methods for Determining Average Grain size

Fig. 2.3.9– example of twin grains (contrast Etchs) from plate III – Grain size 4 at 75X – image taken from ASTM E112. Source: [2.3.6] ASTM E112 – Standard Test methods for Determining Average Grain size

2.3.5.2. *Intercept procedure*

For this procedure, the number of grains intercepted, N, or the number of grain boundary intersections, P, should be counted using a test line of know length on a computer monitor or on a photomicrograph of a representative of a specimen at a known magnification.

The intercept procedure is recommended for all structures that depart from the uniform equi-axed form. For anisotropic structures, procedures are available to make separate size estimations in each of the three principal directions to estimate the average size.

The measuring line may be straight or circular. The grid shall be applied randomly to an adequate number of fields to have a statistical result.

There is no direct mathematical relationship between the ASTM grain size number, G, and the mean linear intercept, unlike the exact relationship between the mean linear intercept, l, and the average grain area is exact for circles but not quite exact for a structure of uniform equiaxed.

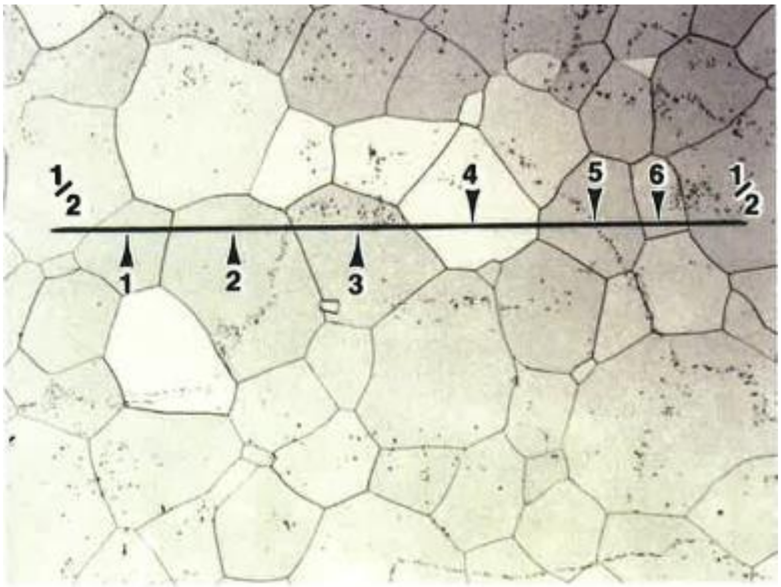


Fig. 2.3.10 – example of interception N (intercepts). [2.3.7]

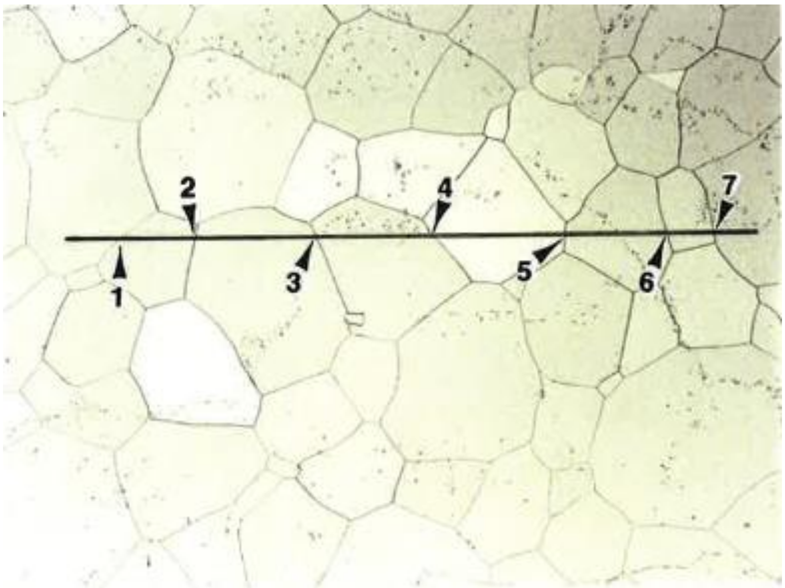


Fig. 2.3.11 – example of intersection P. Source: [2.3.7] *UNI EN ISO 643 – Steels – Micrographic determination of apparent grain size*

Linear intercept segments method:

To estimate the average grain size, it is recommended to count the number of intercepted grains, by one or more straight lines sufficiently long to yield at least 50 intercepts. It is desirable to select a combination of test line length and magnification such that a single field will yield the required number of intercepts.

The Precision of grain size estimates by intercept method is a function of the number of grain interceptions counted.

An intercept is a segment of test line overlaying one grain. An intersection is a point where a test line is cut by grain boundaries. It may also be counted with the same results in the single-phase metal.

The effects of moderate departure from an equiaxed structure may be eliminated by making intercept counts, on a line array, counting the lines having four or more orientations.

Circular intercept method:

The measuring line consists of three concentric circles. The magnification or diameter of the circle shall be selected so that there are 40 to 50 intercepts when the measurement grid surpasses the field to be examined.

The circular intercepted segment method tends to give slightly higher intercepted segment values and thus slightly lower number of intersections. To minimize this, the intersections caused by a triple point shall be counted as two intersections instead of 1,5, as is the case with the linear intercepted segment method.

Results:

The mean value of the number of intercepts $\overline{N}$, or intersection, $\overline{P}$ is calculated with the following formula:

$\overline{N_L} = \overline{N}/\mathrm{LT}$ and $\overline{P_L} = \overline{P}/\mathrm{LT}$

For non-equiaxed grain boundary, three lines oriented parallel to the three principal test planes lines can be used (longitudinal, transverse and planar). The mean number of intercepts, or the mean number of intersections is determined from the cubic root of the product of three measurements, according to:

$\overline{N_L} = \overline{(N}/\mathrm{LTx}\ \mathrm{X}\overline{N}/\mathrm{LTy}\ \mathrm{X}\overline{N}/\mathrm{LTz}\ )1/3$ and $\overline{P_L} = \overline{(P}/\mathrm{LTx}\ \mathrm{X}\overline{P}/\mathrm{LTy}\ \mathrm{X}\overline{P}/\mathrm{LTz}\ )1/3$

Modern methods of grain size measurements, such as automatic image analysis, can be used to measure grain size of applicable materials, providing that accuracy of the methods has previously been proven by an extensive cross correlation.

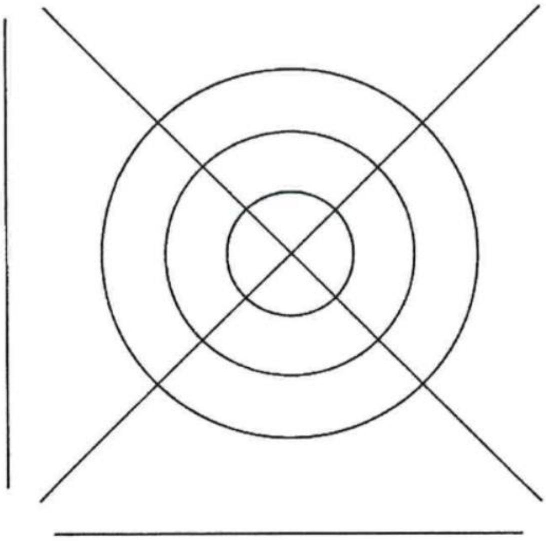
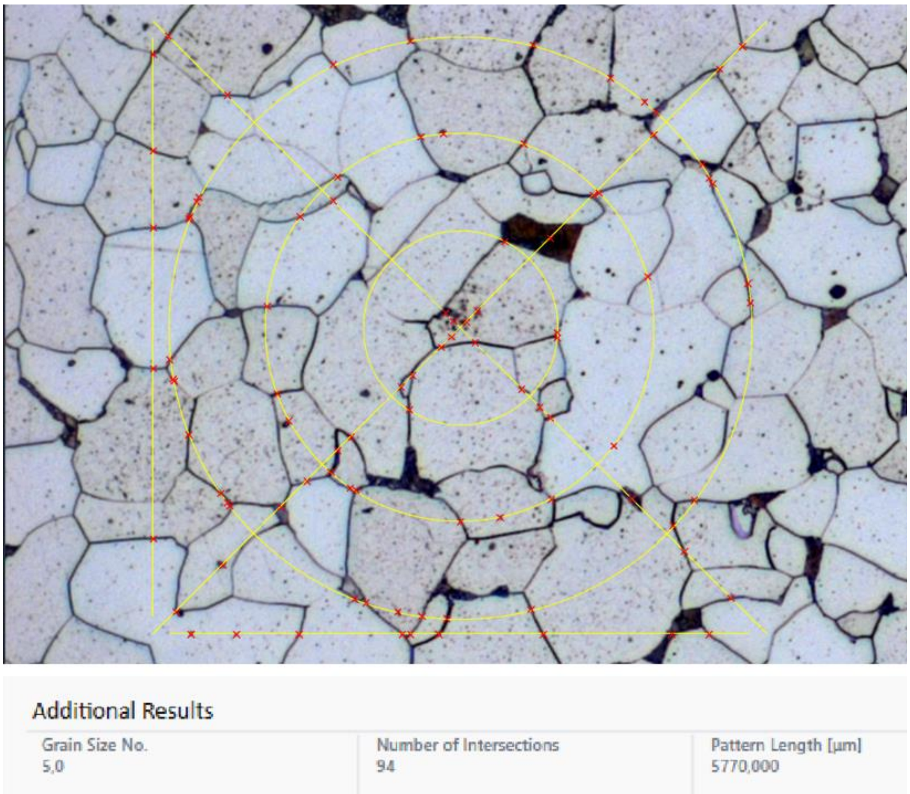


Fig. 2.3.12– recommended measurement grid for intercept segment procedure. Source: [2.3.7] UNI EN ISO 643 – Steels – Micrographic determination of apparent grain size.



Additional Results

Grain Size No.	Number of Intersections	Pattern Length [µm]
5,0	94	5770,000

Fig. 2.3.13– examples of an intercept measurement performed on a carbon steel. Source: IIS laboratory.

2.3.5.3. *Planimetric methods*

The planimetric method is the least used, historically a circle with 79,8 mm in diameter was overlapped on a live image and the real magnification is shown on the equipment's screen. The magnification should be adjusted so that the circular area contains at least 50 grains.

Two counts are made:

- n_1 is the number of grains completely within the test circle
- n_2 is the number of grains intersected by the test circle.

The total number was equivalent to:

$$N100 = n_1 + 0{,}5n_2$$

The number of grains, m, for mm^2 on the specimen surface is computed from:

$$m=2n_{100}$$

or, in the case of any magnification, g:

$$m=(g^2/5000)n_g$$

where 5000 is the test circle area in mm^2.

This approach assumes that, on average, half of the grains intersected by the test circle are within the circle while the remaining half is outside the circle. This assumption is valid for a straight line through grain structure, but not for a curved line. The deviation created by this assumption increases as the number of grains inside the test circle decreases. If the number of grains within the test circle is a least 50, the deviation is around 2%.

A simple way to avoid this problem is to envision a square or rectangle while considering that the grains intersecting each of the four corners, are one fourth within the figure and three-fourth outside it. These four corner grains together represent one grain within the test box. Ignoring the four corners' grains, a count is made on n_1 - the grains completely within the box; and n_2 - the grains intersected by the four sides of the box. The equation becomes:

$$N100 = n_1 + 0{,}5n_2 + 1$$

The number of grains, m, for mm^2 on the specimen surface is computed from:

$$m=(g^2/A_f)n_g$$

where A_f is the apparent area of the test figure used for grain counting in mm^2.

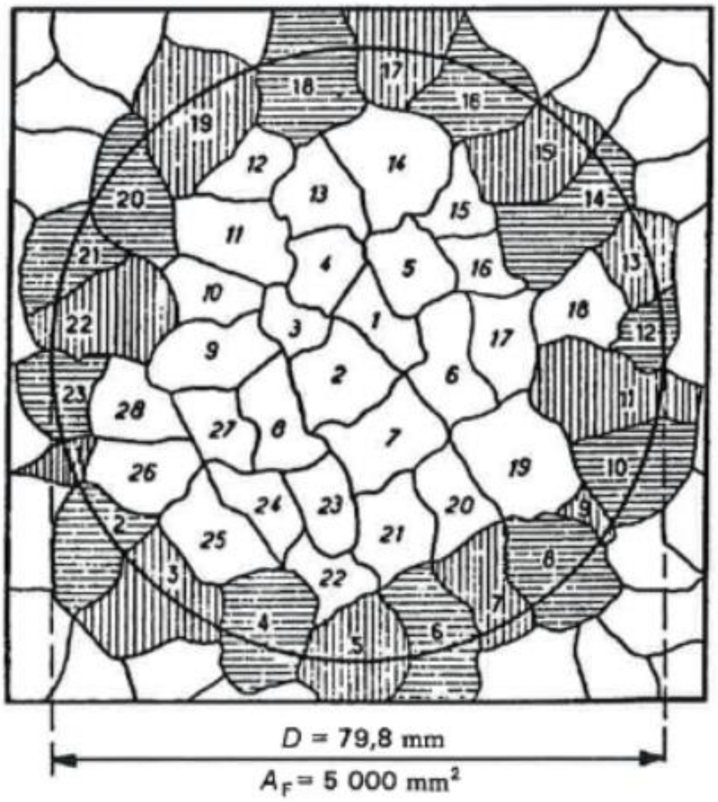
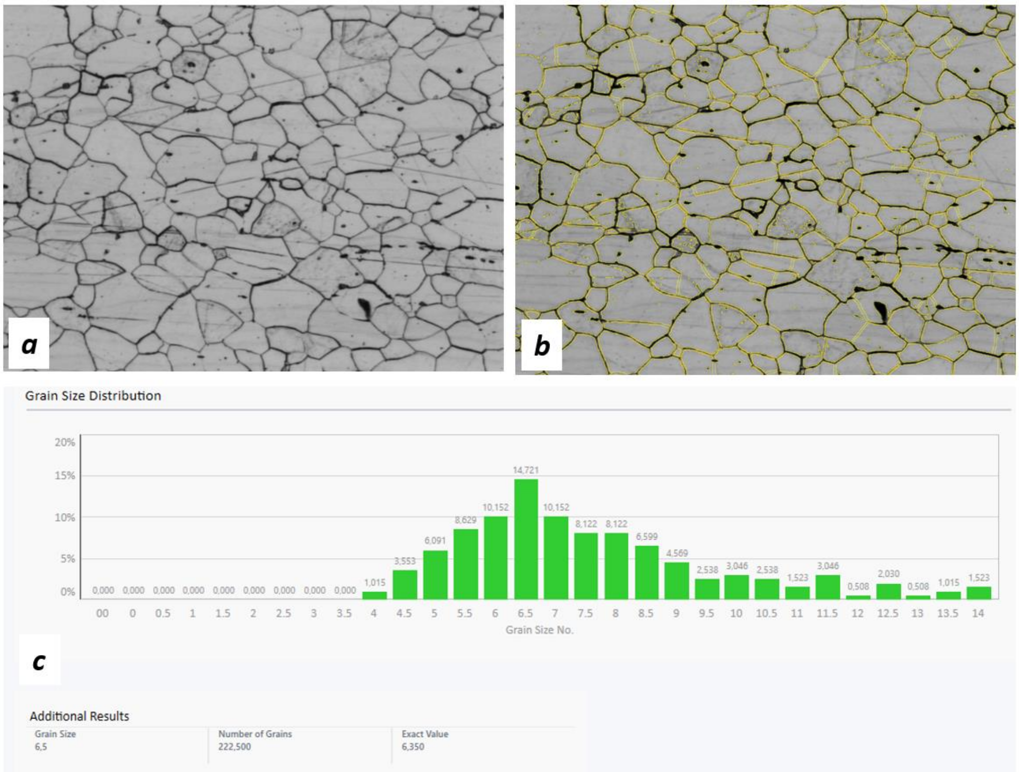


Fig. 2.3.14– evaluation of number of grains in an area enclosed by a circle. Source: [2.3.7] UNI EN ISO 643 – Steels – Micrographic determination of apparent grain size



Additional Results

Grain Size	Number of Grains	Exact Value
6,5	222,500	6,350

Fig. 2.3.15– examples of a planimetric measurement. Detail of the micrographic structure before (a) and after (b) elaboration image by software and the grain size distribution histogram obtained (c). Source: IIS Laboratory

References:

[2.3.1] ASM HANBOOK – Volume 9 – Metallography And Microstructures.

[2.3.2] Metallography – Principles and practice – George F. Vander Voort – Directors, Research and technology Buehler, Ltd. Lake Bluff, Illinois.

[2.3.3] ASTM E562:2011 – Standard Test Methods for Determining Volume Fraction by Sistematic Manual Point Count.

[2.3.4] ASTM E1245:2003 - Standard Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis

[2.3.5] Metallographer's Guide – Practices and procedures for Iron and Steel – Bruce L. Bramfitt, Arlan O. Benscoter.

[2.3.6] ASTM E112 – Standard Test methods for Determining Average Grain size.

[2.3.7] UNI EN ISO 643 – Steels – Micrographic determination of apparent grain size.

2.4. Demonstration of capabilities of the program for image analysis

In this chapter, we will explore the key concepts and tools used to perform metallographic image analysis, outlining the process from image acquisition to their processing and interpretation of the obtained results.

The various stages involved in preparing metallographic samples, essential for obtaining clear and detailed images, will be illustrated. Subsequently, we will examine the importance of microscopes in capturing images and discuss the various illumination techniques that help highlight specific microstructural features. Finally, we will delve into the digital environment, exploring specialized software that enables quantitative analysis of metallographic images, providing an accurate and in-depth interpretation of the observed structures.

2.4.1.1. Samples preparation

In order to obtain a good quality of the analysed surface, a mechanical polishing should be performed, because other polishing process (e.g., electrochemical polishing) may involve rounded edges and artifacts.

The last polishing steps can be performed with diamond suspension of 3 µm or less; it is advisable to finish the last polishing step with colloidal silica based suspensions (diameter of abrasion particles less than 0,1 µm). The grinding and polishing procedure must be chosen based on the type of alloy to be prepared.

A typical procedure for carbon steels or low alloy steels is shown in the following point:

- First step: grinding with SiC grinding papers (grit 220) or equivalent grinding disk for some minutes with water, in order to obtain the correct flatness of the samples.
- Second step: grinding with grinding disk with diamond suspension with particles diameter of 9 µm.
- Third step: polishing using polishing clothes with diamond suspension with particles diameter of 3 µm.
- Fourth step: polishing using polishing clothes with colloidal silica suspension containing particles with diameter lower than 0,1 µm.

If available, automatic systems are preferable to obtain the right final mirror surfaces of the metallic sample.

For some analyses, such as samples characterized by high brittle and deterioration, other precautions can be adopted. Typical precautions include thickness measurements of oxide layers (for example magnetite in the boiler tube) or evaluation of porosity when thermal spray coating, in which the grinding and polishing steps could break the layer generating measurement errors. In those cases, a more precise cutting step (e.g., automatic cutting machine) and a double mounting (after and before the cutting) can be necessary.

Scratches on the micrographic section can be detected as dark areas using image analysis software and causes an overestimation of the measured phase. These kinds of defects demand constant supervision of the image analysis process by the operator/technician.

2.4.1.2. Demonstration of some practical cases of Delta Ferrite determination

The most common applications of the method to determine the volume fraction are listed below:

- Determination of a percentage of Delta ferrite in weld metal of austenitic stainless steels.
- Determination of a percentage of Delta ferrite in a Duplex stainless steel.
- Evaluation of inclusions content in a steel or other alloys.
- Evaluation the percentage of porosity in cast, welding, brazing, and coating.

The amount of Delta Ferrite expected in austenitic stainless steel welded joints is within a range of 5-15 %. A small amount of Delta ferrite reduces the hot cracking sensitivity of the welded joint; however, a higher amount, reduces the toughness at low temperatures and the corrosion resistance.

The micrographic sections observation is performed by Optical Microscope, usually 100x, 200x or 500x objectives are selected.

After a first qualitative evaluation of the amount of Delta Ferrite, a 25 or 49-point grid can be used, as indicated in the standard ASTM E562. Sometimes a preliminary measurement can be performed with ferrite scope in order to select the correct grid.

One example of Delta Ferrite measurements by point count method via software is shown below.

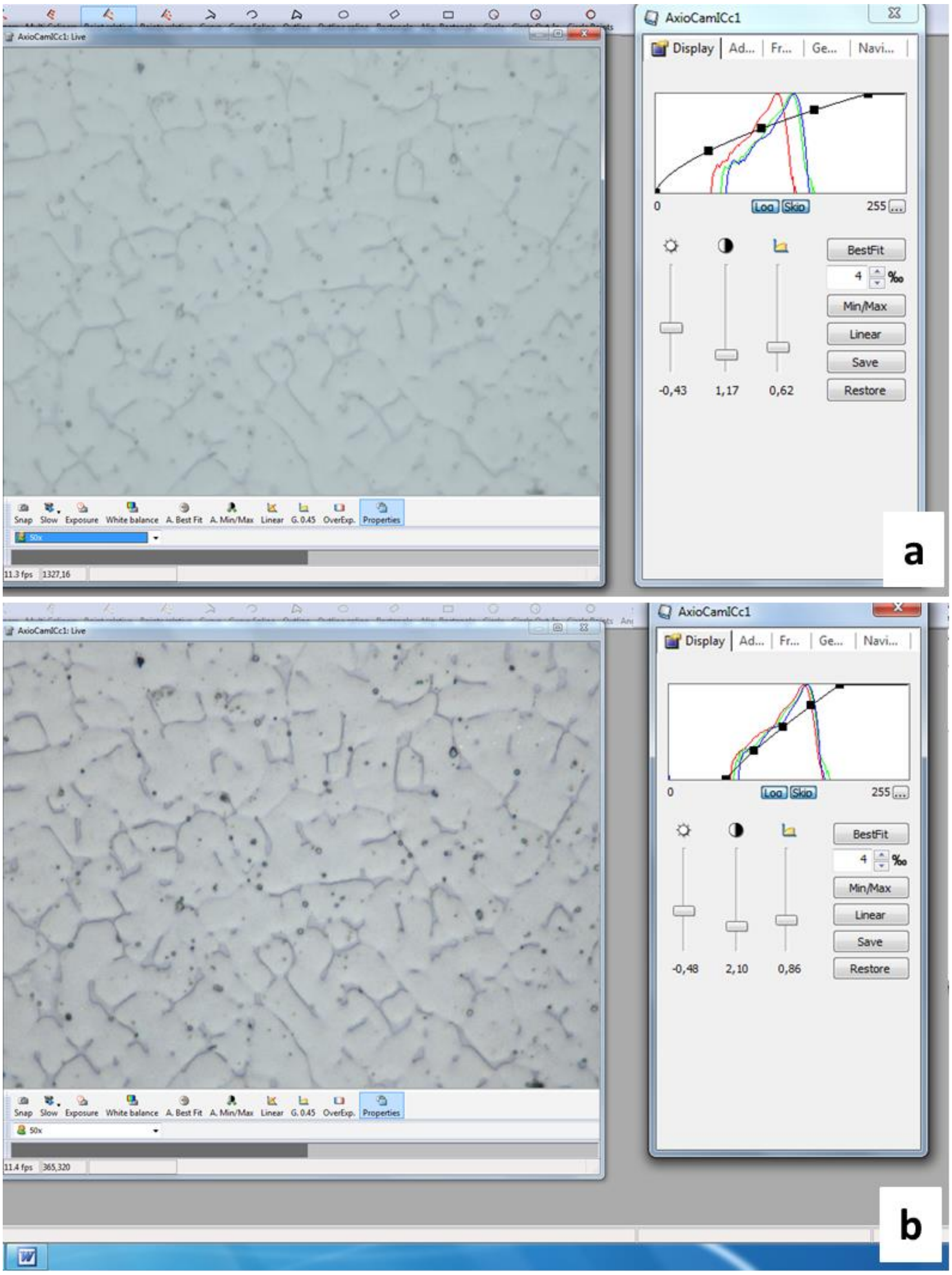


Fig. 2.4.1– Details of the welded zone of an austenitic stainless-steel type 316L. The figures show before (a) and after (b) the Lookup table (LUT) optimization. Source: IIS Laboratory.

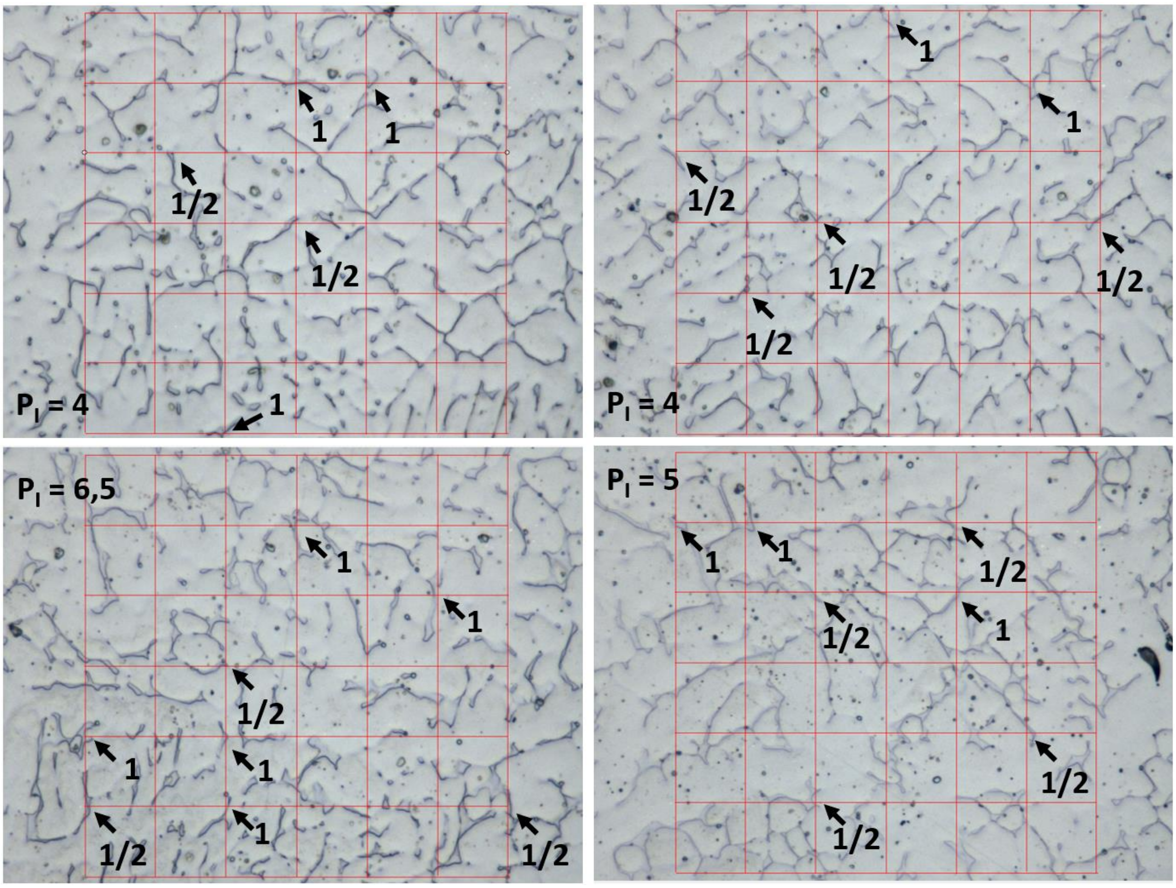


Fig. 2.4.2– Examples of some fields analysed by point count method with a 49 points grid. Source: IIS Laboratory.

P_T = 49			
Number of fields counted n	***10***	***20***	***85***
Percentage of grid points P_p	10,41	10,31	10,17
95% Confidence interval	1,52	1,02	0,57
Relative accuracy % Ra	14,56	9,94	5,65
Standard deviation	2,40	2,29	2,59

Table 2.4.1– The ferrite percentage measured by point count corresponds to P_p. The table shows how the measurement accuracy increases as the number of measured fields increases. Source: IIS Laboratory.

Another application of point count method is the determination of a percentage of Delta ferrite in Duplex stainless steel.

Duplex stainless steels combine the advantages of ferritic and austenitic stainless steels. The properties are being determined by the chemical composition and the phase balance. The high chromium content and alloying with nitrogen, contribute to high strength and the resistance to intergranular corrosion. Stress-corrosion cracking is superior when compared to other austenitic grades.

Duplex stainless steels are based on the Fe-Cr-Ni-N alloy system. The chemical composition of these steels has been adjusted such that the base metal microstructure consists of about 50% ferrite and 50% austenite. However, all duplex stainless steels solidify as virtually 100% ferrite and depend on partial solid state transformation to austenite for balanced microstructure. Nitrogen is added as an alloying element to accelerate formation of the austenite phase and stabilize it.

Generally, duplex stainless steels have a higher ratio of ferrite-promoting elements to austenite-promoting elements than austenitic stainless steels. The following figure indicates that alloys whose WRC-1992 chromium equivalent is about 1.85 times their nickel equivalent, or more, will solidify as 100% ferrite; the duplex stainless steels usually have ratios between 2,25 and 3,5.

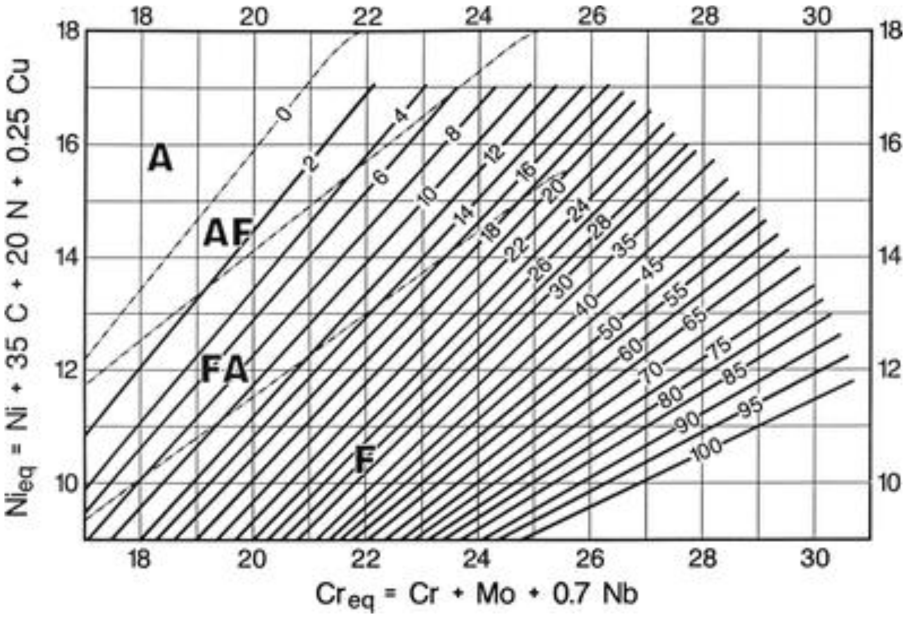


Fig. 2.4.3– WRC 1992 diagram, used to estimate the Ferrite Number based on Creq = Cr+Mo+0, 7Nb and Nieq =Ni+35C+20N+0,25Cu. Source: [2.4.2] WRC-1992 Constitution Diagram for Stainless Steel Weld Metals

At high temperatures (above the ferritic solvus), the alloys remain 100% ferrite. Austenite can only nucleate and grow below the ferrite solvus. Annealing and hot-working operation of these steels are performed at temperatures below ferrite solvus, where ferrite and austenite can coexist. By controlling the processing temperature and cooling rate from that temperature, the proportion and distribution of ferrite and austenite can be controlled in the wrought product.

For these reasons, in addition to chemical analysis, verification of the distribution of ferrite and austenite is very often required to evaluate their quality and performance. Usually, 16 points grid are used, as indicated by the standard ASTM E562. One example of Ferrite/Austenite measurements by point count method via software is shown below.

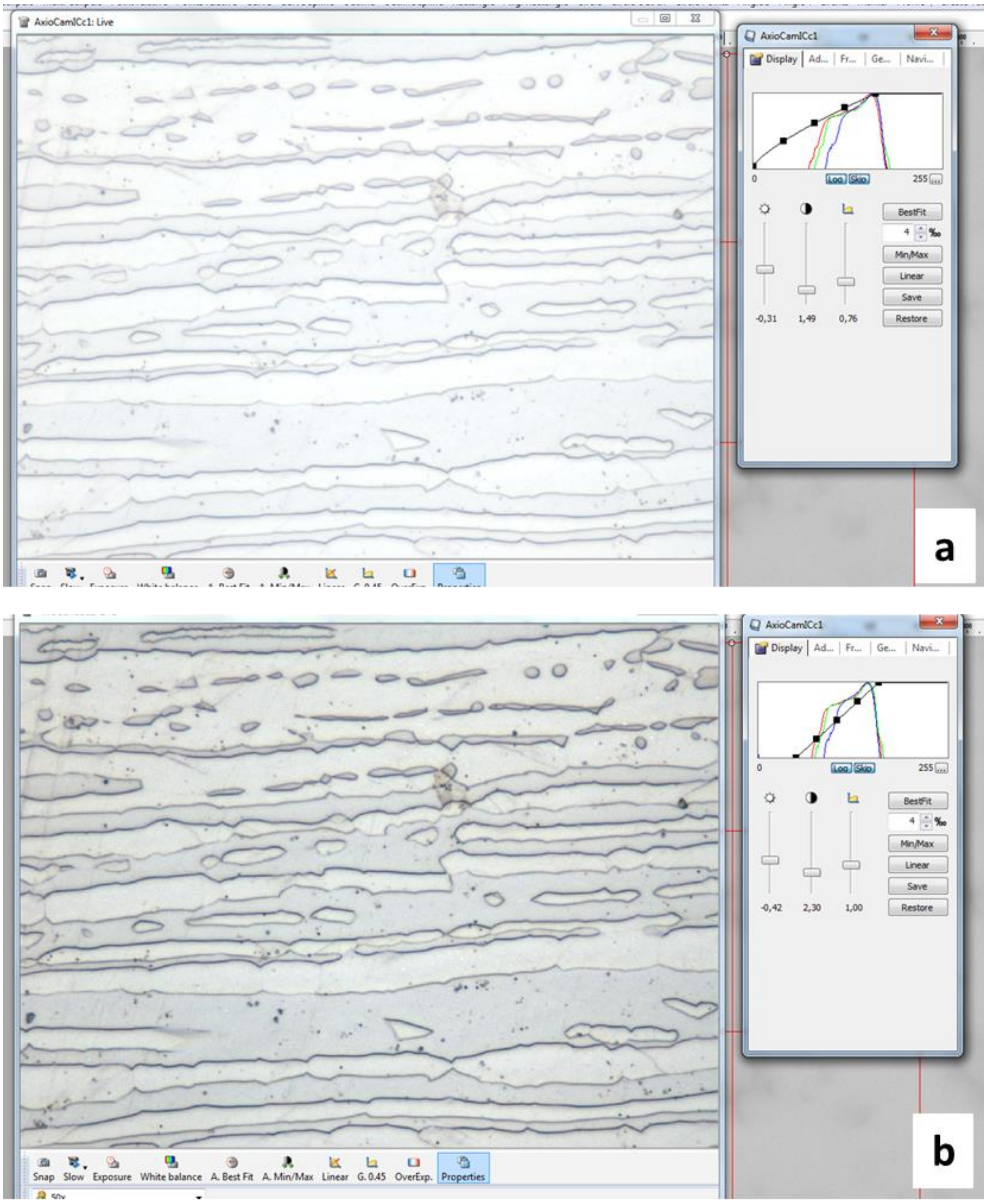


Fig. 2.4.4– Details of a Duplex stainless-steel type 2205. (a) As taken. (b) After the lookup table (LUT) optimization. Source: IIS Laboratory.

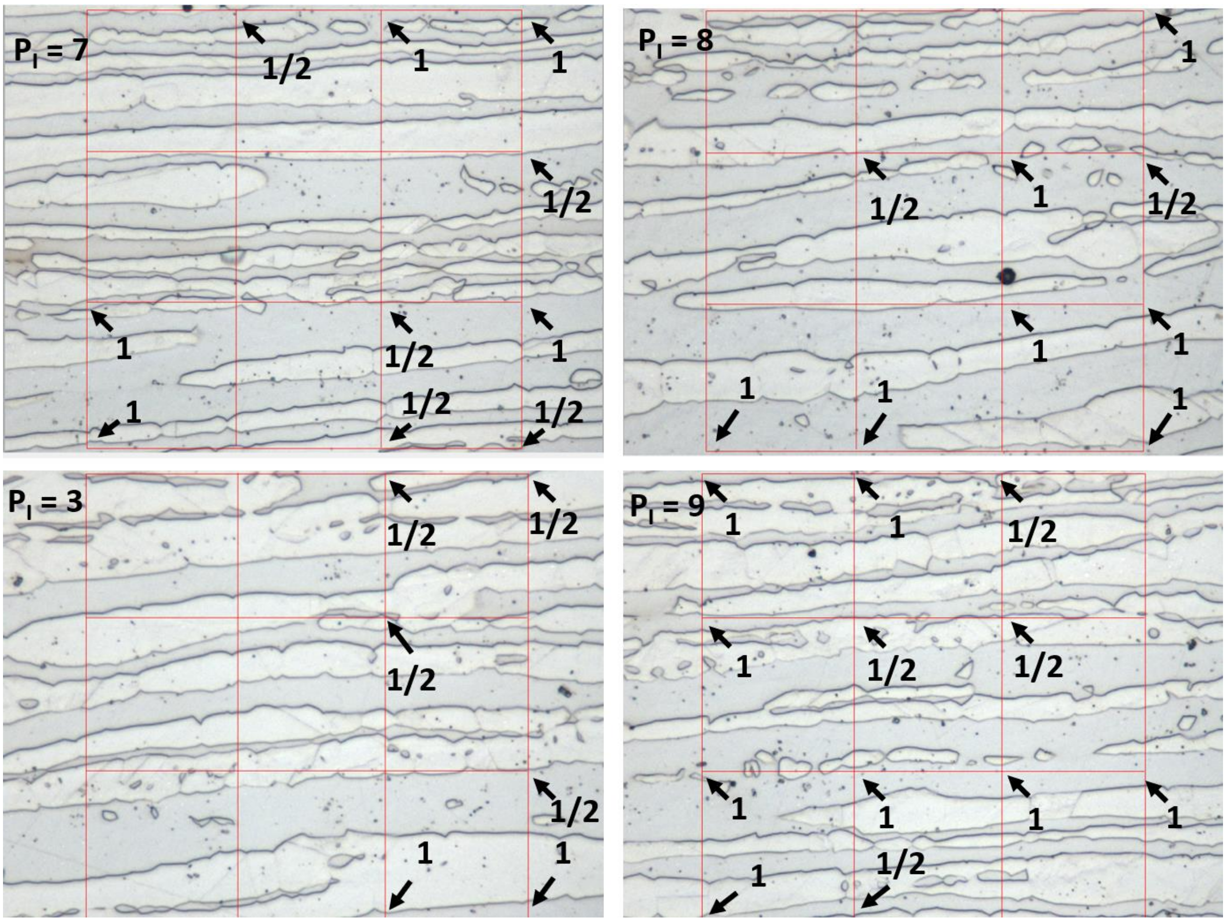


Fig. 2.4.5– Examples of some fields analysed by point count method with a 16 points grid. Source: IIS Laboratory

P_T = 49			
Number of fields counted n	***10***	***20***	***85***
Percentage of grid points P_p	42,19	44,53	44,53
95% Confidence interval	7,17	4,77	2,16
Relative accuracy % Ra	17,00	10,72	4,84
Standard deviation	11,34	10,68	9,65

Table 2.4.2– The ferrite percentage measured by point count corresponds to P_p. The table shows how the measurement accuracy increases as the number of measured fields increases. Source: IIS Laboratory

2.4.1.1. *Demonstration of some practical cases of Porosity percent evaluation*

The analysis image finds another important application, not only in the determination of the phases, but also in the evaluation of porosity and/or discontinuity within a matrix. Porosity may be present in the welding, brazing or in some type of coating (e.g., thermal spray coating).

In welding or brazing joints, porosity is an imperfection (nr. 200 in UNI EN ISO 6520-1) and therefore requires to be evaluated through acceptability criteria selected on the basis of the component.

The evaluation of the percentage of porosity should be carried out on the projected area also evaluated by radiographic examination (see ISO 5817); however, the evaluation by micrographic examination can be carried out and it is complementary.

Image analysis programs allow to obtain linear measurements of the maximum diameter and areas of pores and cavities.

The procedure for measuring porosity can be summarized in the following steps:

- Image Acquisition: Acquire high-resolution images of the interesting area using an optical or electron microscope. Ensure the images are clear and well-focused.
- Image Preparation: load the acquired image into an image processing software (e.g., ImageJ or other specific software) and apply a filter to reduce noise of the image, such as a Gaussian or median filter.
- Binarization: Apply a binarization algorithm to separate the porous areas from other parts of the image and choose an appropriate threshold to distinguish between pores and matrix.
- Porosity Measurement: calculate the ratio of the total pore area to the total area of the brazing zone, expressing porosity as a percentage.
- Validation and Correction: manually review the obtained results to correct any segmentation or measurement errors.
- Reporting and Interpretation: generate a report that includes the processed images, calculated porosity, and any other relevant information.

This procedure provides a general framework for analysing porosity in welding or brazing joints or other details using image processing. However, it's important to tailor this procedure to suit the specific requirements of a specific case and the available equipment.

An example of analysis applied to the porosity present in a brazed joint on a copper alloy is shown in the following images.

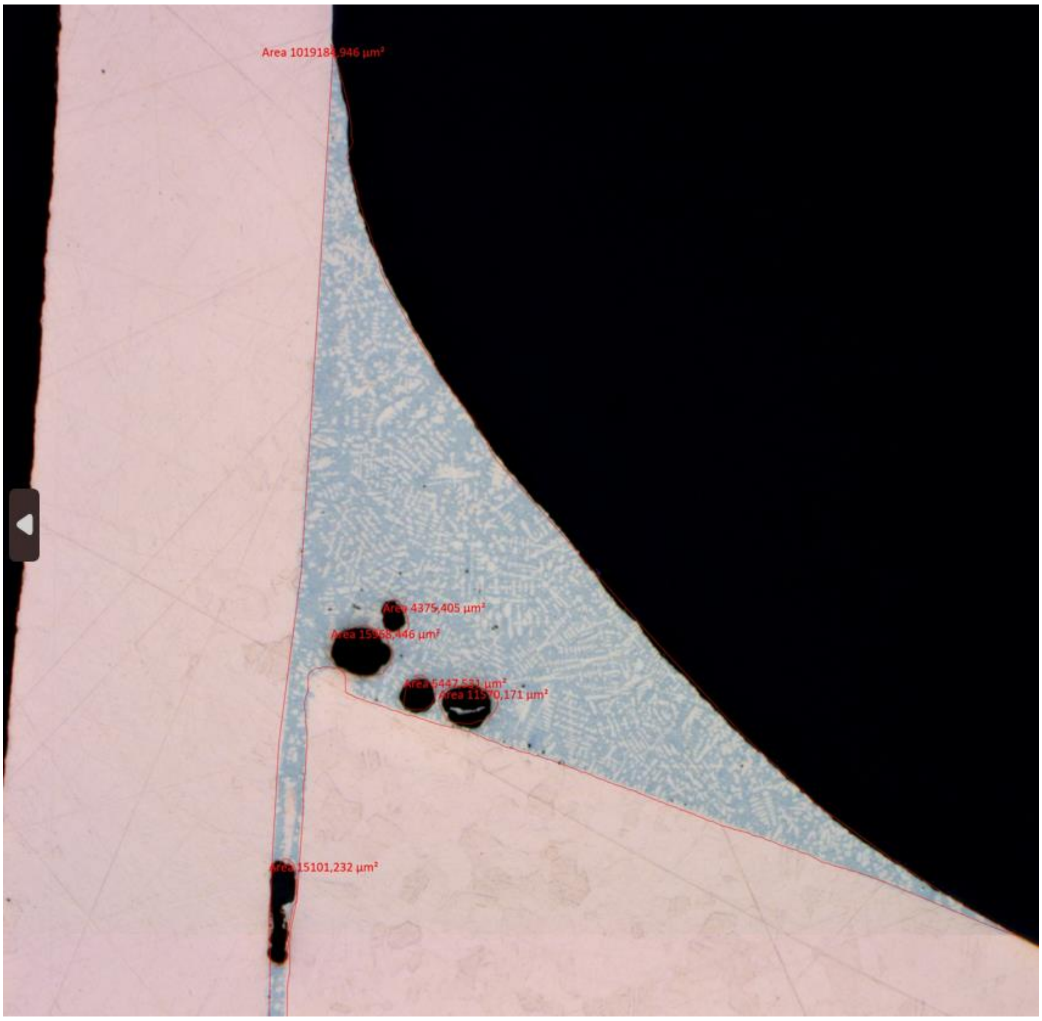


Fig. 2.4.6– Detail of a brazing joint on a copper alloy component. Porosity areas measured by image analysis program. Source: IIS Laboratory

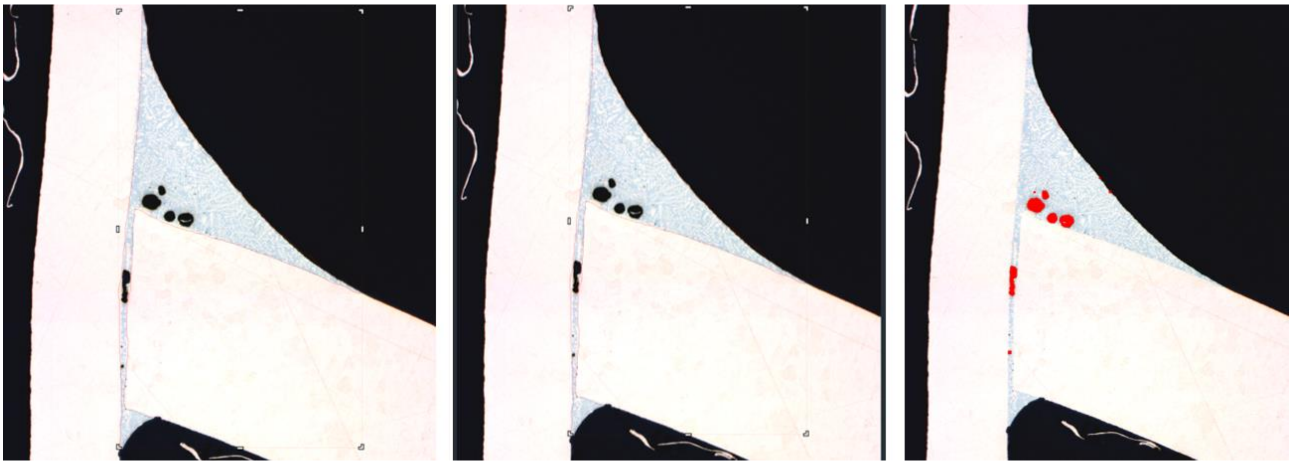
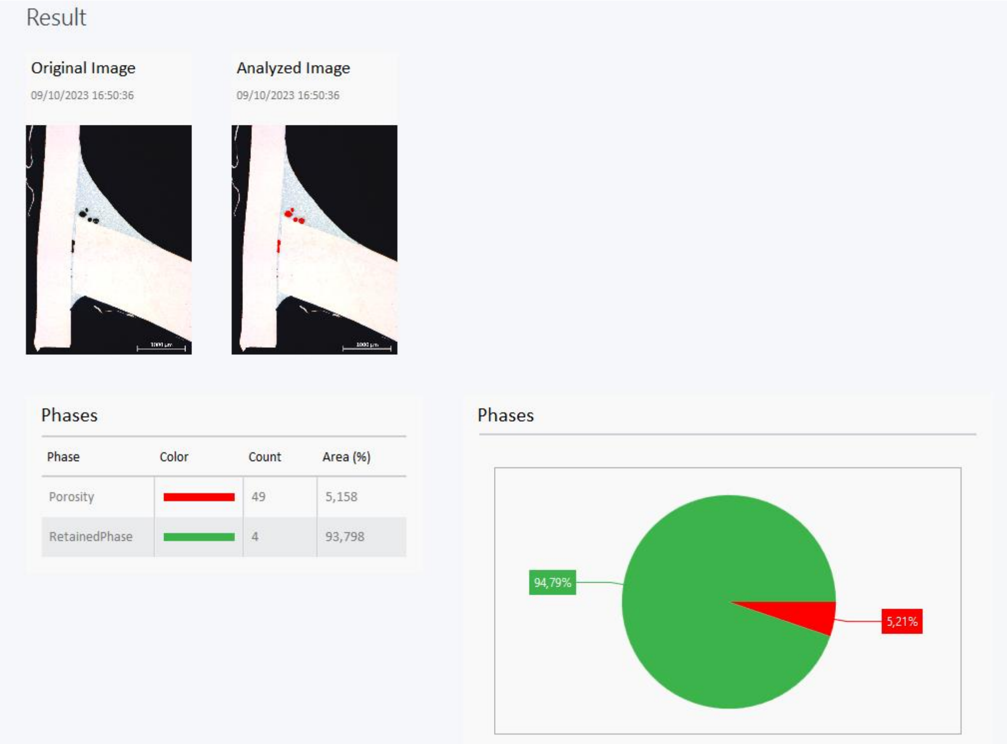


Fig. 2.4.7– Detail of a brazing joint on a copper alloy component. Porosity percent measured by image analysis program. Source: IIS Laboratory



Result

Original Image
09/10/2023 16:50:36

Analyzed Image
09/10/2023 16:50:36

Phases

Phase	Color	Count	Area (%)
Porosity	🟥	49	5,158
RetainedPhase	🟩	4	93,798

Phases

Fig. 2.4.8– Detail of a report obtained by image analysis program. The percentage of porosity on brazing joint is about 5,2 %. Source: IIS Laboratory

The analysis of the porosity presented in a coating obtained with the thermal spray method, observed in section, is shown below.

Evaluating porosity in a coating produced by thermal spray process is critical to ensure optimal coating quality, durability, and performance. This contributes to preserving the integrity and effectiveness of the coated object in its operational context.

Porosity and discontinuity influence the following characteristics:

- Hardness and Strength;
- Adhesion and Cohesion;
- Corrosion Resistance.

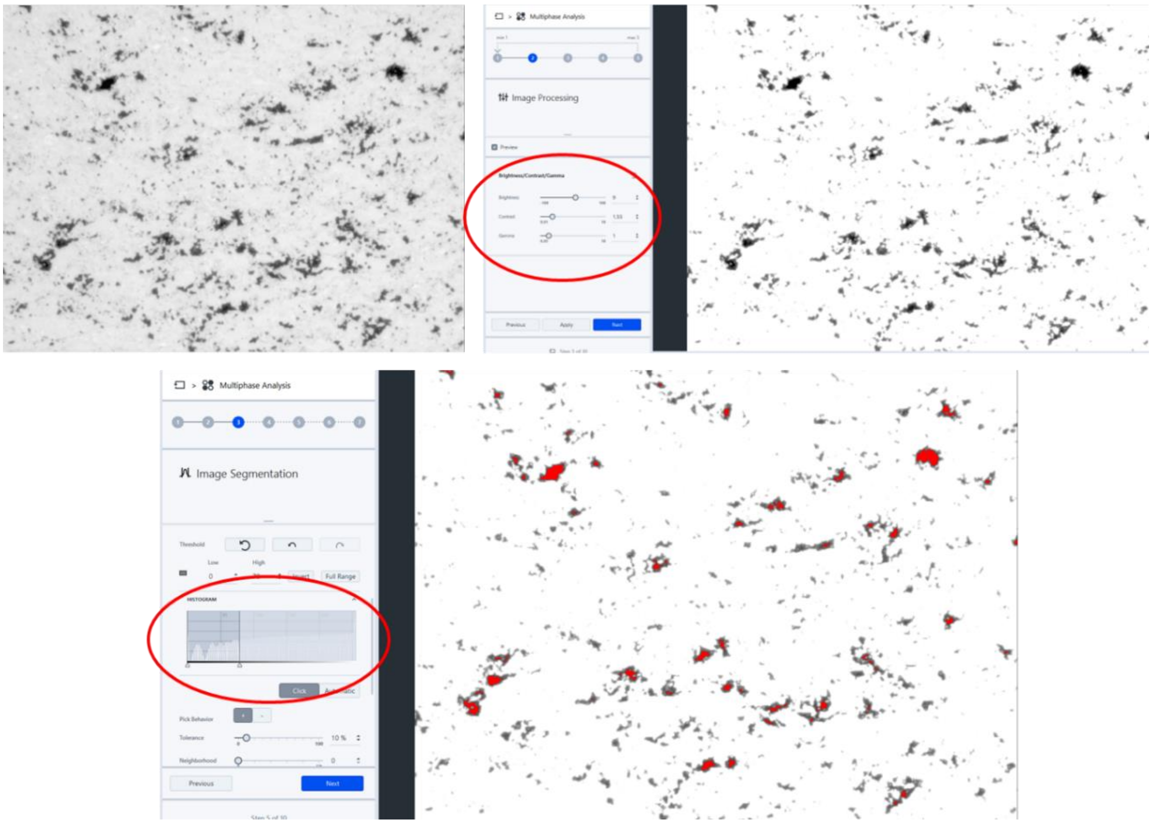


Fig. 2.4.9– Detail of a thermal spray layer. Porosity percentage measured by the image analysis program. The figures show the various steps to optimize the image. Source: IIS Laboratory

Result

Original Image
09/10/2023 16:55:29

Analyzed Image
09/10/2023 16:55:29

Phases

Phase	Color	Count	Area (%)
Porosity	🟥	104	0,531
RetainedPhase	🟩	16	99,469

Phases

Fig. 2.4.10– Detail of a report obtained by image analysis program. The percentage of porosity on thermal spray coating is about 0,53%. Source: IIS Laboratory.

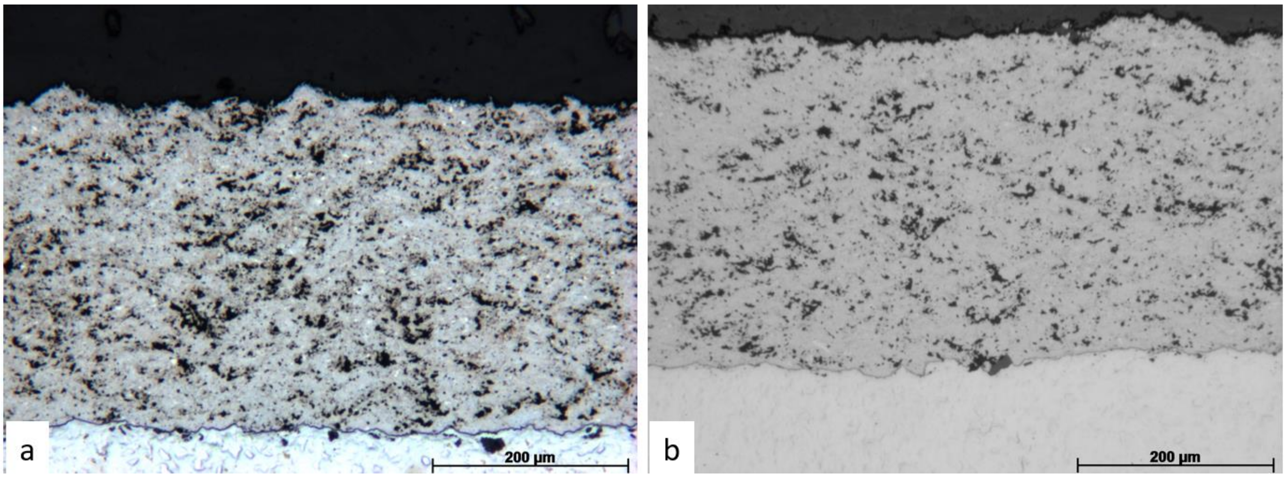


Fig. 2.4.11– Comparison between two micrographic sections taken from the same thermal spray coating with a conventional metallographic preparation(a) and with a more accurate metallographic preparation(b) such as the use of automatic cutting systems, mounting and grinding polishing pressure reduced, etc. Source: IIS Laboratory.

2.4.1.1. Demonstration of some practical cases of Grain size measurements

Grain size measurement in the industry is a fundamental process for several reasons:

- Allowing for quality control during production to ensure the material meets required standards and specifications.
- Optimization of the mechanical and physical properties of materials, as grain size and distribution can influence key factors such as hardness, strength, ductility, and fatigue resistance.
- Development of appropriate processing and heat treatment methods by determining optimal parameters to achieve a desired microstructure and enhance material performance.
- Prediction of the long-term behaviour of the material under different operating conditions and environments.
- Research and development of new materials and optimization of existing ones, enabling the design of materials with superior performance for specific applications.

In summary, the measurement of metallographic grain is crucial for understanding the microscopic structure of metallic materials and optimizing material properties, therefore improving quality, production process efficiency, and advancements in metallurgy and industrial applications.

Grain size can be a specific requirement for some European or American standards, such as the following examples:

- Case-hardening steels often have specifications regarding grain size to ensure a controlled and uniform structure after the case-hardening heat treatment (EN 10084).
- Chromium-molybdenum alloy steels often require controls on grain size to ensure better workability and mechanical performance (ASTM A519).

- Specifications for steels that need to undergo heat treatment may include requirements for grain size to ensure the right microstructure after the treatment (ASTM A304).
- Steels used in high-strength spring production often require strict control of grain size to ensure consistent and reliable performance (EN 10270).

Often, the grain size can be specified as a requirement on product specifications based on company know-how.

The following images show some examples of grain measurement with the various methods described in the previous paragraph.

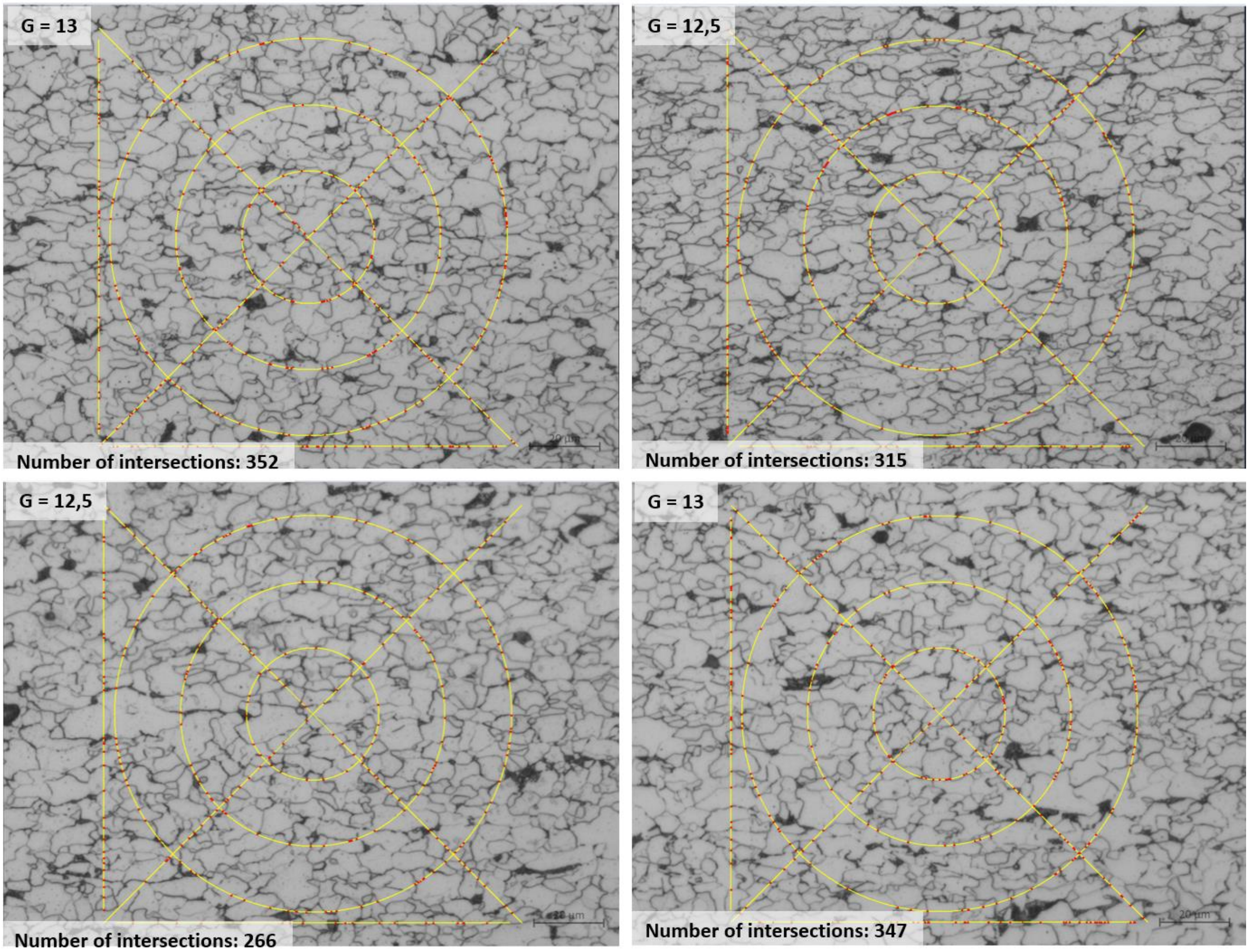
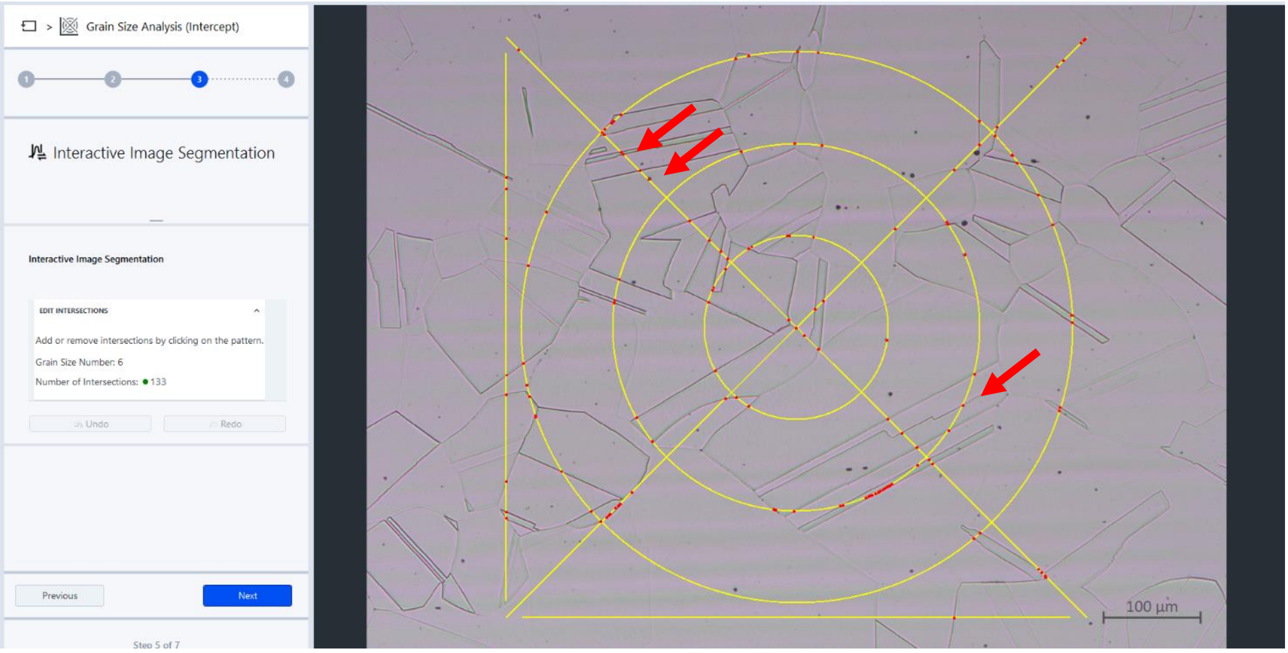


Fig. 2.4.12– Details of some fields analysed by interception method on a fine grain carbon steel, according to *UNI EN ISO 643.* Source: IIS Laboratory.

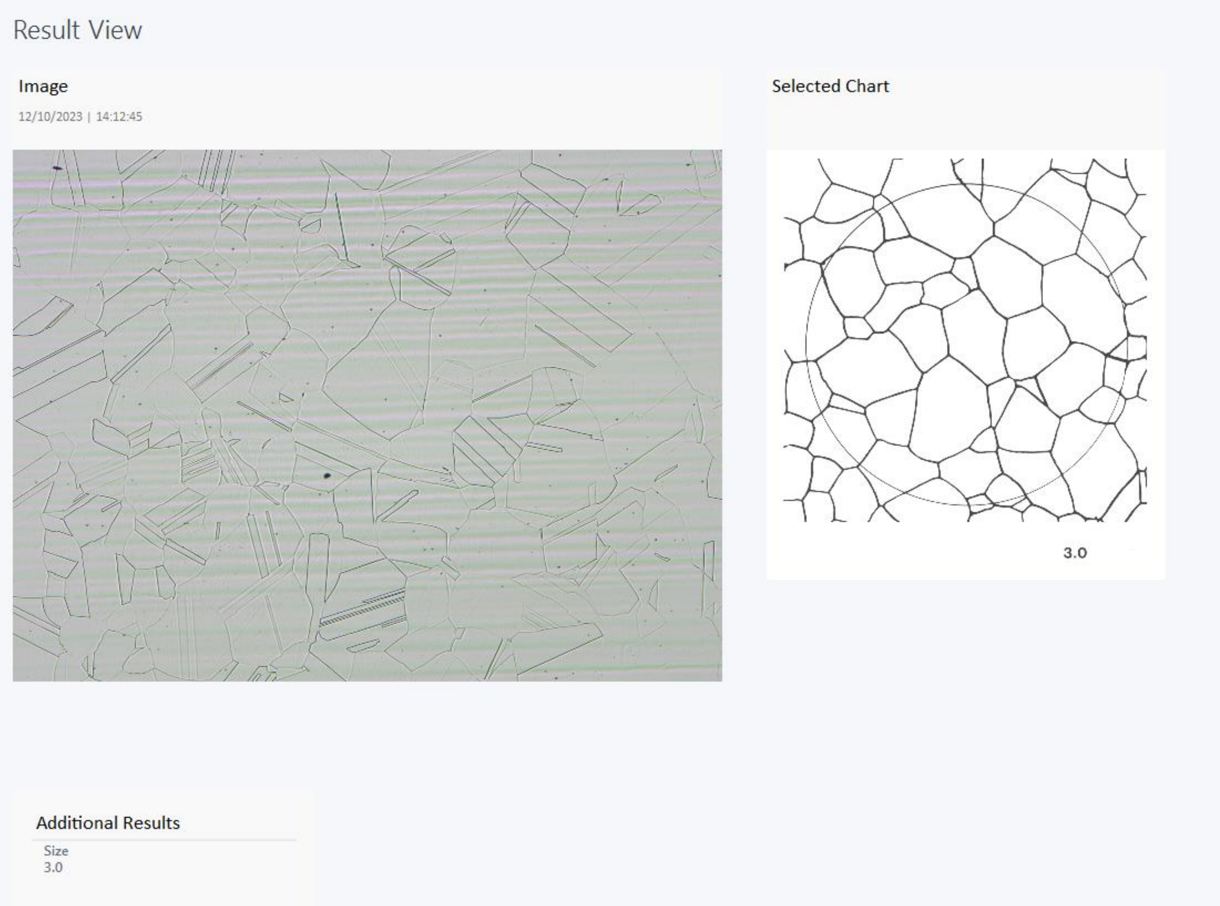


Add or remove intersections by clicking on the pattern.

Grain Size Number: 6

Number of Intersections: ● 133

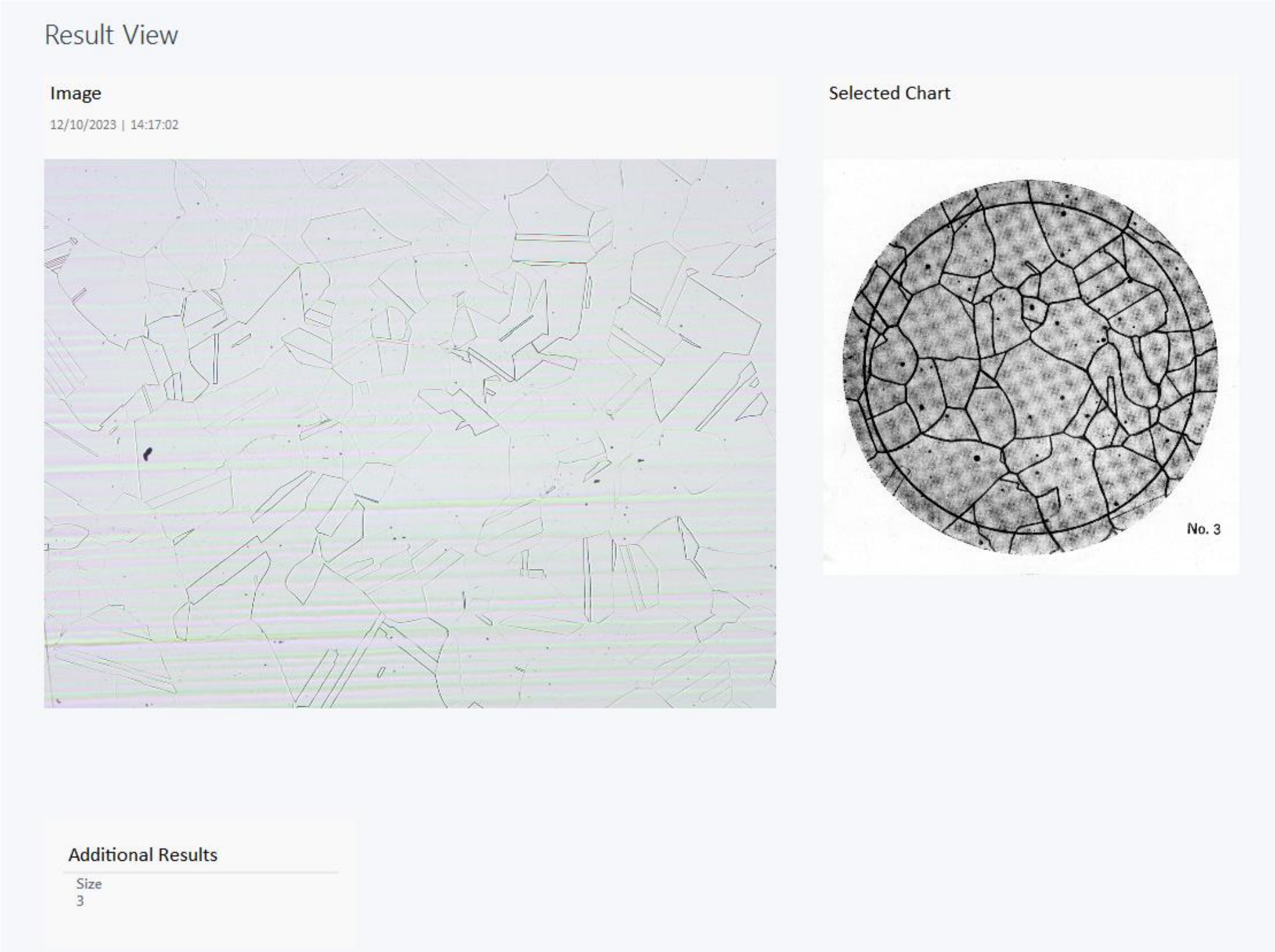
Fig. 2.4.12– Details of grain size measurement by interception method on a super austenitic stainless steel (Type AISI 310LMO), according to *UNI EN ISO 643.* The figure shows a measurement error of the image analysis due to the inability to distinguish grain boundaries from grain twins. Source: IIS Laboratory.



12/10/2023 | 14:12:45

Size
3.0

Fig. 2.4.13– Details of grain size measurement by comparison method on a super austenitic stainless steel (Type AISI 310LMO), according to UNI EN ISO 643. Untwinned grains (Flat Etchs). Source: IIS Laboratory.



Result View

Image
12/10/2023 | 14:17:02

Selected Chart

Additional Results

Size
3

Fig. 2.4.14– Details of grain size measurement by comparison method on a super austenitic stainless steel (Type AISI 310LMO), according to ASTM E112. Twin grain plate used. Source: IIS Laboratory.

References:

[2.4.1] Welding Metallurgy and Weldability of Stainless Steels – John C. Lippold and Damian J. Kotecki.

[2.4.2] WRC-1992 Constitution Diagram for Stainless Steel Weld Metals

[2.4.3] ASTM E562:2011 – Standard Test Methods for Determining Volume Fraction by Sistematic Manual Point Count.

[2.4.4] ASTM E1245:2003 - Standard Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis.

[2.4.5] ASTM E112 – Standard Test methods for Determining Average Grain size.

[2.4.6] UNI EN ISO 643 – Steels – Micrographic determination of apparent grain size.

[2.4.7] UNI EN 10084 – Case hardening steels – Technical delivery condition.

[2.4.8] ASTM A519 - Standard Specification for Seamless Carbon and Alloy Steel Mechanical Tubing.

[2.4.9] ASTM A304 - Standard Specification for Carbon and Alloy Steel Bars Subject to End-Quench Hardenability Requirements.

[2.4.10] UNI EN 10270 – Steel wire for mechanical spring – patented cold drawn unalloyed spring steel wire.

2.5. Introduction to scanning microscopy

The Scanning Electron Microscope (SEM) allows for the observation and characterization of heterogenous organic and inorganic materials on a nanometric to micrometric scale. The SEM is used to observe and characterize surface features over large (or small) areas of the specimen. In metallography, SEM provides information about the microstructures, characterization of secondary/intermetallic phases, failure mechanisms, etc.

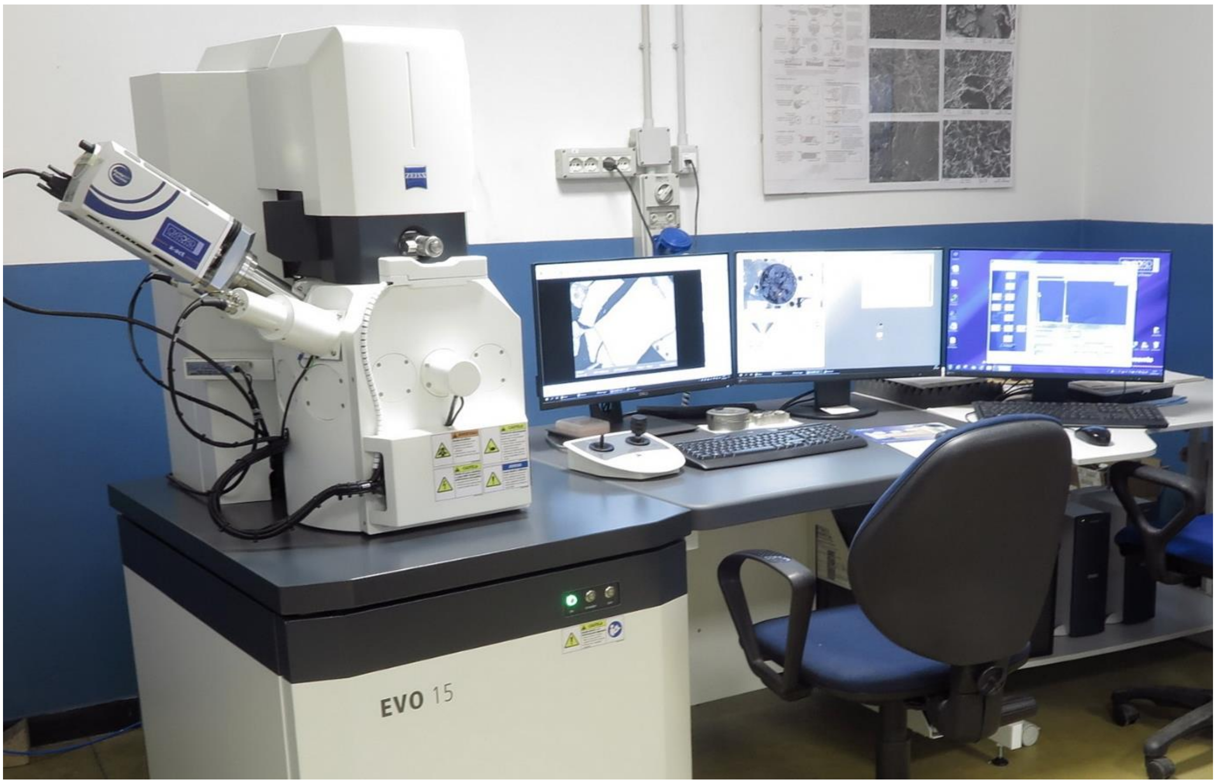


Fig. 2.5.1– overall view of a Scanning Electron Microscope. Source: IIS Laboratory

A SEM produces images of a sample by scanning the surface with a focused beam of electrons. The electron beam is generated from a cathode and is accelerated to the sample by a high voltage (from 1000 to 30000 V); the cathode can be either a hot cathode which emits electrons by thermionic effect (tungsten filament, lanthanum hexaboride LaB6), or a cold cathode which emits by an electrostatic field (field emission gun FEG).

When electrons (primary electrons) strike the metal surface of the specimen, the following signals are generated:

- Secondary electrons (SE) generated by the inelastic interactions between the electron beam and the sample, affect only the superficial or subsurface layers. They allow for the formation of a morphologic and topographic image of the sample surface.
- Backscattered electrons (BSD) generated by elastic collisions between electrons and atoms, induce changes in the trajectory of electrons, large atoms scatter more electrons than small

atoms, giving a greater signal as the atomic number (Z) increases. This principle makes it possible to obtain images with qualitative information of the chemical compositions.

- X-ray photons generated by the interaction of electrons with atoms. Each X-ray photon is characterized by a frequency (ν) and a wavelength (λ) that are characteristic of the atom of the element from which it was generated.

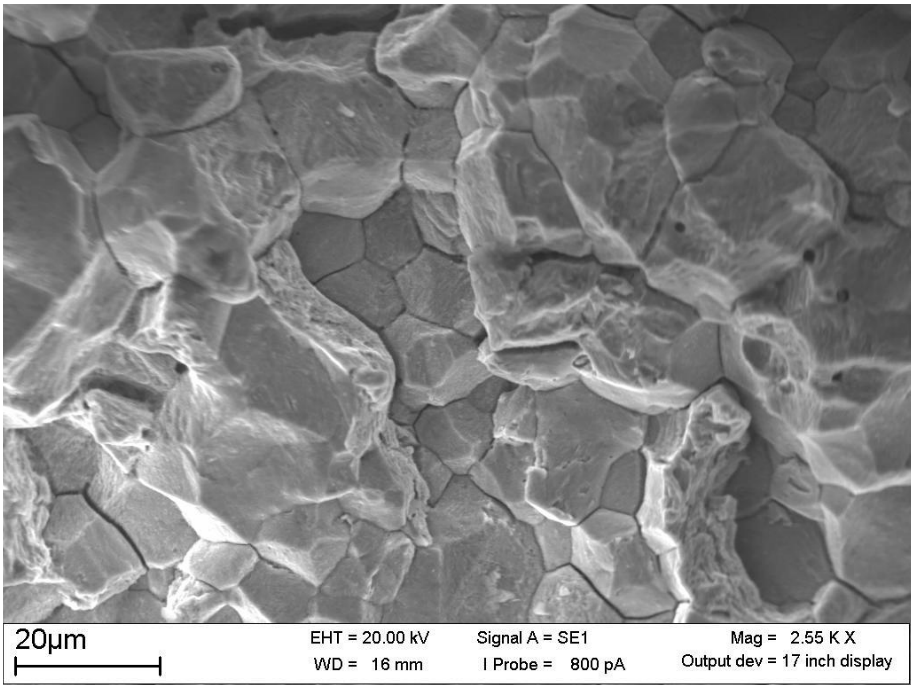


Fig. 2.5.2– detail of an intergranular fracture obtained by SEM with secondary electrons (SE). Source: IIS laboratory.

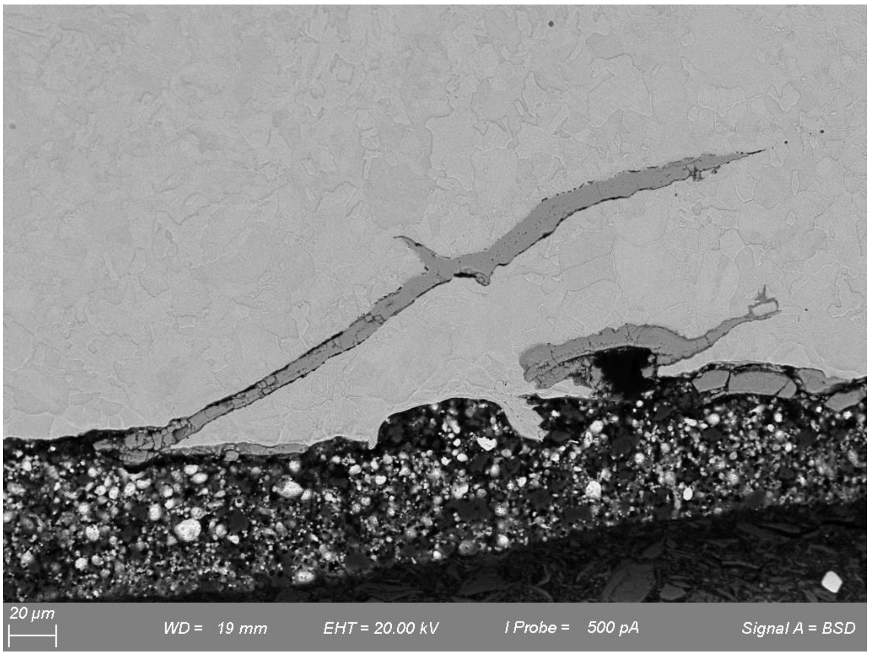


Fig. 2.5.3– detail of a micrographic section obtained by SEM with backscattered electrons (BSD). Source: IIS laboratory.

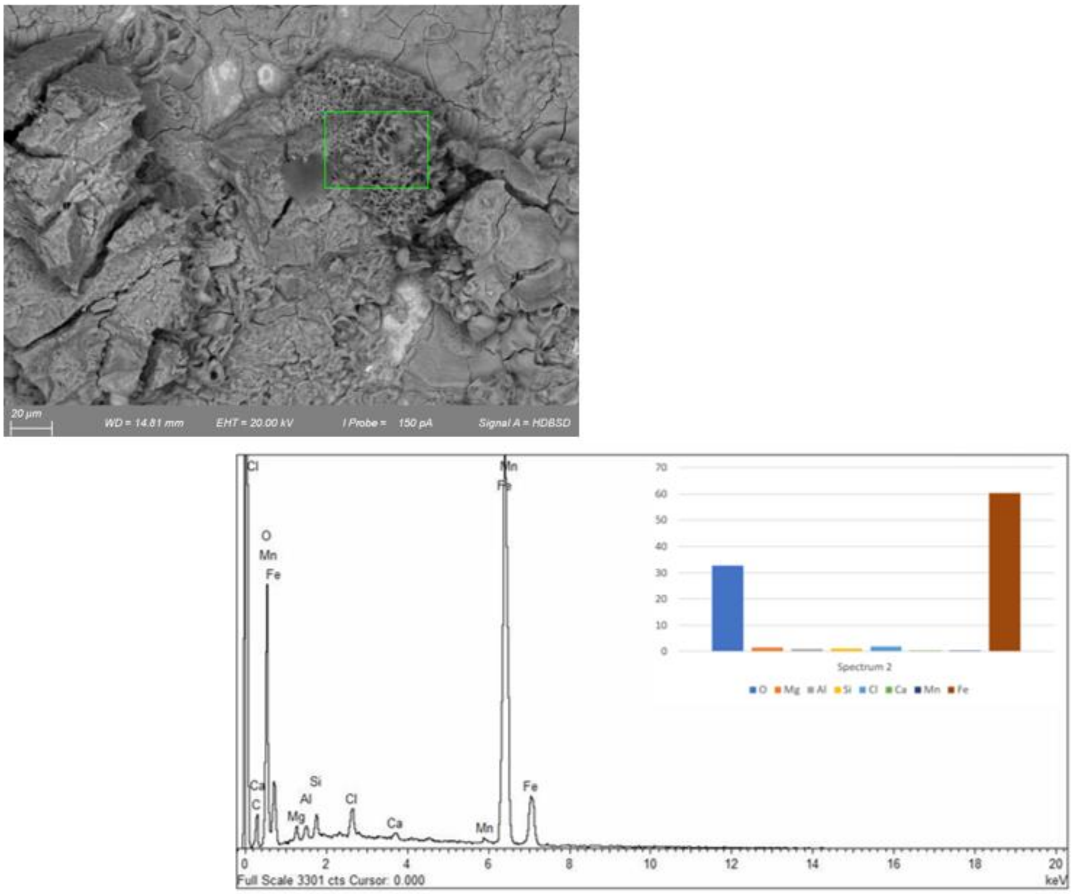


Fig. 2.5.4– example of EDS analysis of deposits. Source: IIS Laboratory.

The probes in the SEM capture the signal and send the data to the acquisition system (control console) where they are processed to obtain three-dimensional images and information on the composition.

Advantages of SEM in comparison to optical microscopy (OM), are reported in the following points:

- High depth of field (DOF) - 3D images with topographical information.
- Very high magnifications (> 150000x).
- High resolution images (3-10 nm).

Furthermore, SEM instrumentation is often equipped with an EDS (Energy Dispersive X-Ray Spectroscopy) system that allows for the chemical composition analysis of features that are observed on the monitor.

SEM can be equipped with other types of probes such as:

- EBSD (Electron Back Scattered Diffraction)
- SAN (Scanning Auger Nanoprobes)
- WDS (Wavelength dispersive X-Ray Spectroscopy).

2.5.1. Modes of operation

The two major components of a SEM are the electron column and the control console. The electron column consists of an electron gun and electron lenses, which influence the paths of electrons travelling down the vacuum column. The base of column is usually taken up with vacuum pumps that produce a vacuum of about 10^{-4} Pa. The control console consists of a cathode ray tube (CRT), viewing screen, knobs and computer keyboard that controls the electron beam.

The electron gun generates electrons and accelerates them to an acceleration voltage in the range of 1000-30000 V. The spot size from a tungsten filament gun is too large to produce a sharp image unless electron lenses are used to lower the magnification it will create a much smaller focused electron spot on the specimen.

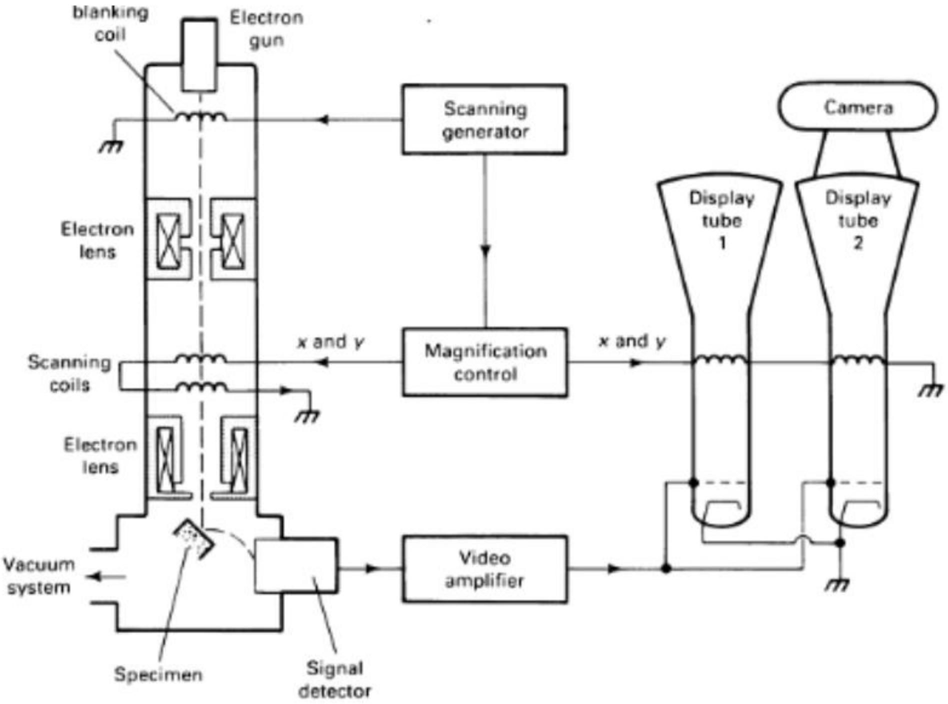


Fig. 2.5.5– Typical design of the Scanning Electron Microscope. Source: [2.5.1] ASM HANBOOK – Volume 9 – Metallography and Microstructures

The scanned image is formed point by point. The deflection system causes the beam to move to a series of discrete locations along a line, then along another line which is below the first, and so on, until a rectangular "raster" is generated on the specimen.

At the same time, the same scan generator creates a similar raster of on the viewing screen.

Two pairs of electromagnetic deflection coils are used to sweep the beam across the specimen.

The first pair of coils deflects the beam off the optical axis of the microscope and the second pair bends the beam back onto the axis at the pivot point of the scan. The magnification M of the image is the ratio of the length of the raster on the viewing screen to the corresponding length of the raster on the specimen. For example, a 100 µm-wide raster on the specimen displayed on a 10 cm wide viewing screen generates an image of 10000x magnification.

When the operator needs an increase in the image magnification, the scan coils are less excited, and the beam deflects across a smaller distance on the specimen.

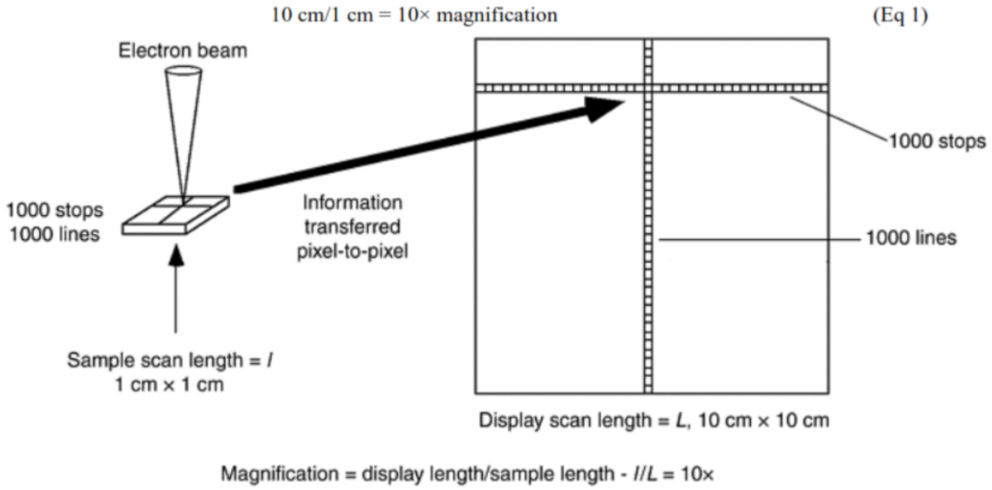


Fig. 2.5.5 – illustration of how the information obtained from a square raster on the sample is transferred to the viewing screen to produce magnification. [2.5.2] Scanning electron microscopy and X-Ray microanalysis – Third edition – Joseph Goldstein. Dale Newbury, David Joy, Charles Lyman, Patrick Echlin, Eric Lifshin, Linda Sawyer, and Joseph Michael.

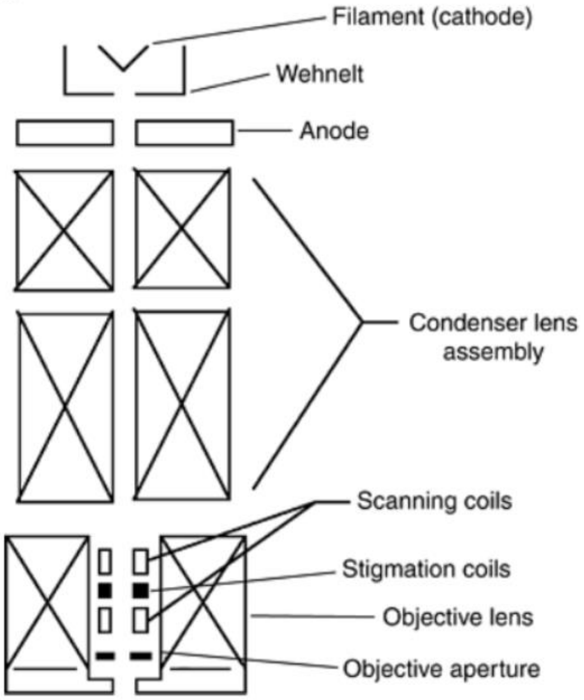


Fig. 2.5.6– sketch of a typical scanning electron microscope. Source: [2.5.3] ASM HANBOOK – Volume 11 – Failure analysis and prevention

The two signals most used to produce SEM images are secondary electrons (SE) and backscattered electrons (BSE); both signals are collected when a positive voltage is applied to a collector screen in front of the detector. Electron captured by the scintillator/photomultiplier are then amplified for display.

As the beam electrons strike the specimen, they interact as negatively charged particles with the electrical field of the specimen atoms. The positive charge of the protons is concentrated in the nucleus, whereas the negative charge of the atomic electrons is much more dispersed in a shell structure beam electron. The interaction between electron beam and atoms can deflect the beam electrons along a new trajectory ("elastic scattering" with no kinetic energy loss), the elastic scattering can leave the specimen (backscattering); the probability of elastic scattering increases in corelation with the atomic number (Z). Heavier atoms have a much stronger positive charge, the electron energy increases as the atomic number (Z) decreases, approximately as $1/E^2$.

Simultaneously with elastic scattering, the beam electrons lose energy and transfer it in various ways to the specimen atoms (inelastic scattering), generating useful imaging signals such as secondary electrons and analysis signal such as X rays.

The back scattered electrons are those electrons that are scattered from the specimens and can be collected as the primary beam scans the specimen surface. The collected back scattered electron image of the specimen surface is displayed on a cathode ray tube (CRT) and can be captured. There is some electron energy loss during the backscattering process. The higher the atomic number of the elements in the sample is the grater the degree of backscattering (less energy loss). This means that elements with higher atomic numbers, such as iron (Z = 26), will appear brighter on the backscattered electron image of the CRT than elements with lower atomic numbers, such as oxygen (Z = 8). An example of a backscattered electron image of oxide scale penetration is shown in figure 2.5.5. Steel appears as a light gray constituent, and the oxide appears as two darker gray constituents.

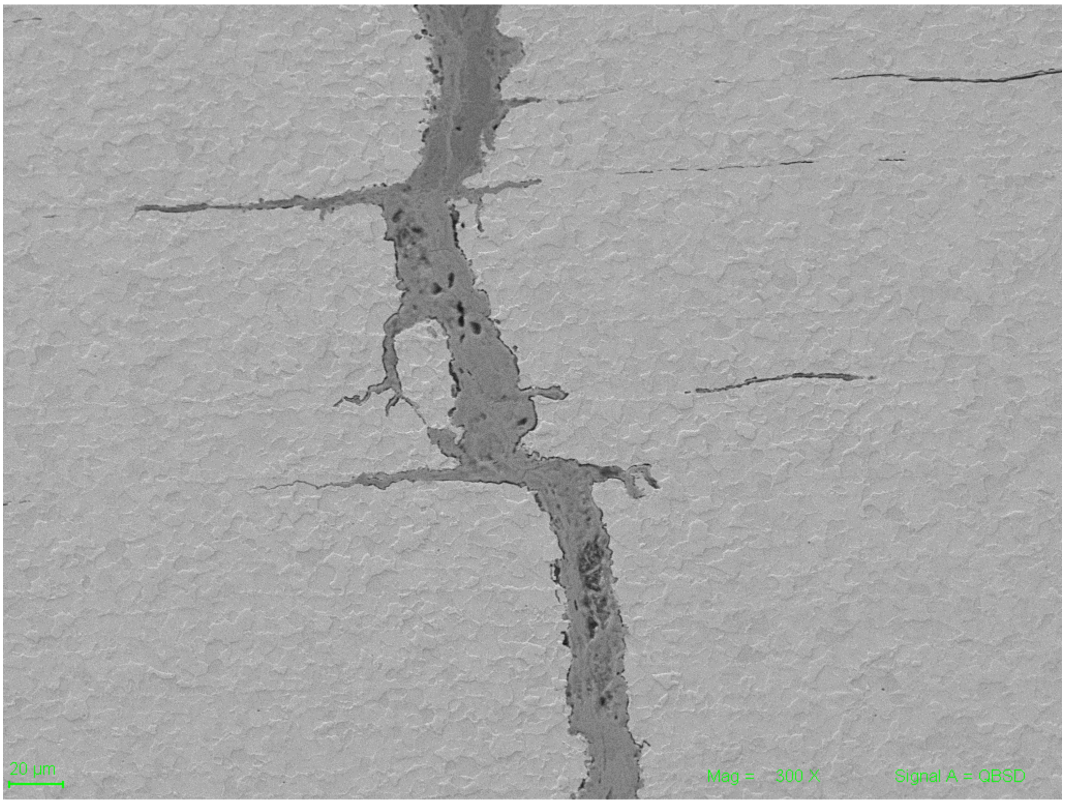


Fig. 2.5.7– detail of a crack obtained by Scanning Electron Microscope with BSE signal. Source: IIS laboratory.

Secondary electrons, on the other hand, are the results of the interaction of primary (beam) electrons with those electrons contained within the atoms in the sample. The primary electrons can displace the loosely held orbital electrons from the atoms. These displaced electrons are called secondary electrons. The SE have a much lower energy than the backscattered electrons, previously described. This means that secondary electrons are only detected from the surface and near-surface regions of the specimen, because those from deeper regions are easily absorbed by the sample. Thus, secondary electrons yield a secondary electron image on the cathode ray tube that reveals surface topography and produces an image with enhanced depth of field.

In this micrograph, inclusions can be seen in many of the voids on the cracked surface.

X-rays are also emitted from the sample, because of the interaction of the primary and backscattered electrons with the inner shell electrons of atoms in the sample. The primary/backscattered electrons have sufficient kinetic energy to displace inner shell electrons out of orbit. Where an electron is displaced from a particular inner electron shell, an X-ray photon is emitted when an electron moves into its place. These X-rays have a characteristic energy (and Wavelengths) for particular atoms. Because of this, they are called characteristic X-rays. This means that every X-ray that is collected has an energy and wavelength that is unique to the particular element present in the sample. The importance of collecting these X-rays is that compositional information can be thus obtained.

Generally, in a SEM, only the X-ray energy is analysed. The technique is called energy dispersive spectroscopy, or EDS. The intensity is plotted on vertical axis, and the X-ray energy is plotted on the horizontal axis. This analysis indicates the elements present but does not reveal the distribution of elements. Some SEMs can provide an EDS X-ray map of the elements contained on the surface of the sample.

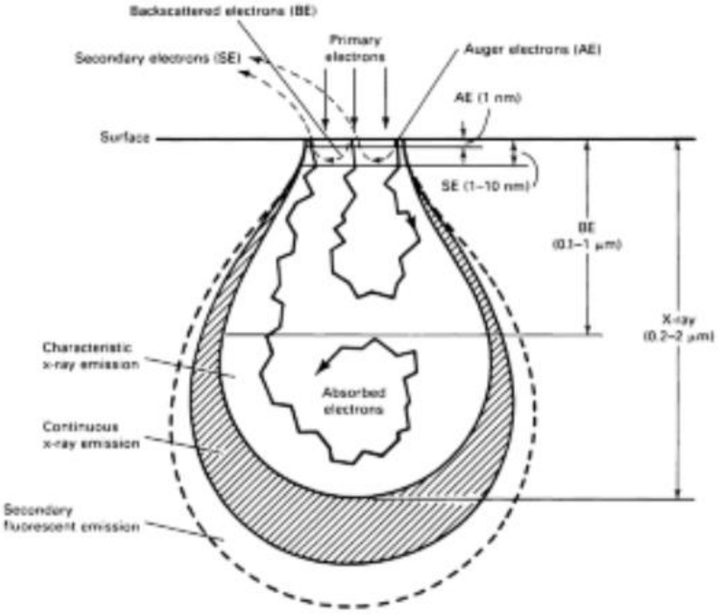


Fig. 2.5.8 – types of electron-beam-exited electrons and radiation used in SEM. Source: [2.5.1] ASM HANBOOK – Volume 9 – Metallography and Microstructures

2.5.2. Forms of contrast

2.5.2.1. Topographic contrast

The most pronounced topographic contrast effects results from the dependence of secondary electron yield and backscattering coefficient on the angle between the surface element and the primary electron beam. The resulting contrast is analogous to an optical image in which the light is generated from the detector and the observation direction is that of the incident electron beam. The stereoscopic impression in enhanced by shadows in regions hidden from the detector.

2.5.2.2. Material contrast

Material contrast, or atomic number contrast, affects results from the anelastic interaction between the backscattered electron and the atoms in the specimen.

Material contrast is useful for qualitative identification of phases and is especially suitable for quantitative evaluation of microstructural geometry by image analysis. The atomic number contrast is usually obscured by topology contrast and, for secondary electrons, is sufficiently strong only in favourable cases, such as ideally flat surfaces.

The topographic contrast for backscattered electrons is much less pronounced than for secondary electrons and can be significantly reduced by using ring-shaped detectors, even if the surface is rough. Edge effects may remain a problem, and ideally flat polishing is necessary for metallographic specimens where the phases have similar chemical composition.

A difference in average atomic number smaller than 1 is sufficient for slight material contrast, at least in the lower range of atomic numbers where the contrast is more pronounced.

2.5.2.3. Electron-channelling patterns

The primary electrons penetrate the crystal lattice to a depth that depends on the atomic packing density along different crystallographic directions. If electrons follow the channels between atom rows, the probability of their escaping is decreased. Many Bragg conditions are met with small angular variations. Rocking the primary electron beam around a point on the specimen creates diffraction lines from a small area (< 10 µm). These selected-area electron-channelling patterns are like Kikuchi patterns obtained using TEM.

Information on the crystal orientation, grain boundary, twins and other crystal features are obtained from a surface layer less than 50 nm in thickness.

2.5.2.4. Edge contrast

At edges, the region of electron escape is deformed, and more secondary electrons and backscattered electrons can exit the specimen at the positive edges through the higher area of the truncated surface; at negative edges, the electron yield is reduced by additional absorption. This effect blurs the exact shape of the edges and can be lessened by reducing the energy of the primary electrons. However, edge contrast is sometimes advantageous. Edges can be detected between facets and phases of identical brightness and small particles. Grain boundaries appear as dark lines after etching. Contrast between phases of different hardness can be achieved by producing various degrees of microroughness by polishing.

2.5.3. Resolution and contrast enhancement

Each element of SEM has its own characteristics regarding resolution and noise, which determine image quality. The SEM operator must know the details in order to optimize the image quality for a specific specimen and purpose of investigation.

For example, the signal-to-noise ratio of the secondary electrons increases alongside the intensity of the primary electron beam then leads to increasing electron yield and scanning time.

The current of the condenser lenses controlling the spot size, the acceleration voltage, the working distance, and the scanning time can be set by the operator.

2.5.3.1. Secondary electron imaging

The depth resolution of images produced in SE mode is due to the small volume from which secondary electrons are excited. The resolution of an SE image obtained by a scanning microscope depends on the width of the energy distribution of primary electrons controlling chromatic aberration, lens aberration, precision of the scanning system, the efficiency of the detectors, and the quality of the amplifiers. Other factors as vibration from the floor or pumping system can deteriorate the image quality.

Factors to be optimized by the operator include the intensity (I) and acceleration voltage (EHT) of the primary beam, the size of the final aperture and its distance to the specimen surface (WD), the scan velocity and the tilt of the specimen surface. For a large depth of focus, which is usually required for fractured surfaces at low magnification, a small aperture and a large working distance are selected. For higher magnification, a short working distance and high lens current must be used to minimize the spot size.

A small spot size results in the emission of a small current of electrons from the specimen surface, which reduces the signal-to-noise ratio.

The optimum choice of acceleration voltage of the primary beam depends by the kind of specimen. At low acceleration voltages, the beam becomes sensitive to contamination and tray field aberration. However, high beam energy is less suitable for producing topographic contrast at high resolution.

Typically, it is necessary to reduce the acceleration voltage in order to observe non-conductive specimens or to improve the topographic resolution.

2.5.3.2. Backscattered electron imaging

The resolution of backscattered mode is much less than resolution of secondary mode because of larger width and depth of escape of electrons. The information depth depends strongly on the acceleration voltage and decreases with atomic number or density.

High resolution BE images can be obtained by reducing the acceleration voltage; however, the depth of information from metal surfaces using BE is at least one order in magnitude larger than that for secondary electron at 5 KeV, and the acceleration energy must be reduced further. This requires high efficiency detectors.

2.5.4. Specimen requirements

Special techniques are not usually necessary in preparing the specimen for SEM. Fracture specimens need to be cut to a size, without contaminating the surface, that will fit within the specimen chamber of the microscope.

The specimen can be glued to a metallic stub that fits into the specimen chamber. Usually, more than one specimen can be placed in the camber.

Mounted metallographic specimens are also used in SEM, specially prepared conductive dyes are used to “bridge” the metallic specimen to the metallic specimen holder in order to electrically ground the specimen. If not electrically grounded, the electrical charges will build up on the specimen surface and cause imaging problems. In fact, on certain specimens, such as gold, because its excellent electrical conductivity, is deposited on the surface by sputtering to drain away the excess electrons and enhance the secondary image. Conductive sputtering is essential for nonconducting surface.

If EDS analysis is required to qualitatively analyse the composition of non-metallic inclusions, it is important not to polish the specimen with alumina or silica polishing compounds, because they can contaminate the specimen. A diamond polishing compounds is usually more appropriate unless one is analysing for carbides.

2.5.5. EDS analysis

EDS (Energy-Dispersive X-ray Spectroscopy), also known as energy-dispersive X-ray analysis, is an analytical technique used in scanning electron microscopes (SEM) to determine the chemical composition of samples. This technique is particularly useful for obtaining information about the distribution of elements within a sample and to identify the elements present.

When high-energy electron beam electrons strike the sample, they can remove electrons from the orbits of the sample's atoms. This interaction creates vacancies in the atomic shells.

The missing electrons in the atomic shells are replaced by electrons from higher shells. When this occurs, energy is released in the form of X-rays that are characteristic of the elements involved. Electrons falling to fill the inner vacant orbits release X-rays with specific energies for each element.

X-ray spectra, consisting of peaks of specific energy, are detected by an energy-dispersive detector. This detector measures the energy of the X-rays emitted from the sample after interaction with the electron beam.

Each element has a unique X-ray spectrum with distinct energy peaks (its own identifying peak). The measured energy peaks in the detector allow for the identification of elements present in the sample.

The X-rays analysed in SEM usually have energies between 0.1 and 20 KeV.

The incident beam electron leaves the atom having lost at least E_K, where E_K is the binding energy of the electron to the K shell. The ejected orbital electrons leave the atom with a kinetic energy of a few eV to several KeV, depending on the interaction. The atom itself is left in an excited state with a missing inner shell electron. The atom returns to its ground state (lowest energy) within approximately 1 ps through a limited set of allowed transition of outer shell electron filling the inner shell vacancy. The energies of the electrons in the shells (atomic energy levels) are sharply defined with values, characteristic of a specific element. The energy difference between electron shells is a specific or characteristic value for each element.

The excess energy can be released from the atom during relaxation in one of two ways. In the Auger process, the difference in shell energies can be transmitted to another outer shell electron, ejecting it from the atom as an electron with a specific kinetic energy. In the characteristic X-ray process, the difference in energy is expressed as a photon of electromagnetic radiation which has a sharply defined energy.

Electrons of an atom occupy electron shells around the atom that has a specific energy. In order of increasing distance from the atomic nucleus, these shells are designated by K, L, M etc.

Characteristic X-ray lines results from transitions between subshells.

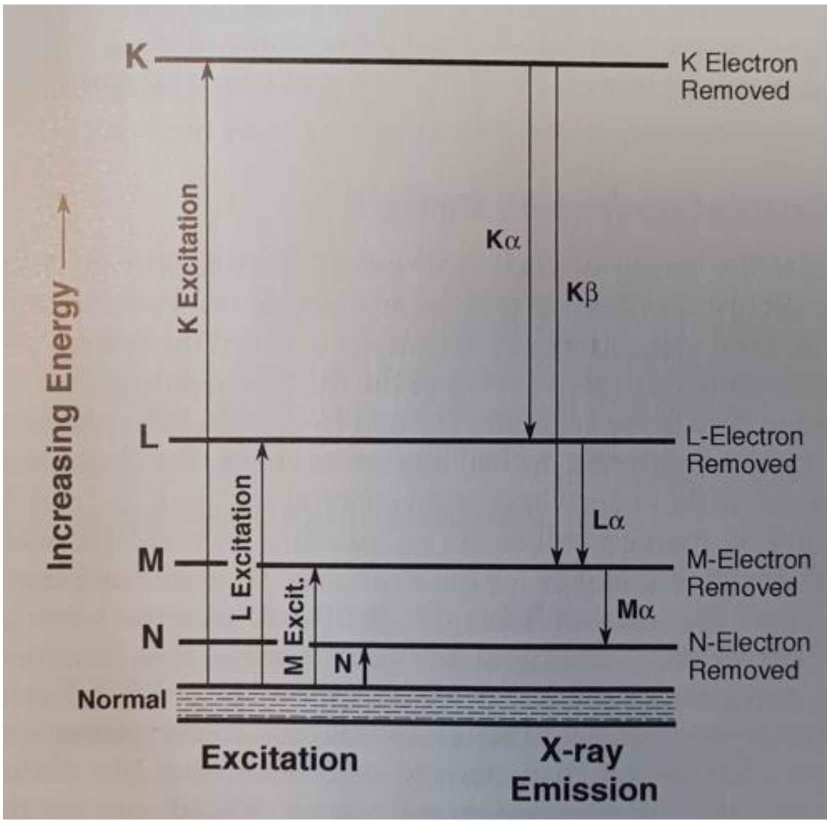


Fig. 2.5.9 – energy level diagram for an atom. The energy of the atom increases upon ionization of the K,L,M or N shell (excitation). Source: [2.5.2] Scanning electron microscopy and X-Ray microanalysis – Third edition

– Joseph Goldstein. Dale Newbury, David Joy, Charles Lyman, Patrick Echlin, Eric Lifshin, Linda Sawyer, and Joseph Michael.

In addition to identifying elements, EDS can be used to map the spatial distribution of elements within the sample. This provides information about the chemical composition in different parts of the sample. Furthermore, EDS can offer quantitative information about the percentage of each element present in the sample.

In summary, EDS in a scanning electron microscope allows for the identification of chemical elements present in a sample by analysing the X-ray spectra emitted during the interaction of the electron beam with the sample. This technique is valuable in metallography, fractography and other kind of analyses where understanding the chemical composition of samples at a microscopic level is important.

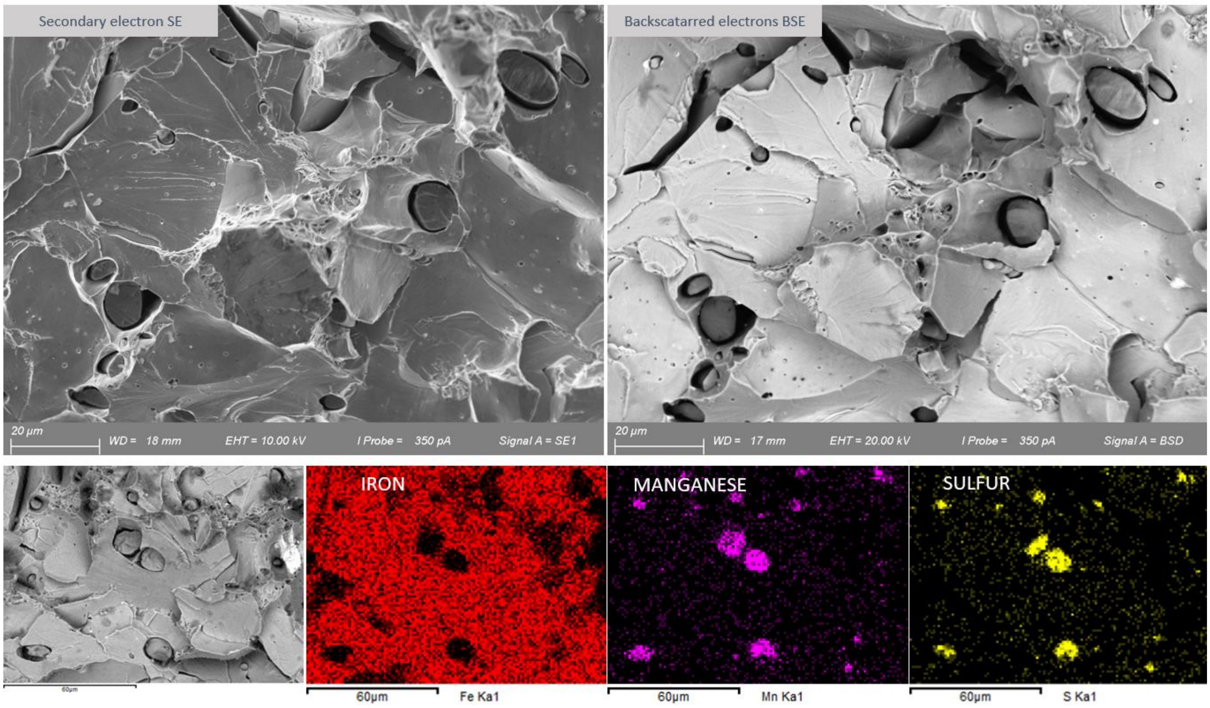


Fig. 2.5.9 – example of a mapping of elements obtained by EDS. Source: IIS Laboratory

References:

[2.5.1] ASM HANBOOK – Volume 9 – Metallography and Microstructures

[2.5.2] Scanning electron microscopy and X-Ray microanalysis – Third edition – Joseph Goldstein. Dale Newbury, David Joy, Charles Lyman, Patrick Echlin, Eric Lifshin, Linda Sawyer, and Joseph Michael.

[2.5.3] ASM HANBOOK – Volume 11 – Failure analysis and prevention

2.6. Demonstration of scanning microscope capabilities

2.6.1. Fracture surfaces examination

Scanning electron microscopy finds in fractography one of its main uses. Fracture surfaces are characterized by jagged morphology and typically should be observed by secondary electrons (SE) to obtain morphology and topography information.

A fracture can be ductile, brittle, intergranular, or propagated due to the mechanical fatigue phenomena. Ductile fractures are characterized by tearing of metal after plastic deformation and internal energy accumulation. Ductile tensile fractures in most materials have a fibrous appearance and are classified on a macroscopic scale as either flat (perpendicular to the maximum tensile stress) or shear (at about 45° to the maximum tensile stress). Ductile fractures occur by micro void formation and coalescence, in the technical literature these micro voids are called “dimples”.

SEM examination of ductile fracture surfaces reveals information about the type of stress, direction of crack propagation, and relative ductility of the material. The shape of the dimples is determined by the type of loading that the component experienced during fracture. The orientation of the dimples reveals the direction of the crack propagation. Equiaxed dimples are cup shaped, and they form under conditions of uniform plastic strain in the uniaxial stress (e.g., tensile test). In comparison, elongated dimples shaped like parabolas results from non-uniform plastic-strain conditions (e.g., bending or shear overload).

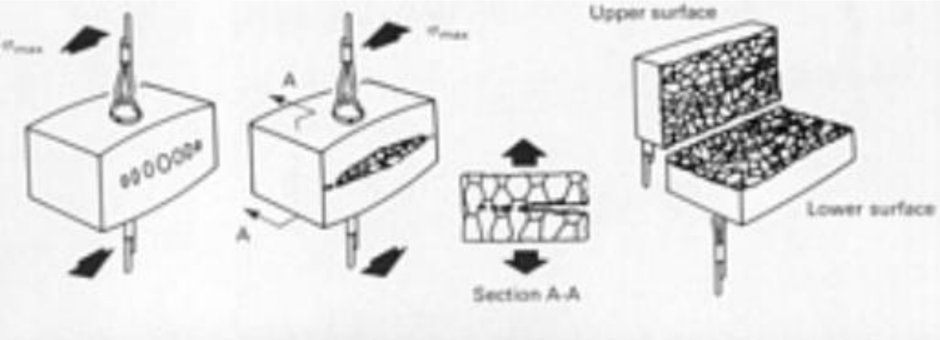


Fig. 2.6.1 – Ductile fracture mechanism. Dimples formation.

Source: [2.6.2] ASM HANDBOOK – Volume 12 – Fractography

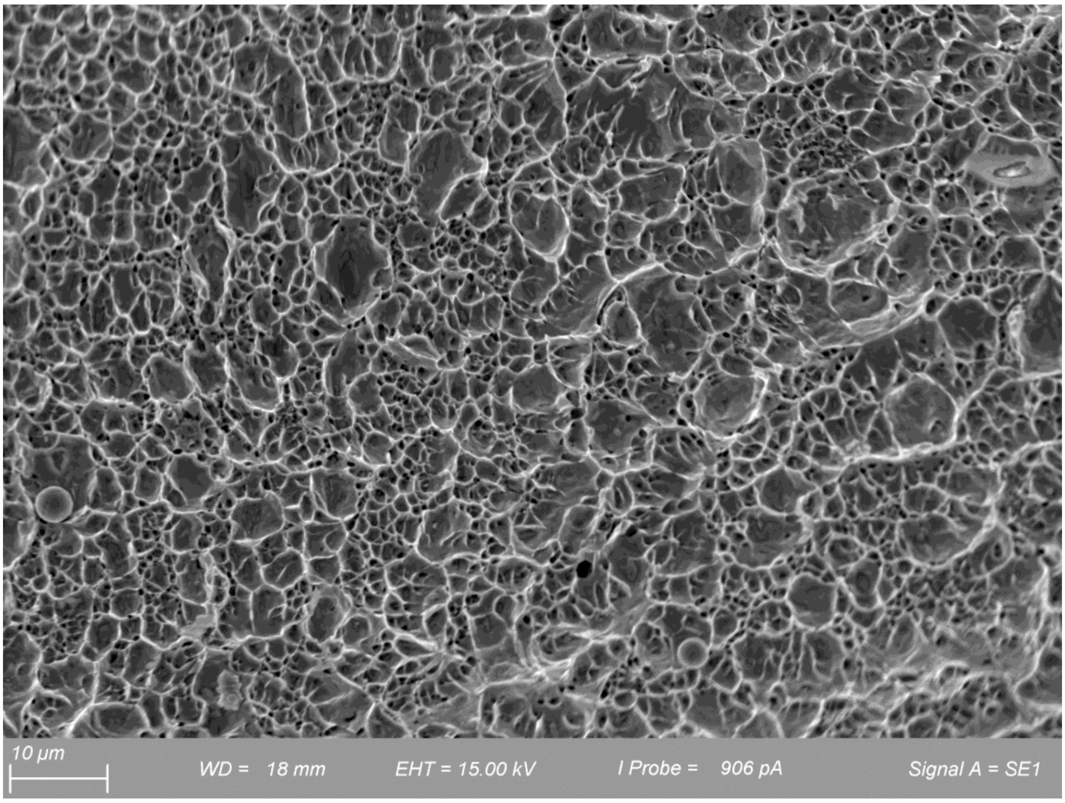


Fig. 2.6.2 – example of a ductile fracture surface observed by SEM (Secondary electrons). Source: IIS Laboratory.

Brittle fractures are characterized by a rapid crack propagation with less use of energy than with the ductile fractures and without plastic deformation. Brittle fractures have a bright, granular appearance and may not exhibit necking; typically, they have a flat path and are perpendicular to the direction of the maximum tensile stress. A chevron pattern may be present on the fracture surface, pointing toward the origin of the crack, especially in brittle fractures of flat platelike components. At high magnification, the observation of brittle fracture by SEM reveals very flat surfaces known as cleavage planes.

Cleavage is a low energy fracture that propagates along well-defined low index crystallographic planes know as cleavage planes. A cleavage fracture should have perfectly machining faces and should be completely flat and featureless. However, metal alloys are polycrystalline and contain grain boundaries, and other imperfections that changes the crystal lattice orientation, such as possible mismatch of the low-index planes across the grains. These alloys produce distinct cleavage planes.

River patterns represent steps between different local cleavage facets at slightly different heights but along the same general cleavage plane. Because local crystallographic structure can modify the local direction of the crack propagation, the overall direction is assigned only after confirming the orientation of the river patterns in several areas on the fracture surface.

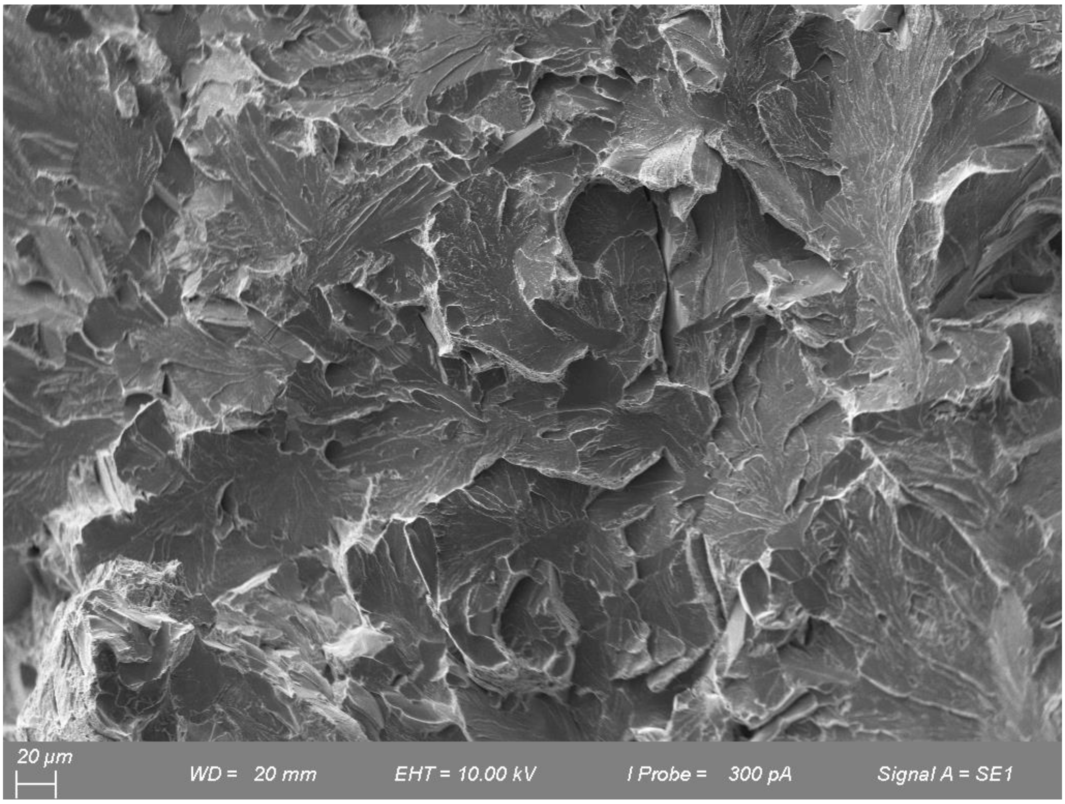


Fig. 2.6.3 – Example of a brittle fracture surface observed by SEM (Secondary electrons). Source: IIS Laboratory.

Intergranular brittle fracture referred to as grain boundary decohesion is promoted by the synergetic effect of the environmental conditions and tensile stress, or it is the result of the presence of the lowest melting point constituents at the grain boundaries. It is easy to recognize intergranular fracture, but the identification of the fracture causes is more complex.

The cohesive process involving the weakening of the atomic bonds, the reduction in surface energy required for localized deformation, molecular gas pressure, protective films depletion and anodic dissolution at active sites is associated with hydrogen embrittlement and stress corrosion cracking (SCC).

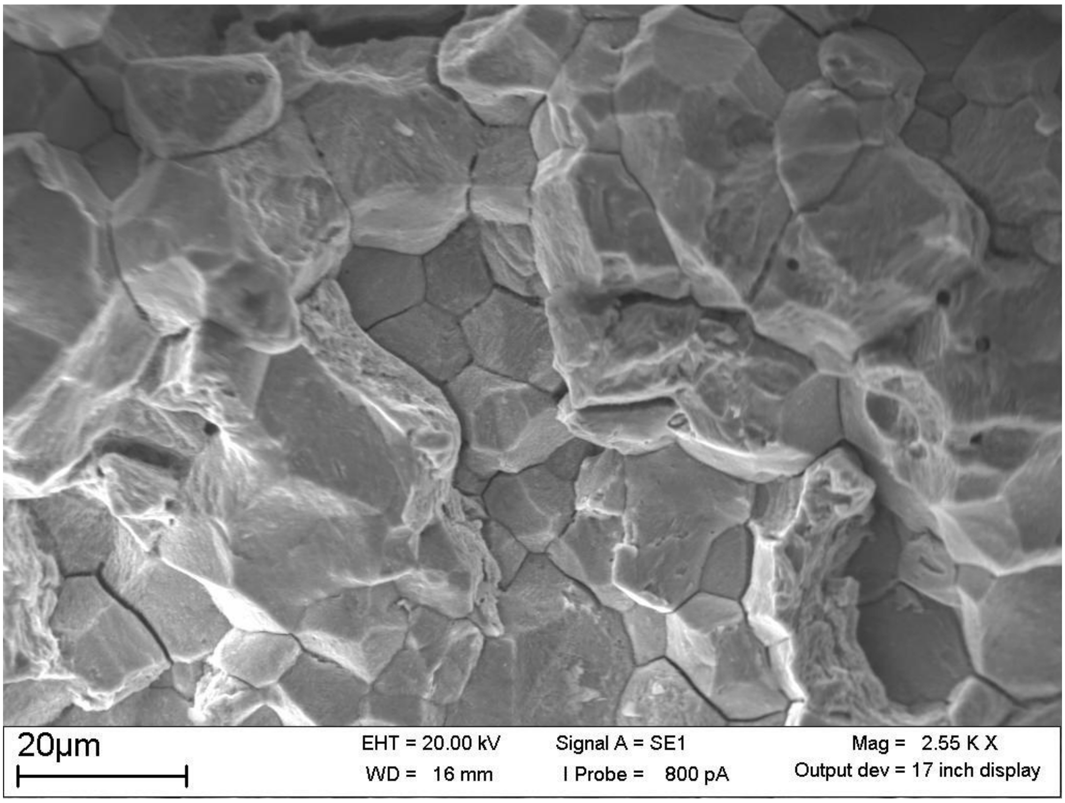


Fig. 2.6.4 – Example of intergranular fracture surface observed by SEM (Secondary electrons). Source: IIS Laboratory.

Fatigue is a time-dependent mechanism that can be separated in three stages that exhibit different features:

- Stage I: crack initiation or crack nucleation.
- Stage II: crack propagation.
- Stage III: final fracture by overload.

The stage I is a point (or points) that is usually at the surface, where the cyclic strain is the greatest or where material defects or residual stresses lessen the fatigue resistance of the component. The crack typically initiates at a small zone and propagates by slip-line fracture, extending inward from the surface at 45° to the stress axis.

The location of the origin is defined by interpreting features of the stage II crack propagation zone. Macroscopic beach marks radiate away from the origin in concentric semicircles. These are the progression mark commonly associated with fatigue. When the propagation zone is examined by SEM at higher magnification (> 5000x), the beach marks can be resolved into hundreds or thousands of fatigue striations.

Striations are characteristically mutually parallel and at right angles to the local direction of crack propagation. They vary in striation-to-striation spacing with cyclic stress intensity, they are equal in number to the number of load cycles. Also, fatigue striations do not cross one another, but may join and form a new zone of a local crack propagation.

If the component has been subjected to uniformly applied loads of sufficient magnitude, a single advance of the crack front (the distance between two adjacent striations, is a measure of the rate of propagation per stress cycle). However, if the loading is non-uniform, there are wide variations between a given stress cycles series and the spacing of the striation, although one stress cycle doesn't exhibit striations.

Despite the presence of striations indicating fatigue as the mode of failure, their absence doesn't eliminate fatigue as a possibility. For example, fatigue striation is well defined in aluminium alloys but is often poorly defined in ferrous alloys; furthermore, oxidation, corrosion, or mechanical damage can eliminate striations.

Fig. 2.6.5 – Example of nickel alloy shaft fractured by fatigue phenomena. Source: IIS Laboratory.

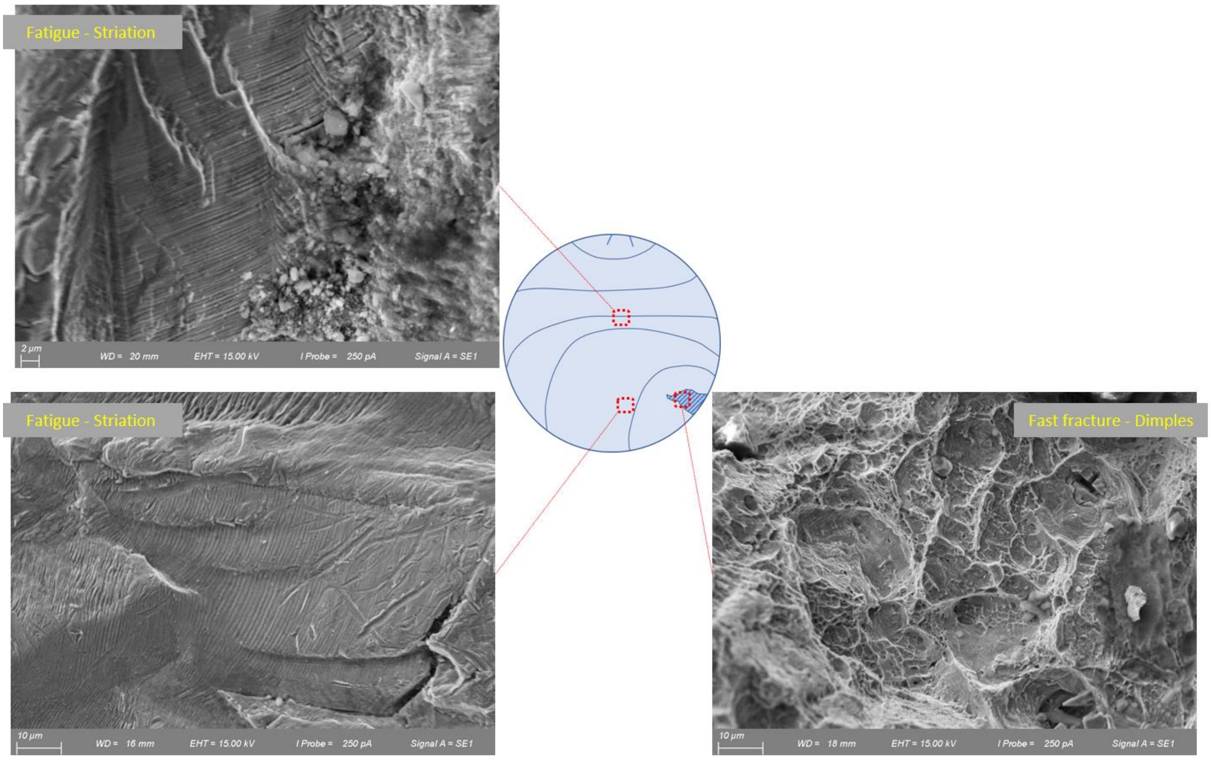


Fig. 2.6.6 – Details of the fracture surface in the propagation area with striation and final fast fracture with ductile mechanism (dimples). Source: IIS Laboratory.

2.6.2. SEM applied to the failure analysis of metal alloys and their welding joints

According to the previous paragraph, it is easy to understand why the SEM is so important for the study of damage mechanisms. In addition to the analysis of the fracture morphology, it allows to analyse phases, intermetallic compounds, corrosion deposits/products, and to perform punctual chemical analyses by EDS probe. Some practical cases are shown below.

Example 1:

Investigation of a crack observed in the weld metal of dissimilar welded joint between carbon steel and super-duplex stainless steel performed adopting a Ni-base alloy filler metal (Inconel 625) and a SAW welding process.

The BSE (Back Scattered Electron) images have revealed the presence of a lamellar (eutectic) white phase, located in correspondence to crack surfaces and crack tips (see photos 2.6.7).

The EDS qualitative microanalysis has shown that these compounds consist of Nb/Mo-rich intermetallic phases. These compounds are characterized by a low melting point.

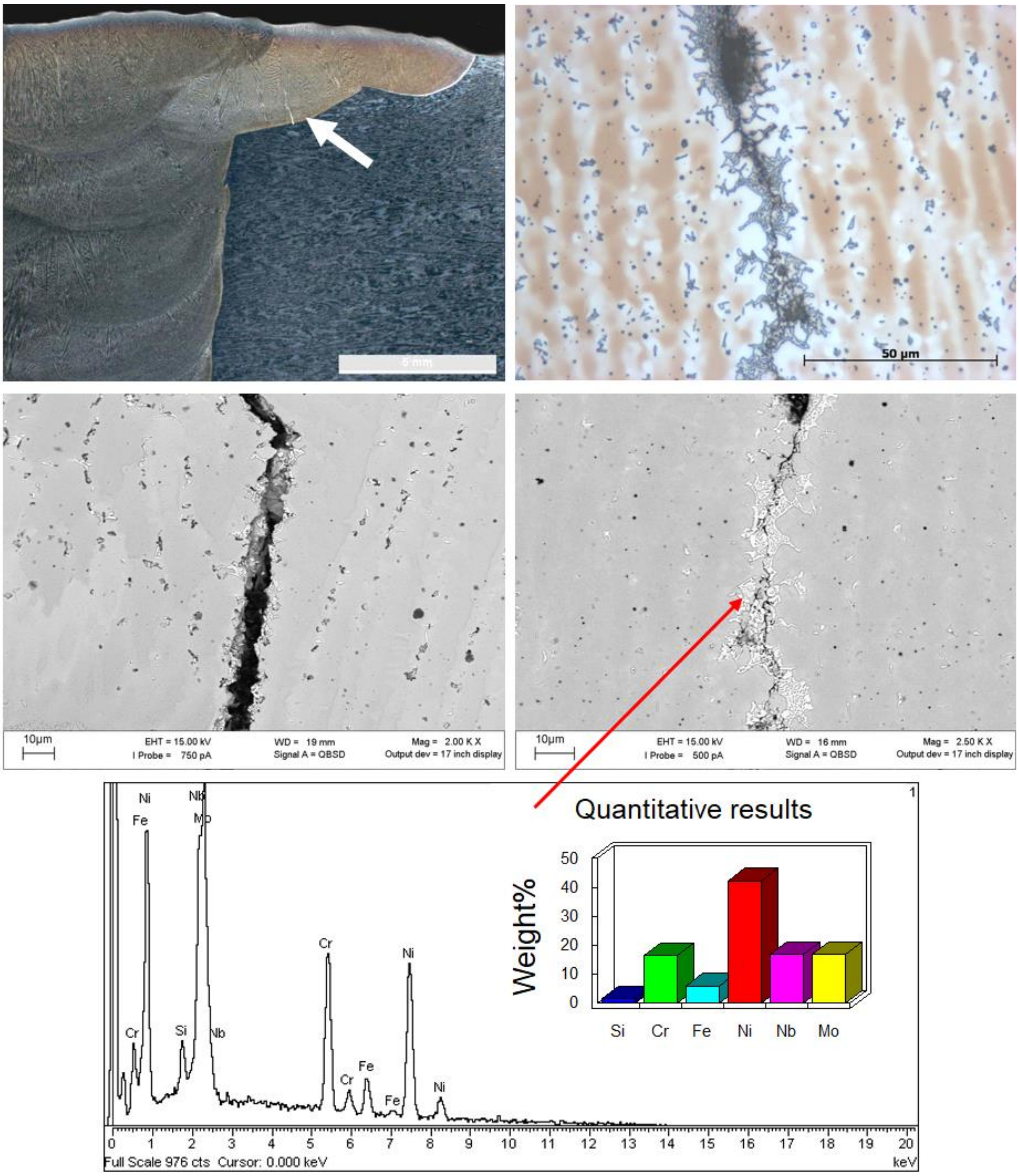


Fig. 2.6.7 – Hot crack observed in the weld metal of dissimilar welded joint between carbon steel and super-duplex stainless steel performed adopting a Ni-base alloy filler metal. Source: IIS Laboratory.

Example 2:

Investigation of a crack observed close to a brazing joint of a component in nickel alloy type Incoloy 800. The brazing alloy was CuAgZn alloy.

The SEM investigation performed on a micrographic section showed the intergranular cracks. The EDS analyses performed inside the cracks mainly revealed cooper, silver, and zinc, attributable to the brazing alloy. The failure mechanism was due to Liquid Metal Embrittlement (LME).

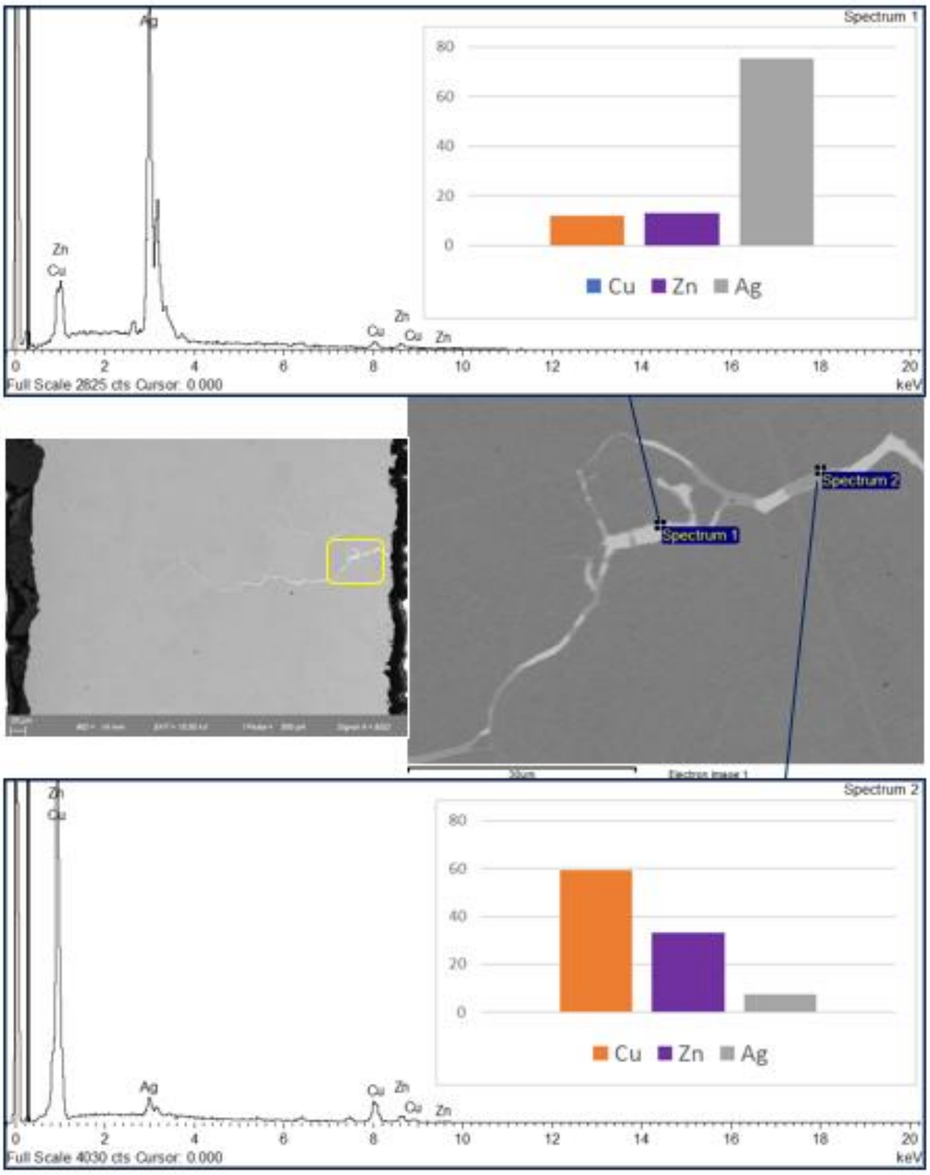


Fig. 2.6.8 – LME observed in a brazed component – Source: IIS Laboratory.

Example 3:

Investigation of a fracture occurred on a heat exchanger tube in austenitic stainless steel alloy type AISI 316L.

The micrographic examination had revealed a network of trans-granular and branched cracks on the whole section investigated. SEM and EDS analyses detected chlorine and other elements attributable to the base material. The failure mechanism was due to chlorine stress corrosion cracking (Cl-SCC).

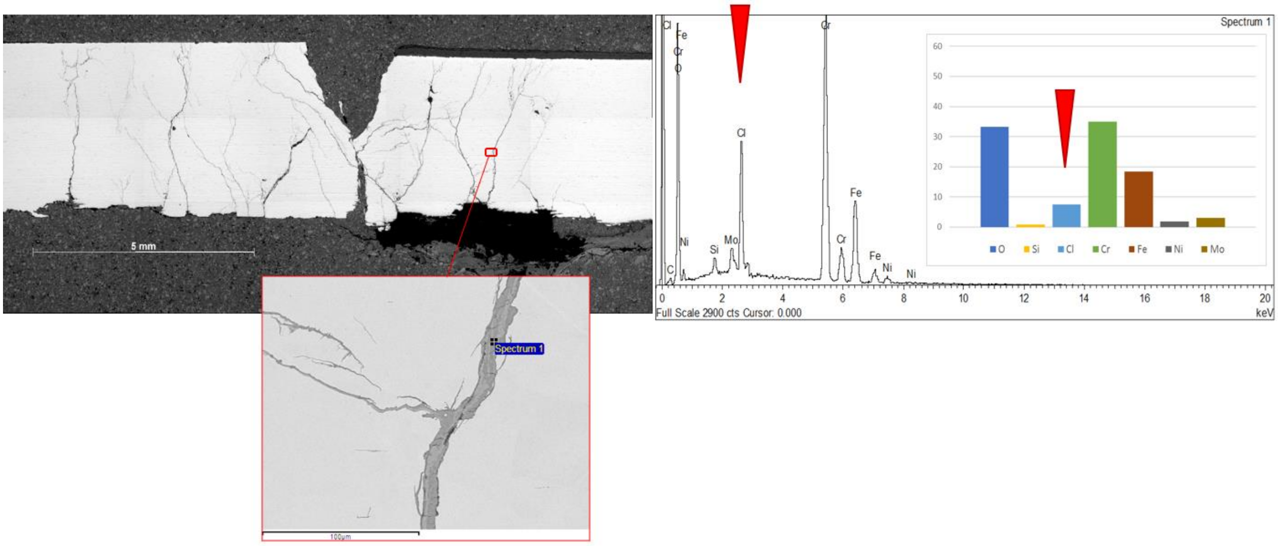


Fig. 2.6.9 – Selective corrosion of a duplex stainless steel in alkaline environment. Source: IIS Laboratory.

Example 4:

Investigation of corrosion phenomena observed on a Duplex Stainless Steel welding joint. Both micrographic and fractographic examination, performed by SEM, showed a selective corrosion of an austenite phases in duplex heat affected zone. This phenomenon is typical of an alkaline environment.

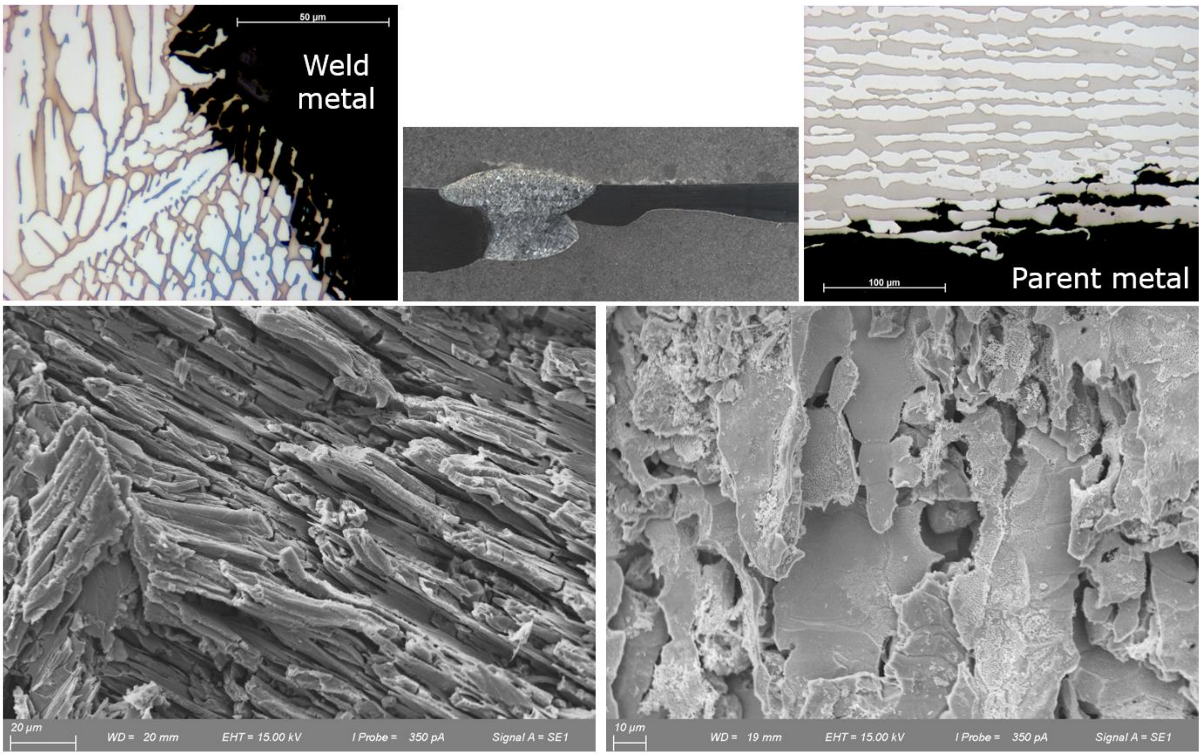


Fig. 2.6.10 – Selective corrosion of a duplex stainless steel in alkaline environment. Source: IIS Laboratory.

Example 5:

Investigation on a sample taken from an "Inlet casing" of a turbine, affected by numerous cracks.

The casing was manufactured in austenitic stainless-steel type AISI 304 H.

The turbine was subject to severe thermal cycles, with numerous start-ups and shutdowns.

The temperature of the inlet flow was about 800 °C; steam was injected inside from the tubes located in the central cone, with the dual purpose of preheating the casing before start-up and cooling it during operation. The service temperature had to be below 732 °C

the steam is injected with a flow rate of approximately 45 Kg/hours, but for some periods, it reached approximately 800 Kg/hours.

Micrographic examinations showed cracks with a branched and intergranular pattern, characterized by the presence of deposits inside them. The EDS analyzes of the deposits revealed elements attributable to the base material and variable quantities of sulphur.

The corrosion phenomena occurred was attributable to stress corrosion cracking caused by polythionic acids (Polythionic Acid Stress Corrosion Cracking - PASCC). The sulfides can react with the humidity present in the steam and form polythionic acids (H_2SnO_6). PASCC typically occurs during shut-down periods, or during operation if condensation is present in the steam. The sensitized grain of austenitic stainless steel also provides a preferential weak point that favors the development of the phenomenon.

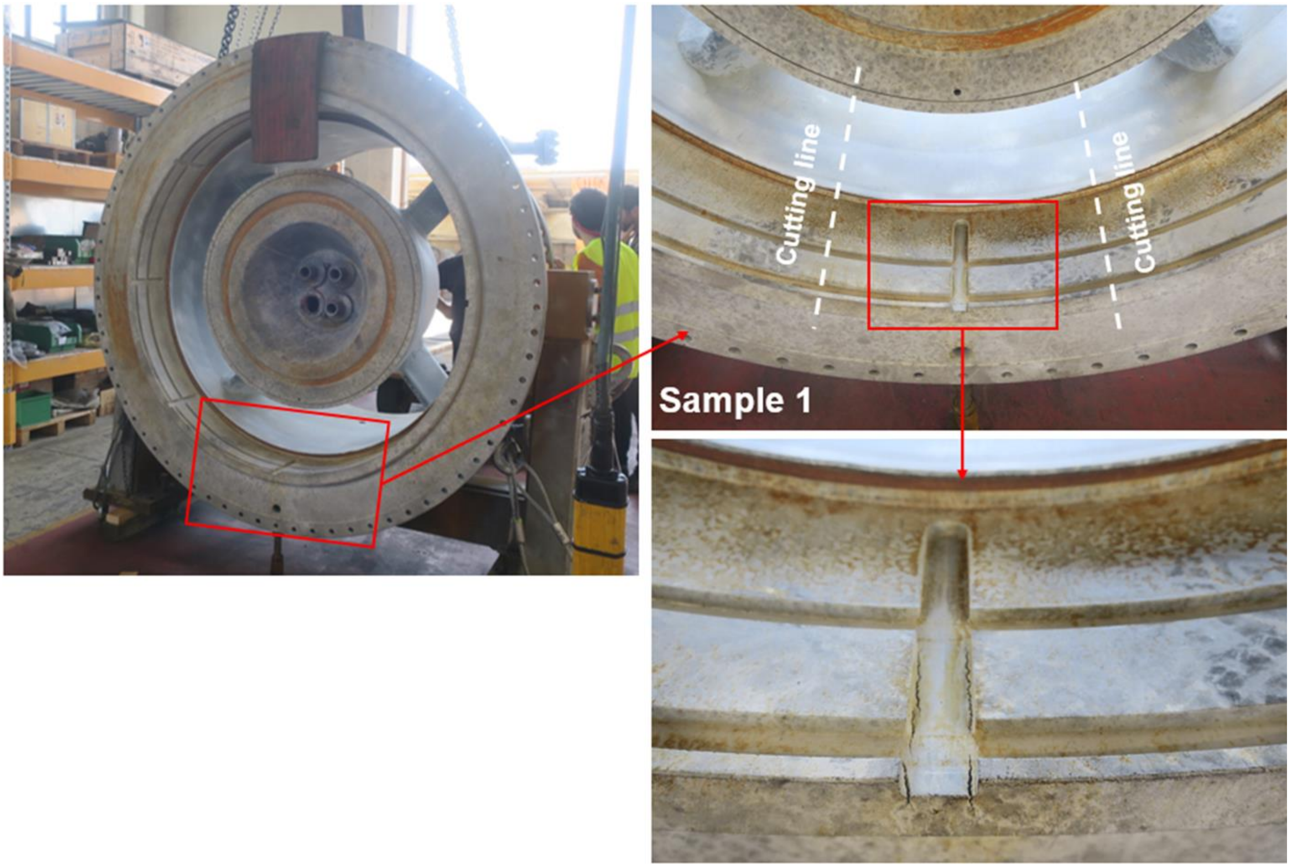


Fig. 2.6.11 – details of the inlet casing of a turbine manufactured in austenitic stainless steel type AISI 304H. Source: IIS Laboratory.

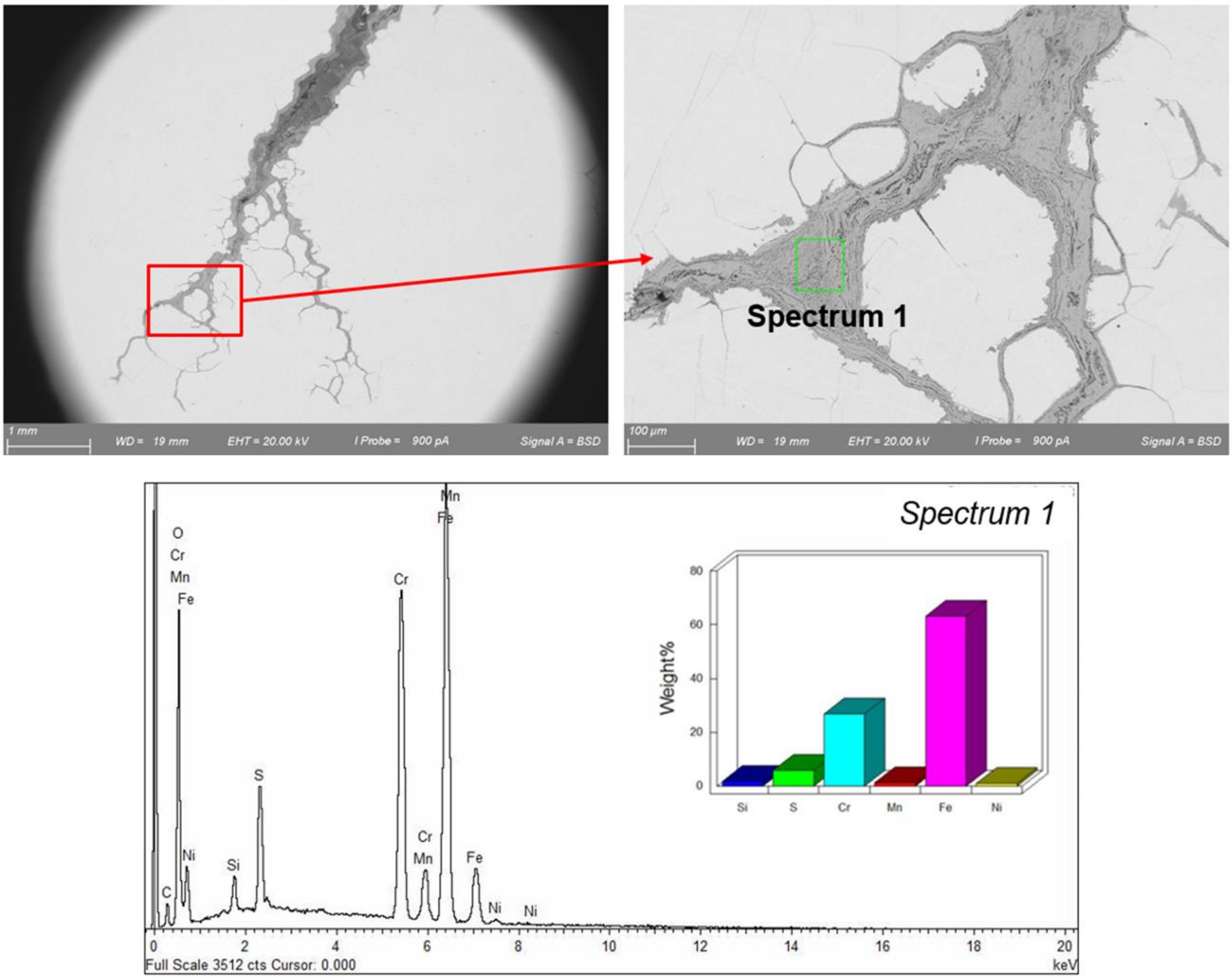


Fig. 2.6.12 – details of the micrographic section taken from the inlet casing at some cracks observed by Scanning Electron Microscope. EDS analysis shows the presence of elements attributable to base metal and sulphur. Source: IIS Laboratory.

References:

[2.6.1] ASM HANDBOOK – Volume 9 – Metallography and Microstructures

[2.6.2] ASM HANDBOOK – Volume 12 – Fractography

[2.6.3] ADM HANDBOOK – Volume 11 – Failure analysis and prevention

[2.6.4] Scanning electron microscopy and X-Ray microanalysis – Third edition – Joseph Goldstein. Dale Newbury, David Joy, Charles Lyman, Patrick Echlin, Eric Lifshin, Linda Sawyer, and Joseph Michael.

3. CU3: Demonstrations in Macro and Microscopic Examination

3.1. Demonstration of assessment of macroscopic specimens of joints

3.1.1. General considerations

Metallography is a part of physical metallurgy that deals with the study of the macrostructure and microstructure of metallic materials.

Macroscopic analysis represents a rapid and simple method for researching materials. It involves the examination of large surfaces, highlighting local or general defects, with details being overlooked. It is the simplest and quickest method for researching metals and metallic alloys. Typically, it precedes any structural analysis and consists of visual observation with the naked eye or at a maximum magnification of 50:1 for the samples being examined.

Through macroscopic analysis, aspects related to the solidification of metallic materials (dendrites, shrinkage cavities, cracks, etc.), their fracture, mechanical, and structural inhomogeneities, and the structure of welded joints can be revealed. It is performed directly on the external appearance of the sample (material) without any prior preparation or on surfaces resulting from fractures (casings), whether unprepared or prepared by grinding and chemical etching. Regardless of the type of macroscopic analysis, its execution involves a series of operations: selecting the sampling location and obtaining the sample, sectioning, grinding, and, if necessary, chemical etching.

3.1.1.1. Preparation of metallographic samples

The preparation of metallographic samples consists of three stages:

1. ***Sampling** (extraction of samples)* - The location for sampling must be chosen so that the sample is representative of the material under study. Since there is no general rule for choosing this location, it will depend on the nature, shape, and size of the specimen, as well as the mechanical or thermal treatments to which the material has been subjected. Instructions on how to take and prepare metallographic samples are provided in ISO/TR 20580:2022 "Preparation of metallographic specimens."
2. ***Preparation of the examination surface*** - The prepared surface of the samples must be flat and free of impact marks, scratches, or foreign bodies introduced during the preparation process. The three stages of surface preparation are:

- levelling - achieved through grinding or polishing. If the sample was cut using machine tools or a hand saw, this step may not be necessary. Care should be taken to avoid overheating and deformation of the surface.
- grinding - carried out using abrasive materials (abrasive papers) with increasingly fine abrasive grain sizes. Grinding of samples can be done manually or automatically.

- polishing - aims to remove the last very fine scratches and create a perfect shine on the prepared surface. This operation can be performed using mechanical grinding machines or through an electrolytic process.

3. ***Metallographic etching*** - Depending on the material of the sample, a wide variety of metallographic etchants are used. When these meet the polished surface of the sample, they dissolve or colour the structural constituents differently, revealing the structure. Etching can be done by immersing the piece in the etchant or by swabbing with cotton wool, soaked in the etchant. Normally, the sample is considered etched when it has lost its metallic lustre. After revealing the structure through etching, the samples are washed with water, then with alcohol, and dried by gentle blotting on filter paper or in a stream of warm air.

3.1.2. Macroscopic analysis on unprepared surfaces

This refers to the examination and analysis of materials at a macroscopic level without any prior surface preparation. In this approach, the surface of the material is observed and evaluated in its natural or as-found state, without any polishing, grinding, or chemical etching.

Macroscopic analysis on unprepared surfaces can provide valuable insights into the overall condition, defects, and features of a material. It allows for a quick assessment of material properties and characteristics without the need for extensive sample preparation.

This method is particularly useful when a rapid assessment is required, or when the material's external features and defects need to be studied without altering its natural state. However, it may have limitations in revealing fine structural details compared to more advanced microscopy techniques that involve sample preparation.

In summary, macroscopic analysis on unprepared surfaces is a valuable tool in materials analysis, offering a straightforward way to assess the condition and characteristics of materials without the need for extensive sample preparation.

Figure 3.1.1. Sample of Annealed Steel. Source: https://sim.tuiasi.ro/wp-content/uploads/2015/03/MF1-Laboratoare.pdf

Carburized and quenched steels have a finer grain structure on the exterior than in the central region of the section, and the shade of grey varies from the exterior to the interior (Figure 2).

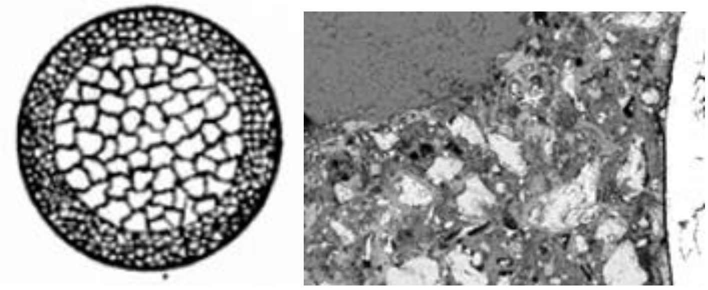


Figure 3.1.2. Sample of Carburized Steel. Source: https://sim.tuiasi.ro/wp-content/uploads/2015/03/MF1-Laboratoare.pdf

The colour of parts that have fractured during use can potentially indicate the cause of the fracture. If in one area the colour is darker than the rest of the surface, it suggests the presence of a pre-existing crack before the final fracture.

The formation and appearance of a cracked surface provide information about the causes of the fracture and the type of stress that led to it. A fracture surface in the form of a cone and cup in the vicinity of necking indicates ductile cracks, while a fracture surface without necking deformations suggests brittle cracks. Fatigue propagations are recognized by their specific appearance on the fracture surface (Figure 3). Under the influence of variable dynamic stresses, small cracks form and propagate from the exterior to the interior, rarely in the opposite direction, reducing the load-bearing cross-section, ultimately leading to sudden material failure. The region where the crack propagates gradually has a smooth and shiny appearance (due to the friction of contacting surfaces), and the final fracture zone appears grainy. Early cracks can originate from surface imperfections (scratches, grooves, abrupt section transitions, etc.), which act as stress concentrators.

Figure 3.1.3. Fatigue Fracture Surface. Source: https://sim.tuiasi.ro/wp-content/uploads/2015/03/MF1-Laboratoare.pdf

By fracturing a metal ingot intergranular, the resulting surface can reveal the three zones formed during primary crystallization.

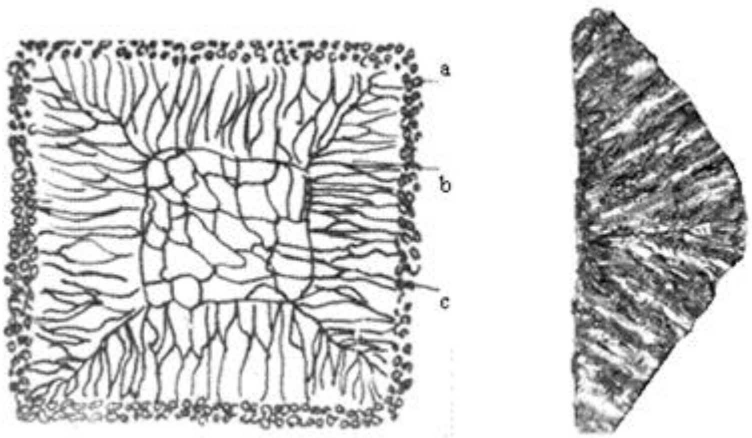


Figure 3.1.4. Zones formed during primary crystallization:

a) Zone of fine equiaxed grains; b) Zone of columnar grains;

c) Zone of coarse equiaxed grains. Source: https://sim.tuiasi.ro/wp-content/uploads/2015/03/MF1-Laboratoare.pdf

Zone 1 consists of small, spherical, and equiaxed grains. In this zone, the cooling rate is very high due to the contact between the liquid (melt) and the cold wall of the mold, resulting in a rapid crystallization rate and a high crystallization capacity. Moreover, roughness and impurity particles on the mold walls act as foreign crystallization nuclei, leading to a fine grain structure.

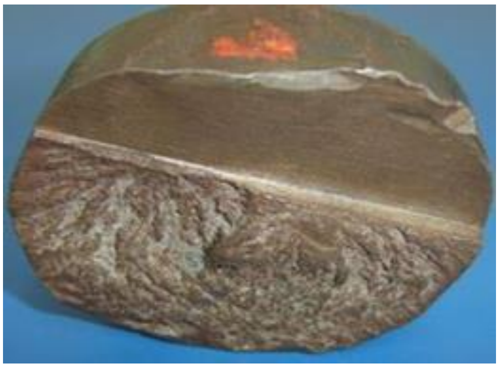
Zone 2 is the region of columnar grains (the zone of trans crystallization). The formation of elongated grains along the temperature gradient is caused by the high cooling rate, resulting in a high linear crystallization rate. However, the crystallization capacity is lower in this zone due to the absence of foreign crystallization nuclei.

Zone 3 consists of coarse, spherical, and equiaxed grains that are not oriented. This zone is found in the centre of the ingot, where the cooling rate is very low due to the solidified outer layer, resulting in a low degree of supercooling, and consequently, low, and uniform crystallization capacity and rate. Metallic material with such an inhomogeneous structure can only be used after undergoing a heat treatment process to anneal and homogenize the structure.

3.1.3. Macroscopic analysis of metallic materials on unprepared surfaces allows for the examinations and assessment of these structural features

No.	*Sample studied*	*Observations*
1		**White cast iron in its raw state** The sample originates from a remelting block; brittle metallic material. - Silver-white fracture with coarse columnar grains (schistose appearance). - Pores (external blowholes) are visible on the exterior surface, and internal blowholes can be seen within the fracture.
2		**Raw gray cast iron** The sample comes from a remelting block. The fracture has a gray colour, and within it, the following can be observed: - Fine equiaxed (globular) grains that are non-uniform in size. - Ellipsoidal voids caused by gases trapped in the molten metal during solidification (internal blowholes). - A chemical inhomogeneity of black colour resulting from the segregation of graphite carbon (zonal segregation). - Pores are visible on the exterior surface.

3		**Mottled Cast Iron** The sample comes from a remelting block. It is a type of cast iron that transitions from white cast iron to gray cast iron, which is confirmed by its fracture: - On the exterior, it exhibits the colour and macroscopic structure typical of white cast iron, while towards the base, it corresponds to gray cast iron (between these two typical zones, there is a mixture of columnar white cast iron grains with equiaxed gray cast iron grains). - Internal and external blowholes (pores) are visible, as well as a spherical-shaped cold shut.
4		**Mechanical Gray Cast Iron** The sample comes from a component produced by casting from secondary melt gray cast iron. - The colour of the fracture is gray, and the grains are equiaxed, very fine, and uniform. - There are no internal or external blowholes (pores).
5		**Cast Steel** - The sample comes from a cast bar. - The exterior surface is irregular. - The fracture has a silver-white colour. - The grains are large and globular in shape.

6		**Hot Rolled Steel** The sample comes from a round profile that has been hot rolled and notched for easier fracture. - The exterior surface is smooth. - The fracture has a silver-white colour and exhibits fine, shiny globular grains. - Within the crack, you can also observe the directions of plastic deformation of the material, which was fractured by bending, arranged radially in the form of clusters of metallic material.
7		**Steel with Defect** - The sample exhibits cracks (fracture surfaces). - The cracks can result either from vigorous quenching (gray fracture with very fine, imperceptible to the naked eye, velvety grains) or from prolonged overheating (burning - intergranular oxidation), followed by plastic deformation.
8		**Sample from a Zinc Block** Silver-white, shiny fracture with coarse grains.

9		**Sample with Mechanical Wear** The sample comes from a sandblasting nozzle used in a cleaning installation for cast parts. - Inside the piece, there is an irregularity in its circular shape due to wear.
10		**Sample with Mechanical Wear** The sample comes from a sliding bearing, specifically a half-bearing. - Exfoliation of the anti-friction alloy is observed.

3.1.4. Macroscopic analysis on prepared surfaces

The purpose of this analysis is to determine the chemical and structural inhomogeneities of metallic materials.

3.1.4.1. Preparation of metallographic samples

The sample should be taken from a region of the material that allows for the maximum acquisition of information about the material, its manufacturing processes, and any treatments it has undergone. This operation is carried out through breaking, cutting (manual or mechanical sawing, etc.), or fracturing (if the material is brittle).

Analysis on metallic materials under a microscope is conducted on small samples, which are prepared in advance to differentiate the metallographic constituents.

Metallic materials are solid, crystalline bodies that are opaque, which necessitate examination under an optical microscope using the principle of light reflection. To enable light rays to be differentially reflected by various metallographic constituents, the study surface of the sample must be prepared. Preparing samples for examination with an optical metallographic microscope involves the following operations: sample extraction, preparation of the working surface, and chemical etching.

Metallographic samples are relatively small pieces with surfaces ranging from (0.5...30) x (30...0.5) mm^2, specially prepared from the metallic materials to be studied under an optical microscope.

Preparation of Samples – Operations

In the laboratory, samples will be taken from various materials using a very thin abrasive disc. A sample surface will be prepared through levelling, grinding, polishing, chemical etching, and finally, it will be examined under the naked eye or a metallographic microscope with the structure being drawn.

For macroscopic analysis, a series of operations must be carried out: selecting the sampling location and taking the sample, levelling, grinding, polishing, and, if necessary, chemical etching.

1. *Sample Collection:* To obtain the most accurate information about the material under investigation, the sample collected must be representative and suitable for the intended purpose. The choice of the sampling location should be made so that the sample includes the characteristic structure of the metallic material, as well as any effects of prior mechanical, thermal, thermochemical processing, existing defects, etc. Metallographic samples should not be too large, depending on the semi-finished products or parts being studied, the samples may have different dimensions. Samples taken from thin sheets, wires, small parts like medical alloys, or samples that need to be studied at the edge should be fixed in a holder or embedded in synthetic resin or easily fusible material. Sample extraction from metallic materials can be done by manual or mechanical cutting using a saw or abrasive disc. Machines such as milling machines, lathes, or other equipment can also be used, ensuring that the sample's initial structure is not altered due to heating or deformation. Therefore, flame oxyacetylene cutting, electric cutting, which produce excessive heating, or shearing and chiselling that cause deformation should be avoided. When no other solution exists, the altered part of the material due to the extraction process should be removed by milling, turning, grinding, etc. Hard materials should be cut with thin abrasive discs with abundant cooling, while for brittle materials, samples should be fractured.
2. *Gliding:* machined metallographic samples are subjected to levelling of the study surface, which is performed using a grinder, lathe, or milling machine, ensuring that the samples do not overheat, especially those that have been quenched or heat-treated.
3. *Grinding* is carried out on metallographic paper, which has abrasive grains of various sizes fixed on one side in decreasing order. This can be done manually through a back-and-forth linear motion (with the paper fixed on a glass plate) or automatically on grinding machines equipped with one or two rotating discs at a speed of 10-15 m/s, to which the metallographic paper is attached. If the sample surface has been finished by a machining operation, grinding should start with medium grit abrasive (25 hundredths of a mm) or No. 60 (the linear mesh size of the screen through which the abrasive was sieved), followed by fine grit (16, 12, 10),

very fine grit (8, 6, 4), and finally, papers containing extra-fine micro-powders (M40, M28...M7). Transition from one paper with a certain grit to another with a finer grit should occur when the scratches from the previous grinding have disappeared, ensuring that the sample is rotated 90 degrees from its previous position. Samples with normal dimensions can be held directly by hand, while smaller ones can be mounted or embedded in synthetic resins or softer materials. During grinding, slight pressure should be applied to the abrasive paper to avoid overheating above 50-60°C and oxidation of the surface.

4. *Polishing* of the sample's study surface is done to remove the final scratches from grinding and obtain a smooth surface with a perfect shine. This is done on a polishing machine, where a rotating disc with a higher speed than in grinding (15-20 m/s) is used, and a woollen (felt) cloth moistened continuously with a 1:20 abrasive suspension solution (aluminium oxide, magnesium oxide, etc.) is attached. For polishing, the sample is held by hand and gently pressed onto the cloth until all scratches disappear (max. 5 min.). During polishing, the sample is slowly rotated to avoid tearing of inclusions and the formation of new scratches. Extending the polishing time causes the structure to become raised due to the wear of the soft structural constituents. After polishing, the sample is rinsed with a jet of water to remove any traces of abrasive, then with alcohol, and sometimes a neutralization of acid traces is performed using a basic solution (sodium carbonate). The sample is dried with a stream of warm air or by gentle blotting with filter paper. The sample, thus prepared, can be subjected to microscopic examination to determine non-metallic inclusions (slag, oxides, sulphides, graphite, etc.), any defects (microcracks, micro reticulations, etc.), or it can be chemically etched to differentiate the structural constituents.
5. *Chemical Etching:* Chemical reagents do not react uniformly with all structural constituents of the metallic material subjected to etching. Some constituents (especially heterogeneous ones) are corroded (dissolved) or coloured, while others remain shiny. If the polished surface contains the same type of metallographic constituents, the chemical reagent will corrode only the boundaries between the grains of the same kind because at the grain boundaries, there are heterogeneous zones (with impurities, inclusions, amorphous layers, etc.) that are not resistant to the chemical action of the reagents. The chemicals commonly used are acids or solutions of acids in water or alcohol (see table), and etching can be performed at either room temperature or with heating. After chemical etching, metallographic samples are rinsed with a water jet, then with alcohol (to remove any acid or grease residues from the study surface). Sometimes, neutralization of acid traces with a basic solution (sodium carbonate) is applied. The sample is dried with a stream of warm air or by gentle blotting with filter paper. Under

the microscope, the boundaries between corroded grains and those coloured toward darker shades appear dark because the threshold rays are absorbed by the black surfaces. The light rays can also be deviated elsewhere (according to the laws of reflection) if compared to the optical system of the microscope, as seen in Figure 5 (with vertical illumination - bright field). Unetched constituents (shiny) (a) reflect light rays into the microscope eyepiece and appear white in colour. Corroded constituents (b) or boundaries between grains can have a shiny surface, in which case the rays are reflected laterally (the angle of incidence equals the angle of reflection), or they can have a matte surface when rays are absorbed totally or partially. In both cases, the constituents appear dark under the microscope. Coloured constituents (c), absorbing light rays, are observed under the microscope as dark with varying degrees of darkness.

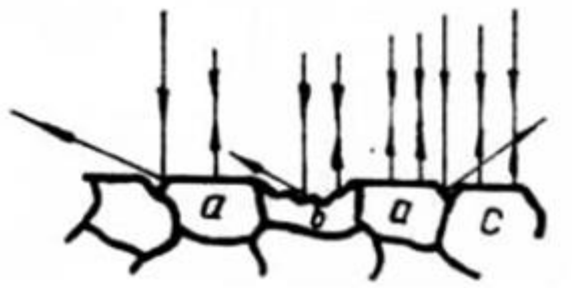


Figure 3.1.5. Highlighting constituents under the optical microscope. Source: https://sim.tuiasi.ro/wp-content/uploads/2015/03/MF1-Laboratoare.pdf

The etching is performed by immersing the sample in the reagent or by wetting the surface with a cotton swab soaked in the chemical reagent until the shiny surface of the sample becomes slightly matte. The action of the reagent is interrupted by rinsing the sample with a jet of water and then with alcohol. The samples are dried using a stream of hot air or by gently pressing a filter paper on the washed surface. Etching can be performed at room temperature or at elevated temperatures (up to 400°C).

Metallographic reagents are selected based on the nature of the metallic material under investigation and the specific aspects being studied:

- For ordinary steels and cast irons, Nital is used (2-5% HNO_3 in ethyl alcohol). For stainless alloyed steels, aqua regia is used (20-30 ml HCl, 10 ml HNO_3, 30-70 ml glycerol).
- Noble, semi-noble, and non-noble medical alloys based on Cr-Ni or Cr-Mo use Aqua Regia reagent (20-30 ml HCl + 10 ml HNO_3 + 30-70 ml glycerol). Another reagent for these types of materials is composed of 3 parts HCl and 1-part HNO_3, or one can use Groesbeck reagent (4 g potassium permanganate + 4 g NaOH + 100 ml water). These reagents can also be used for stainless and refractory steels.

- Copper-based medical alloys use reagents like 50 ml ammonium bi-oxide (NH_4BiO3) + 20-50 ml (H_2O_2) + 50 ml water (H_2O), or 30-50 ml (HNO_3) + 70-50 ml water (H_2O), or 5 g ammonium persulfate ($(NH_4)2S_2O_8$) + 90 ml water (H_2O).
- To highlight the platinum structure, 100 ml HCl + 10 ml HNO_3 + 50 ml water (H_2O) is used. For silver structures, a solution of 50 ml potassium cyanide (KCN) with a concentration of 5% + 50 ml ammonium persulfate ($(NH_4)2S_2O_8$) 5% concentration is used. For the titanium structures, 25 ml HNO_3 + 25 ml HF + 50 ml glycerol ($C_3H_8O_3$) is used.

Macroscopic analysis

As a result of plastic deformation of the metallic material, the grain structure is rearranged according to the direction of the deformation (grain flow). In the process of cold plastic deformation ($T_{deformation} < T_{recrystallization}$) with high degrees of deformation (drawing, rolling, etc.), the grains of the metallic material elongate in the direction of maximum deformation. The obtained longitudinal section (treated with a chemical reagent) is characterized by elongated grains obtained by cold plastic deformation (Figure 6).

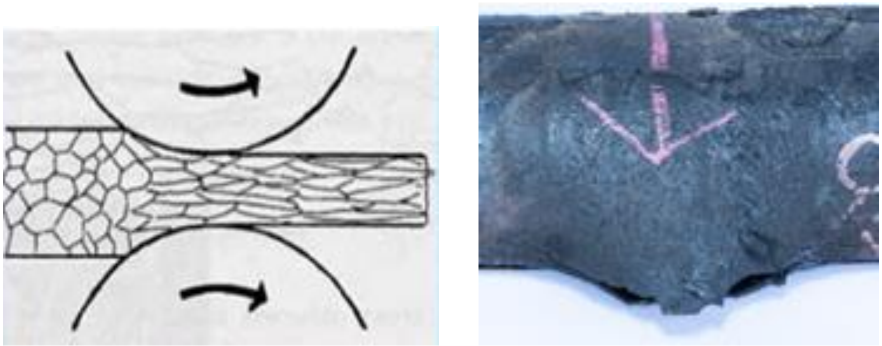


Figure 3.1.6. The grain structure obtained as a result of cold plastic deformation. Source: https://learnmech.com/hot-rolling-vs-cold-rolling-difference-between-hot-rolling-and-cold-rolling/

Following hot plastic deformation ($T_{deformation} > T_{recrystallization}$) the grain flow that appears in longitudinal sections is no longer characterized by the elongated shape of the grains. This is because recrystallization occurs due to the temperature of the plastic deformation exceeding the recrystallization temperature, causing the grains to become equiaxed. In this case, the grain flow is highlighted by the distribution of inclusions in layers. It can be observed in Figure 7 that the grain flow follows the contour of the piece. In the absence of non-metallic inclusions, hot-deformed metallic material does not exhibit a fibrous appearance.

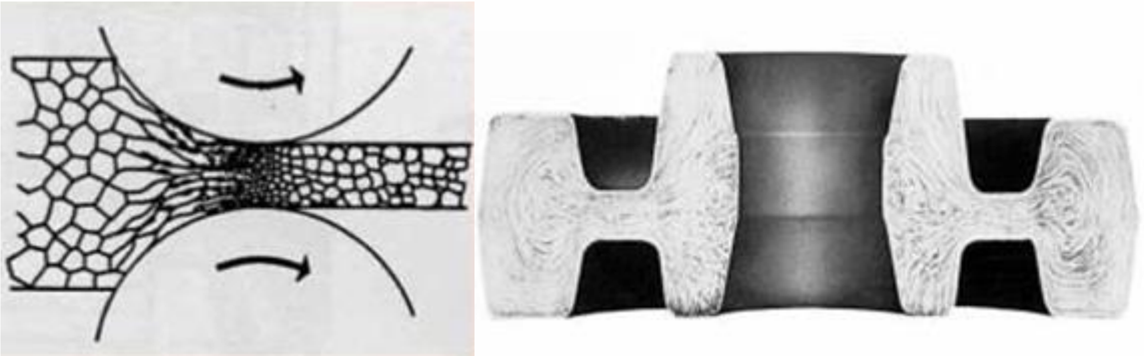


Figure 3.1.7. The grain structure obtained as a result of hot plastic deformation. Source: https://learnmech.com/hot-rolling-vs-cold-rolling-difference-between-hot-rolling-and-cold-rolling/

Aspects of welded joints

Various aspects of welded joints can also be highlighted, such as the shape of the weld bead (in X, V, U, etc. shapes), different weld layers, the heat-affected zone (HAZ) - which appears darker in colour, and the weld metal zone (WMZ). Additionally, the cross-sectional examination of welded joints can reveal characteristic zones (Figure 8) after grinding and chemical etching: the base metal zone (BMZ), the heat-affected zone (HAZ) - which is darker in colour, and the weld metal zone (WMZ). Other features that can be observed include the shape of the weld cross-section (V-shape, X-shape, U-shape, etc.), distinct weld layers, the zone of recrystallization (columnar crystals), and defects (pores, segregations, lack of fusion, cracks, etc.).

The base metal zone is located far enough from the welding operation to prevent any significant physical-chemical changes.

The heat-affected zone represents an area where the thermal effects during a welding process have induced structural and property changes in the base metal.

The weld bead is the molten and solidified area where the maximum thermal effect has temporarily allowed the base material's edges to melt, possibly with the participation of filler material or only the filler material itself.

The different zones in the cross-section of the weld bead are highlighted through proper preparation and chemical etching (for example, Adler reagent is used for steels).

Therefore, macroscopic examination of the cross-sectional area of welded joints reveals the following zones:

- base metal zone (BMZ)
- heat-affected zone (HAZ)
- weld metal zone (WMZ)

The base metal zone, being sufficiently distant, does not undergo any significant physical-chemical changes due to the welding operation. However, the heat-affected zone represents an area where structural and property changes have occurred in the base metal due to the elevated temperature during the welding process. The weld bead (weld seam) is the molten and solidified area where the maximum thermal effect has led to the temporary melting of the edges of the pieces to be welded, either with or without the participation of filler material. The profile and dimensions of these zones may vary for the same metallic material, depending on the welding type and any heat treatments applied before, after, or during welding.

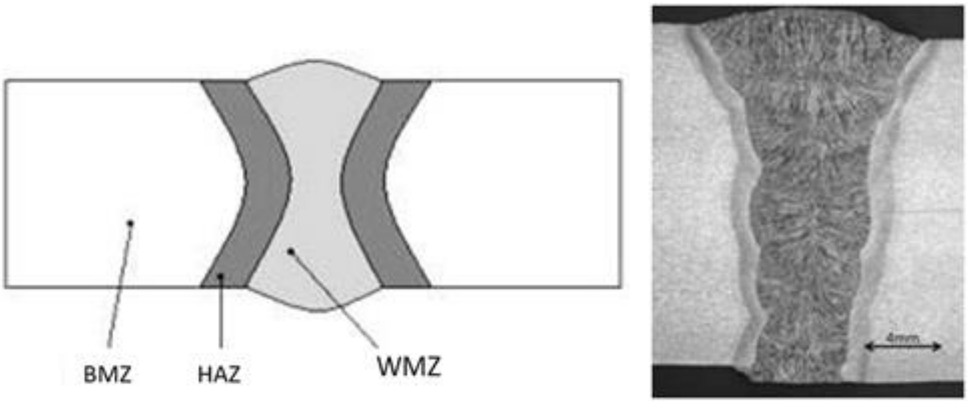


Figure 3.1.8. Zones of the weld joint in classic electric arc welding with filler material. Source: https://sim.tuiasi.ro/wp-content/uploads/2021/05/Laborator_SM_2020.pdf

Chemical inhomogeneity resulting from casting, plastic deformation, or thermochemical treatments can also be highlighted through macroscopic examination. For example, the dendritic structure can be emphasized through appropriate chemical etching (Oberhoffer reagent for steels and Keller reagent for aluminium alloys). Figure 9 shows the primary dendritic structure of an Al-Si alloy (AlSi12), where chemical inhomogeneity resulting from casting is evident.

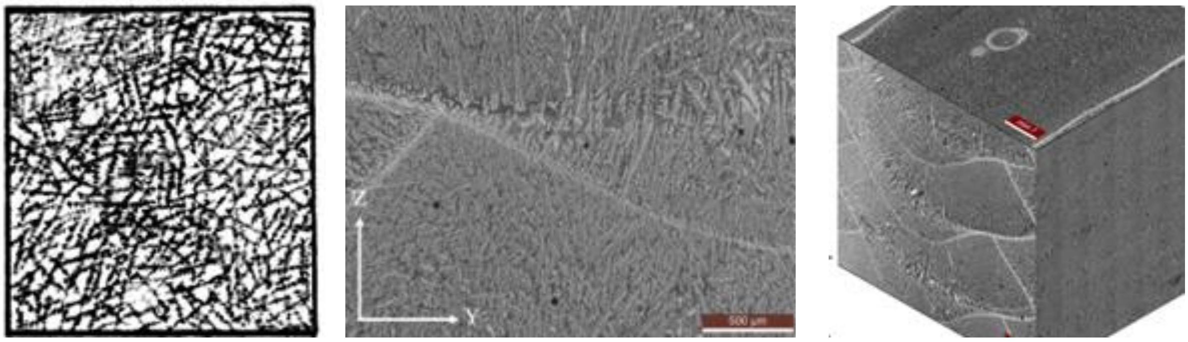


Figure 3.1.9. Primary dendritic structure of an Al-Si alloy. Source: https://sim.tuiasi.ro/wp-content/uploads/2015/03/MF1-Laboratoare.pdf

Additionally, through chemical etching of a cross-sectional surface of a thermally treated piece by carburizing, the outer layer enriched in carbon (having a darker colour) can be observed, allowing the macroscopic assessment of the depth of carbon diffusion into the surface layer of the piece.

During the macroscopic examination of prepared and chemically etched surfaces of metallic materials, certain defects can be detected, such as slag inclusions, material overlays, pores, blowholes, cracks, etc.

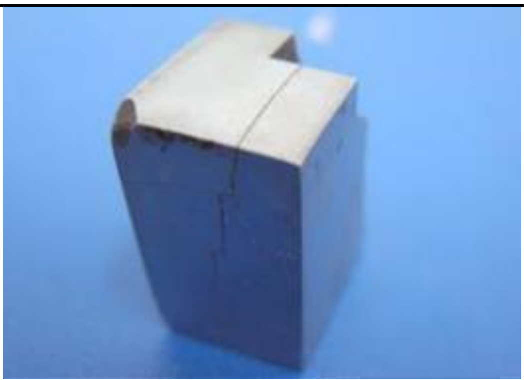
Equipment, materials, and working procedure

Macroscopic analysis involves the visual examination of fractured or prepared surfaces, or chemically etched surfaces of samples made from various metallic materials (cast irons, steels, non-ferrous alloys). It is recommended to use magnifying glasses (Figure 10) to highlight certain macro-structural aspects that are challenging to observe with the naked eye. Metallographic samples will be analysed with an emphasis on their shape, the colour and type of the fracture, structural inhomogeneities, defects resulting from processing or material stresses and so on, or on the welded section.

Figure 3.1.10. Magnifying glasses for macroscopic examination. Source: https://sim.tuiasi.ro/wp-content/uploads/2021/05/Laborator_SM_2020.pdf

Additionally, fracture surfaces of samples made from various metallic materials will be analysed to determine the type of fracture and the material's behaviour under plastic deformation. Based on the conducted analysis, conclusions will be drawn regarding whether the examined material exhibits ductile or brittle behaviour and under what conditions it can be practically used.

3.1.5. Macroscopic examinations of metallic materials on prepared surfaces

No.	*Sample studied*	*Observations*
1		**Casted aluminium alloys** The sample is taken from a small alloy ingot: - On the prepared and chemically etched surface, dendrites are observed. - At the top of the sample, there is a shrinkage cavity and a crack caused by the material's contraction during solidification. - In the centre of the sample, an internal void (blowhole) is visible.
2		**Cracked steel sample** Following the heat treatment of quenching, the sample has cracked due to high internal stresses induced by the presence of mechanical stress concentrators (e.g., an unconnected keyway groove) or impurities arranged in rows.
3		**Sample from a welded component under load** In a cross-sectional view, chemically etched, the three characteristic zones of the weld can be observed: - Base material zone (BMZ). - Heat-affected zone (darker in colour - HAZ). - Weld metal zone (WMZ).

4		**Sample from a V-welded steel** In a cross-sectional view, chemically etched, the solidified material zone and the base material can be observed. - The weld was created by root pass completion.
5		**Cold deformation of sample** The grain structure following the contour of the piece can be observed.

3.1.6. Questions

1. What is the purpose of the Demonstration of assessment of macroscopic specimens of joints in this context?
2. What are the steps involved in the Preparation of metallographic samples?
3. What are the stages of preparing the surface for examination?
4. How does Macroscopic analysis on unprepared surfaces differ from Macroscopic analysis on prepared surfaces?
5. Why is Macroscopic analysis of metallic materials on unprepared surfaces important, and what structural features can be examined through this method?
6. Which are the areas of the cross-section of welded joints that can be highlighted?
7. Are there specific techniques or tools discussed for macroscopic analysis?
8. What are the potential applications or industries where this macroscopic analysis of metallic materials is relevant?

3.2. Practical training in unaided identification of welding imperfections on macroscopic specimens

3.2.1. Introduction to Welding Imperfections

Welding imperfections are deviations or irregularities in a weld that can affect its integrity, strength, and overall quality. These imperfections can arise from various factors, including welding process parameters, material properties, operator skills, and environmental conditions. Identifying and addressing these imperfections is crucial to ensure the reliability and safety of welded structures.

3.2.1.1. Overview of Common Welding Imperfections

- **Porosity** refers to the presence of small cavities or voids within the weld metal caused by trapped gases, such as hydrogen or nitrogen. These voids can weaken the weld and make it more susceptible to cracking.

Fig. 3.2.1. Gas cavities. Source: M.S. Rogers, Welding Inspection Defects/Repairs Course Reference WIS 5, 2003.

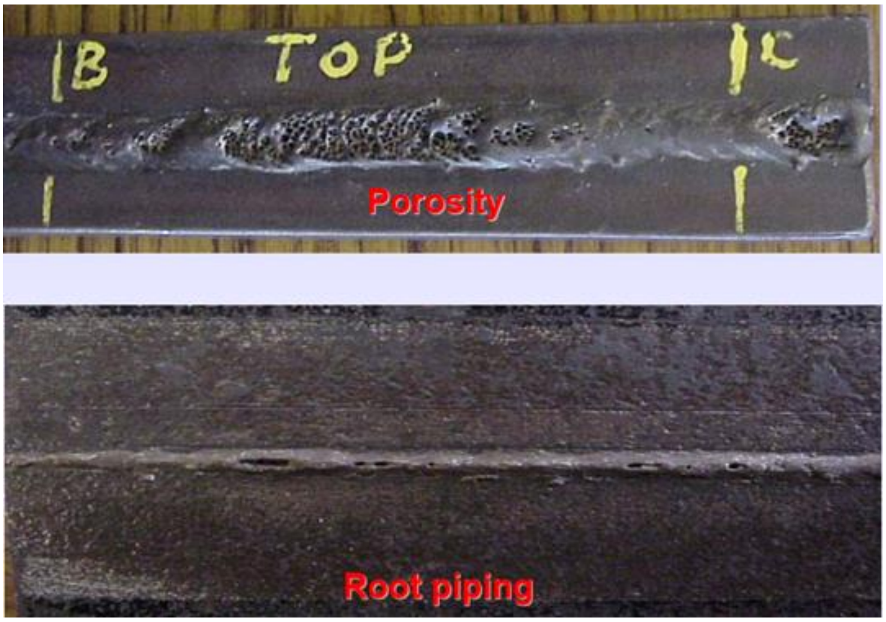


Fig. 3.2.2. Visual characteristics of gas cavities. Source: M.S. Rogers, Welding Inspection Defects/Repairs Course Reference WIS 5, 2003.

- **Lack of Fusion** occurs when there is inadequate bonding between the weld metal and the base metal or between different weld passes. It can result in a weak joint that may not be able to withstand applied forces.

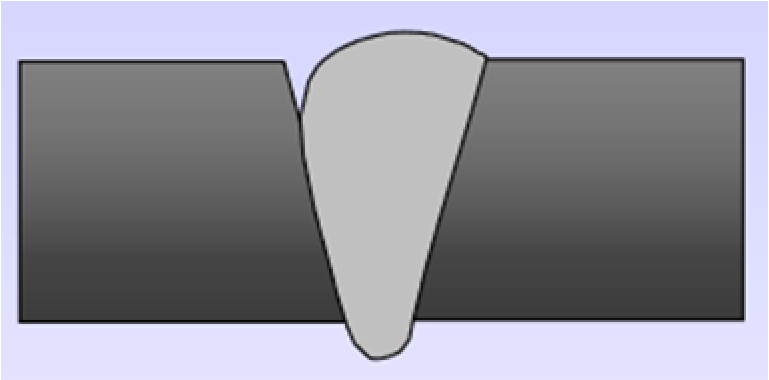


Fig. 3.2.3. Incomplete filled groove. Source: M.S. Rogers, Welding Inspection Defects/Repairs Course Reference WIS 5, 2003

- **Incomplete Penetration** happens when the weld metal does not fully penetrate the joint or extend through the entire thickness of the base metal. This can lead to reduced strength and integrity.

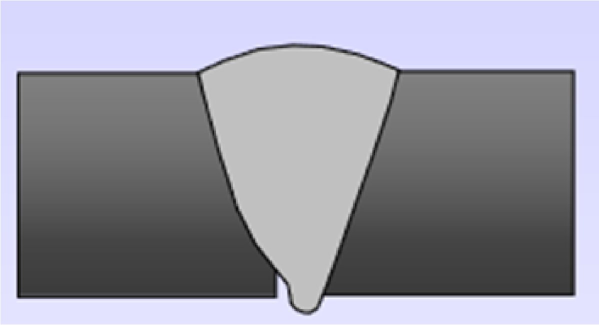


Fig. 3.2.4. Incomplete root fusion. Source: M.S. Rogers, Welding Inspection Defects/Repairs Course Reference WIS 5, 2003

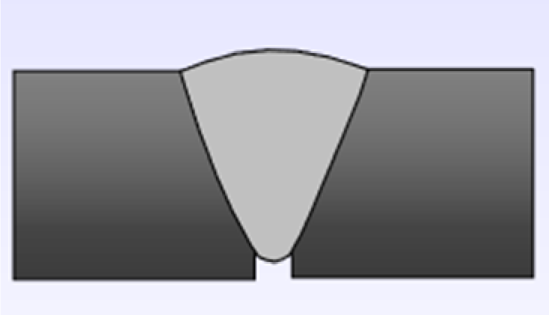


Fig. 3.2.5. Incomplete root penetration. Source: M.S. Rogers, Welding Inspection Defects/Repairs Course Reference WIS 5, 2003

- **Undercutting** is the formation of a groove along the base metal near the weld due to excessive heat or improper welding technique. It can create stress concentrations and weaken the joint.

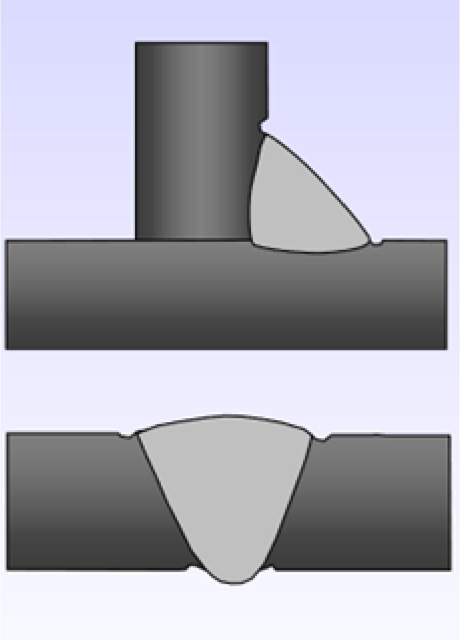


Fig. 3.2.6. Root and cap under cut. Source: M.S. Rogers, Welding Inspection Defects/Repairs Course Reference WIS 5, 2003

- **Overlap** occurs when the weld metal extends onto the surface of the base metal without proper fusion. This can result in a lack of strength and increased susceptibility to cracking.

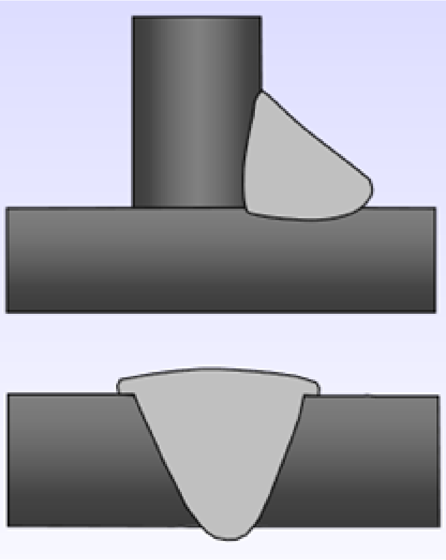


Fig. 3.2.7. Toe overlap in Butt/Fillet weld. Source: M.S. Rogers, Welding Inspection Defects/Repairs Course Reference WIS 5, 2003

- **Cracks** can form in various orientations and locations within the weld. Common types include longitudinal cracks along the weld axis, transverse cracks across the weld, and cracks in the heat-affected zone. Cracks can compromise the structural integrity of the weld.

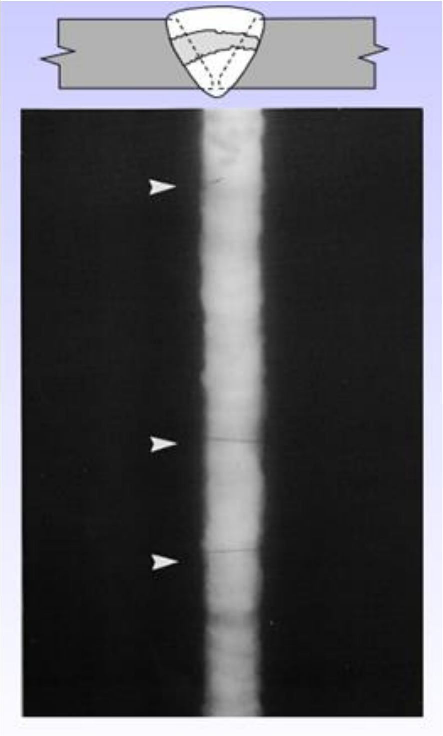


Fig. 3.2.8. Transverse crack

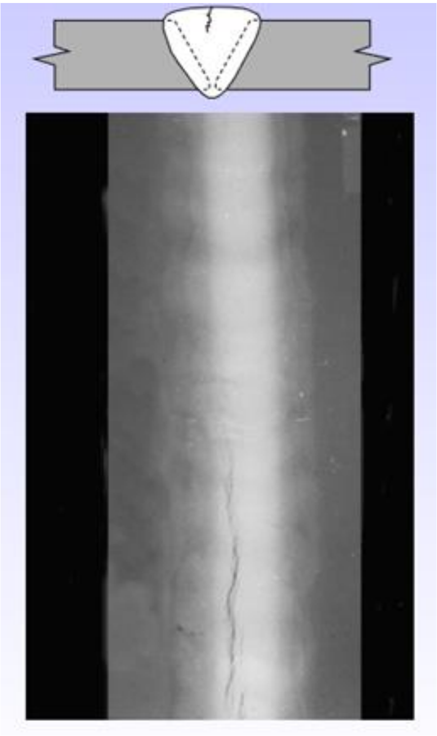


Fig. 3.2.9. Longitudinal crack

Source: M.S. Rogers, Welding Inspection Defects/Repairs Course Reference WIS 5, 2003

- **Weld Spatter** refers to the small droplets of molten metal expelled from the welding arc that can adhere to the surface of the weld. While not always structurally detrimental, excessive spatter can affect appearance and potentially cause weak points.

Fig. 3.2.10. Visual characteristics of spatter. Source: M.S. Rogers, Welding Inspection Defects/Repairs Course Reference WIS 5, 2003.

- **Inclusions** are foreign materials, such as slag, oxide, tungsten, or flux, that become trapped in the weld. They can cause areas of weakness and reduced corrosion resistance.

Fig. 3.2.11. Inter-pass slag inclusions

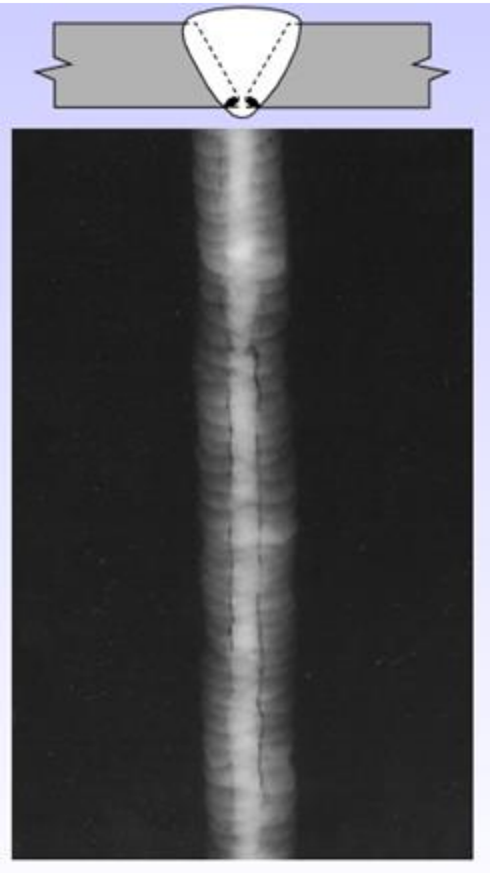


Fig. 3.2.12. slag lines

Source: M.S. Rogers, Welding Inspection Defects/Repairs Course Reference WIS 5, 2003

- **Misalignment** occurs when the weld joint is not properly aligned before welding, leading to uneven and weak welds.

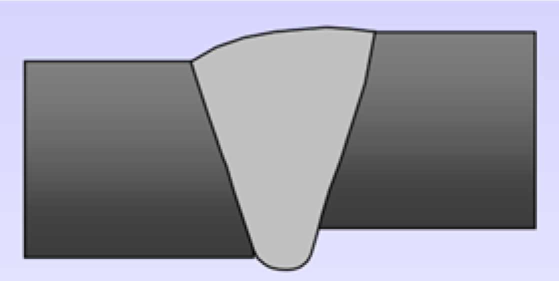


Fig. 3.2.13. Linear Misalignment. Source: M.S. Rogers, Welding Inspection Defects/Repairs Course Reference WIS 5, 2003

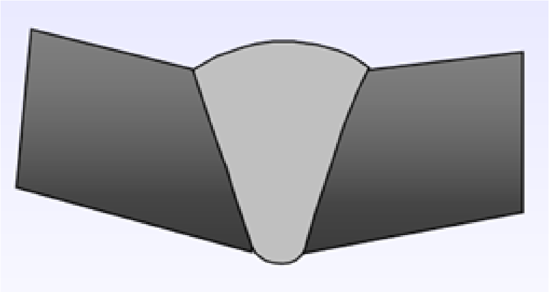


Fig. 3.2.14. Angular Misalignment. Source: M.S. Rogers, Welding Inspection Defects/Repairs Course Reference WIS 5, 2003

- **Excessive Reinforcement** happens when the weld bead extends beyond the required dimensions. This can lead to stress concentrations and may not meet design specifications.

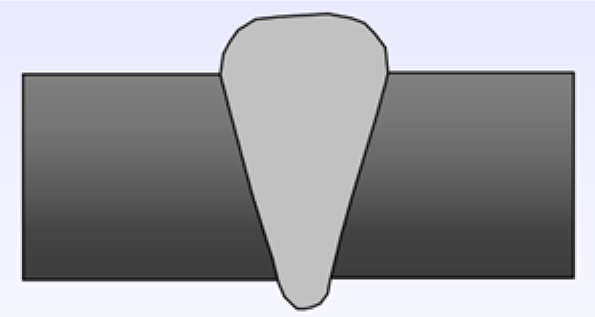


Fig. 3.2.15. Excessive cap height. Source: M.S. Rogers, Welding Inspection Defects/Repairs Course Reference WIS 5, 2003

- **Burn-Through** occurs when the weld heat is excessive, causing melting through the base metal and potentially creating holes.

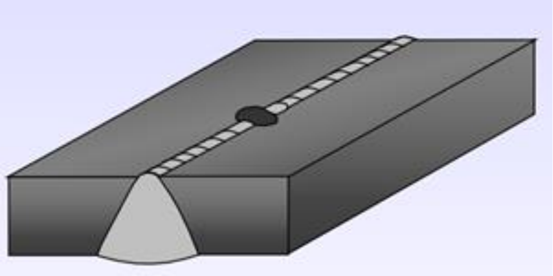


Fig. 3.2.16. Burn-through. Source: M.S. Rogers, Welding Inspection Defects/Repairs Course Reference WIS 5, 2003

- **Distortion** refers to the warping or deformation of the welded structure due to the heat input during welding. While not necessarily a defect, excessive distortion can affect fit and function.

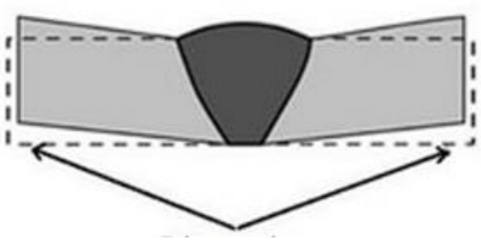


Fig. 3.2.17. Distortion. Source: M.S. Rogers, Welding Inspection Defects/Repairs Course Reference WIS 5, 2003

Welding imperfections can be correlated to the type of welding process used due to the specific characteristics and parameters associated with each welding method. Different welding processes have distinct heat sources, deposition rates and techniques, which can influence the types of imperfections that are more likely to occur.

Shielded Metal Arc Welding can be more prone to slag inclusions, especially if proper cleaning and slag removal are not performed between passes. Porosity and lack of fusion can also occur due to the manual nature of the process, which can lead to inconsistent coverage and inadequate heat control.

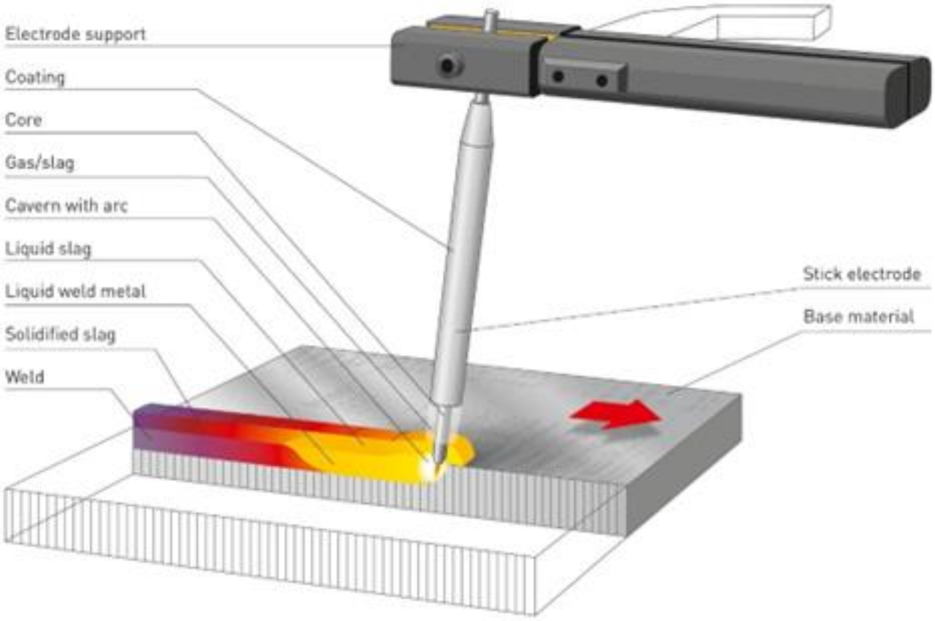


Fig. 3.2.18. Shielded Metal Arc Welding. Source: https://www.kjellberg.de/gas-shielded-metal-arc-welding.html

Gas-shielded metal arc welding is generally less prone to slag-related imperfections, but it can have issues with porosity and incomplete fusion if the shielding gas is not adequately controlled. Weld spatter might also be present due to the use of consumable electrode wire.

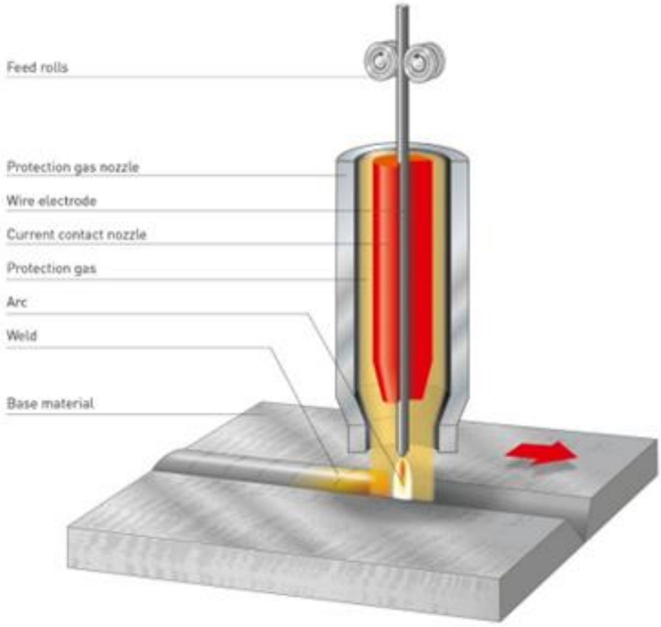


Fig. 3.2.19. Gas-shielded metal arc welding. Source: https://www.kjellberg.de/manual-metal-arc-welding.html

Gas Tungsten Arc Welding is known for producing high-quality welds, but lack of fusion and incomplete penetration can occur if the heat input is not controlled properly. Tungsten inclusions can also be a concern if the tungsten electrode contacts the weld pool.

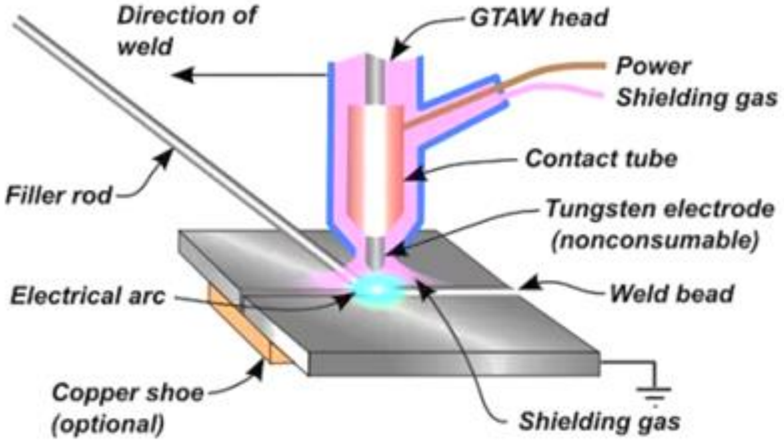


Fig. 3.2.20. Gas Tungsten Arc Welding. Source: https://www.weldingis.com/gtaw-welding/

Flux-Cored Arc Welding can experience slag inclusions and porosity if the flux core is not properly compatible with the base metal or if there's insufficient shielding gas. Similar to GMAW, spatter might be an issue due to the use of consumable filler wire.

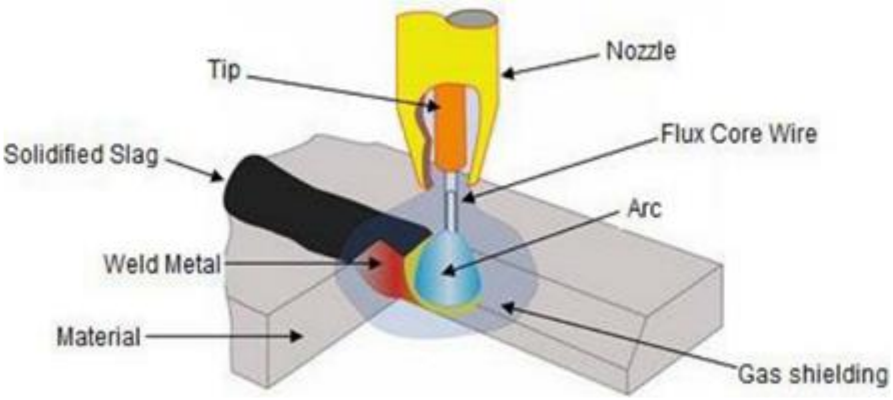


Fig. 3.2.21. Flux-Cored Arc Welding. Source: Celso, Alves Correa; Niederauer, Mastelari (2014). Effect of welding parameters in flux core arc welding (FCAW) with conventional and pulsed current in the efficiency and fusion rate of melting coating. Scientific Research and Essays.

Submerged Arc Welding is generally associated with deep, high-quality welds, but incomplete fusion and lack of sidewall fusion can occur if the joint preparation and alignment are not well-controlled.

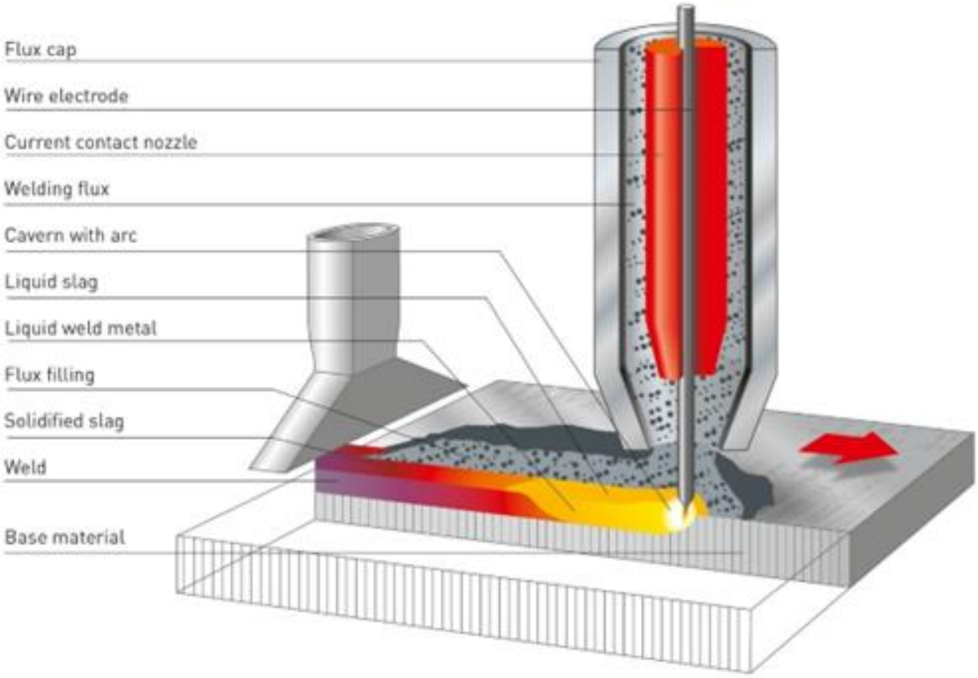


Fig. 3.2.22. Submerged Arc Welding. Source: https://www.kjellberg.de/submerged-arc-welding-2.html

Plasma Arc Welding reduces precise and controlled welds, but inadequate penetration and lack of fusion can arise if the parameters aren't appropriately set. Tungsten inclusions and improper keyhole control can also lead to defects.

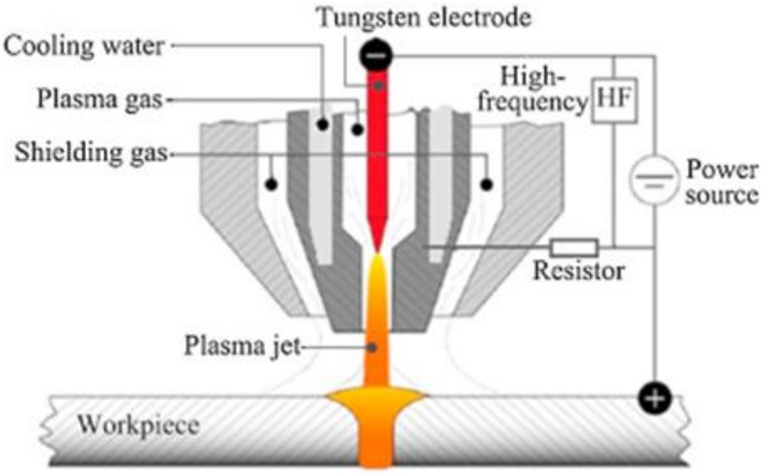


Fig. 3.2.23. Plasma Arc Welding. Source: Liu, Zu Ming; Cui, Shuang Lin; Luo, Zhen; Zhang, Chang Zhen; Wang, Zheng Ming; Zhang, Yu Chen (2016). Plasma arc welding: Process variants and its recent developments of sensing, controlling, and modelling. Journal of Manufacturing Processes.

Electron Beam Welding is known for high-energy density process, usually result in minimal imperfections. However, inadequate joint fit-up, keyhole instability, and beam focusing issues can lead to weld defects like lack of fusion and porosity.

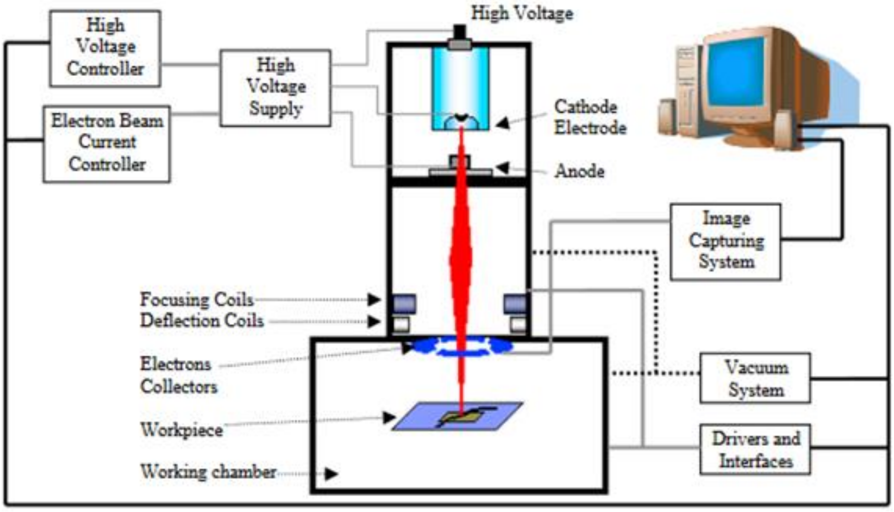


Fig. 3.2.24. Electron Beam Welding. Source: Oltean, Stelian-Emilian (2018). Strategies for monitoring and control with seam tracking in electron beam welding. Procedia Manufacturing, 22(), 605–612.

3.2.2. Visual Inspection Techniques for Welding Imperfection Identification

Visual inspection is a crucial method for identifying welding imperfections. It involves a detailed visual examination of the weld and its surrounding area to detect irregularities, defects, and imperfections.

Preparation and cleaning: before beginning the visual inspection, ensure that the weld area is clean and free of any debris, paint, or contaminants that could obscure imperfections. Cleaning the surface with a wire brush or appropriate solvent may be necessary.

Proper lighting: adequate lighting is essential for accurate visual inspection. Use bright, diffused lighting to minimize shadows and reflections that can hide imperfections.

Angle of observation: examine the weld from multiple angles, including perpendicular and oblique views. Changing the angle of observation can reveal imperfections that may not be visible from a single perspective.

Reference standards: have reference standards or samples of acceptable and unacceptable welds available for comparison. These standards can help determine if the observed imperfections exceed acceptable limits.

Visual aid tools: depending on the size and detail of the weld, use visual aid tools like magnifying glasses, binoculars, gauges, or borescopes to enhance your ability to spot fine imperfections.

Systematic inspection: follow a systematic inspection procedure, examining the weld joint in a structured sequence. Start from one end and work your way to the other, ensuring no area is overlooked.

Distinguishing acceptable and unacceptable imperfections: develop the ability to differentiate between acceptable imperfections, like surface irregularities, minor ripples or spatter, and actual defects, like cracks, undercut or lack of fusion, function to the information provided in the appropriate standard.

Knowledge of welding processes: familiarize yourself with the specific welding process used, as each has unique characteristics and potential imperfections or defects associated with it. Understanding the process can aid in identifying defects.

Code and standard compliance: be aware of relevant industry codes and standards that specify acceptable tolerances for welding imperfections. Compliance with these standards is mandatory.

Training and experience: experience plays a significant role in becoming proficient in visual inspection. Continuous training, practice, and exposure to various types of welds and imperfections enhance your skills.

Non-Destructive Testing (NDT): understand when to complement visual inspection with more advanced NDT techniques, such as radiographic testing, ultrasonic testing, magnetic particle testing, or dye penetrant testing, for a more comprehensive assessment, especially in critical applications.

3.2.2.1. Inspection Procedures and Best Practices

Effective inspection procedures and best practices are crucial to ensuring accurate and thorough assessments of welds and other materials. Proper inspection procedures help maintain quality, safety, and compliance with industry standards.

Safety:

- Prioritize safety by wearing appropriate personal protective equipment (PPE) such as safety glasses, gloves, helmets, and clothing.

- Be aware of any potential hazards in the inspection environment, especially when inspecting in industrial settings.

Plan and Prepare:

- Develop a clear inspection plan that outlines the scope, objectives, and acceptance criteria for the inspection.
- Gather all necessary inspection equipment, tools, reference standards, and documentation before beginning the inspection.

Visual Inspection Sequence:

- Follow a systematic sequence ensuring that all areas of the weld are thoroughly examined.
- Start from one end and work your way to the other to avoid missing any sections.

Cleaning and Preparation: Ensure that the surfaces to be inspected are clean, free from contaminants, and properly prepared. Remove dirt, rust, paint, or other debris that could obscure imperfections.

Appropriate Lighting:

- Use suitable lighting conditions that provide an adequate illumination of the inspection area.
- Angle the light to minimize shadows and reflections.

Inspection Aids: Utilize appropriate inspection aids such as magnifying tools, borescopes, and mirrors to enhance visibility and access to hard-to-reach areas.

Reporting and Record Keeping:

- Generate detailed inspection reports that include inspection results, photographs, drawings, and any recommendations for repairs or corrective actions.
- Maintain a comprehensive record of all inspections for future reference.

3.2.3. Macroscopic Examination of Welded Joints

Macroscopic inspection of welding joints is a destructive testing technique used to visually assess the overall quality, integrity, and characteristics of a welded joint. This inspection method involves a detailed visual examination of the weld and its immediate surroundings using the naked eye or low-magnification aids. The primary purpose of macroscopic inspection is to identify and evaluate macroscopic defects, irregularities, or discontinuities in the weld, ensuring that it meets the specified standards and requirements.

Preparation and Evaluation of Macroscopic Specimens

Preparing test specimens for macroscopic inspection involves carefully preparing and sectioning representative samples of the welded joints or components that need to be examined. The goal is to create cross-sections that allow for a clear and detailed visual assessment of the weld and any potential defects. The steps to prepare test specimens for macroscopic inspection are as follows:

Select Representative Samples: Choose test specimens that accurately represent the type of welded joints or components you want to inspect. The specimens should contain the welds or areas of interest.

Cutting:

- Use appropriate cutting equipment, such as a bandsaw, hacksaw, or abrasive cutoff wheel, to make clean and precise cuts through the test specimens. The goal is to create cross-sections of the welds or components.
- Ensure that the cutting process does not induce additional damage or defects to the specimens.

Orientation:

- Mark the specimens to indicate the orientation of the weld joint or area of interest. This helps maintain consistency during inspection and analysis.
- If possible, cut the specimens to reveal both the weld bead and the adjacent base material and heat-affected zone (HAZ).

Mounting: Depending on the size and shape of the test specimens, you may need to mount them in a suitable material to facilitate handling, grinding, and polishing. Common mounting materials include epoxy resin or acrylic.

Grinding: Use a series of abrasive grinding papers or wheels with progressively finer grits to remove excess material and create a flat, smooth surface on the test specimens. This step is critical for achieving a polished surface that allows for clear inspection.

Polishing: Use a fine grit polishing compounds or diamond pastes to further enhance the surface quality. Continue polishing until the surface achieves a mirror-like finish.

Etching (Optional): In some cases, applying an appropriate etchant to the specimen's surface can reveal microstructural details and highlight features like weld boundaries, grain structure, and the presence of defects. The choice of etchant depends on the material being inspected.

In macroscopic inspections, etchants are chemicals used to reveal and highlight microstructural details in metallic materials. These etchants are applied to the surface of prepared test specimens to differentiate between various structural constituents, grain boundaries, phases, and potential defects. The choice of etchant depends on the material being inspected and the specific microstructural features of interest.

Video 3.2.1: Macroscopic Techniques Part 1. Source: Prof. Dr. Ing. Rainer Schwab, Hochschule Karlsruhe, University of Applied Sciences, Karlsruhe, Germany [https://www.youtube.com/watch?v=fc8zrgYJCJw]

Video 3.2.2: Macroscopic Techniques Part 2. Source: Prof. Dr. Ing. Rainer Schwab, Hochschule Karlsruhe, University of Applied Sciences, Karlsruhe, Germany [https://www.youtube.com/watch?v=UuHofNW40Yw&t=14s]

Etchant classification:

1. Nital (Nitric Acid Solution): Nital is a commonly used etchant for revealing grain boundaries and the overall microstructure of ferrous materials, such as steel. It typically consists of nitric acid (HNO_3) mixed with ethanol or methanol.
2. Picral Etchant, which consists of picric acid dissolved in ethanol or methanol, is often used for revealing grain boundaries in aluminium and aluminium alloys.
3. Keller's Reagent is a mixture of nitric acid and alcohol, and it is commonly used to reveal the grain structure and dendritic structure in aluminium and magnesium alloys.
4. Vilella's Reagent is a mixture of water, hydrochloric acid, and nitric acid. It is used for revealing the macrostructure of copper and copper alloys.
5. Marble's Reagent is used for etching brass and copper alloys. It is composed of a solution of copper chloride in hydrochloric acid.
6. Aqua Regia is a mixture of concentrated nitric acid and concentrated hydrochloric acid. It is used for etching gold and some platinum-group metals to reveal macroscopic features.
7. Sodium Hydroxide Etchants are used for aluminium and its alloys to reveal macroscopic features. They typically consist of a solution of sodium hydroxide (NaOH) or caustic soda.
8. Fry's Reagent is a mixture of acetic acid and hydrogen peroxide and is used for revealing the macrostructure of stainless steel and some nickel-based alloys.
9. Nital-Ethanol is a mixture of nital, and ethanol is often used for examining welds, as it helps highlight the weld structure and potential defects in the weld area.
10. Kalling's No. 2 Etchant is used to reveal the macrostructure of cast irons.

It's essential to choose the appropriate etchant based on the material being inspected and the specific macrostructural features of interest. The etching process should be carefully controlled to achieve the desired results without over-etching or damaging the specimen. Proper safety precautions should be observed when handling etchants, as many of them are corrosive or potentially hazardous.

Cleaning: Thoroughly clean the prepared specimens to remove any residues from the cutting, grinding, and polishing processes. Ensure that the surface is free from contaminants.

Storage and Labelling:

- Properly label each specimen with essential information, including the date of preparation, the type of specimen, orientation, and any relevant details about the weld or component.
- Store the specimens in a controlled environment to prevent corrosion or degradation until they are ready for inspection.

Documentation: Maintain detailed records of the specimen preparation process, including photographs and notes, to provide a complete history of each test specimen.

Once the test specimens are prepared, they are ready for macroscopic inspection. During inspection, use appropriate lighting, magnification tools, and inspection procedures to thoroughly examine the cross-sections and assess the weld quality and the presence of any defects. Properly prepared specimens are essential for obtaining accurate and reliable inspection results.

Video 3.2.3: Metallography Sample Preparation. Source: https://www.youtube.com/watch?v=ok42dVq2CU0

3.2.4. Practical Training in the Identification of Welding Imperfections

The identification of welding imperfections is a fundamental component of ensuring the quality, safety, and reliability of welded structures and components. This hands-on training program provides participants with the essential skills and knowledge required to identify welding imperfections without the assistance of advanced tools or technology. The program is structured to provide a comprehensive understanding of welding imperfections, inspection techniques, safety measures, and documentation practices.

The practical training program comprises several key components. Firstly, participants are introduced to the critical task of identifying welding imperfections and understanding their potential structural impact, with an overview of common imperfections like porosity and cracks. Then, they delve into the world of visual inspection tools and equipment, learning how to effectively utilize instruments such as lighting, magnifiers, and examination booths for thorough assessments. Best practices for consistent and reliable inspections are covered, followed by an in-depth exploration of macroscopic examination to spot welding imperfections. Hands-on exercises offer real-world experience in identifying and characterizing imperfections. The program also guides participants in examining macroscopic specimens and interpreting their findings to make informed assessments of welding quality. Emphasis is placed on the importance of accurate documentation and transparent reporting, along with the adherence to safety precautions, including Personal Protective Equipment (PPE) requirements and hazard mitigation measures during weld inspections.

3.2.5. Hands-on Practice with Visual Inspection Techniques

1. Visual Inspection Equipment Familiarization:

Participants begin by becoming familiar with the visual inspection equipment and tools they will use during the exercises. This includes lighting, magnifiers, mirrors, and examination booths.

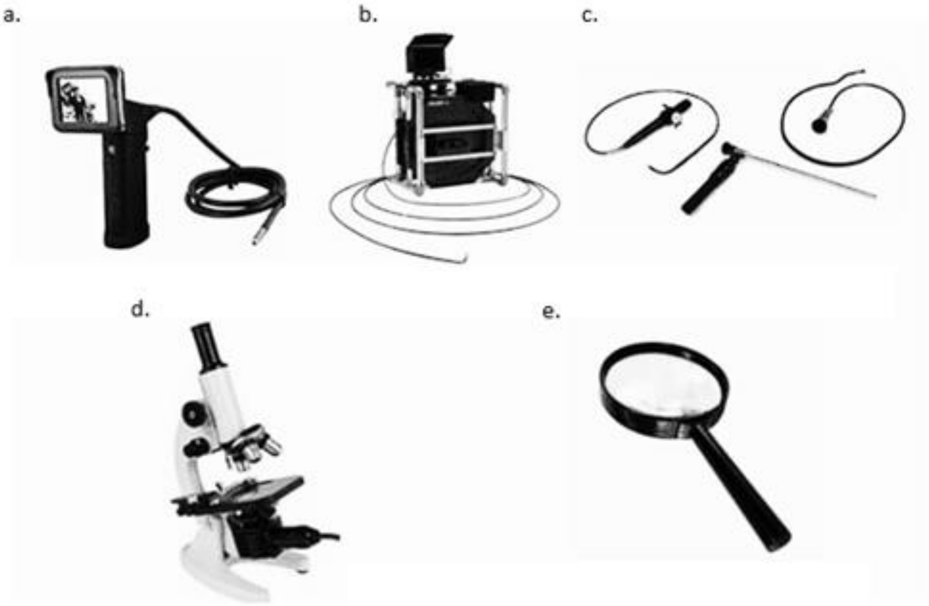


Fig 3.2.25: Videoscope (a); advanced videoscope (b); borescopes(c); magnifying glass (d); microscope (e). Source: https://worldofndt.com/introduction-to-visual-testing/

2. Examples of Welded Joints:

Physical examples of welding joints or specimens with intentionally introduced welding imperfections are provided for inspection. These specimens represent real-world scenarios and serve as the basis for hands-on practice.

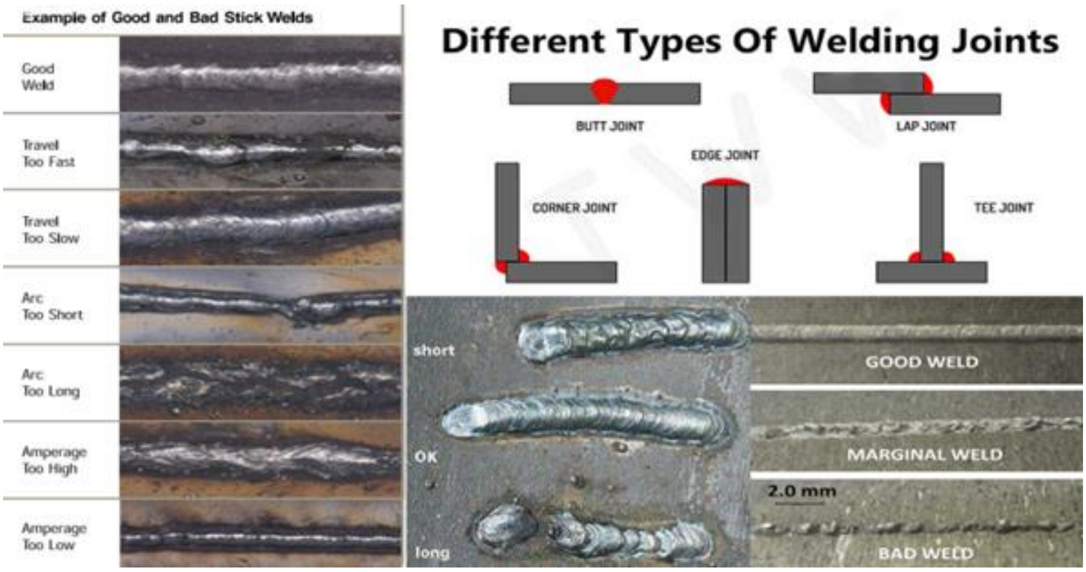


Fig. 3.2.26: Different types of welding joints. Source: https://engineeringdiscoveries.com/different-types-of-welding-joints/

3. Practical Exercises:

Participants are guided through a series of practical exercises, each focusing on specific aspects of visual inspection. These exercises can include:

- **Porosity Identification:** Participants learn to recognize and differentiate between different types of porosity, such as pinholes, surface porosity, and subsurface porosity. They practice assessing the extent and severity of porosity in welds.

Fig. 3.2.27: Porosity. Source: https://www.mechanicwiz.com/porosity-in-welding/

- **Crack Detection:** Participants practice identifying cracks, both surface and subsurface. They learn to distinguish between crack types, such as longitudinal, transverse, and crater cracks.

Fig. 3.2.28: Crack. Source: https://www.rapiddirect.com/blog/types-of-welding-defects/

- **Lack of Fusion and Incomplete Penetration:** Exercises involve spotting areas where lack of fusion or incomplete penetration has occurred. Participants assess the implications of these imperfections on welding quality.

Fig. 3.2.29: Lack of fusion. Source: https://www.onestopndt.com/ndt-articles/what-is-lack-of-fusion-in-welds

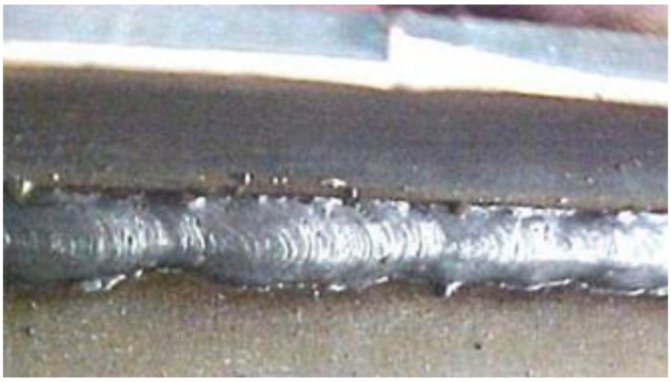


Fig. 3.2.30: Incomplete penetration. Source: https://allgas.us/b/what-are-welding-defects--types-causes-and-remedies

- **Undercut Evaluation:** Participants learn to identify undercut, its location on welds, and its potential impact on structural integrity.

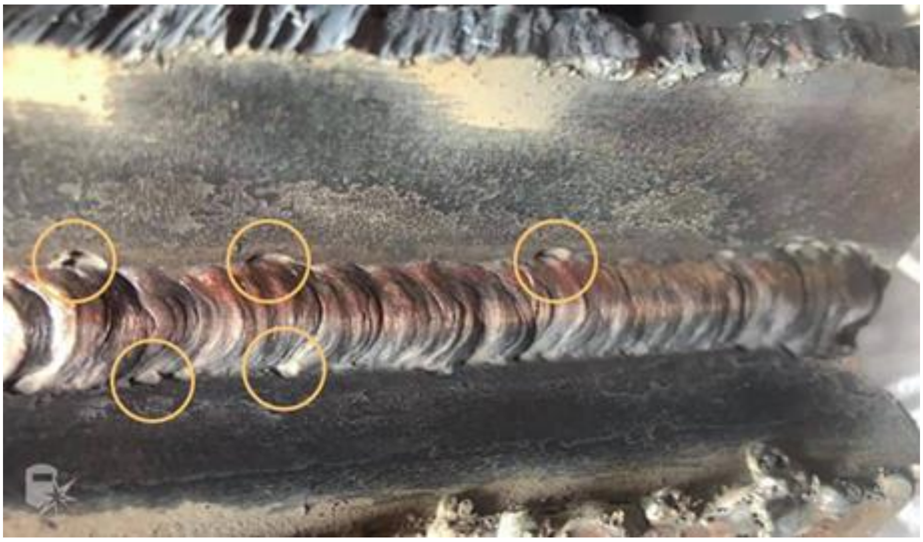


Fig. 3.2.31: Undercuts. Source: https://weldguru.com/undercut-in-welding/

- **Weld Profile Examination:** Participants analyse weld profiles to ensure they meet specified standards and dimensions.

Video 3.2.4: Welding inspection. Source: https://www.youtube.com/watch?v=nKHl7JI9p7E

4. Severity Assessment:

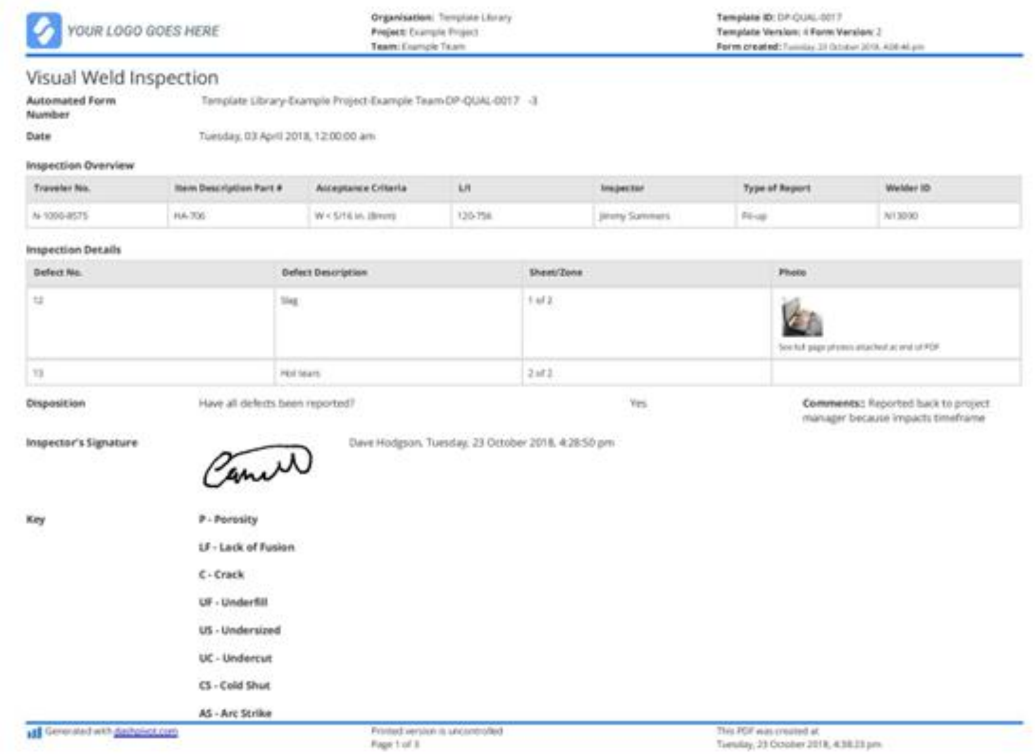
Participants not only identify imperfections but also practice assessing their severity. This is crucial for determining whether a weld is acceptable or requires corrective action.

Fig. 3.2.32: Difference between good and bad weld, example 1. Source: https://waterwelders.com/good-weld-vs-bad-weld/

Fig. 3.2.33: Difference between good and bad weld, example 2. Source: https://blog.daihen-usa.com/why-welding-parameters-are-so-important-to-finished-quality

5. Documentation Practice:

Participants document their findings during the inspection exercises. This includes recording the type and location of imperfections, their dimensions, and any relevant observations.



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Organisation: Template Library
Project: Example Project
Team: Example Team

Template ID: DP-QUAL-0017
Template Version: 4 **Form Version:** 2
Form created: Tuesday, 23 October 2018, 4:08:46 pm

Visual Weld Inspection

Automated Form Number Template Library-Example Project-Example Team-DP-QUAL-0017 -3

Date Tuesday, 03 April 2018, 12:00:00 am

Inspection Overview

Traveler No.	Item Description Part #	Acceptance Criteria	L/I	Inspector	Type of Report	Welder ID
N-1000-8575	HA-706	W < 5/16 in. (8mm)	120-756	Jimmy Summers	Fit-up	N13000

Inspection Details

Defect No.	Defect Description	Sheet/Zone	Photo
12	Slag	1 of 2	See full page photos attached at end of PDF
13	Hot tears	2 of 2	

Disposition Have all defects been reported? Yes **Comments:**: Reported back to project manager because impacts timeframe

Inspector's Signature Dave Hodgson, Tuesday, 23 October 2018, 4:28:50 pm

Key
P - Porosity
LF - Lack of Fusion
C - Crack
UF - Underfill
US - Undersized
UC - Undercut
CS - Cold Shut
AS - Arc Strike

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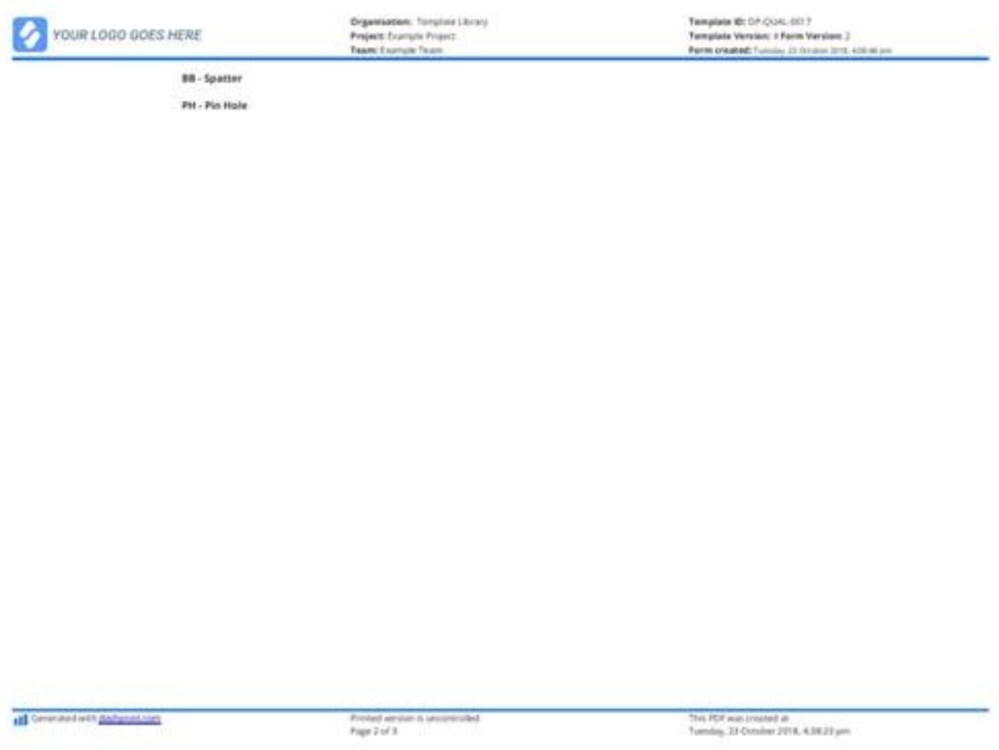
Fig. 3.2.34: Documentation practice example 1/3. Source: https://sitemate.com/templates/quality/forms/visual-weld-inspection-template/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Organisation: Template Library
Project: Example Project
Team: Example Team

Template ID: DP-QUAL-0017
Template Version: 4 **Form Version:** 2
Form created: Tuesday, 23 October 2018, 4:08:46 pm

BB - Spatter

PH - Pin Hole

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Fig. 3.2.35: Documentation practice example 2/3. Source: https://sitemate.com/templates/quality/forms/visual-weld-inspection-template/

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Organisation: Template Library
Project: Example Project
Team: Example Team

Template ID: DP-QUAL-0017
Template Version: 4 **Form Version:** 2
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Comments:

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Fig. 3.2.36: Documentation practice example 3/3. Source: https://sitemate.com/templates/quality/forms/visual-weld-inspection-template/

6. Instructor Guidance:

Throughout the exercises, experienced instructors provide guidance, answer questions, and offer feedback to help participants improve their inspection skills.

7. Error Recognition and Correction:

Participants are encouraged to recognize and correct any errors they may make during the inspection process. This promotes continuous improvement and accuracy.

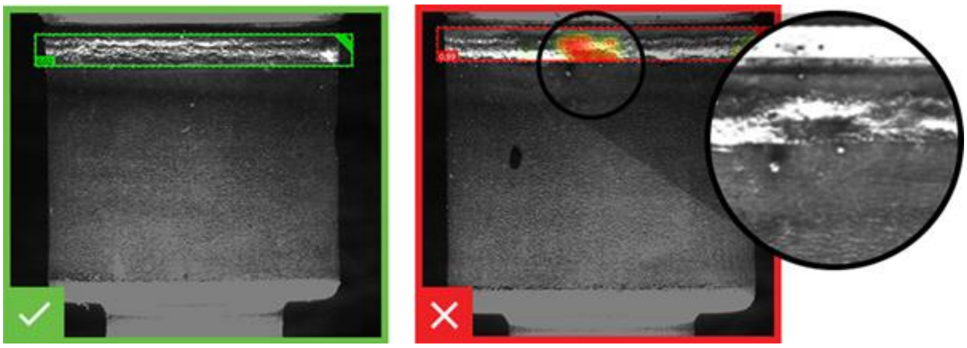
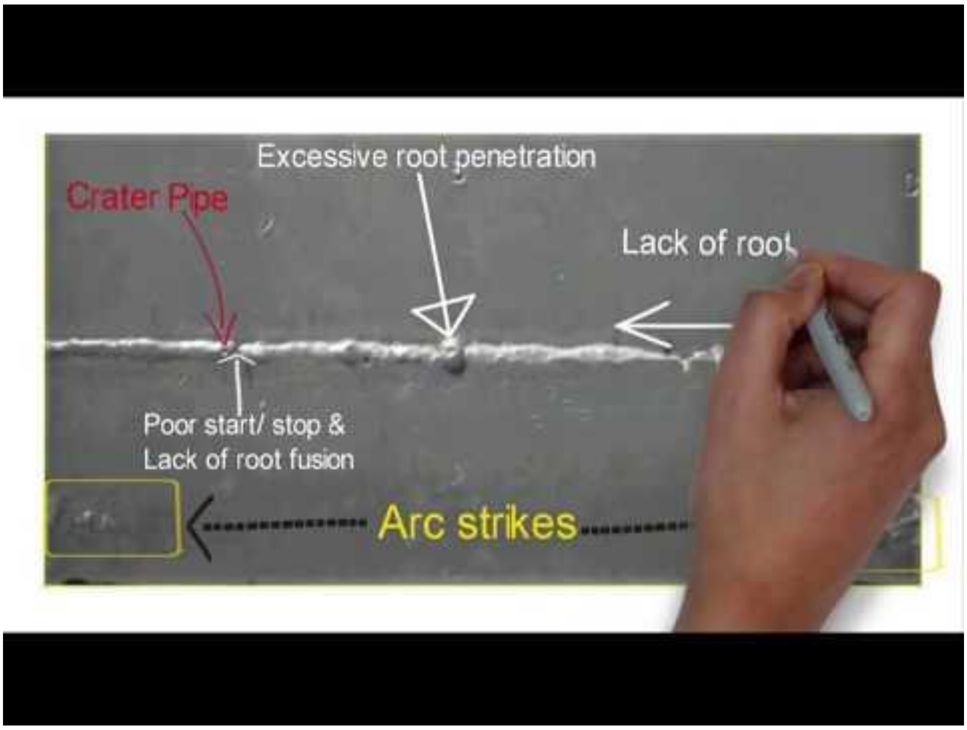


Fig. 3.2.37: Welding Seam Inspection. Source: https://www.cognex.com/industries/automotive/powertrain-systems/welding-seam-inspection

3.2.6. Examination and Analysis of Macroscopic Specimens

The examination and analysis of macroscopic specimens, particularly in the context of material science and engineering, involves the thorough visual inspection and assessment of prepared samples or components to gather information about their physical characteristics, defects, and overall quality.

The process of material examination comprises several key steps. It commences with the preparation of specimens, involving cutting, mounting, grinding, and polishing to ensure well-prepared surfaces for analysis. Subsequently, a visual inspection is conducted to identify visible features and anomalies. Microstructural examination may follow, using microscopes to scrutinize grain sizes, boundaries, and material composition. Photographs are taken for documentation, which proves invaluable for later analysis and comparison. Defects, irregularities, and dimensions are meticulously recorded, followed by an evaluation of the overall structure and surface condition. The findings are then compared to industry standards, codes, and specifications, determining compliance with quality and safety requirements. Based on this analysis, an assessment of the specimens' suitability for their intended purpose is made. A comprehensive inspection report is created, encompassing descriptions, measurements, photographs, and recommendations. Finally, the specimens and related documentation are archived and stored for future reference, maintaining a robust quality control and records management system.



Video 3.2.5: Welding Defects, their appearance and identification. Source: https://www.youtube.com/watch?v=IgeUT5EIH5o

3.2.6.1. *Documentation and Reporting of Welding Imperfections*

Documentation and reporting of welding imperfections are essential aspects of quality control and assurance in welding projects. Properly documenting and reporting imperfections help ensure transparency, traceability, and compliance with industry standards and project specifications.

Documentation guide:

Start by utilizing standardized forms or templates created for the purpose of documenting welding imperfections/defects. These forms typically encompass fields for vital details and checklists to identify prevalent defects.

Clearly mark the inspection or test report with crucial information, which includes the project's name, location, date, the inspector's identity, and a unique identifier for the examined item.

Make a specific remark on each identified welding imperfection or defect. This remark should encompass specific information regarding the defect type, its precise location on the weld, dimensions, and any pertinent contextual observations.

Categorize each imperfection/defect in accordance with industry-recognized standards or codes. Different defects might carry varying levels of severity, and this categorization aids in evaluating their potential impact on structural integrity.

Incorporate explicit photographs or visual documentation of every imperfection. Visual evidence frequently proves more effective in elucidating the nature and extent of each defect.

Utilize sketches or diagrams to visually represent the positioning and measurements of welding imperfections. These graphical aids enhance comprehension.

Refer to the acceptance criteria or standards employed for appraising the imperfections. This facilitates a clear determination of whether the defect falls within acceptable parameters.

Document the inspection methods and equipment used to detect and measure welding imperfections. This contextual information provides insight into the inspection process.

Propose recommendations for corrective actions, repair procedures, or further assessments based on the identified imperfections. These suggestions serve as guidance in decision-making.

Incorporate designated spaces for the inspector's signature and any mandatory endorsements from relevant parties. This step validates the report's accuracy and authorizes proposed actions.

Submit inspection reports promptly upon the inspection's conclusion. Timely reporting ensures that required measures can be taken without undue delays.

Convey the findings and recommendations to all pertinent stakeholders, including welders, supervisors, engineers, and project managers.

Establish an efficient system for the orderly storage and archiving of inspection reports and related documentation. Adequate retention guarantees the accessibility of historical records.

Maintain a comprehensive record of all inspection reports, both for future reference and as an integral component of a quality control and assurance initiative.

Periodically review inspection reports to identify trends, recurring issues, or areas where process enhancements can be applied. Utilize this information to refine welding practices.

3.2.6.2. Reporting Formats and Requirements

Reporting formats and requirements can vary widely depending on the specific industry, organization, and the nature of the information being reported. However, there are some common elements and considerations that apply to many types of reports.

- **Title and Cover Page**: Reports typically start with a title page that includes the report's title, date of submission, the name of the author or organization, and any relevant logos or branding.
- **Executive Summary**: Many reports begin with an executive summary, which provides a concise overview of the report's key findings, recommendations, and conclusions. It's often the first section that decision-makers read.
- **Table of Contents**: A table of contents lists the major sections and subsections of the report, along with page numbers. It helps readers navigate the document easily.
- **Introduction**: It sets the stage for the report by explaining its purpose, scope, and objectives. It also provides background information and context for the report's content.

- **Methodology**: In technical or research reports, the methodology section explains how data was collected, analysed, and any relevant research or testing methods used.
- **Findings or Results**: This section presents the main findings, results, or observations of the report. It often includes data, graphs, charts, or other visual aids to support the findings.
- **Discussion**: This section interprets the findings and provides analysis. It may address the implications of the results, trends, comparisons, and any relevant theories or frameworks.
- **Recommendations**: If applicable, needs to provide clear and actionable recommendations based on the findings. Recommendations should be specific, practical, and aligned with the report's objectives.
- **Conclusion**: Summarize the key points of the report and restate its main conclusions. It reinforces the report's main takeaways.
- **Formatting Requirements**: Follow formatting guidelines such as font size, line spacing, margins, and page numbering. Consistency in formatting ensures a professional and polished appearance.
- **Graphics and Visuals**: Use graphics, charts, tables, and other visuals when appropriate to enhance the presentation of data and information. Ensure that visuals are labelled and referenced properly.
- **Language and Style**: Use clear, concise language and maintain a consistent writing style throughout the report. Avoid jargon or technical terms that may be unfamiliar to the audience.
- **Audience Consideration:** Tailor the report's content, tone, and level of technical detail to the needs and expectations of the intended audience.
- **Distribution**: Determine how the report will be distributed to its intended audience. This may involve electronic distribution, physical copies, or presentations.

www.Inspection-for-Industry.com
Quality Control Form
**WELDING DAILY REPORT
(PIPING)**

Project:
Client:
Contractor:
Ref. Standard:
Doc. No.:
Report No.:
Date:
Page 1 of 1

Area/ Location:
Ref. Drawing:
Rev.

Line No.	Spool No.	Joint No.	Joint Type	O.D	Thk.	Welder (s) ID			Type of Material/Material Spec.		WPS No.	Pre-heat	PWHT	Fit up Report No.	VT Result	Remarks
						Root	Hot	Cap	....	to						

Technical Comments:

Abbreviation:
Acc. : Accept   Rej. : Reject   RC : Re-Cap   NA: Not Applicable
B: Butt Weld, S: Socket Weld, T: Seal Weld (for Threaded Joints), M: Miter
For Example: Pipe / 5L X52 to Flange / A105

Quality Control	TPI Inspection	Client
Name: Sign: Date	Name: Sign: Date	Name: Sign: Date:

Fig. 3.2.38: Welding inspection report template example. Source: https://in.pinterest.com/pin/welding-inspection-report-template-1-templates-example--504543964505475585/

3.2.7. Safety Precautions during Weld Inspection

Safety precautions during weld inspection are critical to ensure the well-being of personnel involved in the inspection process. Welding operations can produce hazardous conditions and inspecting welds, especially in industrial settings, requires careful attention to safety protocols.

Safety requirements:

- **Personal Protective Equipment (PPE)**: Appropriate PPE should always be worn, including safety glasses or goggles, a welding helmet or face shield, flame-resistant clothing, gloves, and steel-toed safety boots. The specific PPE requirements may vary based on the inspection environment.

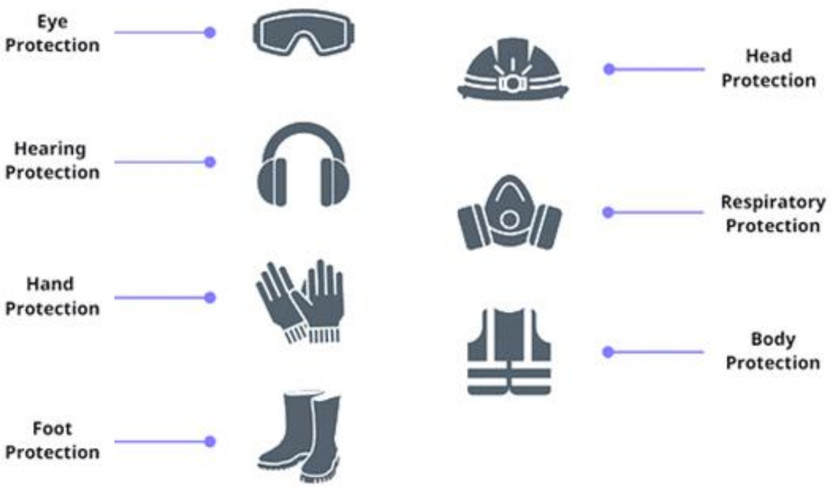


Fig. 3.2.39. Personal Protective Equipment. Source: https://safetyculture.com/topics/ppe-safety/

- **Respiratory Protection and ventilation measures** are essential when dealing with potential exposure to welding fumes, gases, or particulate matter. Properly selected respirators should be worn to address the specific type and level of contaminants present, while ensuring that the inspection area maintains good ventilation to lower the concentration of welding fumes, thereby reducing the risk of inhalation exposure.
- **Noise Protection**: Welding processes can generate high levels of noise. It is mandatory to use hearing protection, such as earplugs or earmuffs, in order to prevent hearing damage.
- **Eye Protection**: To protect from potential hazards, such as flying debris, sparks, and ultraviolet (UV) or infrared (IR) radiation, welding goggles or helmets with appropriate lenses are essential.
- **Proper Lighting**: Adequate lighting is crucial for accurate inspection; the inspection area should be well-lit; it is recommended to use a portable work light or inspection lamps when needed.
- **Electrical Safety**: Electrical hazards associated with welding equipment should be considered; power cords, plugs, and electrical connections must be inspected for damage. Proper grounding of welding equipment is mandatory.
- **Falling Object Prevention**: When working at heights or in elevated positions, fall protection equipment such as harnesses and safety lines must be used to prevent accidents.
- **Handling of Tools and Equipment**: Tools and equipment should be carefully used to avoid cuts, abrasions, or injuries; tools should be kept in good working conditions.
- **Fire Safety**: Fire hazards must be avoided; thus, fire extinguishers need to be nearby the workspace, and personnel must be instructed on their utilisation; all flammable materials should be removed from the workspace.
- **Hot Surfaces**: Welded components and nearby surfaces can become extremely hot, contact with these hot surfaces should be avoided in order to prevent burns; thermal barriers or insulating materials must be used when necessary.
- **Safe Access**: Safe access to inspection areas must be provided, using stable platforms, ladders, or scaffolding as needed to reach elevated workspaces.
- **Chemical Exposure**: Chemicals or cleaning agents used in the inspection process are potential dangers, thus it is encouraged to follow proper handling and disposal procedures, as well as wear appropriate gloves when necessary.
- **Communication**: Clear communication with all personnel in the vicinity of the workspace should be ensured, the use of signals, signs, or verbal communication is encouraged to ensure everyone is aware of ongoing activities.

- **Emergency Procedures**: Personnel should be familiarized with the emergency response plan (emergency exits location, first aid kits, eye wash stations, and emergency showers).
- **Training**: Regular safety training updates are essential; it is mandatory that the personnel operating in the workspace have received proper training in welding safety and inspection protocols.
- **Risk Assessment**: Risk assessment is required before initiating any activities, namely, the need to identify potential hazards and implement control measures that will mitigate risks.

3.2.7.1. Personal Protective Equipment (PPE) Requirements

Personal Protective Equipment (PPE) requirements for weld inspection, like those for welding itself, are crucial to ensure the safety and well-being of individuals involved in the inspection process. The specific PPE requirements can vary based on factors such as the type of welding being inspected, the inspection environment, and the potential hazards present.

Eye Protection:

- **Safety Glasses or Goggles**: These protect the eyes from flying debris, sparks, and other potential eye hazards.

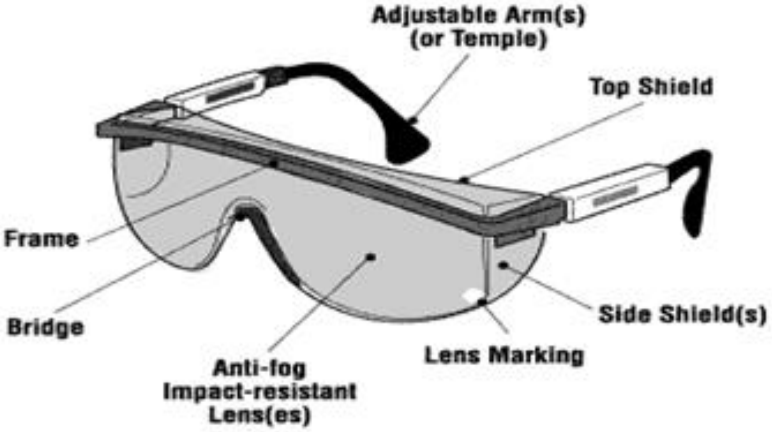


Fig. 3.2.40. Welding Goggles. Source: https://www.ccohs.ca/oshanswers/prevention/ppe/glasses.html

- **Welding Helmet**: A welding helmet with a suitable lens shade protects against the intense light, UV, and IR radiation produced during welding inspections.

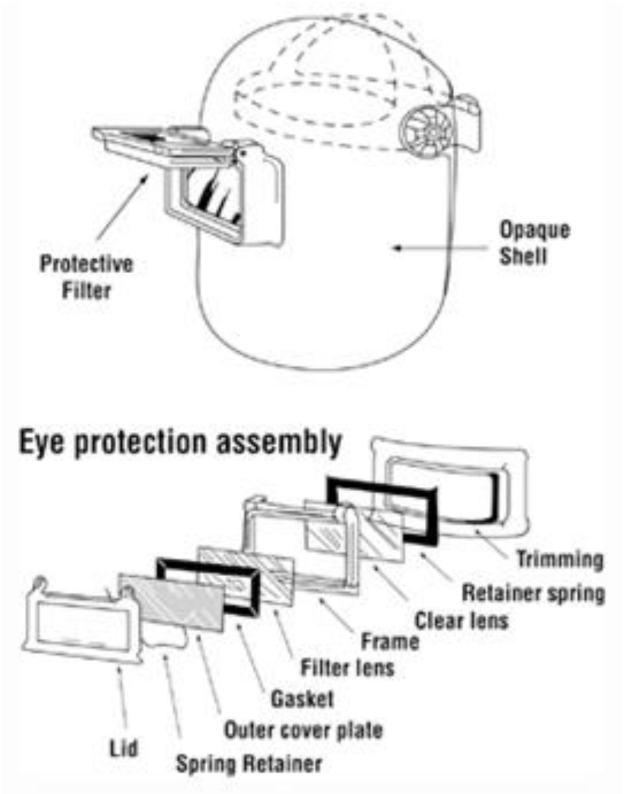


Fig. 3.2.41. Welding Helmet. Source: https://www.ccohs.ca/oshanswers/safety_haz/welding/ppe.html

- **Face Shield**: When additional face protection is needed, a face shield can be worn over safety glasses or goggles.

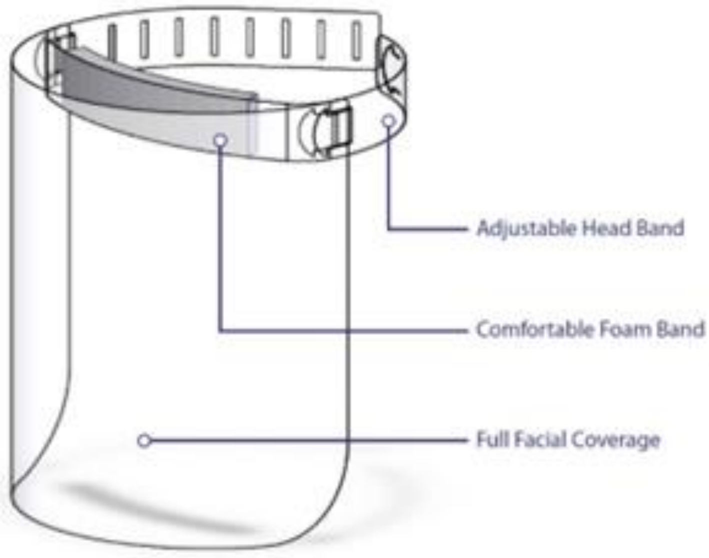


Fig. 3.2.42. Face Shield. Source: https://www.ardentdisplays.com/protective-face-shields/

Respiratory Protection:

- **Respirators**: Depending on the inspection environment, weld inspectors may need respirators to protect against welding fumes, gases, or particulate matter. The type of respirator required depends on the specific contaminants present.

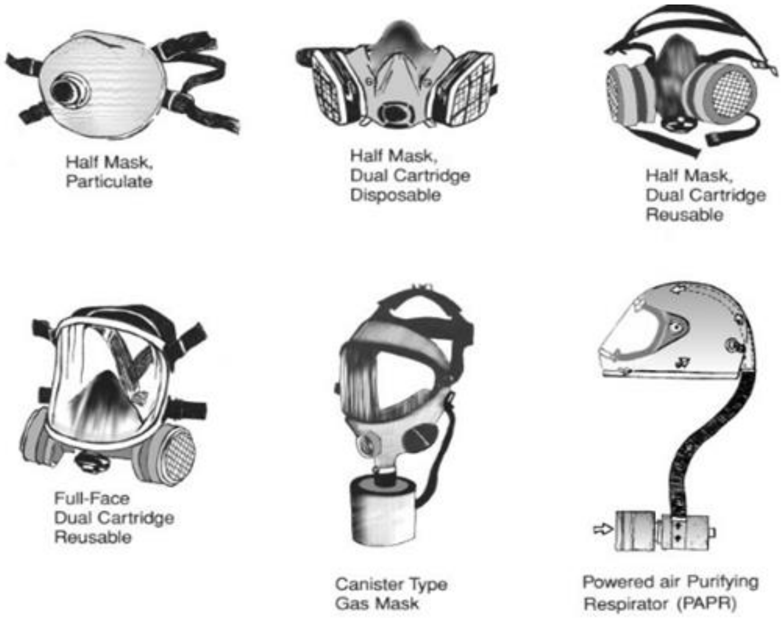


Fig. 3.2.43. Respiratory protection. Source: https://pesticidestewardship.org/respirators/air-purifying-respirators/

Hearing Protection:

- **Earplugs or Earmuffs**: Welding inspections can occur in noisy environments. Hearing protection helps prevent hearing damage from prolonged exposure to high noise levels.

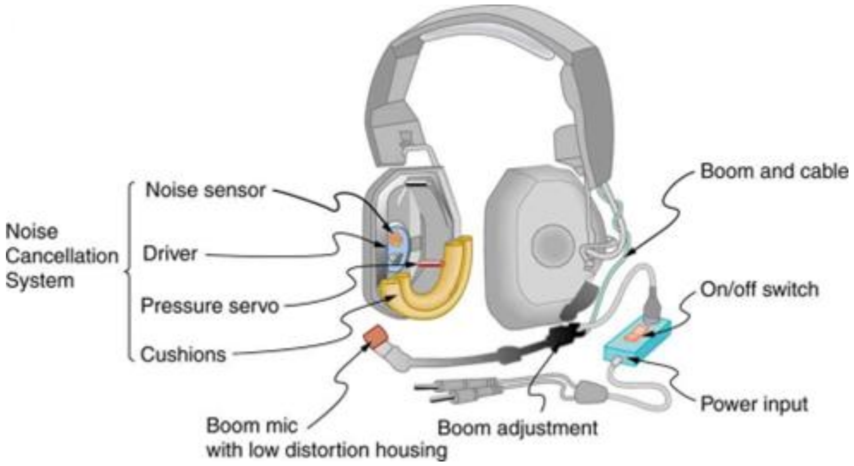


Fig. 3.2.44. Ear Defenders for Hearing Protection. Source: https://uk.rs-online.com/web/content/discovery/ideas-and-advice/ear-defenders-guide

Hand Protection

- **Welding Gloves**: These are essential for protecting the hands from burns, sparks, and hot surfaces. Insulated gloves may be necessary for handling hot components.

Fig. 3.2.45. Welding Gloves. Source: https://www.amazon.de/YEZIHJ-Schwei%C3%9Fhandschuhe-H%C3%A4ndeschutzschwei%C3%9Fhandschuhe-Lederarbeitsschwei%C3%9Ferhandschuhe-Hitzebest%C3%A4ndiger/dp/B0B7R39GGC

Body Protection:

- **Flame-Resistant Clothing**: Inspectors should wear flame-resistant clothing to protect against burns and sparks. This includes flame-resistant shirts, pants, and coveralls.

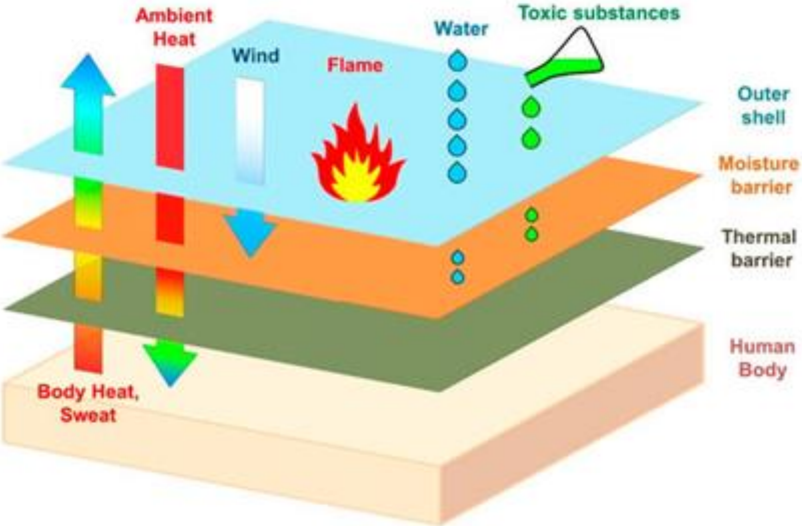


Fig. 3.2.46. Scheme of the construction of a multilayer assembly used in firefighters' protective clothing. Source: Renard M, Puszkarz AK. Modeling of Heat Transfer through Firefighters Multilayer Protective Clothing Using the Computational Fluid Dynamics Assisted by X-ray Microtomography and Thermography. Materials. 2022

- **Welding Jacket**: A welding jacket provides additional protection for the upper body.

Fig. 3.2.47. Welding Jacket. Source: https://www.eskosafety.com/shop/esko-fusion-leather-welders-jacket/

Foot Protection:

- **Steel-Toed Safety Boots**: Safety boots with steel toes protect the feet from falling objects and hot metal.

Fig. 3.2.48. Toed Safety Boots. Source: https://www.ctcgroupe.com/en/your-markets/personal-protective-equipment/ce-marking-current-footwear-ppe-and-standards-162-1.html

Head Protection:

- **Hard Hat**: In environments with overhead hazards or the risk of falling objects, wearing a hard hat is necessary.

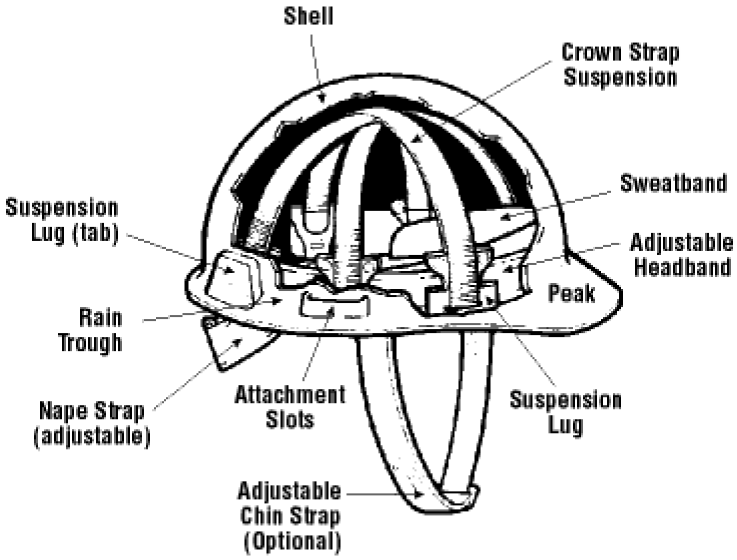


Fig. 3.2.49. Welding Hard Hat. Source: https://www.ccohs.ca/oshanswers/prevention/ppe/headwear.html

Fall Protection:

- If the inspection involves working at heights, fall protection equipment such as a safety harness and lanyard may be required.

Fig. 3.2.50. Components of a Fall Arrest System. Source: https://www.karam.in/blog/revisiting-concept-fall-protection

It's important to note that PPE requirements can vary depending on local regulations, industry standards, and company policies. Before starting any welding inspection, individuals should receive training on the proper use and maintenance of PPE, as well as an understanding of the specific hazards associated with the inspection task. Additionally, PPE should always be in good condition, regularly inspected, and replaced if damaged or worn.

3.3. Practical training in specimen preparation

Practical training for common materials preparation is hereby presented according to the methodology presented in figure 1.2.2.

3.3.1. Carbon steels

In the case of carbon steels, the preparation needs to follow the basic methods (see Chapter 1.2.1.). Example of a carbon steel preparation below according to ISO 16060:2003.

Sampling: Cutting a suitable size sample (as a function of the mounting). Suitable cutting methods are water jet cutting, mechanical cutting under cooling, etc. Figure 3.3.1. shows a cutting machine used for microscopy tests.

Figure 3.3.1.1. Metcon Servocut 302

Mounting: Hot mounting or cold mounting is also suitable (see Chapter 1.2.2.), the mounting cylinder diameter is usually 25-50 mm. During hot mounting, the hot mounting machine manual needs to be followed. An example of a hot mounting machine is shown in Figure 3.3.1.2.

Figure 3.3.1.2. Hot mounting machine

During the cold mounting process, the manual of the mounting material needs to be followed (two component resin).

Figure 3.3.1.3. Cold mounting process example

Grinding: when grinding under water colling use a suitable grinding machine.

Fine grinding: Use SiC grinding papers or foils from 200 -1000 grit. In the case of manual grinding follow Chapter 1.2.5, while for the use of the grinding machine(fig. 3.3.1.4) refer to the user manual. Use grinding papers or foils as a function of the machine requirement with the same grit as the manual grinding process.

Figure 3.3.1.4. Example of a grinding machine

Polishing: polishing is made by a polishing machine using a polishing pad with adhesive particles. The polishing pads can be in several forms and quality (felt, velvet, linen, etc.). Abrasive can be diamond or alumina (9-0.05µm) in water suspension.

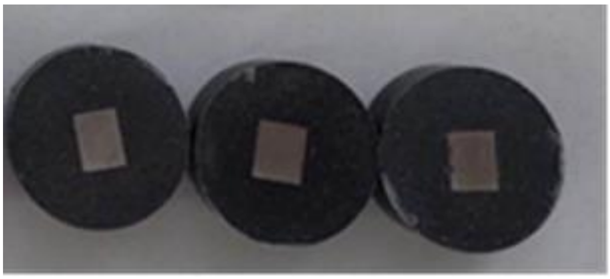


Figure 3.3.1.5. Polished test samples

Cleaning: the carbon steel surface needs to be cleaned by Methanol in order to remove all contamination and water (see Chapter 1.2.4.)

Drying: drying should take place in a stream of mild air.

Etching: suggested Nital (see Table 1.2.8.1.)

3.3.2. Austenitic stainless steel

The preparation of test samples of stainless steel is the same as in the case of carbon steel. It needs to be taken care of when cutting because the austenitic stainless steels have low hardness. Cutting with a water jet, if available, is recommended. During the grinding and polishing, the preparation needs to be carried carefully, to maintain the austenitic stainless steel microstructure. The preparation of the stainless steel should be done according to ISO 16060:2003.

Etching: suggested Oxalic acid solution (see Table 1.2.8.1.)

3.3.3. Copper and alloys

The preparation of test samples of copper and alloys is usually the same as for steels and other metals, but during polishing it is suggested the use of diamond abrasive particles (3-0.05µm). The cleaning and drying process is the same as in the case of carbon steels. The preparation of copper and its alloys should be done according to ISO 16060:2003.

Etching: suggested Hydrochloric acid-iron chloride (see Table 1.2.8.1.)

3.3.4. Aluminium and alloys

The preparation of test samples of aluminium and its alloys is the same as in the case of carbon steel. It needs to be taken care when cutting because the aluminium and alloys have low hardness. Cutting with a water jet, if available, is recommended. Make the aluminium preparation on the base of ISO 16060:2003.

Etching: suggested Kroll etchant (see Table 1.2.8.1.)

3.3.5. Titanium and alloys

The preparation of test samples of titanium and its alloys is usually the same as for steels and other metals, but during polishing the usage of diamond abrasive particles is suggested (3-0.05µm). In the last step of polishing diamond suspension combined with H_2O_2 should be used. The cleaning and drying process is the same as in the case of carbon steels. Make the titanium and alloy preparation on the base of ISO 16060:2003.

Etching: Keller's etchant (see Table 1.2.8.1.).

3.3.6. Polymers

The preparation of polymers samples is different from metals; thus, it demands a more careful preparation.

Sampling: a wide range of polymers with different hardness in ambient temperature can be found. Therefore, the recommendation is to cut the polymers under glass transition temperature, which is in the case of the weldable polymers over the room temperature. Mechanical cutting methods with water cooling or low-rate hand cutting can be used.

Mounting: it is very important to know the polymer melting or softening temperature along with the polymerization temperature. The hot mounting temperature needs to be lower than the polymerization, softening, and melting temperature. The mounting material should be chosen in accordance with the polymer, so that there is no chemical reaction in-between each other. The mounting process takes into account the chemical ability and the reaction temperature of the mounting material.

Grinding and fine grinding: Use SiC grinding papers or foils from 200 -1000 grit. In the case of manual grinding follow Chapter 1.2.5. In the case of the grinding machine follow the manual of the machine. Use grinding papers or foils as a function of the machine requirement with the same grit as the manual grinding. Figure 3.3.1.4. shows a grinding machine. Use low rotation speed during the polymer preparation.

Polishing: polishing is made by a polishing machine, use a polishing pad with adhesive particles. The polishing pads can be in several forms and quality (felt, velvet, linen, etc.). The abrasive can be diamond or alumina (9-0.05µm) in water suspension.

Cleaning: only water cleaning.

Drying: drying should take place in a stream of ambient air (check room temperature).

Etching: no etching.

References:

[3.3.1] Bramfitt B. L.., Benscoter A. O.: Metallographer's Guide, Practices and Procedures for Irons and Steels. 1st. Ed. ASM International, Materials Park, 2002. 169–244.

[3.3.2] McCall J. L.., Mueller W. M.: Metallographic Specimen Preparation, Optical and Electron Microscopy 1st. ed. Plenum Press, New York, 1973. 1–348.

[3.3.3] Vander Voort G. F.: Metallography Principles and Practice. 4th. ed. ASM International, Materials Park, 2007. 1–258.

[3.3. 4] ISO/TR 16060:2003 Destructive tests on welds in metallic materials — Etchants for macroscopic and microscopic examination, 2003

3.4. Standards for the quality assessment of joints made by welding and related techniques

3.4.1. EN ISO 17639 Destructive tests on welds in metallic materials — Macroscopic and microscopic examination of welds

This standard provides recommendations for specimen preparation, test procedures, and their main objectives for macroscopic and microscopic examination. It defines macroscopic and microscopic examination, as described below:

- macroscopic examination: the examination for the prepared test specimen needs to be made by visual test without magnification or maximum with 50x magnification. The examined surface of the specimen can be etched or unetched.
- microscopic examination: the examination for the prepared test specimen needs to be made by microscope and uses a magnification between 50x – 500x. The examined surface of the specimen can be etched or unetched.

The standard defines the personal condition, the people who doing the test is the examiner.

The standard provides all instructions for test specimen preparation including surface finishing and etching methods with referred etchants. It lists the safety rules to be observed during examination. Examination of the prepared surface can be done on the etched or unetched specimens in accordance with the relevant standards. The standard suggests the form of the experimental report.

3.4.2. ISO/TR 15608 Welding — Guidelines for a metallic materials grouping system

This standard includes a uniform system for classification of metallic materials for welding. The grouping system can be used for heat treatment or materials testing.

The materials which are referred to in this standard are the following: steels, aluminium and alloys, copper and alloys, nickel and alloys, titanium and alloys, zirconium and alloys, and cast irons.

Steels are classified as a function of the minimum yield strength, on the base of the manufacturing or heat-treating processes and according to their Chromium and Nickel alloy content or based on the microstructure (e.g. austenite, ferrite, martensite).

Aluminium, copper, nickel, titanium, zirconium and their respective alloys, are classified based on their alloying elements. The cast irons are classified according to the microstructure. This standard refers to the following standards ISO/TR 20172 Welding – Grouping system for materials- European materials, ISO/TR 20173 Welding – Grouping system for materials- American materials, ISO/TR 20174 Welding – Grouping system for materials- Japanese materials.

3.4.3. ISO/TR 17671-2 Welding – Recommendations for welding of metallic materials – Part 2: Arc welding of ferritic steels

This standard provides guidance for manual, semi-mechanized, mechanized, and automatic arc welding of ferritic steels, excluding ferritic stainless steels, in all product forms. Details concerning the possible detrimental phenomena that can occur are given with advice on methods by which they can be avoided.

This part of ISO/TR 17671 is generally applicable to all ferritic steels and is appropriate regardless of the type of manufacturing involved, although the application standard can have additional requirements.

3.4.4. ISO/TR 16060 Destructive tests on welds in metallic materials — Etchants for macroscopic and microscopic examination

This standard provides a review of the most important etchant materials (with chemical composition) for macroscopic and microscopic examination of the welded material testing in accordance with EN ISO 17639. The standard suggests the surface preparation methods before etching and gives additional requirements.

3.4.5. EN ISO 17637 Non-destructive testing of welds - Visual testing of fusion-welded joints

This standard specifies visual testing of fusion welds in metallic materials. It may also be applied to visual testing of the joint prior to welding. The non-destructive testing of welds is applicable to:

- Fusion-welded joint manufacturers
- Inspection authorities in various sectors such as construction, automotive, general engineering, aerospace, and marine
- Authorised training and qualification bodies
- Certification bodies

The standard ensures that visual testing of fusion welds in metallic materials is uniform and repeatable, irrespective of new processes, revised standard practices, newly developed equipment or changes to the materials used. It can thus help the improvement of weld quality by precise and efficient visual testing of fusion-welded joints.

3.4.6. ISO 15614-1 Specification and qualification of welding procedures for metallic materials — Welding procedure test — Part 1: Arc and gas welding of steels and arc welding of nickel and nickel alloys

This standard specifies how a preliminary welding procedure specification is qualified by welding procedure tests. ISO 15614-1 applies to production welding, repair welding, and build-up welding. ISO 15614-1 defines the conditions for the execution of welding procedure tests and the range of qualification for welding procedures for all practical welding operations within the qualification of this document.

The primary purpose of welding procedure qualification is to demonstrate that the joining process proposed for construction is capable of producing joints having the required mechanical properties for the intended application.

Two levels of welding procedure tests are given in order to extend the range of application to welded joints. In level 2, the extent of testing is greater, and the ranges of qualification are more restrictive than in level 1. Procedure tests carried out to level 2 automatically qualify for level 1 requirements, but not vice-versa. When no level is specified in a contract or application standard, all the requirements of level 2 apply.

This standard applies to the arc and gas welding of steels in all product forms and the arc welding of nickel and nickel alloys in all product forms.

Arc and gas welding are covered by the following processes in accordance with ISO 4063.

111 - manual metal arc welding (metal-arc welding with covered electrode);

114 - self-shielded tubular-cored arc welding;

12 - submerged arc welding;

13 - gas-shielded metal arc welding;

14 - gas-shielded arc welding with non-consumable electrode;

15 - plasma arc welding;

311 - oxy-acetylene welding.

The principles of this document may be applied to other fusion welding processes.

It is worth mentioning that a former process number does not require a new qualification test according to this document. Specification and qualification of welding procedures that were made in accordance with previous editions of this document may be used for any application for which the current edition is specified. In this case, the ranges of qualification of previous editions still remain applicable.

It is also possible to create a new WPQR (welding procedure qualification record) range of qualification according to this edition based on the existing qualified WPQR, provided the technical intent of the testing requirements of this document has been satisfied. Where additional tests have to be carried out to make the qualification technically equivalent, it is only necessary to perform the additional test on a test piece.

3.4.7. ISO 15614-2 Specification and qualification of welding procedures for metallic materials — Welding procedure test — Part 2: Arc welding of aluminium and its alloys

This standard specifies how a preliminary welding procedure specification is qualified by welding procedure tests.

It applies to the arc welding of wrought and cast aluminium and its alloys. It does not apply to finishing welding of aluminium castings, which is mentioned in ISO 15614-4.

3.4.8. EN 10247 Micrographic examination of the non-metallic inclusion content of steels using standard pictures.

This draft European Standard defines a method of microscopic non-metallic endogenous inclusion assessment using picture charts. The method does not apply to particles of a length or diameter less than 3,0 µm or a width smaller than 2,0 µm. If defined by a product standard or agreement between the involved parties for certain special products, inclusions with a width below 2,0 µm can be evaluated by length alone. Inclusions with dimensions exceeding the upper limits are summarised in this standard. It is assumed, if particles are elongated or if there are stringers of particles, that they are parallel to each other. Other arrangements are not covered by this draft standard. This draft European Standard applies to samples with a microscopic precipitation approaching random distribution.

From the data of measurements obtained by this method, evaluation according to other standards can be established. This draft European Standard does not apply to free cutting steels.

It is worth mentioning that the basic principle of this draft European Standard allows the determination of non-metallic inclusion content by image analysis techniques.

3.4.9. ISO 6520-1, Welding and allied processes — Classification of geometric imperfections in metallic materials — Part 1: Fusion welding

This standard serves as the basis for a precise classification and description of weld imperfections.

In order to avoid any confusion, the types of imperfection are defined with explanations and illustrations where necessary. Metallurgical imperfections are not included.

Another system for the designation of imperfections is possible according to ISO/TS 17845. Some correspondence is given between the existing classification of imperfections according to ISO 6520-1 and the designation system according to ISO/TS 17845.

3.4.10. ISO/TS 17845 Welding and allied processes — Designation system for imperfections

This standard provides a system for the designation of imperfections in welding and allied processes. It applies to metallic and non-metallic materials (e.g. thermoplastics). It can also be applied for other purposes (e.g. founding).

3.4.11. ISO 6520-1 Welding and allied processes — Classification of geometric imperfections in metallic materials — Part 1: Fusion welding

This standard serves as the basis for a precise classification and description of weld imperfections. In order to avoid any confusion, the types of imperfection are defined with explanations and illustrations where necessary. Metallurgical imperfections are not included. Another system for the designation of imperfections is possible according to ISO/TS 17845. Some correspondence is given between the existing classification of imperfections according to ISO 6520-1 and the designation system according to ISO/TS 17845.

References:

[3.4.1] EN ISO 17637 Non-destructive testing of welds - Visual testing of fusion-welded joints

[3.4.2] EN ISO 17639:2022 Destructive tests on welds in metallic materials — Macroscopic and microscopic examination of welds 2022.

[3.4.3] ISO 15614-1:2017 Specification and qualification of welding procedures for metallic materials — Welding procedure test — Part 1: Arc and gas welding of steels and arc welding of nickel and nickel alloys.

[3.4.4] EN 10247 Micrographic examination of the non-metallic inclusion content of steels using standard pictures

[3.4.5] ISO 6520-1 Welding and allied processes — Classification of geometric imperfections in metallic materials — Part 1: Fusion welding

3.5. Practical training for microscopic specimen structure identification.

3.5.1. Carbon and low alloy steel

Carbon and alloy steel can be manufactured with a wide range of properties. To describe these microstructures, it is necessary to identify the constituents present. Single phase constituents include austenite, ferrite, δ ferrite, cementite, various alloy carbides, and martensite, as well as various inter-metallic phases, nitrides, and no metallic inclusions.

The ferritic grains can be equiaxed or elongated (in lamination direction). In carbon steel, fully ferritic steels are obtained only when the carbon content is low (C < 0,08 % wt). Ferrite is a soft low-strength phase and if the grain size is fine, then good ductility and formability are obtained.

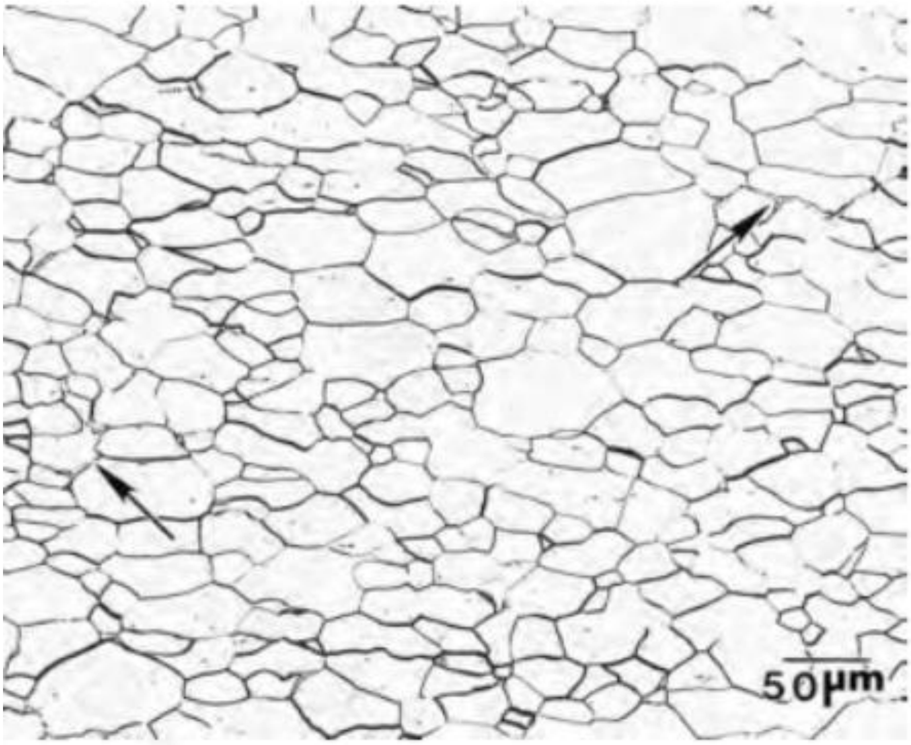


Fig. 3.5.1 – Low alloy steel (C<0,08 % wt). Fully ferritic structure. Etchant: Nital 2%. Source: IIS Laboratory

Most type of non-alloyed steel for structural or pressure applications (C content between 0,08 and 0,4 % wt) consist of ferrite and pearlite. The amount of pearlite is qualitatively indicative of the carbon content in the alloy. A higher carbon content beyond the Fe solubility limit (0,08 % wt) means higher precipitation of cementite (Fe_3C) and consequently a higher presence of pearlite (lamellar structure of cementite and ferrite).

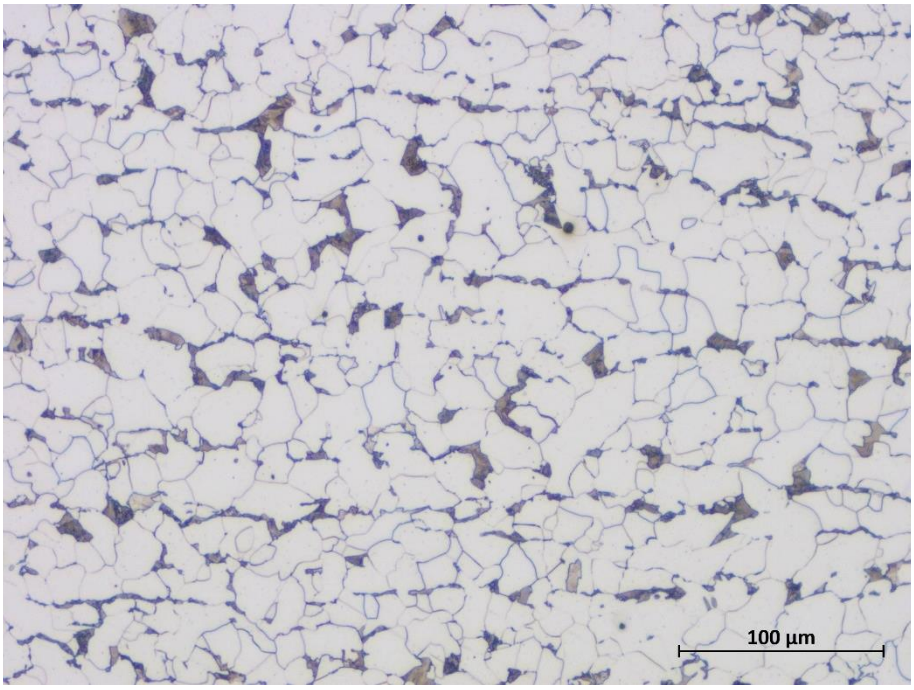


Fig. 3.5.2 – Carbon steel for boiler and superheater tube (C = 0,2 % wt). Ferrite and pearlite. Etchant: Nital 2%, Source: IIS Laboratory

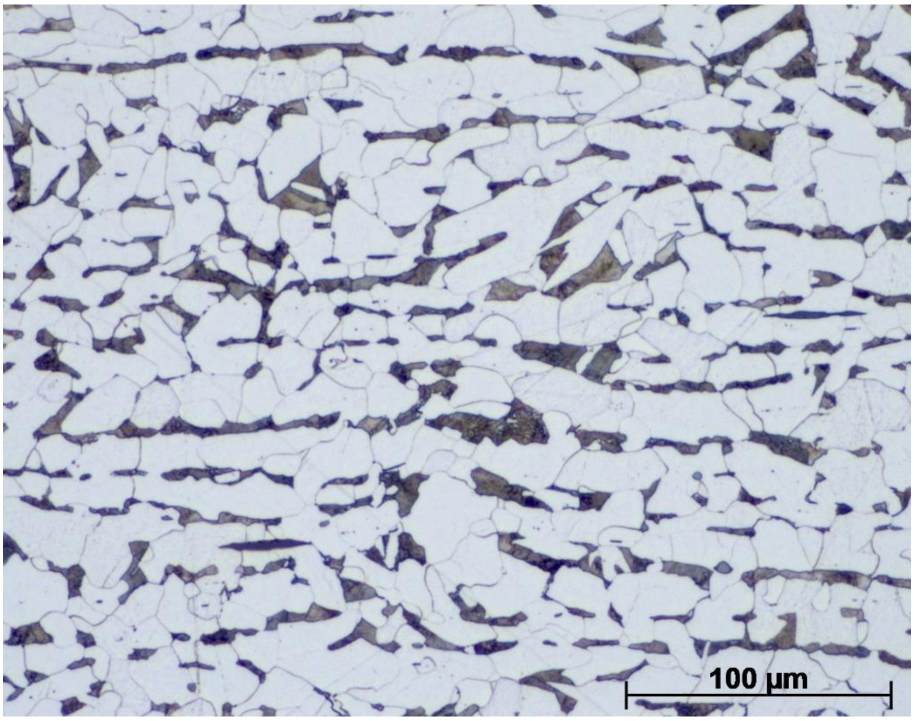


Fig. 3.5.3 – Carbon steel for structural application type S355J0 (C = 0,25 % wt). Ferrite and pearlite. Etchant: Nital 2%. Source: IIS Laboratory

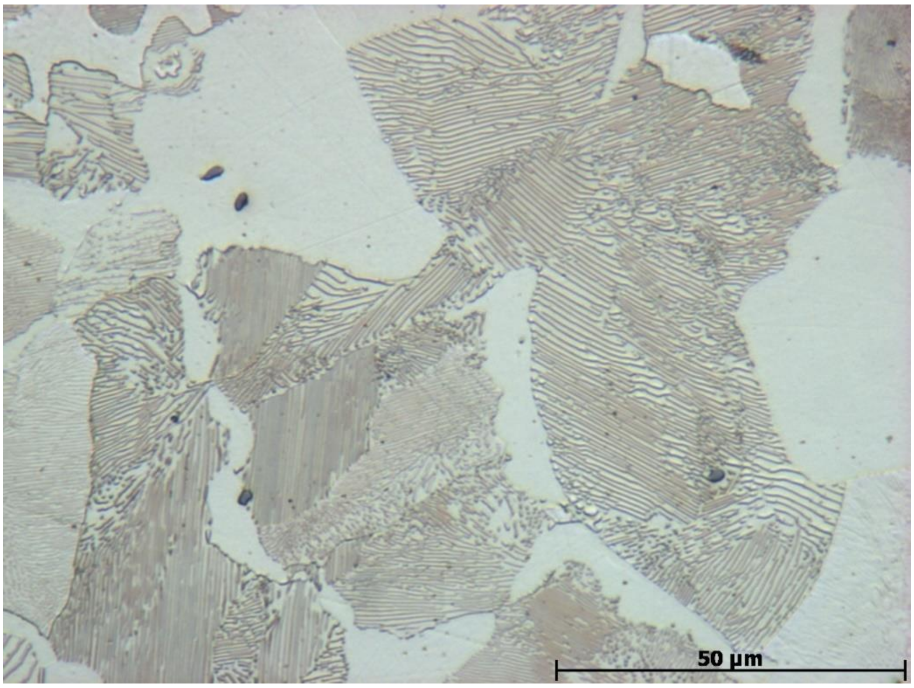


Fig. 3.5.4 – Detail of pearlite at high magnification (500x) – Pearlite consists of alternating flakes of ferrite (white) and cementite (grey/brown). Etchant: Nital 2%. Source: IIS Laboratory

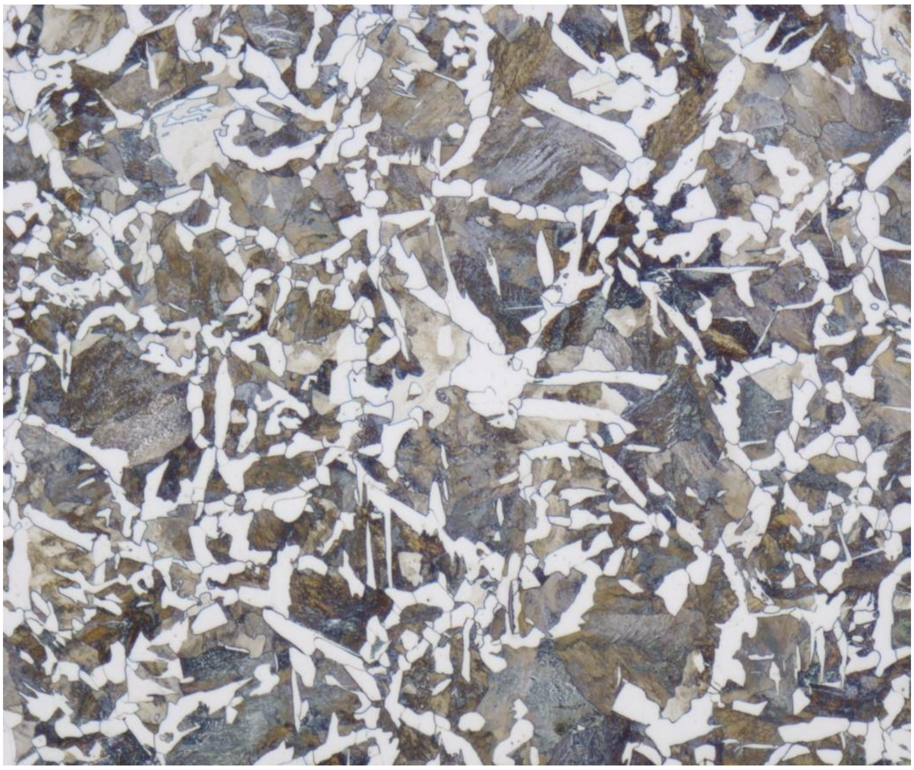


Fig. 3.5.5 – Detail of a medium carbon steel type C45 (C = 0.45 % wt) – Pearlite and ferrite isles. Etchant: Nital 2%. Source: IIS Laboratory

The different allotropic forms of iron and the presence of carbon and other alloying elements allow transformations of the structure subjected to thermal cycles due to heat treatments or the execution of welding.

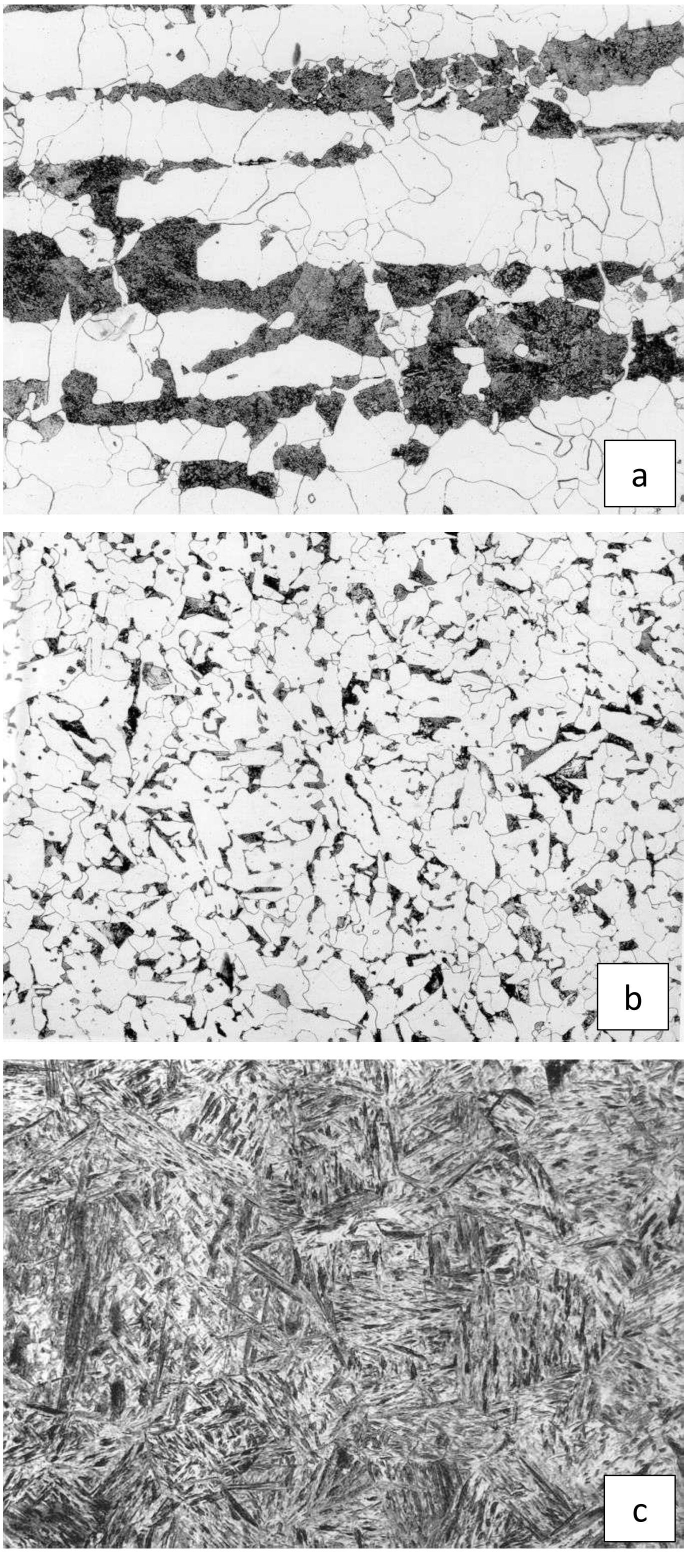


Fig. 3.5.6 – Details of structural steel type S355J2 with different heat treatments. (a) Annealed; (b) Normalized; (c) Quenched. Etchant: Nital 2%. Source: IIS Laboratory.

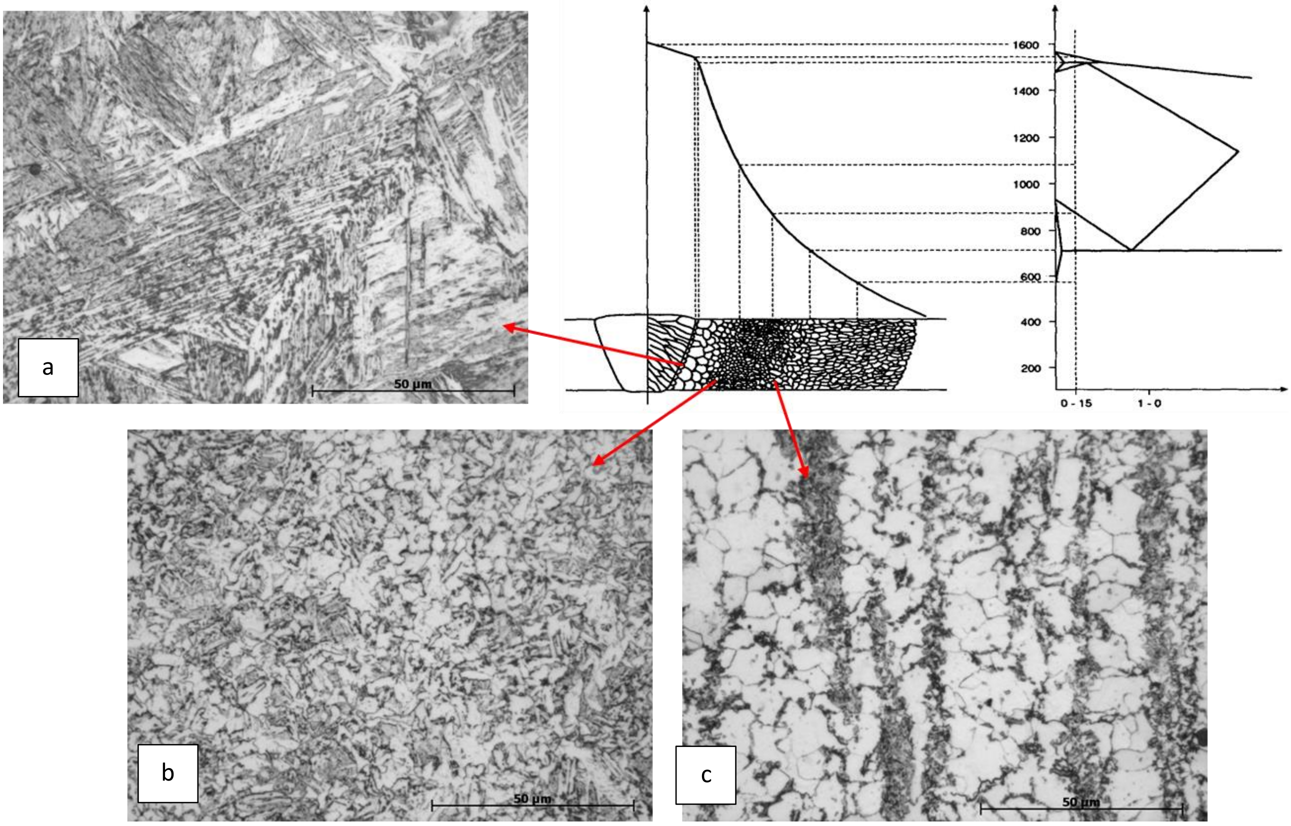


Fig. 3.5.7 – Details of heat affected zone of a low alloy steel welding joint. (A) Coarse grain zone; (b) Fine grain zone; (c) Partial trasformation zone - Nital 2%. Source: IIS Laboratory.

3.5.2. Austenitic Stainless Steel

By adding alloying elements to the steel, the balance of the phases and the final structure can be modified. Some elements stabilize the ferritic phase (Chromium equivalent), while others stabilize the austenitic phase (Nickel equivalent). The most important family of high alloyed steels are stainless steels.

If large amounts of austenite stabilizing elements (carbon, nickel, manganese, nitrogen) are present, then austenite can be observed at low temperature.

Austenitic stainless steels and austenitic manganese steels are fully austenitic alloys. These are single phase alloys. Austenite is also a soft low strength phase; however, cold working produces hardening.

In austenitic stainless steel (AISI 300 series), solid-state allotropic transformation is suppressed; and there aren't any transformations due to thermal cycling; thus, only a grain enlargement is observed in the heat affected zone (HAZ).

Typically, the weld zone has a dendritic structure with about 5-10 % in delta ferrite. The presence of delta ferrite is very important because it improves the hot cracking resistance when welding.

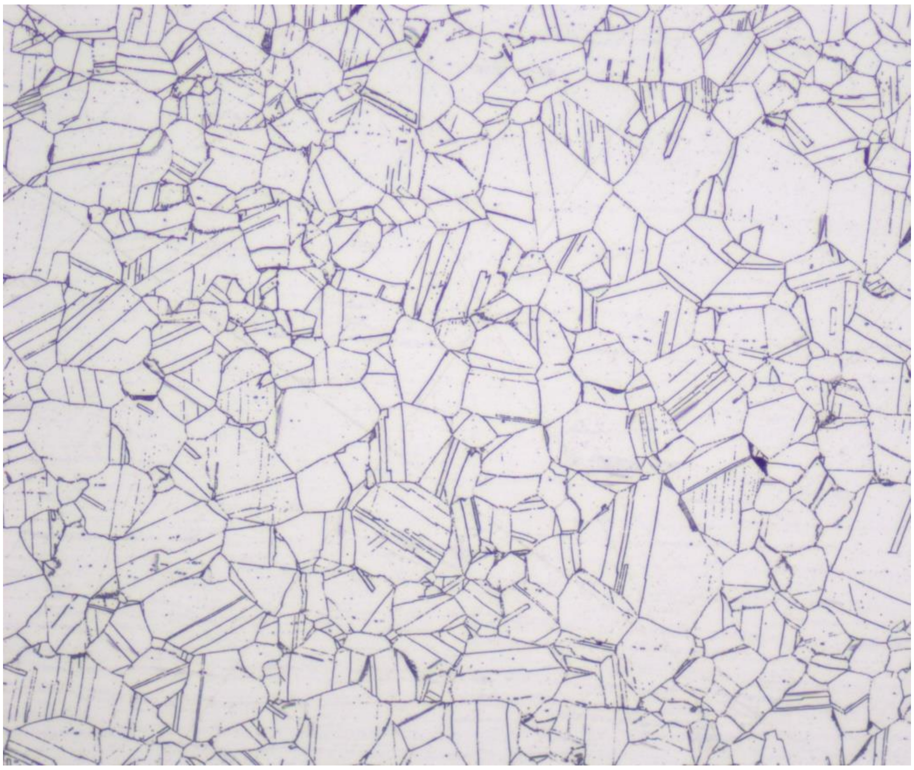


Fig. 3.5.8 – Detail of fully austenitic structure (100x) – Polygonal austenitic grains. Etchant: Electrolytic Oxalic acid solution. Source: IIS Laboratory

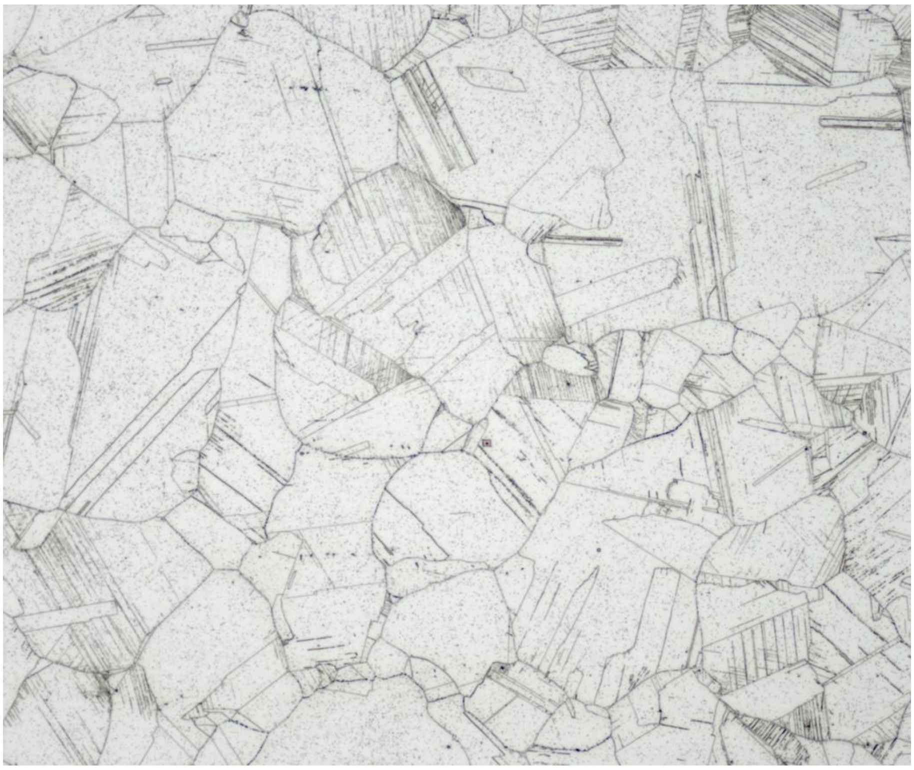


Fig. 3.5.9 – Detail of fully austenitic structure (100x) with hardening signs – Polygonal austenitic grains. Etchant: Electrolytic Oxalic acid solution. Source: IIS Laboratory

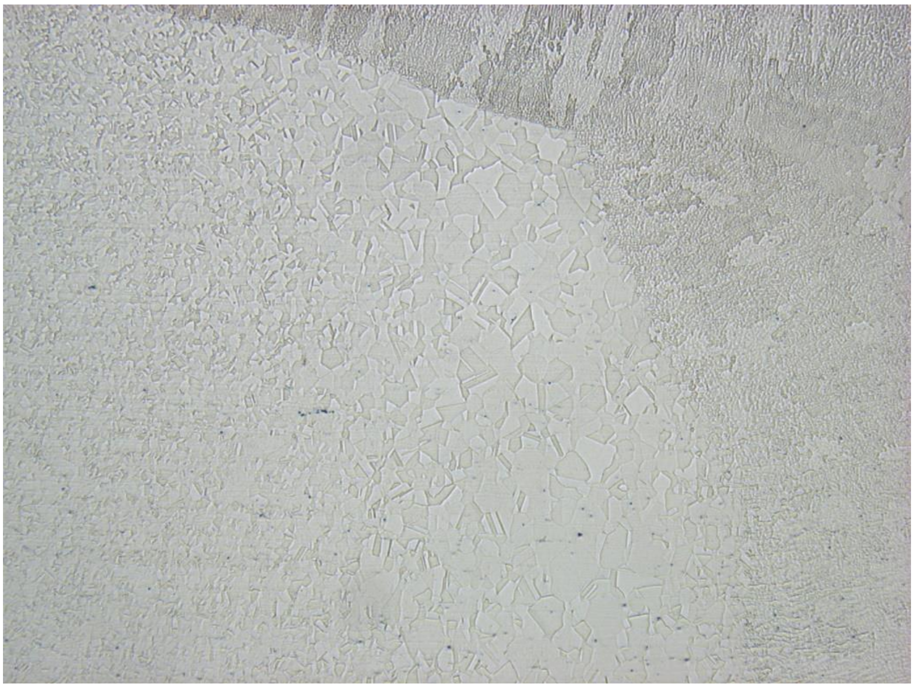


Fig. 3.5.10 – Heat affected zone (HAZ) of austenitic stainless-steel type AISI 316 (25 x). Coarsened grains in HAZ. Source: IIS Laboratory

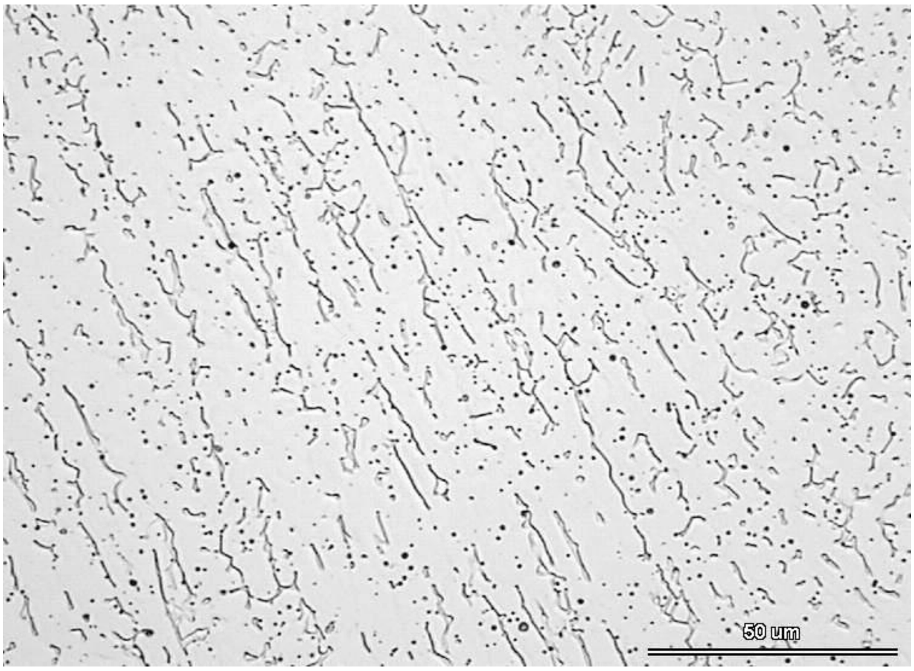


Fig. 3.5.11 – Weld zone (WZ) of austenitic stainless-steel type AISI 304. Austenite dendritic structure with delta ferrite. Etchant: Electrolytic Oxalic acid solution- Source: IIS Laboratory

Austenitic stainless steels may become sensitised if they are heat-treated or used at temperatures in the range 500 – 850°C. The heat-affected zones of welds may also be sensitised in some circumstances. Chromium carbides are visible as dark points at the grain boundaries.

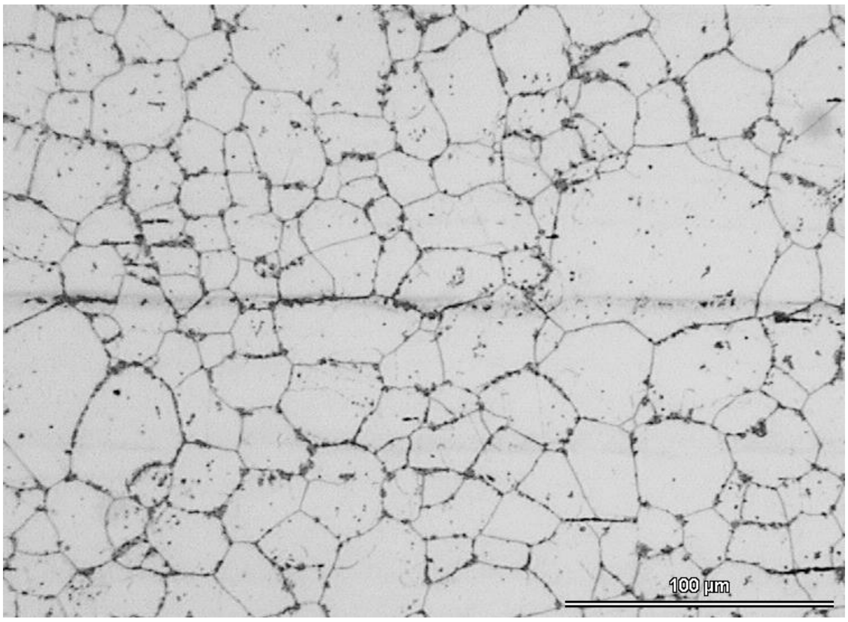


Fig. 3.5.12 – Detail of fully austenitic structure (200x) with chromium carbides at the grain boundaries (sensitization). Etchant: Electrolytic Oxalic acid solution- Source: IIS Laboratory

Sigma phase is a non-magnetic intermetallic phase composed mainly of iron and chromium, which forms in ferritic and austenitic stainless steels during long exposure at 550 – 950 °C; this is a problem that occurs during operation or heat treatments and not during welding.

Sigma phase is hard and brittle. and when present in stainless steels is usually undesirable. By electrolytic caustic etching (e.g. NaOH or KOH solution) we can detect the sigma phase, thus exhibiting dispersed globular morphologies.

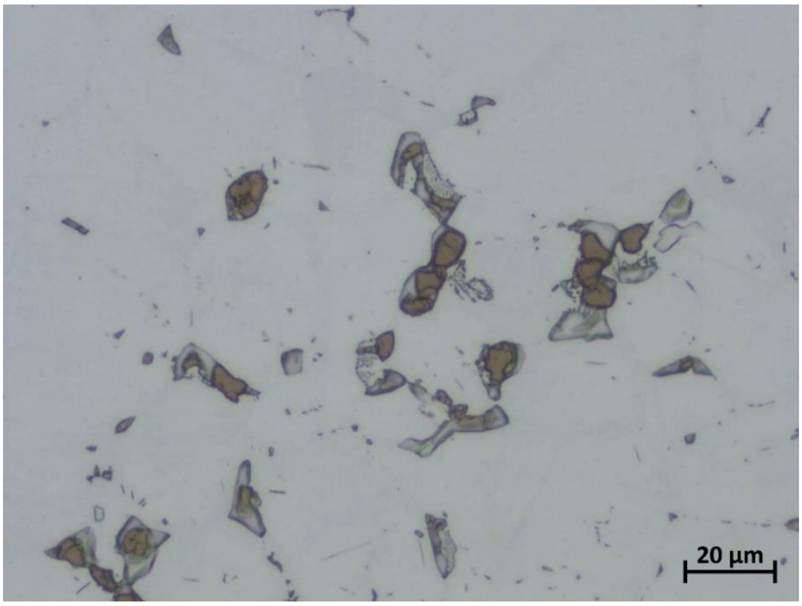


Fig. 3.5.13 – Sigma phase on austenitic stainless-steel type 304H. Etchant: Electrolytic NaOH solution. Source: IIS Laboratory

3.5.3. Duplex Stainless Steel

Duplex stainless steels are based on Fe-Cr-Ni-N alloy system. The chemical composition of these alloys has been adjusted such that the base metal microstructure usually consists of about 50% ferrite and 50% austenite.

Because of the complex alloying of the duplex grades, a number of precipitation reaction can occur over a range of temperatures from below approximately 1000 °C. Many of these precipitates embrittle the duplex alloys and are to be avoided. These include sigma (σ), chi and alpha (a) prime, as well as chromium nitrides.

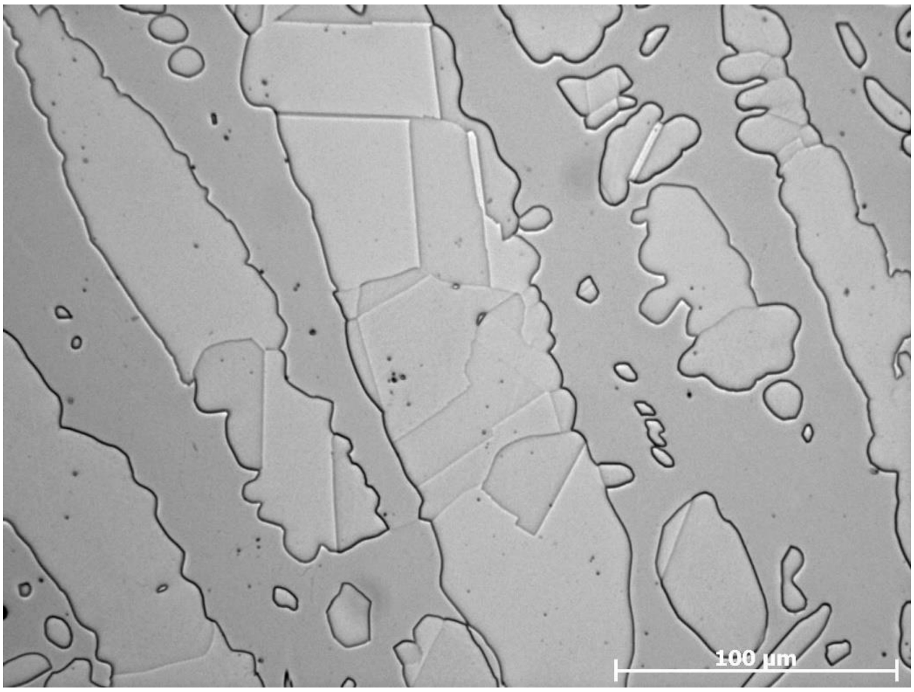


Fig. 3.5.14 – Duplex stainless steel – biphasic structure of ferrite and austenite. Etchant: Electrolytic Oxalic acid. Source: IIS Laboratory

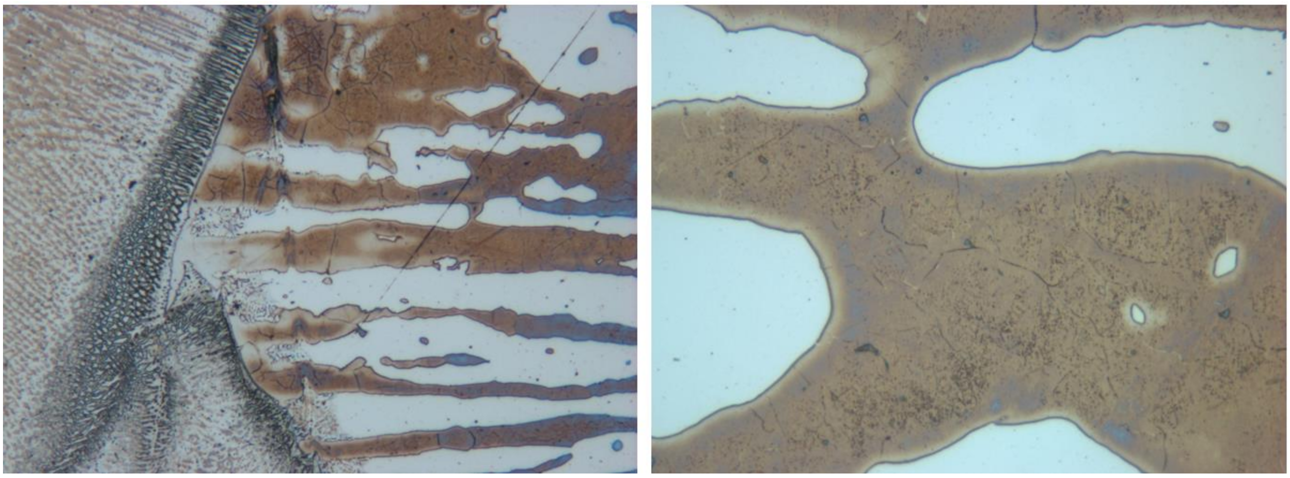


Fig. 3.5.15 – Details of the Heat Affected Zone of a duplex stainless (2205). Presence of intragranular chromium nitrides in ferritic phase. Etchant: Electrolytic NaOH solution. Source: IIS Laboratory)

3.5.4. Martensitic structure

Martensite is the generic term for microstructures formed by diffusion less phase transformation. Martensitic transformations occur in steel and iron-base alloys as well as several nonferrous systems. Martensite usually is produced in steel or iron-base alloys by rapid quenching of austenite and forms by a shear mechanism. The final structure is fine and acicular; in some case it is possible to observe the primary austenitic grain boundary.

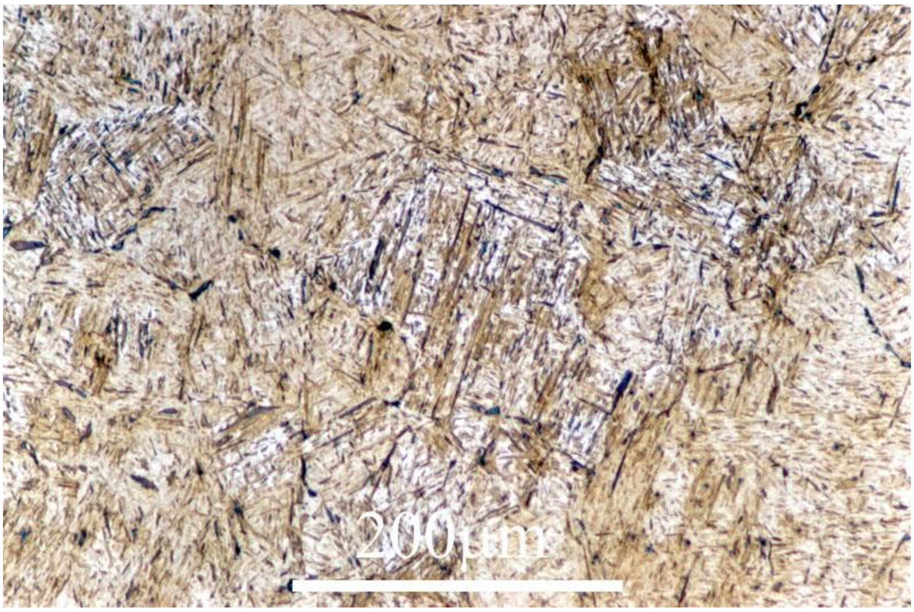


Fig. 3.5.16 – Details of tempered medium carbon steel. Etchant: Nital 2%. Source: IIS Laboratory

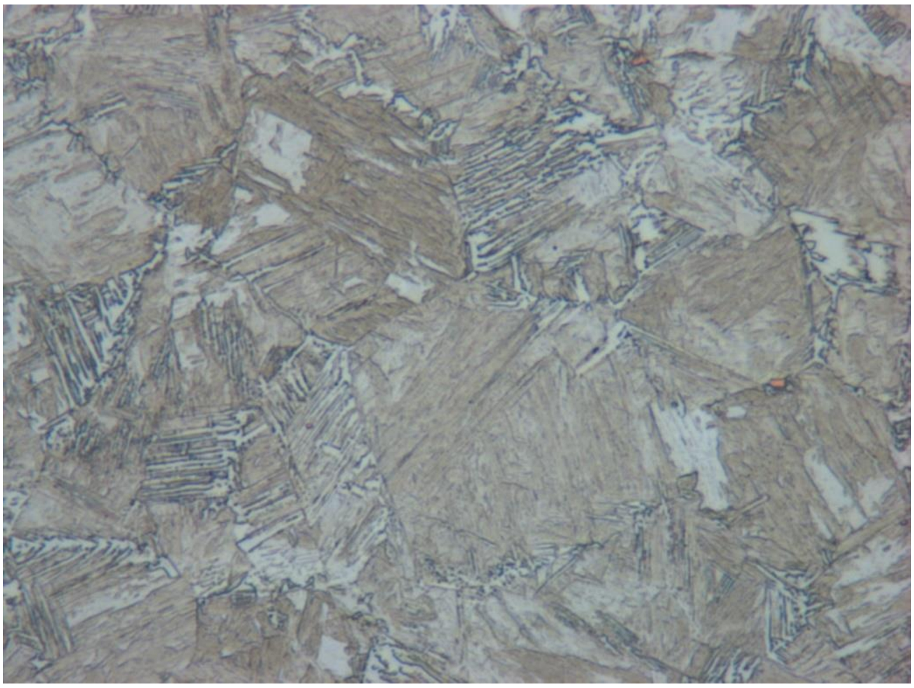


Fig. 3.5.17 – Details of Chromium Molybdenum steel (P91). Etchant: Vilella. Source: IIS Laboratory

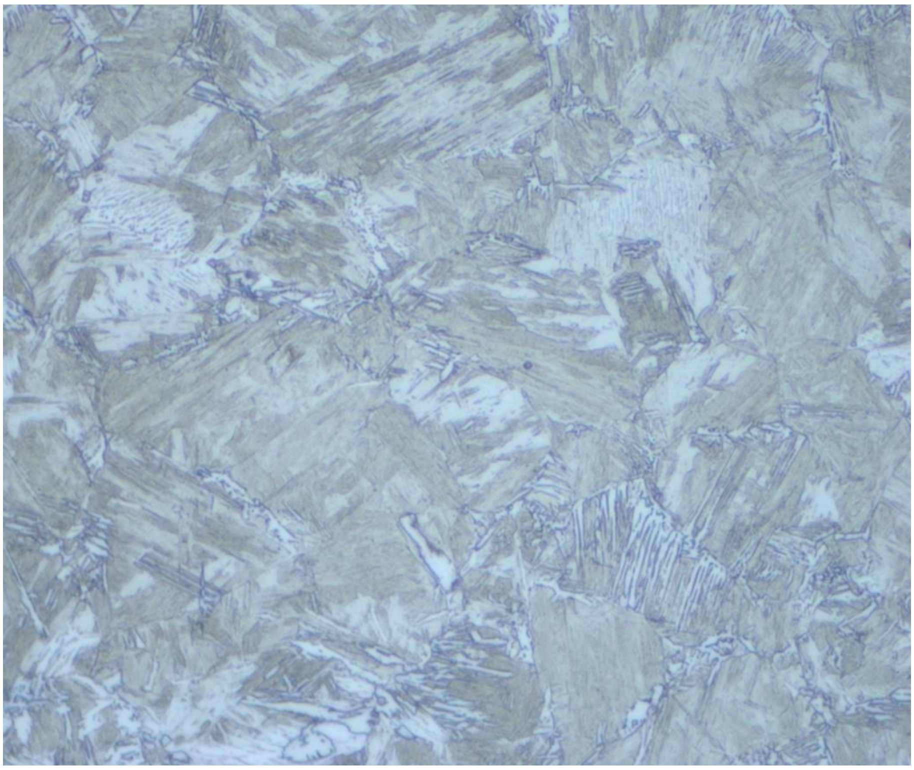


Fig. 3.5.18 – Details of a ballistic steel (Secure 500). Etchant: Nital 2%. Source: IIS Laboratory.

References:

[3.5.1] ASM HANDBOOK – Volume 9 – Metallography And Microstructures

[3.5.2]Welding Metallurgy and Weldability Stainless Steels – John C. Lippold and Damian J. Kotechi.

3.6. Demonstration of different kinds of microscopic structures

3.6.1. Weld analysis

Macro examination is commonly carried out on unmounted cross-sections through welded joints and involves cutting and coarse/fine grinding techniques. The resultant finish is adequate for etching, followed by an examination of the macro features of the weld joint.

Some of them are listed below:

- Weld geometry
- Number and size of passes
- Depth of penetration
- Extent of HAZ (heat affected zone)
- Surface imperfections such as cracks, undercut, excessive throat, convexity, and weld toe angle.
- Internal imperfections such as cracks, porosity, metallic inclusions, lack of fusion, lack of penetration, and slag
- Joint geometry imperfections such as root gap, root face, angle of chamfer, and misalignment

For micro examination techniques, the provision of a polished, optically flat surface will be required. Microscopy examination is carried out for studies of weld imperfections and microstructural features such as:

- Voids (porosities, shrinkage cavities, micro-cracks)
- Special phases (sigma-phase in stainless steel)
- Grain sizes/structure
- Weld structure
- Segregation
- HAZ and base structure

3.6.2. Phase analysis

The definition of the phase in the case of the metallic material is the next: the part of the system that is homogeneous, has chemically and physically identical properties and can be separated by an independent boundary surface is called a phase. Metal phase analysis is required to characterize different phases in metallic materials. Phase analysis is a special technique used in welding engineering and materials science to determine a sample's phases by way of different methods. It can be conducted via several methods, including coloured etching, X-ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy (TEM), depending on the required outcomes. Metal phase analysis is vital for gaining information about specific welded and base materials.

The phase analysis result of the base metal, the HAZ and the joint gives information on the waited-for mechanical properties and the welded structure appropriateness. Example: in the HAZ of a welded low-carbon steel, the martensite phase is unwanted. Another example is the precipitations in HAZ of an Austenitic stainless steel. The phase analysis can also inform us in the case of carbon steels about the austenitization method (austenite grain sizes) because after the cooling even if it happens a phase transformation (ferrite, perlite) the grains keep the grain size of the previous austenite.

3.6.3. Porosity analysis

In the welded joint several times can be porosity. The reason for the porosity of the absorption of nitrogen, oxygen, and hydrogen in the molten weld pool which is then released on solidification to become trapped in the weld metal. The best way to avoid porosity the clean the workpiece before the welding. The Table summarizes some kinds of metals and the gases causing porosity.

Table 3.6.1. The gases causing porosity

Metal	Gas
C-Mn steel	H, N, O
Stainless steel	H
Aluminium and alloys	H
Copper and alloys	H, N
Nickel and alloys	N
Titanium and alloys	H, N, O

The porosity can be found in the joint (Figure 3.6.1.) or near the surface (Figure 3.6.2.) of the welded joint.

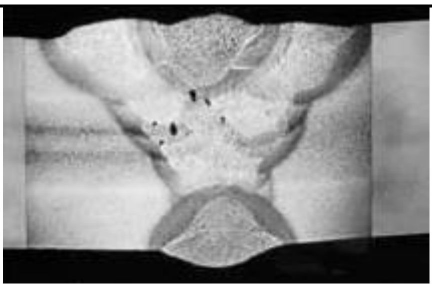


Figure 3.6.1. Uniformly distributed porosity

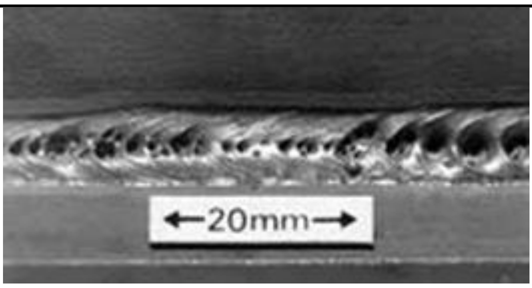


Figure 3.6.2. Surface breaking porosity

Several non-destructive tests are suitable as penetrant or magnetic particle inspection, ultrasonic inspection, X-ray test, etc. The destructive test as a microscopic examination is suitable (Figure 3.6.1.).

3.6.4. Decarburization

Decarburization is a chemical process when the Carbon of the steel joins with oxygen. This process is established at high temperatures (above 700 °C) (Figure 3.6.3.). The problem with this process the steel carbon content decreases. The process is made by diffusion and diffusion is also a limit of this process. The decarburization usually can happen on the steel surface and in a thin layer under the surface. The decarburized layer has a negative impact on the microstructure and mechanical properties of the welded joint and can cause cracking in the border of the joint and the base metal.

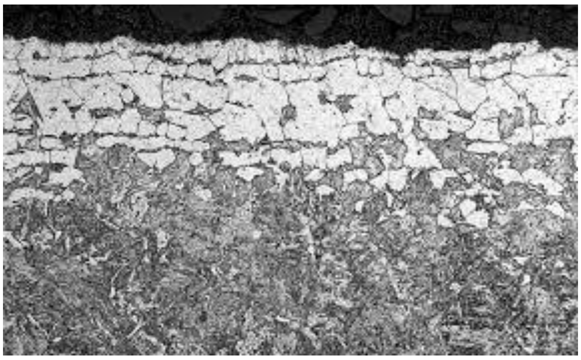


Figure 3.6.3. Decarbonized surface of C-Mn steel

3.6.5. Copper structures

The copper and alloy structure depends on the manufacturing process (casting, rolling, etc.). The casted copper structure shows a dendritic structure, and the rolled and annealed structure shows a small grains structure. The copper can form oxides in the structure which is detectable by microscopy test.

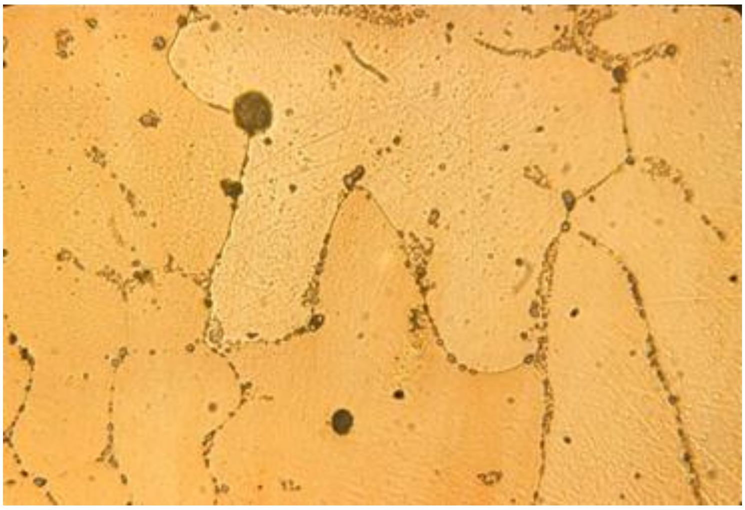


Figure 3.6.4. Copper microstructure with oxides

3.6.6. Aluminium structures

Aluminium and alloy structures depend on the manufacturing process (casting, rolling, etc.). The casted copper structure shows a dendritic structure, and the rolled and annealed structure shows a small grains structure. Aluminium forms an oxide layer on the surface of the workpiece. In the case of casted aluminium, it can find porosity on the surface of the porous, thin oxide layer. During the welding, the melted aluminium reacts with oxygen and establishes oxides or porosity. Oxide layer thickness with time, temperature and available oxygen increase with quantity. The cross-section of an aluminium workpiece is shown in Figure 3.6.5.

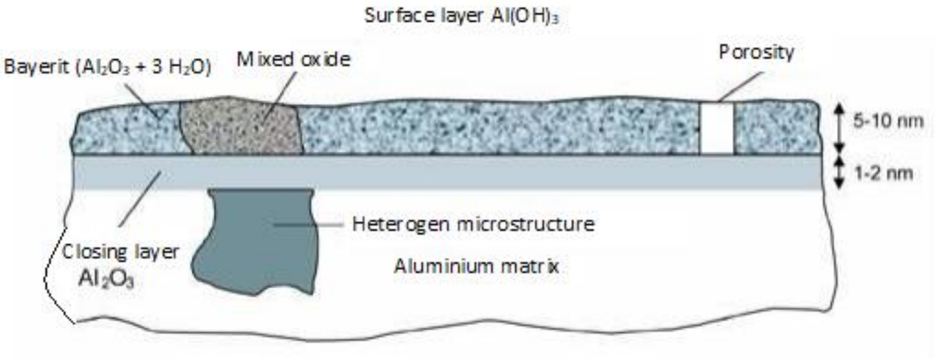


Figure 3.6.5. Cross section of an aluminium workpiece

The heat treatment in the case of the special aluminium alloy can modify the surface structure. The solution treatment and the age precipitation hardening heat treatment illustration are shown in Figure 3.6.6.

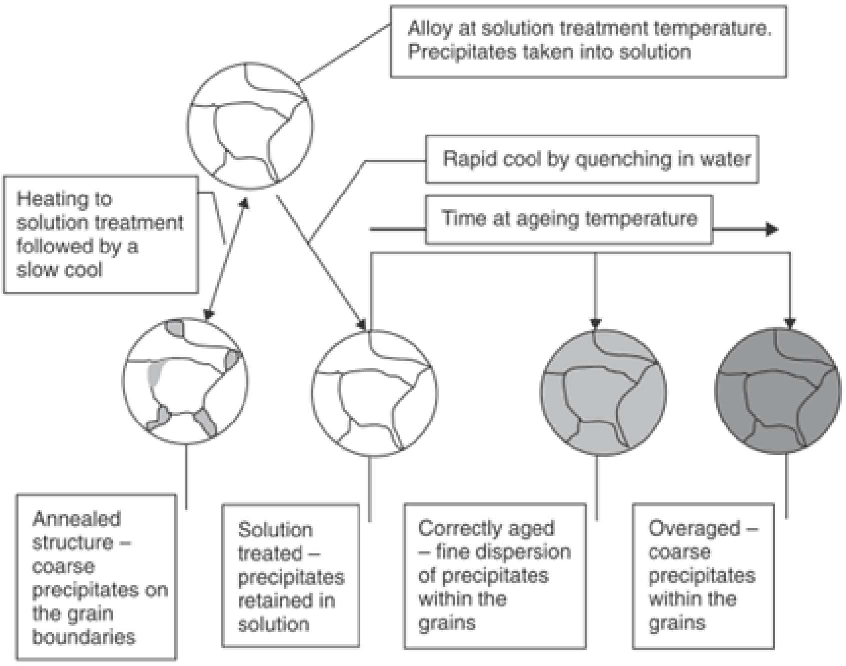


Figure 3.6.6. The heat treatment affected structures

3.6.7. Cast iron structures

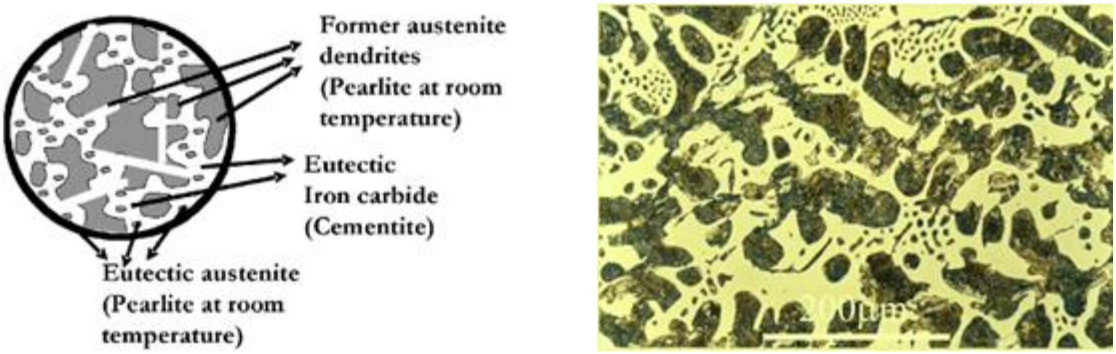
The metallurgy of cast iron is much more complex than its economics. Cast iron is one of the more complex metallurgical systems. It is interesting to note that iron-carbon alloys (with less than

2% carbon) are metastable. The true stable system is the iron-graphite phase transformation system (Fe-C). The solid-state transformations, on which cast-iron heat treatments are based, are similar to those applied to steels and can be characterized as:

- On the cooling of hypoeutectic irons, at a temperature just below 1147 °C the microstructure consists of proeutectic austenite and ledeburite. On further cooling, excess carbon comes out as cementite from proeutectic and eutectic austenite. Finally, at 727 °C both eutectic and proeutectic austenite decomposes into pearlite with a carbon content of approximately 0.77%.
- On cooling of eutectic cast irons, ledeburite is formed. On further cooling, excess carbon comes out of the solution as cementite from eutectic austenite. Finally, at 727 °C eutectic austenite decomposes into pearlite.
- On the cooling of hypereutectic irons, at a temperature just below 1147 °C, the microstructure consists of proeutectic austenite and ledeburite. On further cooling, excess carbon comes out as cementite in the form of proeutectoid cementite and eutectic austenite. Finally, at 727 °C eutectic austenite decomposes into pearlite.

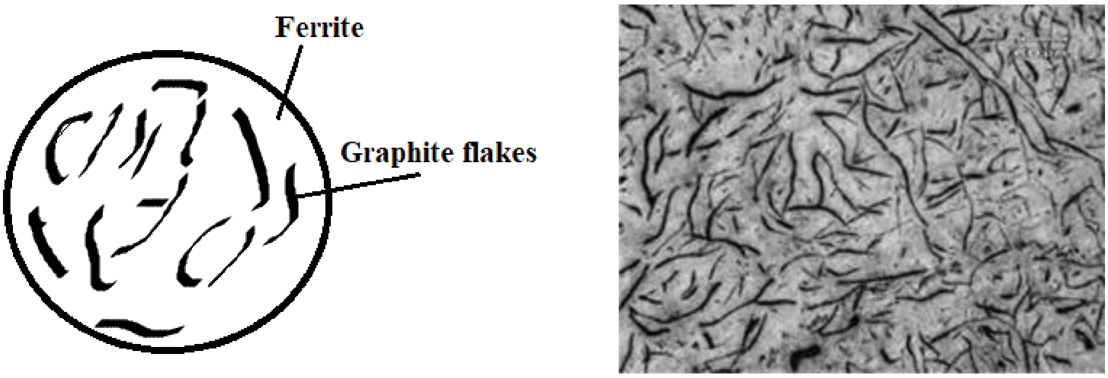
If an iron alloy exceeds 2.11% carbon, the carbon does not have to nucleate from the decomposition of austenite, but it can instead form directly from the melt by a eutectic reaction. Note that cementite (Fe_3C) can still nucleate at the eutectic more readily than graphite, but on sufficiently slow cooling, graphite itself can form and grow.

On the base of the solidification process, we divided into two groups. White cast irons (Figure 3.6.7.) crystallize according to the metastable Fe-C system and the grey cast irons (Figure 3.6.8.) crystallize according to the stable Fe-C system.



a) b)

Figure 3.6.7. White cast iron a) schematic representation, b) microscopy picture



a) b)

Figure 3.6.8. Grey cast iron a) schematic representation, b) microscopy picture

The white cast iron structure can be modified with a heat-treating process. The malleable cast iron is made by heat treatment. This conversion, in usual practice, is a two-step process commonly referred to as the first and second stages of graphitization. The first stage of a typical heat-treating cycle consists of heating the castings at a temperature between 900 to 950°C. The pearlite transforms into austenite by a process of rapid diffusion of the carbon from cementite into the iron to form a solid solution of carbon in gamma iron. The massive carbides are virtually unaffected by heating. Therefore, above the eutectoid range (but below the eutectic) the iron consists of massive carbides in austenite. After the first stage of graphitisation, the structure of a malleable cast iron consists of austenite and graphite (Figure 3.6.9.).

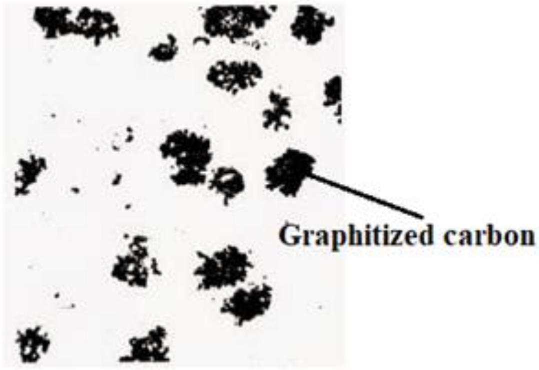


Figure 3.6.9. Ferritic malleable cast iron

References

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3.6.2. Welding for design engineers, CWB Group, Canadian Welding Bureau 2006. ISBN 0-9739175-0-4

3.6.3. John C. Lippold; Welding Metallurgy and Weldability, John Wiley & Sons, Inc, Canada, 2015.

3.7. Demonstration of specimen preparation manners for metallographic examination

3.7.1. Macroscopic testing of metallographic specimen preparation steps

1. Sampling: A suitable size test specimen needs to be cut (see Chapter 1.2.1.) slowly under water cooling (Figure 3.7.1.). Water jet cutting can be used, if available.

Figure 3.7.1. Mechanical cutting under water cooling

2. Mounting: It depends on the specimen size and geometry. In the case of macroscopic examination, the sample, which can be held in hand, can be prepared without mounting (mounting is seen in Chapter 1.2.2.).

3. Marking: The test specimen needs to be marked to be identified during the preparation process (see Chapter 1.2.3.).

4. Grinding: Grinding with SiC paper step by step on finer grain paper as described in chapter 1.2.5.

Figure 3.7.2. Grinding

5. Cleaning: washing under water, followed by air blowing (see Chapter 1.2.4.)

6. Etching: if needed, according to the type of the intended analysis (see Chapter 1.2.8.).

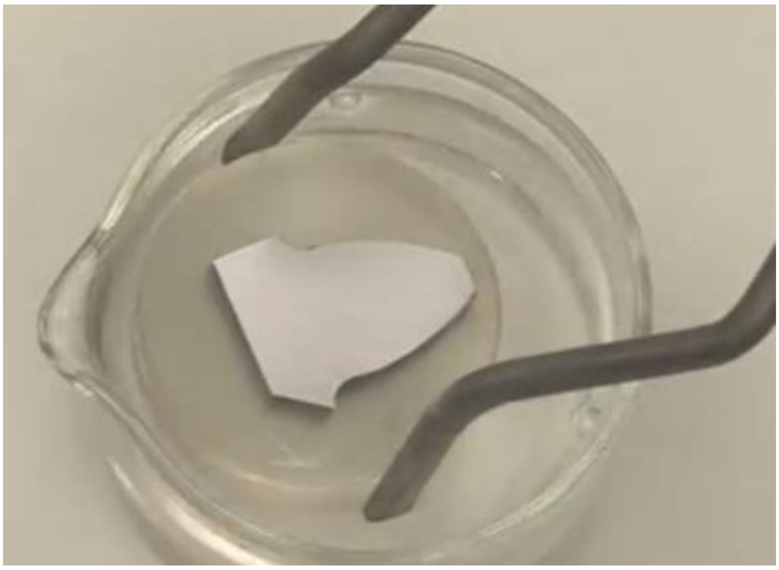

Figure 3.7.3. Etching

7. Cleaning: washing with water and followed by cleaning with alcohol (see Chapter 1.2.4.). In the case of polymer specimens only water cleaning should be used.

Cleaning with water

Cleaning with Methanol

Figure 3.7.4. Cleaning

8. Drying: Drying should take place in a stream of mild air, in the case of polymers with ambient air temperature (see Chapter 1.2.4.).

3.7.2. Microscopic testing of metallographic specimen preparation steps

1. Sampling: A suitable size test specimen needs to be cut, usually a maximum 25x25x25 mm^3, or a maximum 25 mm diameter and 25 mm high cylinder (see Chapter 1.2.1.), cut slowly under water cooling (Figure 3.7.1.) or use water jet cutting.

2. Cleaning: washing with water and after with Methanol (see Chapter 1.2.4.), in the case of polymer specimens only water cleaning is required. It can also be cleaned by ultrasonic bath.

3. Drying: Drying should take place in a stream of mild air (see Chapter 1.2.4.).

4. Mounting: hot or cold mounting (mounting is seen in Chapter 1.2.2.).

Figure 3.7.5. Mounting

5. Marking: The test specimen needs to be marked to identify it during the preparation process (see Chapter 1.2.3.).

6. Grinding: Grinding with SiC paper, step by step on finer grain paper (Figure 3.7.6.) (see Chapter 1.2.5.).

Figure 3.7.6. Grinding

7. Cleaning: washing with water (see Chapter 1.2.4.).

8. Polishing: Polishing in a polishing pad with alumina or diamond abrasive particles, step by step on finer abrasive particles, electrolytic polishing can also be used (see Chapter 1.2.6. and Chapter 1.2.7.).

9. Cleaning: washing with water and after with alcohol (see Chapter 1.2.4.).
10. Drying: drying should take place in a stream of mild air (see Chapter 1.2.4.).
11. Etching: with a suitable etchant (Figure 3.7.3.) (see Chapter 1.2.8.).
12. Cleaning: washing with water and after with alcohol (see Chapter 1.2.4.).
13. Drying: drying should take place in a stream of mild air (see Chapter 1.2.4.).