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Preparation of metallographic specimens

Confection des éprouvettes métallographiques

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Foreword

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This document was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 7, *Methods of testing (other than mechanical tests and chemical analysis)*.

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Preparation of metallographic specimens

1 Scope

This document presents a list of common practices in preparation methods of metallographic specimens for optical and scanning electron microscopy, including preliminary preparation, grinding and polishing of specimens as well as microstructure revelation methods covering the optical method, etching methods (chemical, electrolytic, constant potential, ion sputtering and high temperature relieving) and the interface layer method [1][2].

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Preliminary preparation

4.1 Selection of metallographic specimens

4.1.1 General

Because the metallographic examination serves a specified purpose that differs from case to case, there is no one single way to select and prepare specimens. However, it is the accepted state of the art to select specimens that are representative of the material that is being studied. The location, type of section and number of the specimens to be studied are usually dictated by the manufacture method of metals, examination intent or purpose, related standards or agreement upon enquiry.

4.1.2 General studies or routine work

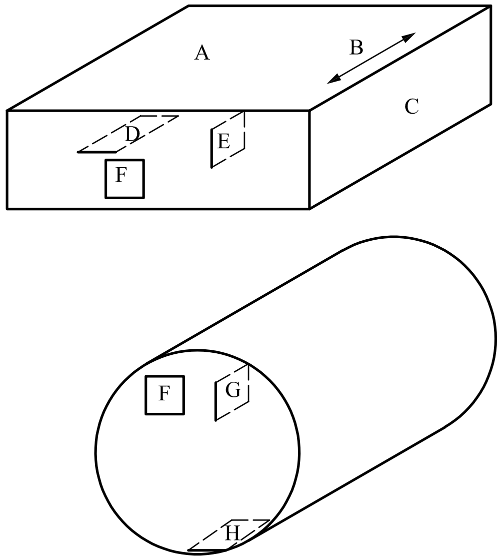
Specimens are generally chosen from locations most likely to reveal the maximum variations within the material under study [3]. For example, specimens could be taken from a casting in the zones wherein maximum segregation might be expected to occur as well as specimens from sections where segregation could be at a minimum [4]; in the examination of strip or wire, test specimens are often taken from each end of the coils [5]; heat or surface treated specimens are often taken so as to include all the heat or surface treated layers [6]; welding specimens often incorporate the welding seam, heat affected zone and base metal [7].

4.1.3 Study of failures

In nearly all situations, test specimens are taken as closely as possible to the fracture or to the initiation of the failure. Before taking the metallographic specimens, study of the fracture surface is completed, or at the very least, the fracture surface is documented [8]. In many cases, specimens are taken from a sound area for a comparison of structures and properties.

4.2 Selection of type of section to be examined

4.2.1 Having established the location of the metallographic samples, the type of section to be examined is decided. The locations of surfaces examined are always given in reporting results and in any illustrative micrographs. A suitable method of indicating surface locations is shown in Figure 1.



Key

A rolled surface
B direction of rolling
C rolled edge
D planar section
E longitudinal section perpendicular to rolled surface
F transverse section
G radial longitudinal section
H tangential longitudinal section

Figure 1 — Method of designating location of area shown in photomicrograph [9]

4.2.2 Transverse sections or cross sections (Surface F) taken perpendicular to the main axis of the material are often used to reveal the following information:

a) Variations in microstructure from surface to center;

b) Distribution of nonmetallic impurities across the section [10][11][12];

c) Distribution of carbide net;

d) Depth of surface imperfections;

e) Depth of coatings;

f) Depth of decarburization [13][14];

g) Depth of corrosion;

h) Surface chemical heat treatment and microstructure and thickness of coating.

4.2.3 Longitudinal sections (Surface D, E, G and H) taken parallel to the main axis of the material are often used for revealing the following information:

a) Inclusion content of steel [10][11][12];

b) Degree of plastic deformation, as shown by grain distortion;

c) Banding in the structure [15];

d) The microstructure attained with any treatment.

4.2.4 In hot-worked or cold-worked metals, transverse and/or longitudinal sections are studied. Special investigations require specimens with surfaces prepared parallel to the original surface of the product. In the case of wire and small rounds, a longitudinal section through the centre of the specimen proves advantageous when studied in conjunction with the transverse section.

4.3 Size of metallographic specimens

Specimens to be polished for metallographic examination are generally not more than 400 mm^2 in area for the section to be prepared[16]. The height perpendicular to the section to be prepared is generally no greater than the transverse size of the specimen and is often dictated by the sample preparation equipment available.

4.4 Cutting of metallographic specimens

There is no single ideal technique to section specimens. Possible techniques include wheel cutting, linear cutting, mechanical machining (turning, milling, planing, grinding), sawing, shearing, flame cutting, hammering for the hard and brittle metal, etc. It is the accepted state of the art to select a technique that minimizes alterations to the structure of the metal, such as deformation and overheating of specimens. Aside from the choice of technique, strategies to reduce sample damage include adapting the machine parameters, using coolant or lubricant, and removal of this damage by subsequent preparation steps (such as by grinding wheel).

4.5 Marking of metallographic specimens

Marking of metallographic specimens is made right after the cutting in order to trace the specimens during their preparation. Marking is not made on the observed surface. Care is taken to avoid the degradation of the marks in the following processes, such as cleaning and heat treatment. The specimen is re-marked after each step that degrades or obscures the previous marking.

4.6 Cleaning

All foreign material on the specimen, such as greases, oils, coolants and residue from cutoff blades, are removed by a suitable solvent (such as ethanol, acetone, etc.). Ultrasonic cleaning is effective in removing the last traces of residues on a specimen surface. Any coating on metal that interferes with the subsequent treatment, etching, or observation of the base metal is removed before polishing.

4.7 Mounting

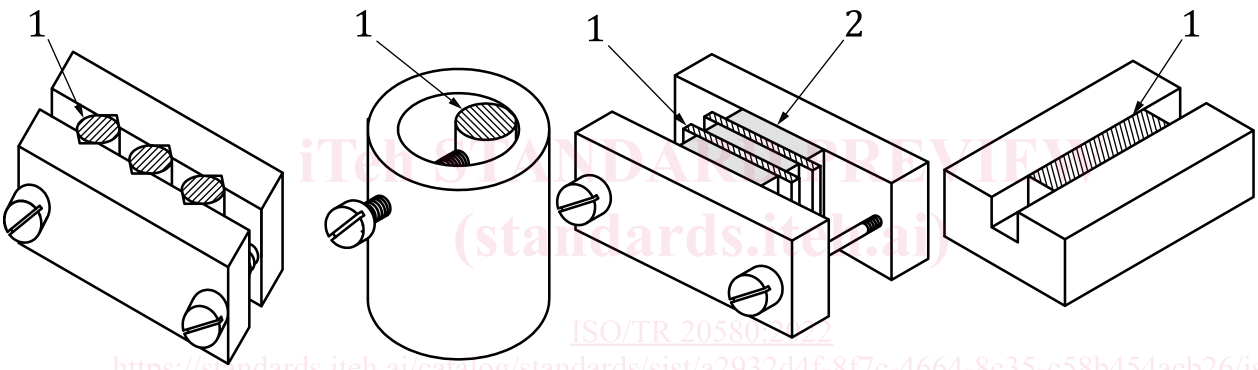
4.7.1 General

Mounting of the specimen is usually performed to simplify and improve the sample preparation. Reasons to mount a specimen can include its handling (small, fragile, soft, or oddly shaped specimens), a need to fixate separate parts (e. g. fractures), a need for edge retention, and a desire for specimen standardization (e. g. for automatic grinding and polishing). The mounting method is chosen so as not to change the microstructure of the specimen. The observed surfaces are generally placed facedown. Specimens are either mechanically mounted, mounted in a support material (usually plastic), or a combination of the two.

4.7.2 Mechanical mounting

4.7.2.1 Mechanical mounting refers to the tight joining of specimens in suitable clamps by bolts and screws, to prevent absorption and subsequent exudation of polishing materials or etchants (see Figure 2). Strip and sheet specimens are mounted by binding or clamping several specimens into a pack held together by two end pieces and two bolts. The clamp is often selected to be of similar hardness and composition as the specimens to minimize the rounding of the edges of the specimens during grinding and polishing as well as any galvanic effects that would affect the polishing process or inhibit etching. Mechanical mounting is often avoided in cases where the clamping pressure presents a risk of specimen alteration.

4.7.2.2 A common aid to minimize the seepage of polishing materials and etchants is the use of filler elements of a softer material. Use of filler material is especially advantageous if the specimens have a high degree of surface irregularities. Filler material is chosen so as not to react electrolytically with the specimen during polishing or etching. Thin pieces of plastic, lead, or copper are typical materials used for this purpose. Copper is especially good for steel specimens since the usual etchants for steels do not attack the copper. Alternatively, the specimens are coated with a layer of epoxy resin before being placed in the clamp in order to minimize the absorption of polishing materials or etchants.



Key

1 specimen

2 filler material

Figure 2 — Mechanical mounting clamps

4.7.3 Plastic mounting:

4.7.3.1 General

Plastic mounting is the most common method for mounting metallographic specimens. The choice of a mounting compound influences the extent of edge rounding observed during the grinding and polishing operations. Strategies to minimize rounding include grouping small pieces of similar hardness around the specimen, reinforcing the plastic with hard filler (e. g. alumina or glass), and plating specimens with metals of lower hardness and resistance to electrochemical reaction. Specimens with a thin surface layer (diffusion layer, metal coating, plating, etc.) are sometimes tilted before mounting to enlarge the visible area of the layer in a specific direction. Plastic mounting is divided into two classes — compression and castable [17].

4.7.3.2 Compression mounting

Compression mounts are prepared in the mould of a mounting press with the observed surface facedown. The height of the plastics introduced into the mould exceeds the height of the specimen before and after mounting. After sealing, heating, pressing, cooling, hardening and opening the mould, a compression mount is accomplished. The temperature and pressure curve of compression mounting are determined based on the plastics selected and the equipment used. Generally, the mounting temperature is not higher than 180 °C and the mounting press not more than 30 MPa (300 bar). The cured mount is usually cooled under full pressure to below 30 °C before ejection from the press. There are 2 types of compression mounting plastics used in the metallographic laboratory:

a) Thermosetting: Diallyl phthalate, Epoxy, Phenolic, etc.

b) Thermoplastic: Acrylic, Polyester acrylate, Epoxy, Polyester, Polystyrene, Polyvinyl chloride, methylmethacrylate, etc.

4.7.3.3 Castable mounting

Castable mounts are usually prepared for specimens sensitive to being altered by heat or being deformed through pressure, such as specimens with a specific thermal history or a low melting point, intricate and porous specimens, or specimens with a large specific surface area. The specimen is placed in the castable mould with the observed surface facedown, then resin and curing agent are combined without forming any bubbles just prior to injection or pouring into the castable mould, after which the mixture cures to form a castable mount at room temperature. Acrylic, Polyester and Epoxy are common castable plastics in use, and the moulds for castable plastics are often simple cups made of hard rubber, polytetrafluoroethylene or cardboard, etc. Porous or intricate specimens are often vacuum impregnated in order to fill voids, prevent contamination and seepage, and prevent loss of friable or loose components.

In special cases, molten metal is substituted for the polymer mix; the mould material is adapted accordingly.

5 Grinding

5.1 Planar or rough grinding

Planar or rough grinding (240 grit and coarser) is performed on belts, rotating wheels or stones, preparing specimens ready for fine grinding. Specimens are usually cooled by water while doing rough grinding in order to avoid changing the microstructure through heat.

5.2 Fine grinding

5.2.1 General

In fine grinding, damage to the specimens incurred from the planar or rough grinding is removed. The specimen is either ground on successively finer abrasive papers or foils (using water or another liquid to wash away grinding debris and to act as a coolant) on a rigid disc usually made of glass or metal, or on a cloth charged with a suitable abrasive.

NOTE The main difficulty in the metallographic preparation of cast iron is to retain the true shape and size of the graphite in its flake, nodular or tempered form. In particular, cast irons with a soft ferritic matrix tend to smear and are prone to deformation and scratching during any stage of the preparation process, as are other soft metallic materials. In these cases, both the samples and the preparation process are often more thoroughly checked because of the increased possibility of inducing artefacts.

5.2.2 Manual methods

When grinding manually, the specimen is moved across the abrasive paper to allow for even wear. Between grinding steps, the specimen is rotated, usually by about 90°. At the end of grinding on each paper, the surface of the specimen and its mount, if any, are flat with one set of unidirectional grinding scratches. Each grinding stage is followed by careful cleaning of the specimen (by water or another liquid or ultrasonic cleaning) to prevent contamination and artefacts from entrained coarser abrasive.

5.2.3 Automated methods

Major advantages of automated grinding and polishing procedures are the consistent quality of specimen preparation and the substantial decrease in time. Abrasive papers from coarse to fine are placed on mechanical grinding device, then the specimen is ground successively. The specimen surface shows uniform scratches before proceeding to the next step. Cleaning between stages is needed to prevent carryover of abrasives and contamination of subsequent preparation surfaces.

6 Polishing

6.1 General

During polishing, damage to the specimens incurred from grinding is removed. There are many polishing methods, such as mechanical polishing, electrolytic polishing, chemical polishing, vibration polishing, etc. The available equipment and operator skills play a significant role in the polishing result and are factored into the specific process definition.

6.2 Mechanical polishing

6.2.1 Rough polishing

Rough polishing is often sufficient for routine evaluations such as micro-indentation hardness and grain size. Typical polishing surface supports include nylon, wool fabric or fine canvas. Abrasives are usually diamond, alumina, magnesium oxide, chromium oxide, ferric oxide, silicon carbide, etc. of usually 1 to 9 μm in size. They are typically supplied as polishing suspension liquids, spray polishing agents or polishing pastes. A typical polishing time is 2 min to 5 min, but it depends on the specimen and the equipment used. The specimens are cleaned by water or another liquid and dried after polishing.

6.2.2 Fine polishing

6.2.2.1 In fine polishing, typical polishing surface supports include nylon silk, velvet, or other fibre velvet. The size and type of abrasive are typically decided in accordance with the hardness of the specimen and the desired end result. Other parameters that influence the end result include the polishing time, the amount of force applied, the rate and direction of movement of the specimen, in order to avoid edge rounding and relief. The specimen is polished until the scratches are completely removed and the observed surface presents a mirror effect. After all polishing has been done, the specimen is cleaned thoroughly by water or another liquid, then cleaned by absolute ethyl alcohol or another suitable liquid with a high vapor pressure and dried, to avoid water stains and contamination [18].

6.2.2.2 Fine polishing can be performed by manual or automated method. When using manual method, the specimen is lightly pressed on the polishing disc and moved back and forth in the direction of the diameter of the polishing disc. The typical duration for the specimen and polishing surface support to be in contact is 10 s to 20 s. The humidity of the support is usually controlled in such a way as to ensure the surface liquid film completely evaporates in 2 s to 3 s after the supply of liquid stops. Excess humidity brings out artifacts such as tailing. Lack of humidity causes a temperature rise of the specimen, decreases lubrication and in certain situations damages the specimen surface. Automated polishing devices move the specimen on the polishing disc following a set track [19]. The clamping force, rotating speed and direction can be adjusted to achieve efficient polishing.

6.3 Electrolytic polishing

For the electrolytic polishing of metal specimens in an appropriate electrolyte, the metal specimens work as anodes, on the surfaces of which the selective corrosion occurs due to the electrolytic reaction and fine polishing is obtained. Satisfactory polishing is the result of a combination of appropriate voltage, current, temperature and polishing time.

NOTE Electrolytic polishing is not generally used for cast irons and other composite microstructures.

6.4 Chemical polishing

The principle of chemical polishing is to unevenly dissolve the surface of a metal specimen using chemical reagents and thus to obtain a mirror surface. This polishing method can only make the specimen surface smooth, but not planar. The chemical polishing is effective to pure iron, aluminum, copper, silver, etc.

6.5 Vibratory polishing

Vibratory polishing is conducted by a system that induces the specimen to move roughly circumferentially on the disc and rotate around its own axis as well. The force applied to the specimen is merely that of its own weight and that of the mounting system. Polishing several specimens simultaneously usually introduces an added element of randomness to the path of the specimen on the disc. This polishing method is usually used to remove residual stress or residual deformation layer on the surface of the specimen [20].

7 Microstructure revelation

7.1 General

There are many ways to reveal microstructures. Some microstructures are revealed right after mechanical polishing. Some microstructures are only properly revealed after physical, chemical or heat treatment. Typical approaches to revealing the microstructure include the optical method, the etching method, and the interference layer method.

7.2 Optical method

The microstructure can be revealed without any treatment after mechanical polishing if different phases exhibit a contrast in colour or reflection characteristics. Specimens can be observed without treatment if the optical microscope is equipped with polarized light, phase contrast, or differential interference accessories. Features such as graphite in gray iron and nodular cast iron, primary silicon of foundry Al-Si alloy, non-metallic inclusions, and micro defects such as cracks and pores, can be directly observed after mechanical polishing.

7.3 Etching method

7.3.1 Chemical etching [13]

The microstructure is revealed by partial chemical or electrochemical dissolution of the specimen surface caused by etchants.

Note 1 to entry The principal etchants for common metals are listed in Annex A.

7.3.2 Electrolytic etching [21]

The metal specimens work as anodes immersed in an appropriate electrolyte. The specimens are etched when inputting a low current and the microstructure is revealed. The etching effect is usually affected by voltage, current, temperature, etching time, etc.