Metallography

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1 INTRODUCTION

The internal structure of metallic materials contains useful information about composition, processing, quality, service history and mechanical properties of these products. Metallographic examination permits determination of macro- and micro-structural details and can yield information not readily obtainable by other methods.

2 SCOPE

This document applies to case working personnel who perform metallography in support of metallurgy examinations.

Techniques for successful metallographic specimen preparation and examination depend on the form and composition of the metal under investigation. Appropriate preparation sequences often must be developed empirically. This procedure outlines the general concerns to be addressed during all metallographic investigations and cites references for specific preparation techniques that have been found to be successful on particular alloy systems. Specimen preparation is typically destructive and requires thoughtful integration into an examination sequence.

3 PRINCIPLE

The internal structures of metal objects retain information on composition, processing history, environmental exposure, and deformation. Metallographic preparation used to reveal these structural details may include the size and shape of the crystals (grains) comprising the material, the presence of multiple phases, casting defects (e.g., gas porosity), and material flow lines from plastic deformation, as well as many other characteristics. Large scale features may be visible to the unaided eye while examination of smaller microstructural features may require magnification using appropriate instrumentation.

In the simplest cases, metallographic preparation may require nothing more than buffing a specimen to remove surface contamination and expose the underlying surfaces to the unaided eye. In more complex situations, it may require sample selection, sectioning, mounting, grinding, polishing, and etching steps to facilitate high magnification observation of features that may be as small as a few nanometers. Metallographic preparation is also used to produce test specimens for microhardness testing. A wide variety of techniques are available for every step of the preparation process, and many tools are available for the examination process. It is not uncommon for several different metallographic sequences to be used to expose all of the pertinent information contained in one specimen.

4 SPECIMENS

Metallographic techniques can be applied to any size specimen; however, depending on the information desired, specimens may require sectioning to permit examination. The orientation and location of specimens taken from parent material must be recorded. This will allow the orientation of microstructural features to be related back to the original object orientation.

5 EQUIPMENT

A list of items commonly used for metallographic examinations follows. Not every item is used for every metallography investigation. The instrumentation and equipment to be employed will depend on the nature of the items to be examined.

- Sectioning equipment: band saw, shears, abrasive cutters, drills, cutting lubricants
- Mounting equipment: epoxy resin systems, thermosetting or thermoplastic mounting media, mold release agents, specimen support clips, compression mounting press, vacuum desiccator
- Grinding equipment: handheld, bench, or belt grinders; silicon carbide (SiC), zirconia or diamond abrasive disks or papers; coolants
- Polishing equipment: SiC or alumina (Al_2O_3) abrasive paper of various grit sizes, diamond or Al_2O_3 polishing compounds or solutions, colloidal silica, polishing cloths of various naps, coolants, clean compressed air
- Chemical etchants for exposing macro or micro features: see ASTM standards E340 and E407 and other references
- Electrolytic polishing/etching equipment: electrolyte, power source, connectors, cathodes
- Rinse solutions: reagent grade alcohol, deionized water
- Ultrasonic cleaner with cleaning solution
- Observation equipment: stereomicroscope, metallograph, scanning electron microscope (SEM)

6 STANDARDS AND CONTROLS

Suitable reference materials, known materials, or reference photomicrographs may be used to evaluate the identity and distribution of constituent(s).

Calibrated gage blocks are used to verify micrometers and calipers for measuring sufficiently large features. Examinations that report microconstituent size will be performed on a microscope with a calibrated reticle or compared to a micron marker that has been verified against a calibrated rule. See CHEM-100 for measurement uncertainty and traceability requirements.

7 SAMPLING

Whole components, or sections from a component, can be examined metallographically.

7.1 Sample Selection

Identification of a suitable region of an object to examine is typically non-statistical in nature and requires consideration of the inhomogeneity of the material and the features of interest. The nature and orientation of features of interest will guide the location(s) of etching and sectioning for observation.

A. To study the macro-scale properties of a metal, a representative sample of the bulk material must be selected from a region some distance away from free surfaces (e.g., metal plate edges) and other metallurgically heterogeneous regions.

B. To study individual characteristics associated with production or service, the region(s) of interest may be examined in-situ or sectioned from the evidence if destructive testing is allowed.

C. If sectioned, the orientation of the section to the original object must be recorded.

7.2 Sampling Plan

If large numbers of physically indistinguishable objects are received for testing, a sampling plan may be employed as detailed in METAL-200 or METAL-210.

A. If the sampling plan will be used to make an inference about the population, then the plan will be based on a statistically valid approach.

B. All of the objects may be tested at the examiner's discretion.

C. Any sampling plan used will be documented in the case notes.

8 PROCEDURE

The following steps describe elements of typical sequences for preparing and observing metallographic specimens. These are guidelines only and not all steps may be necessary; however, As-Received Documentation is mandatory. The exact method(s) used, or developed empirically, depend on many variables including alloy system, specimen size, surface finish required, desired feature(s) to observe and numerous other factors. The preparation procedure must produce a surface that accurately reveals the structure as it existed in the metal before sectioning and/or grinding. Any localized damage/artifacts introduced in the surface should be removed in subsequent preparation steps. If removal of induced artifacts is not possible, limitations to the observation of features of interest will be recorded in the case notes.

8.1 As-Received Documentation

Photodocument the evidence in the as-received condition (ARC) before beginning any metallographic procedures.

8.2 Sectioning

A. Record, by sketch or photograph, the intended section to be removed in a manner that documents its orientation to the original object.

B. Choose a suitable sectioning method to minimize damage to both the retained portion of evidence and the piece to be metallographically examined. Cutting damage depends on the material being sectioned, the cutting device and parameters used, and the amount and type of coolant. Employ a sectioning method that produces minimal surface damage that can be removed during subsequent grinding and polishing. Coolant can be critical because elevated temperature may locally alter the microstructure near the cut.

C. Section the object and document the sectioning method in the case notes. Deburr as needed.

8.3 Mounting

Mounting material must protect and preserve the specimen, preventing physical damage and microstructural alteration. The medium ideally will penetrate into or flow around physical features of the specimen, providing edge retention without pores (air bubbles). The grinding and polishing characteristics of the medium should be similar to those of the specimen, and the mount must chemically resist any solvents, lubricants and etchants used. Consequently, the choice of mounting method will depend on the specimen material and on the desired features to be revealed. For embedded specimens, placing shot or small bearings of material similar in hardness to the specimen around the edge of the mount may assist in maintaining flatness during grinding and polishing.

A. Prior to mounting, remove any residue from the specimen by immersing in cleaning solution in an ultrasonic cleaner for 2-5 minutes for hard materials or <30 seconds for soft materials. Rinse with deionized water and dry in air, heated air, or clean pressurized air. An alcohol rinse after water rinse may be used to aid drying.

B. To mount mechanically, devise a clamping system to securely hold and expose the desired surface for grinding and polishing. For ferromagnetic materials, a strong permanent magnet may be used.

C. To embed the specimen in castable resin, prepare a mold form over a glass plate and coat with mold release. Place the specimen face down within the mold and support with a clip or other fixture if necessary. Choose a resin system with desired characteristics and prepare according to the system instructions, agitating as gently as possible to minimize air bubble production. Pour carefully to cover the specimen while minimizing introduction of bubbles. Readjust specimen alignment if necessary. Vacuum impregnation can remove air bubbles and allow epoxy to enter crevices, assuring complete bonding. Place mold in a vacuum chamber and evacuate, cycling from vacuum to air pressure in one-minute intervals until bubbles are removed. Five cycles are usually sufficient to assure impregnation; however, total time under vacuum should not be extended any more than necessary since some resin system constituents evaporate more readily than others. Allow to harden according to resin system instructions before removing from mold.

D. To compression mount the specimen, coat the interior of the mounting press compression chamber with mold release agent. Place the specimen face down on the piston and support with a clip if necessary. Lower the piston to about double the depth of the specimen to accept the mounting material. Pour powder carefully to avoid misaligning the specimen. Lower the piston completely. Secure the piston head and run the operation cycle.

8.4 Grinding

Use successively finer abrasives to create a flat, smooth surface for polishing, etching or immediate examination. Assure that each step in the sequence removes material to a depth sufficient to eliminate scratches and underlying deformed layer created by the previous step. Several typical sequences follow:

A. To reveal macro features, such as weld heat-affected zones, on large components that have not been sectioned, grind off the surface scale or cladding with a hand grinder and buff with a rotating tool. Proceed with etching and examination.

B. To reveal macro features, such as obliterated serial numbers, on small components or sections of components, grind the surface area using coolant if possible. Perform the minimum amount of grinding required to produce a flat, smooth surface since number restorations typically depend upon the layer of disturbed material created when the number was imparted to the material. If too much material is removed, the characters cannot be restored. Proceed with polishing, etching and examination.

C. To reveal microstructural features such as grain size or phase distribution, grind the specimen on a wet belt grinder to create a flat surface. Sequentially grind through a series of Al_2O_3 or SiC abrasive papers of 240, 320, 400, and 600 grit size. Starting with the coarsest grit (e.g., 240), grind in a direction perpendicular to the scratches imparted by the belt grinding operation. Grinding should continue until the scratches from the previous step are no longer visible. Next, rotate the specimen 90° to the previous grinding direction and remove the scratches left by the prior grinding step using the next finer grit size in the series. Care should be taken to maintain a flat surface. If beveling occurs and is objectionable, the specimen should be reground to remove the bevel. An automatic system can also be used for grinding and polishing.

8.5 Mechanical Polishing

Use successively finer abrasives on polishing paper or cloth to create a smooth surface for etching or immediate examination. Assure that each step in the sequence removes material to a depth sufficient to eliminate scratches and the deformed layer created by the previous step. Several typical sequences follow:

A. For revealing macro-scale features, such as serial number restorations, on small components or sections of components, polish the surface of the area of interest by either applying part to abrasive or abrasive to part. Rinse polishing compound off with suitable solvent (e.g., water or alcohol) and dry with forced air. Proceed with etching and/or examination.

B. For revealing microstructural features such as grain size or phase distribution, sequentially polish through a series of abrasive polishing compounds typically ending with a final polish with 0.05 micron Al_2O_3. These polishing agents are typically applied with lubricant to a dedicated polishing cloth mounted on a rotating wheel. Remove scratches from each previous step using one of the following techniques:

1. Turn the specimen so that the scratches from the previous grit size are perpendicular to the wheel motion and polish until the scratches from the previous grit size are removed.

2. Rotate the specimen counter to the polishing wheel direction until all scratches appear to be of uniform depth.

C. Rinse the polishing compound off with suitable solvent (e.g., water or alcohol) and dry with forced air between each step and when polishing is complete. It may be necessary to ultrasonically clean the specimen between each polishing step for about one minute to prevent cross-contamination. Proceed with etching and/or examination.

D. Should an initial polishing attempt prove inadequate, revert to the previous particle size and repeat the polishing steps. If necessary, go back to the final grinding step (e.g., to 600 grit) then repeat the polishing procedure. Change polishing cloths if any cross-contamination occurs.

E. Chemical/mechanical polishing: Final polishing to produce a totally scratch-free surface is rarely necessary for forensic examinations; however, this may be accomplished by lapping on a polishing wheel or on a vibratory polishing system using an appropriate polishing solution. Acidic polishing solutions are typically used for ferrous materials and basic solutions (such as colloidal silica) for non-ferrous material.

8.6 Electropolishing

Electrolytic polishing is useful for metallographic preparation of metals that are difficult to polish by the mechanical methods detailed above (e.g., Mg, Zr). Electropolishing is not typically appropriate for preparation of multiphase materials. This method can be used to eliminate mechanical deformation induced by conventional mechanical polishing or directly after the final grinding step to replace mechanical polishing.

A. Select an appropriate electrolyte solution and starting combination of current and voltage for the particular metal under examination. ASTM E 340, E 407, and other listed references (see 12 References) offer guidance for selecting electrolyte solutions and power conditions. Additional resources available from metal producers, users, and professional organizations can also provide selection guidance.

B. Establish a DC variable voltage current source, electrical circuit, and electrolytic cell. This requires a DC power supply, wires with alligator clips or other electrical connectors and an electrolyte container. Typically, commercially manufactured electrolytic equipment is used.

C. Prepare specimen surface to a 600 grit or finer finish.

D. Provide stirring or air agitation if necessary to prevent localized heating of the surface (e.g., a magnetic stir bar in the electrolyte solution over a stir plate).

E. Connect electrodes so that the specimen is the anode (connected to the " + " side of the DC power source) and the cathode is a metal component connected to the " - " side of the power source, and both are immersed in the electrolyte.

F. Adjust the voltage to achieve adequate current. "Adequate" will be determined empirically for each specimen because the current density (current/unit area) that is generated depends on the area of the exposed surface, (i.e., the surface of the specimen in contact with the electrolyte.)

G. Should an initial polishing attempt prove inadequate, regrind the specimen to 600 grit or finer and repeat the electropolishing procedure using a different applied voltage.

H. Should artifacts (such as furrowing or dimpling due to gas evolution) be created, regrind the specimen and repeat the electropolishing procedure with additional solution agitation.

8.7 Chemical Etching

Etching proceeds by selective dissolution according to the electrochemical characteristics of the microstructural constituents. Be sure to follow safety and hazardous waste disposal guidelines for dealing with the chemicals used. A typical chemical etching sequence follows:

A. Assure that the specimen surface has been appropriately prepared. Macroetching may only require grinding to a 600 grit finish then buffing. Microetching usually requires a finer finish (e.g., 0.05 micron).

B. Select an appropriate etchant for the type of metal and the feature(s) to be exposed. ASTM E 340, E 407, and other listed references (see 12 References) offer guidance for selecting etchant solutions. Additional resources available from metal producers, users, and professional organizations can also provide selection guidance.

C. Follow safety and hazardous waste disposal guidelines for dealing with the chemicals used.

D. Swab with, or immerse specimen in, the etchant until sufficient contrast is produced. Rinse with appropriate solvent to halt the reaction, typically with deionized or distilled water. Multiple steps of etching and rinsing may be required.

E. Should an initial etching attempt prove inadequate, regrind the specimen to remove the etched surface (e.g., to 1 micron abrasive for microetched materials or 600 grit

for macroetched materials). Sometimes, specimens etch more readily immediately after polishing. Etching can also happen preferentially depending on whether the sample is immersed or swabbed. If two metals are present it may be necessary to mask one so it will not etch to the exclusion of the other.

8.8 Electrolytic Etching

An electrical potential is applied to the specimen using an electrical circuit to promote removal of metal ions from the specimen surface.

A. Select an appropriate electrolyte solution and a starting combination of current and voltage for the particular metal under examination. ASTM E 340, E 407, and other listed references (see 12 References) offer guidance for selecting electrolyte solutions and power conditions. Additional resources available from metal producers, users, and professional organizations can also provide selection guidance.

B. Follow safety and hazardous waste disposal guidelines for dealing with the chemicals used.

C. The basic steps follow those used for electropolishing (see Electropolishing section), but different parameters are used to effect etching rather than polishing.

D. Document the parameters used in the case notes.

8.9 Observation

Identification of microstructural features and their significance requires metallurgical knowledge of the metal being examined. Reference micrographs are provided in ASM Handbook Vol. 9, Metallography and Microstructures. Additional resources available from metal producers, users, and professional organizations may also assist in interpreting microstructures. Identify and use the appropriate ASTM procedure for quantifying microstructural features (such as grain size or inclusion density) if required. See 12 References.

A. Observe the features of interest with instrument(s) that provide sufficient magnification to resolve the features (e.g., eye, stereomicroscope, metallograph, SEM.)

B. Photodocument the as-polished and/or etched features of interest and retain the images in the case notes.

C. Retain the references used for interpretation (in whole, in part, or by citation) in the case notes.

9 ACCEPTANCE CRITERIA

9.1 Instrument Performance

A. Equipment and conditions are selected by the user to produce an artifact-free polished or etched surface for viewing.

B. Adequate function of imaging equipment is demonstrated by acquisition of a photograph in which the features of interest are distinguishable.

9.2 Qualitative Analysis Conclusions

The conclusions derived from this procedure are based on careful interpretation of all of the data gathered from examination. It is often possible to identify the phases present in a material by careful comparison with published microstructures. As part of a metallurgy examination, SEM/EDS can be used to confirm phase identification if doubt exists. (See METAL-210 or METAL-220.)

The degree of certainty to be applied to test results will depend on the material homogeneity and how well the prepared specimen represents the sample, among other factors. Microstructural development is influenced by solidification, phase transformations, heat treatment, and plastic deformation. Production defects, corrosion products and the effects of microstructural constituents and their distribution on mechanical properties may also be important in an investigation. Careful consideration of the specimen geometry and its other physical features generally allows an experienced examiner to select appropriate, representative samples for metallographic purposes. Where doubt exists, additional sampling should be performed.

9.3 Quantitative Analysis Conclusions

Quantitative analyses of microstructures are typically limited to estimations of phase distribution, case depth, or grain size. A validated method will be used to determine such quantities, and its use will be documented in the case notes.

10 LIMITATIONS

10.1 Destructiveness

Metallographic preparation is, by nature, destructive. Destructive tests on evidence should only be conducted if absolutely necessary and must be pre-approved by the contributor and principal investigator.

10.2 Artifact Generation

There are numerous potential sources for artifact generation when preparing metallographic specimens. These include overheating during sectioning, embedded abrasive material, smearing of soft materials (e.g., pure Al or Cu), and pitting or preferential attack during polishing and/or etching. The examiner analyzing the revealed microstructure must also be cognizant of the possibility of stress- or thermally-induced phase transformations when evaluating the material.

Whenever possible, grinding operations should be performed with adequate coolant such as water, glycerin, or lubricating oil to avoid potentially misleading microstructural changes due to localized heating.

11 SAFETY

A. Review all pertinent Safety Data Sheets (SDSs) before using any potentially hazardous chemicals. For the components of an etchant or electrolytic solution, always follow the safety guidelines for the mixture's most hazardous component.

B. Wear appropriate personal protective equipment when preparing specimens.

   1. Wear protective gloves and eyewear when handling evidentiary materials of a hazardous nature.

   2. Wear impact-resistant eye protection during sectioning and rough grinding operations to protect from flying debris

   3. Wear an acid-resistant apron, gloves and chemical splash goggles when polishing and/or etching with hazardous chemicals.

C. Dispose of hazardous chemicals according to the guidelines established by the FBI Laboratory.

D. Any materials that have contacted hydrofluoric acid (HF) containing solutions must be thoroughly rinsed before handling or observing under glass optics because of the potential for HF-containing liquid and fumes to attack bones (decalcify) and etch glass. HF containing rinses must be disposed of as hazardous waste.

E. To apply the above procedures to materials with significant concentrations of hazardous metals such as Pb, Be, Te, Ag, Hg and Cd, rinses and etchants contaminated with these metals must be collected and disposed of as hazardous waste.

   1. Do not cut or grind these metals on machines with closed-loop cooling systems. Collect the chips generated from sawing or grinding and any grinding belts, abrasive papers, polishing cloths, and other solid materials embedded with metal debris, and dispose of them as hazardous waste.

   2. When using automatic polishing equipment, run the drain line into a collection vessel and dispose of it as hazardous waste.

12 REFERENCES

- ASM Handbook, Vol. 9 – Metallography and Microstructures, ASM International, USA, current revision
- Vander Voort, G. F., Metallography: Principles and Practice, McGraw-Hill Publishers 1984
- ASTM Annual Book of Standards, Vol. 03.01 (current revision), multiple standards for metallographic preparation and analysis, ASTM International, West Conshohocken, PA
- ASTM Method E3 (current revision), Standard Guide for Preparation of Metallographic Specimens, ASTM International, West Conshohocken, PA
- ASTM Method E7 (current revision), Terminology Relating to Metallography, ASTM International, West Conshohocken, PA
- ASTM Method E45 (current revision), Standard Test Method for Determining the Inclusion Content of Steel, ASTM International, West Conshohocken, PA
- ASTM Method E112 (current revision), Standard Test Methods for Determining Average Grain Size, ASTM International, West Conshohocken, PA
- ASTM Method E340 (current revision), Test Method for Macroetching Metals and Alloys, ASTM International, West Conshohocken, PA
- ASTM Method E407 (current revision), Practice for Microetching Metals and Alloys, ASTM International, West Conshohocken, PA
- ASTM Method E883 (current revision), Guide for Reflected-Light Photomicrography, ASTM International, West Conshohocken, PA
- ASTM Method E930 (current revision), Standard Test Method for Estimating the Largest Grain Observed in a Metallographic Section (ALA Grain Size), ASTM International, West Conshohocken, PA
- ASTM Method E1077 (current revision), Standard Test Methods for Estimating the Depth of Decarburization of Steel Specimens, ASTM International, West Conshohocken, PA
- ASTM Method E1181 (current revision), Standard Test Methods for Characterizing Duplex Grain Sizes, ASTM International, West Conshohocken, PA
- ASTM Method E1351 (current revision), Standard Practice for Production and Evaluation of Field Metallographic Replicas, ASTM International, West Conshohocken, PA
- ASTM Method E1558 (current revision), Guide for Electrolytic Polishing of Metallographic Specimens, ASTM International, West Conshohocken, PA
- ASTM Method E2014 (current revision), Standard Guide on Metallographic Laboratory Safety, ASTM International, West Conshohocken, PA
- CHEM-100, Chemistry Unit, Federal Bureau of Investigation, Laboratory Division, latest revision
- METAL-210, Chemistry Unit, latest revision
- METAL-220, Chemistry Unit, latest revision

13 REVISION HISTORY

Revision	Issued	Changes
04	09/15/2022	Revised to comply with new formatting requirements. Distributed information from previous Instrumental Conditions and Decision Criteria sections into new Acceptance Criteria section. Informational references removed.