# Metallographic Examinations

## 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 caseworking personnel who perform metallurgy analyses. Techniques for successful metallographic sample preparation and examination depend highly on the form and composition of the metal under investigation. Appropriate preparation techniques are often 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. Sample preparation is typically destructive and requires thoughtful integration into an examination sequence.

## 3 Principle

Metallographic preparation is a technique used to reveal the internal structure of metals and alloys. These structural details 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 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 microstructure features to be related back to the original object orientation.

## 5 Equipment/Materials/Reagents

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.

a. Sectioning equipment: band saw, shears, abrasive cutters, drills, cutting lubricants

b. Mounting equipment: epoxy resin systems, thermosetting or thermoplastic mounting media, mold release agents, specimen support clips, compression mounting press, vacuum desiccator

c. Grinding equipment: handheld, bench or belt grinders; silicon carbide (SiC), zirconia or diamond abrasive disks or papers; coolants

d. 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

e. Chemical etchants for exposing macro or micro features: see ASTM standards E340 and E407 and other references

f. Electrolytic polishing/etching equipment: electrolyte, power source, connectors, cathodes

g. Rinse solutions: reagent grade alcohol, deionized water

h. Ultrasonic cleaner with cleaning solution

i. Observation equipment: stereomicroscope, metallograph, scanning electron microscope (SEM)

## 6 Standards and Controls

Certified reference materials (CRMs) 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 should be performed on a microscope with a calibrated reticle or compared to a micron marker that has been verified against a NIST traceable rule.

## 7 Sampling

### 7.1 Sample Selection

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

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. Refer also to section 10 Decision Criteria.

### 7.2 Sampling Plan

If large numbers of physically indistinguishable samples are received for testing, a sampling plan may be employed. 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. All of the samples may be tested at the examiner's discretion. Any sampling plan and corresponding procedure used will be documented in 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, step 8.1 As-Received Documentation is mandatory. The exact method(s) used, or developed empirically, depend on many variables including alloy system, sample size, surface finish required, desired feature(s) to observe and numerous other factors. The preparation procedure must produce a surface that accurately represents the structure as it existed in the metal before sectioning and/or grinding. Any localized damage/artifacts introduced in the surface must be removed in subsequent preparation steps.

### 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 case notes. Deburr as needed.

### 8.3 Mounting

Mounting material must protect and preserve the specimen, preventing physical damage and microstructural alteration. The medium must 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 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 sample drying.

b. To mount mechanically, devise a clamping system to securely hold and expose the desired surface for grinding and polishing. For ferromagnetic samples, 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 without introducing 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 sample to accept the mounting material. Pour powder carefully to avoid misaligning the specimen. Lower the piston completely. Secure the piston head and start 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 (e.g., on a metallographic belt grinder). 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 sample 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 sample 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:

   i. Turn the sample 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.

   ii. Rotate the sample 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 sample 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 procedure. 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 mechanical methods detailed in 8.2-8.5 (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. See section 15 References for optimal current/voltage relations and electrolyte solutions to use for the particular metal under examination. Similar information can often be found in literature from metallographic supply companies.

a. 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.

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

c. 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).

d. 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.

e. Adjust the voltage to achieve adequate current density. "Adequate" will be determined empirically for each metal because the current density generated depends on sample size.

f. 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.

g. 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.

h. Complete any necessary photodocumentation of the as-polished specimen surface prior to etching.

## 8.7 Chemical Etching

Etching proceeds by selective dissolution according to the electrochemical characteristics of the microstructural constituents. Suggested solutions for chemical etching are given in ASTM E 340 or E 407 and in 15 References. 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. 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.

c. 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 samples 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. Refer to ASTM E 340 or E 407 or other appropriate guidelines (see 15 References) for suggestions of optimal current/voltage relations and electrolytes for the particular metal under examination. Follow safety and hazardous waste disposal guidelines for dealing with the chemicals used. The basic steps are identical to those in section 8.6 Electropolishing, but different parameters are used to effect etching rather than polishing. 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. For unknown materials, reference micrographs are provided in ASM Handbook Vol. 9, *Metallography and Microstructures* as well as in many additional resources. Identify and use the appropriate ASTM procedure for quantifying microstructural features (such as grain size or inclusion density) if required (see section 15 References).

Photodocument the as-polished and/or etched macro and/or micro-structures as needed for case notes.

## 9 Instrumental Conditions

The cooling reservoirs of wet cutting and grinding equipment must be maintained to assure adequate coolant levels and cutting fluid cleanliness according to procedures found in the appropriate instrument manuals.

Microscopes and cameras must be maintained to prevent artifacts from being introduced during observation and photodocumentation. Annual maintenance is usually sufficient; however, if artifacts are observed, the instrument(s) should be cleaned immediately.

## 10 Decision Criteria

The conclusions derived from this procedure are based on careful interpretation of all of the data gathered from examination. In general, it is possible to identify the phases present in a material by careful comparison with published microstructures. When this is not possible, additional testing methods such as x-ray diffraction, transmission electron microscopy or SEM/EDS may be required for phase identification.

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. 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 considered.

Accurate microstructure interpretation requires a well-trained, experienced metallurgist. It is essential that the analyst has received training in microstructural development as it is influenced by solidification, phase transformations, heat treatment, and plastic deformation. The analyst must also recognize production defects and corrosion products and must understand the effects of microstructural constituents and their distribution on mechanical properties. Training may consist of attending topical courses and/or possessing equivalent experience prior to attempting to interpret microstructural information.

## 11 Calculations

Calculations regarding microstructures are typically limited to estimations of phase distribution, case depth or grain size. When required, an appropriate method will be validated, applied, and documented in the case notes.

## 12 Measurement Uncertainty

Normally, metallography is non-quantitative in nature. However, quantitative analysis of a set of metallurgical features is sometimes possible and could potentially be used in a comparison of one metal sample to another or to a published specification, or to establish its approximate mechanical properties and/or processing history. Should it be required, the measurement uncertainty associated with such a procedure will be established in accordance with the *Chemistry Unit Procedures for Estimating Measurement Uncertainty*.

## 13 Limitations

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

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 potential false microstructure changes due to localized heating.

## 14 Safety

Standard safety precautions, such as wearing protective gloves, and eyewear, must be observed when handling evidentiary materials of a hazardous nature. In addition, wear an acid-resistant apron and chemical splash goggles when polishing and/or etching with hazardous chemicals Wear impact-resistant eye protection during sectioning and rough grinding operations to protect from flying debris. When consulting Safety Data Sheets (SDS's) for the components of an etchant or electrolytic solution, always follow the safety guidelines for the mixture's most hazardous component. Review all pertinent SDS's prior to using any potentially hazardous chemicals.

Dispose of hazardous chemicals according to the guidelines established by the FBI Laboratory, consulting the Chemistry Unit Safety Officer for proper procedures.

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.

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. Do not cut or grind these metals on machines with closed-loop cooling systems. Collect 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. When using automatic polishing equipment, run the drain line into a collection vessel. Transfer waste from this collection to the proper container in the 90-day waste accumulation site established by the Safety Officer.

## 15 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

*Chemistry Unit Quality Assurance and Operations Manual*, Federal Bureau of Investigation, Laboratory Division, current revision

*FBI Laboratory Operations Manual*, Federal Bureau of Investigation, Laboratory Division, current revision

*FBI Laboratory Quality Assurance Manual*, Federal Bureau of Investigation, Laboratory Division, current revision

| Rev. # | Issue Date | History |
|---|---|---|
| 2 | 04/24/2014 | Updated required equipment in section 5. Minor grammatical changes made to text in sections 3, 5 and 9.1 and 9.2. Section 13 has been rewritten to reflect the current measurement uncertainty requirements. Equipment referenced in section 15 has been updated. References have been updated in Section 16. |
| 3 | 12/21/2018 | Renumbered Metallurgy SOP Manual documents. This document was formerly Metal 17 and is now designated Metal 800. Added personnel to section 2. Made minor editorial corrections throughout document. Revised throughout to remove specific brand names/ equipment. Incorporated section 7 into section 6 and renumbered subsequent sections. Added requirement for sampling plan retention in section 7. Updated optional procedures in section 8. Augmented section 14. Added additional references to section 15. |

Redacted - Signatures on File

**Approval**

Metallurgy Technical Leader _____ Date: 12/20/2018

Chemistry Unit Chief _____ Date: 12/20/2018

**QA Approval**

Quality Manager _____ Date: 12/20/2018