Metallographic Preparation of Art & Archaeological Specimens:

A Basic Step in the Microscopic Examination of Objects

Martha Goodway, *Metallurgist, Conservation-Analytical Laboratory, Smithsonian Institution*

An object is an object is an object, but what can be learned from it depends upon how it is viewed. A casual observer, for instance, might look at an artifact and perceive it as some type of old metal vessel, probably Oriental in origin, while the more experienced eye would see it as a bronze Chinese *ting,* dating from several centuries B.C. The conservator, curator, or other professional, with the aid of some analytical instrumentation, however, would be able to discern much more – the object's structural characteristics, elemental composition, method of fabrication, various other physical properties, and the environments to which it had been exposed.

Combining this analytical data with a knowledge of the aesthetics of the object can provide valuable clues to the object's history (time and place of origin, previous restorations, etc.), as well as to the economic and social history of an area (where an object was excavated might help delineate trade route patterns, for example). In addition, this type of examination can reveal material characteristics of the object that are important in carrying out a suitable conservation/preservation procedure.

The person treating an object, therefore, is wisely advised, after making a macroscopic examination with the unaided eye, to take a microscopic view of the object . . . and to imagine the effect of the treatments to be applied. How might these influence the metal's mechanical properties or alter surface characteristics? But, unless a specimen is properly prepared, little can be determined by microscopic analysis regarding a material's properties or surface behavior.

A Scratch-Free Surface is Necessary to "Scratch the Surface" for Information

If the material of an object is not transparent, how can one see into the material's structure to get a cross-sectional view, the view that provides the interpretive information? A little more than a century ago, Sorby solved this problem for the study of metal by taking advantage of a special property of metals – their high reflectivity. Thus, if a material is cut open, or sectioned, and the exposed surface is carefully polished so that none of the metal is displaced from its original position, the surface of the section is made into a mirror which can reflect light into the lens of a microscope. And, this reflected light, when passing back through the microscope's objective lenses, forms an enlarged image of the illuminated area. Not only is a scratch-free, mirror-like surface necessary, but also, a very flat surface. That is, the numerical aperture of a high-power objective requires an optically flat surface since its depth of field is so shallow. These requirements place severe limitations on the preparation of the surface.

Simple in Theory, More Difficult in Practice

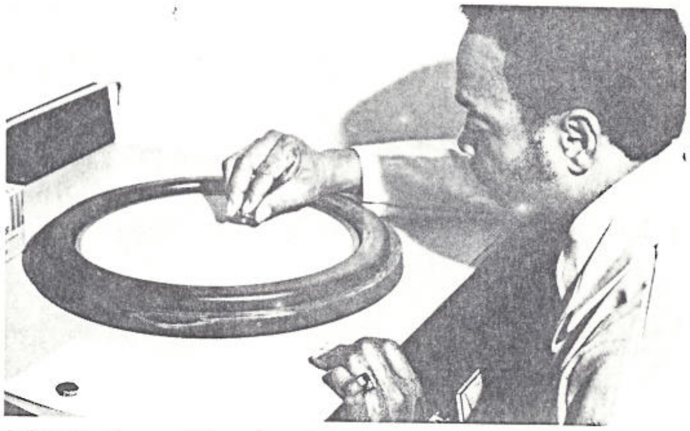
Surface preparation is more of a technical art than a science. It requires a sequence of steps: the sample is mounted, faced off, ground, polished, and etched before it can be observed. Some of these steps occasionally can be omitted or combined.

All of the procedures, however, must be conducted with great care if the examination of the object is to be meaningful. Improper preparation can destroy many of the very features that are the key to understanding a particular material. Excessive grinding, for example, might remove inclusions or dislodge soft secondary phases such as lead particles in leaded brasses. The wrong abrasive material might cause surface distortion and metal flow, eroding grain boundaries.

What and How to Sample

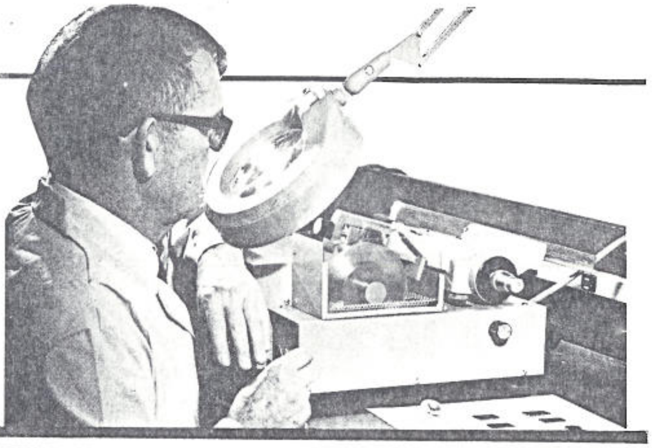
The first step in surface preparation, obviously, is to choose the sample. (Naturally, this applies only to those artifacts where sampling is allowed.) How to select an appropriate sample and mount, or otherwise prepare them for the grinding step, however, often can pose problems.

Samples from art objects or from archaeological material differ in several ways from samples of modern industrial materials. Nearly always, these artifacts are very small and, many times, they are heterogeneous. The very corrosion products which would be cleaned off before proceeding if standard metallographic practice were used are often the features of interest to a conservator or curator. Such considerations lead to modifications in the standard methods recommended by the American Society for Testing Materials (ASTM) and the American Society for Metals (ASM).



Polishing of a sample's surface to an optically flat finish requires care in order not to dislodge inclusions or cause metal flow and surface distortion. Richard Johnson of the Smithsonian's Department of Mineral Sciences demonstrates the correct grip on the specimen when applying it to the polishing wheel.

Some objects are so fragile that a small fragment is easily detached. It may be already broken or damaged in such a way as to offer an area for sampling which may be concealed by later restoration. (Naturally, it is prudent to confer with the conservator who is to make this restoration to determine beforehand what, in fact, may be sampled and later concealed.) Even if a sample is not an accurate representation of the whole object, it may provide some useful data. However, no sample



The progress of the low-speed diamond saw through a section of coarse ceramic is monitored by Grover Moreland, chief of the preparation laboratory for the Smithsonian's Department of Mineral Sciences.

should be taken if it does not promise to be informative.

Once a sample is chosen, it should be mounted so that it can be easily gripped for grinding and polishing. Mounting also creates a larger surface area, making it simpler to obtain a flat surface finish during grinding and polishing.

The selection of a mounting medium depends upon a number of considerations. Several non-reactive plastic mounting materials are available. For a fragile, heavily corroded sample, the selection factors can be quite similar to those faced by a conservator making a choice of consolidant. That is, the mounting medium must function to keep the sample from collapsing under the pressure of grinding and polishing.

When it is desirable to see the entire specimen in the mount or to observe the precise amount of sectioning being done during grinding and polishing, a transparent mounting material should be used. Thermoplastic resins such as Lucite (or other methyl methacrylates), polystyrene, and cellulose-base materials offer the desired clarity. They, however, can become temporarily soft under certain temperature and pressure conditions, such as those of too vigorous grinding.

The conservators' principle of reversibility does not usually apply to the mounting of a sample, but if the sample is to be recovered for some further analyses, one of the methacrylates might be chosen. These plastics resist most etchants, but are readily soluble in organic solvents such as acetone.

Cases may arise where the taking of a sample is not allowed. This in itself need not prevent a metallographic study from being carried out. There are several approaches that should be considered.

A surface can be prepared *in situ,* if the object offers a suitable protuberance. R. M. Organ has described several ingenious designs for jigs adapted to specific museum objects. (For more details see *Recent Advances in Conservation,* Butterworths, London, 1963.) These jigs allow manipulation of the artifacts so that they can be aligned to the grinding surface. A small, but flat and correctly polished surface, therefore, can be produced.

The dendrites of cast brass have etched unevenly in a phenomenon called "coring." Cooling the casting quickly, or chilling, produces a composition gradient across each grain. This is commonly seen in the bronze castings of antiquity. The small round inclusions are drops of lead which, like drops of oil in water, do not dissolve in this alloy. 200X.

If the object is small enough, it can be put under the microscope directly. If it is too large, a replica of the surface can be cast in a plastic film, and the replica observed microscopically, a versatile technique borrowed from transmission electron microscopy.

Obtaining the Appropriate Surface . . . & Surface Finish

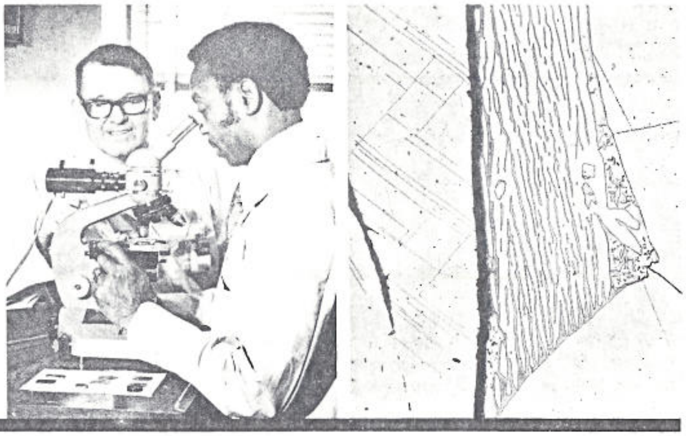
Various surface preparation techniques can be employed to help extract as much information as possible from latter observations. Orientation of a sample, for example, often can affect what structural features are exposed. Therefore, if a specific orientation might be useful to study, care should be taken to insure that this is the surface that is exposed. By mounting the sample in a transparent medium, the sample can be closely watched during grinding and polishing, the exposed section monitored, and the exact surface orientation achieved. In other words, the sample will be precisely faced off.

Another facing off method is utilized when studying a thin layer as in gilding, or a series of layers as in a paint film. The layers can be made to appear broader and easier to distinguish by facing off the sample in a plane just slightly oblique to the layers. This useful device is called a taper section.

To perform the facing off operation, several conservation laboratories have adopted the use of a low-speed saw, equipped with a diamond wafering blade. Compact units, supplied by firms such as Buehler and Leco, feature a saw which is operated at 100 to 200 rpm, with a load of 10 to 50 grams, and a blade which produces a kerf only 3 or 6 mils in width, depending upon the choice of blade. The sample is held securely in a jig, a necessary fixturing item since it may take as long as an hour for the pass to be completed. The extreme hardness of diamond allows the abrasive to cut rather than to plow the surface. Used without excessive force, the saw will produce a surface which needs no grinding and which can be polished directly.

Elimination of the grinding step also can be achieved using a method developed by Grover C. Moreland and Richard Johnson of the Smithsonian Institution. In this procedure, a low-speed polishing wheel (up to 600 rpm) is faced with heavy paper, rather than with a lapping cloth, and is charged with a 3 micron-size diamond compound. An optically flat surface is produced in from 10 to 20 minutes. Omitting the grinding stage can offer a number of advantages. The "plucking" of inclusions, the production of excessive relief and edge-rounding, and the fault of double-facing or faceting (lack of flatness) in the surface that can occur if grinding is improperly carried out are avoided.

The next step is a final polish with a fine abrasive, such as gamma-alumina (0.05 micron, such as Linde B) on a low-speed



Proof of the polishing is in the viewing. A wide range of material characteristics can be revealed when a properly polished specimen is examined through the microscope.

Specimen preparation photographs courtesy of Dr. William G. Melson, Chairman. Department of Mineral Sciences, Smithsonian Institution.

(150 rpm) wheel faced with a napped cloth. With a cloth of deep velvet pile, a fine finish can be produced by adding the gamma-alumina to a froth of water and a neutral detergent (such as is stocked for textile conservation).

Since abrasives as fine as these aluminas tend to stick to the surface of the specimen and fill any crevices it may have, the surface usually requires a cleaning. This can be done by lapping on a fresh, dry napped cloth at low speed or by wiping with soft cotton and rinsing under a strong jet of water. Ultrasonic cleaning here has nearly no effect.

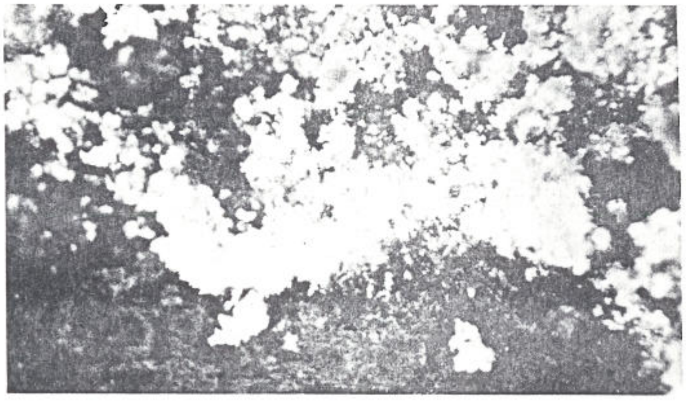
To Etch or Not to Etch

Every sample should be thoroughly examined microscopically before being etched. Minerals, for example, which may be present as products of corrosion or as inclusions in the sound metal, need no etching. In fact, they may be dissolved by the etching reagent if the etchant is not prudently applied.

In the case of metals, there are several ways that microscopic detail in the surface can be seen without etching. If the sample is a copper or copper-based material, oxide films will form immediately upon the freshly polished surface. The films will make the grain structure of the metal or alloy visible under cross-polarized light or dark-field illumination. Inclusions or corrosion layers of cuprous oxide, for instance, will appear ruby-red under these conditions.

Phases of differing hardness will polish to microscopically different heights. These can be distinguished quite beautifully by interference systems of Nomarski or Smith design.

If the sample is entirely mineralized, or if the above-mentioned methods of observation are sufficiently revealing, it may not be necessary to etch. Etching should be done to bring out more detail, without destroying any already visible.



Conservation begins at home: cuprous chloride in the corrosion layers of copper from an ox-hide ingot from the Cape Gelidonya shipwreck is breaking out in "bronze disease." The metallographic specimen requires the same careful storage (i.e., in an environment free of excess humidity and sulphide fumes) as do museum objects. 125X. (Sample courtesy of the late Rutherford John Gettens, Freer Gallery of Art.)

In heterogeneous samples, overetching occurs readily. However, what is overetching may depend somewhat on the resolution of the lenses used. If overetching does occur, it can be erased only be regrinding the sample with 600 grit abrasive and repeating the whole polishing process. Therefore, using a reagent more dilute than is usually recommended can be a time-saving precaution.

Four etching reagents are used most often in art and archaeological conservation activities. Nital (a dilute solution of nitric acid and alcohol) is suitable for iron and steel, while ammonia/hydrogen peroxide, potassium bichromate, and ferric chloride are the three most useful etchants for copper and copper alloys. Better, more reliable results are obtained from the potassium bichromate and ferric chloride if they have been allowed to age before use. Their formulas are given in such references as the *Metals Handbook, Volume 8* ("Metallography, Structures, and Phase Diagrams"), published by the American Society for Metals. These etches, their appropriate methods of application, and the time, temperature, and other variables involved in producing a satisfactorily etched sample have been familiar to metallurgists for many years, and their results can be securely interpreted.

The same method of producing a metal section – sampling, mounting, surface finishing – can be used for paint-layer analysis. For this type of analysis, a very small sample of paint layers can be taken from a painting by inserting a hypodermic needle into the area of interest. The needle with the sample (a plug-shaped volume) still inside is mounted and cross-sectioned.

What You See Is . . .

Interpretation of these visible structures is a matter of experience, plus a gift for pattern recognition. Once cross-sectioned, it is easy to see, for example, the slag stringers of a wrought iron nail which easily differentiate it from a modern low-carbon steel nail.

The iron of meteorites is found in a surprising number of museum objects, usually in knives and daggers. The typical structure of meteoritic iron can persist even when the bulk of the iron has turned to rust. This was clearly shown by Dr. Roy Clarke of the Smithsonian's Division of Meteorites in his work with W. Thomas Chase III and the late Rutherford John Gettens on two ceremonial Chinese axes in the collections of the Freer Gallery of Art.

Sources of meteoritic iron are not always small. The Cape York meteorite, Greenland, weighed at least eight tons, and for centuries has served the Eskimos as a kind of mine. Knives of Cape York iron have been found widely scattered across the north, often at great distances from the original meteorite site. Some idea of migrating/trading patterns emerges from the location of these knives.

Aspects of other cultures, similarly, can be studied by observing the structural characteristics of their metal artifacts. The North American Indian copper culture which flourished

Left photograph:
Wrought iron of the American Civil War not only shows the slag remaining from the smelting process as stringers, but also the microstructure within the slag. 250X.

Far left photograph:
The distinctive microstructures of meteorites, as illustrated here, differ markedly from those of early smelted iron. (Specimen courtesy of Dr. Roy S. Clarke, Jr., Smithsonian Curator of Meteorites.)

around the Great Lakes was dependent upon local supplies of native copper, since the Indians never learned to smelt copper. The microstructures of the native copper and smelted copper differ in a number of ways, most conspicuously by the inclusions of cuprous oxide which result from the smelting process. If these inclusions of cuprous oxide are found in the copper of an Indian object, it may indicate that the copper was obtained in trade with the white man, i.e., a post-contact date. On the other hand, it may indicate that the object was manufactured to fill the demand for American Indian artifacts and is a forgery.

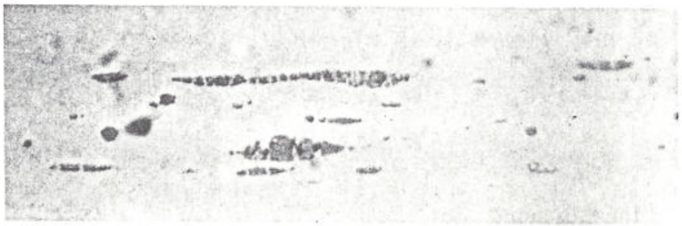
Detection of forgeries, based on microscopic examination, is sometimes easier. Several decorative coatings when studied in cross-section have produced surprises. A small bronze figure which had been described as classical was sampled; the section showed copper plate over lead. The uniform thickness of the plating layer, the structure of it, and the sharp interface between the plating layer and the underlying metal all pointed to a modern electroplate. It was conjectured that this object may have been plated with copper because the copper would have presented fewer problems of producing a convincing patina.

However, even on objects of undoubted antiquity, the decorative layer can be something unexpected. A small bronze bull in the Museum of Fine Arts, Boston, was sampled for sectioning by William J. Young. The bull dates from the third millennium B.C. and was found to have a plated layer of arsenic which had been mistaken for silver.

Another example are gilded objects from South America, whose characteristic alloy is tumbaga (copper and gold). These objects were depletion-gilded by dissolving away the copper at the surface and leaving the gold. Yet sections from similar objects occasionally will show a layer of gold foil instead.

Structural analysis not only can be used for tracing the movement of a certain group of people and for authenticating an item, but also for composition determination. There are certain microstructures which allow an estimate of the composition of the alloy as precise as any other method. A particularly interesting example of this is a ring of the first millennium A.D. from northeastern Thailand which was so corroded that it is now entirely mineralized. Pseudomorphic replacement of the original metallic microstructure allowed Professor Cyril Stanley Smith of M.I.T. to identify the alloy as a bronze, estimate the content of tin to within 2%, and even to add that the ring had been hot-forged and quenched.

The cuprous oxide (dark spots) of tough pitch copper which has been drawn lengthwise demonstrates the relative ductility of the copper metal as compared with its inclusions. The more brittle cuprous oxide inclusions have been shattered by the forces of the drawing die into many smaller inclusions. 500X.

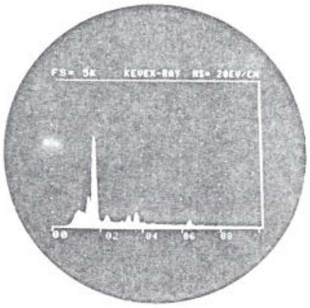


Native copper, here shown at only 100X, is free of oxide inclusions and is conspicuous for its large grain size. Native copper was the only copper available to the pre-contact American Indians. (Specimen courtesy of Prof. Ursula Franklin, University of Toronto.)

Cross-sectioning of bronzes and other metal artifacts also has produced results of interest for the history of technology. Until technical examination was undertaken, it had been assumed that the fine bronzes of China and of the classical world had been cast by the lost-wax process. In fact, they were cast in piece molds. Arthur Steinberg of M.I.T. has shown that the large classical bronze typically was cast in a large number of separate pieces which then had to be joined. Also in the area of joining techniques, Professor Smith has studied Luristan iron weapons of 800 B.C. in cross-section and can find no evidence that the iron had been welded or hardened; rather, they had been skillfully forged to produce secure mechanical joints.

In addition to using the microscope to examine the structure and fabrication of a sample/object, other analytical tools can be used. A sample can be analyzed with the electron beam microprobe to quantitatively and qualitatively determine elemental composition. Areas as small as a few microns in diameter can be investigated. The preparation of a sample (which can be of glass, ceramic, metal, or almost any material) for probe analysis is the same as before. A thin conducting layer, using a few nanometers of carbon, applied to the prepared surface is the only additional step required. Typical of the results that can be obtained was the probe analysis of paint layers of several paintings suspected of being forgeries. The analyses showed titanium (from titanium dioxide, a pigment of 20th century invention) at levels too deep in the paint level to be confused with inpainting. Also, analyses of metal composition can be compared with microstructure for consistency of the two results.

Thus, the well-prepared surface can be an unusually informative device. But, it can be something much more. Some materials present microstructures of great aesthetic appeal which can be appreciated for their own sake and also, as another expression of the beauty of nature.



Cover: The historic adobe brick building at Tumacocori has mud mortar laid between the bricks and walls covered with adobe plaster. Left: SEM-EDXA of clay material in adobe from there indicates presence of silicon, aluminum, potassium, and iron. Since no large amounts of sodium are found, the clay is not the expansive, dimensionally-unstable sodium montmorillonite.

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Adobe materials have a wide range of compositions and mechanical properties. All types of adobe, however, are susceptible to damage from water. Structural integrity can be maintained by techniques to control rain water absorption on tops of walls, erosion by capillary action of ground water, and moisture penetration on vertical surfaces.

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