Routine and High-Volume Preparation of Embedded Coatings Crosssections

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Coating, crosssections, sample preparation, materials, embedding, molds, disk mold

ABSTRACT
This paper outlines the methods for embedding and preparing very small coatings crosssections. Paint and varnish materials, often of widely varying physical properties, are applied to many types of substrate. This varied nature, degradation, and restricted sampling possibilities, from historic and important objects, makes microscopy in the museum world unique. However, the techniques presented can be used to systematically prepare high-quality samples of various materials. They include low material cost, low volume preparation methods as well as rapid, high volume, higher capital investment methods. From these descriptions, decisions can be made within the individual laboratory's budget and needs. Though conceptually different, each of these reproducible methods will produce high quality crosssections. And while the methods and materials are described for coatings sections, a broader applicability to any small sample is apparent.

SAMPLE PREPARATION
Sample preparation is the determining factor in the success of most analytical techniques. This is also true for light microscopy. In many cases, decisions about whether to embed will be made before the sample has been removed from the object of interest. Depending on the level of magnification, successful analysis of microstructure may be totally dependent upon embedding and preparation of an optically flat surface. To prepare such a surface, embedding in hard resin is normally undertaken (Figure 1). For historic coating materials, it is required. Coating materials range in hardness from quartz to air; that is, there may be hard fillers, as well as porosity. Coatings may

Figure 1. Samples embedded in epoxy, cast in 8mm diameter molds, then polished using abrasives. Original magnification 100x: left, reflected bright field; center, reflected dark field; right, fluorescence.

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be thermoplastic, and therefore soft or soluble. They may be cross-linked and brittle. There may be little or no binder in paints, making them friable even if cross-linked. Historic coatings are often soluble in water and polar organics. There is more to gain than the preservation of morphology in an optically flat and perpendicular surface. In order to preserve true microstructure, embedding is required in nearly all cases. But while limiting in one sense, specifications for embedding technique and materials are indicated by the material properties.

Embedding preserves the microstructure because the sample is surrounded and supported by the resin. Furthermore, the resin may impregnate the sample. This may also be a problem, since pores and spaces in under-bound paints can be filled. Obviously, the benefits should outweigh the "cost" of embedding. Theoretically, the analysis of the organic chemistry of embedded samples should be possible. In practice, this has not been successful on historic or degraded samples. The resulting organic analysis, such as infrared and gas chromatography, is usually a generic characterization of organics. In part, this is because the amount of embedding resin is proportionately so great that it overwhelms the small amounts of original organic binder or varnish.

The method used to reveal this true microstructure is another major factor in preparation. The major techniques fall into two groups: microtomy, and grinding and polishing. The microtome produces relatively thin sections, but places enormous stress on the sample. The thin sections are literally split from the matrix. Brittle and porous samples are not good candidates for this method. Lower stress is placed upon an abrasive polished specimen. Microtomed sections are ideal for transparent samples. However, opaque paints will have to be very thin (a few microns) to transmit light. Microtoming a porous sample with quartz filler is exceptionally difficult. Another theoretical advantage of microtomy is in the production of serial sections. However, again due to the nature of most samples, serial production is highly unlikely. The successful use of the microtome is not a quickly acquired skill. On the other hand, abrasive grinding and polishing can produce excellent results even from the first hand-prepared samples. Consequently, I will describe the method best suited to virtually all samples: abrasive grinding and polishing.

**Increasing Efficiency by Embedding Format**

Sample embedding format (size, shape, mold material) is also important for routine and high-vol-

![Figure 2. At left, the holder for four 8mm diameter capsules; at right, the 20mm diameter epoxy disk.](image-url)
The second is a boat-shaped tablet 6 x 5mm by 14mm long. The sample is mounted to a tablet with slow-set cyanoacrylate. Permanent object data is placed on the tablet, directly or on a paper label (direct labeling with permanent markers has rarely been successful). The aluminum holder for the embedded samples will accommodate up to four at a time. With several holders, efficiency increases tremendously. The holder has an epoxy ring to offer a broad surface to maintain planarity. It is important to keep the ring face parallel to the back face of the holder. The ring can be dressed on the lathe, or disposable. In practice, the samples can remain in the holder throughout analysis and documentation. The great advantage of this is that the sample planes are parfocal, and by rotating the holder, each sample can be viewed in turn. The embedding form (since the holder is not in the path) also transmits light, which is useful for thin samples or at edges. Bright field, dark field, UV fluorescence, and transmitted light are typically used during examination.

The samples in holders can be prepared on abrasive papers and films, by hand or on motorized platens.

A further advantage comes from the holder when used in semi-automatic preparation equipment. I chose the general size of the holder to fit into two types of machine. The two main suppliers of semi-automatic equipment in the world today are Struers Inc. and Buehler Ltd. Molds from these companies served as the models for machining prototypes. Since the Struers mold was slightly smaller in size (30mm diameter), I chose that as the basic size of the holder. With a simple adaptor (a 35mm film can or analog), the holder will fit in the Buehler Minimet. For a current project with thousands of samples, I have chosen to use a combination of Struers semi-automatic equipment. The RotoPol-15/RotoForce-1 can prepare up to 12 samples at one time, and average machine time is about two minutes per sample. In this case, the efficiency of machine preparation is necessary, and easily justifies the cost.

The second embedding format was developed for use in teaching week-long coatings microscopy courses. The intention was to take advantage of the strengths of the capsule and holder, and a more low-tech path. The result was a disk form that was produced in two parts and would be prepared by hand. This form would produce high quality results, sacrificing only high through-put possible with the holders. The low start-up costs are also appealing to those who only occasionally need to prepare samples.

Most advantages come from a mold that produces the mounting piece and final form. This uniform round shape is easily held for hand preparation. The disk can be rotated on the microscope stage for the best orientation. If the disk is cast on a level surface, the front (sample) plane will be parallel to the back, thus keeping the sample in focus across its entire surface. The 20mm disk (Figures 2 and 4) fits onto a common microscope slide and can be permanently mounted. The disk is thin enough to transmit light, which can be very useful. Since they will ultimately be about the same height after polishing, it is easy to have several on a slide for rapid comparison. And since they are a standard size they can be easily stored in boxes or slide storage trays.
EXAMPLES OF THE PROCEDURES

A. Sample Holder

There are six main steps in the preparation of samples when the holder is used.

1. Mount the sample on the tablet. This requires the use of custom-made molds to produce the tablet. The sample is oriented close to the tip of the tablet to reduce the material removed to expose it. Samples are tacked in place with cyanoacrylate. (upper right in image, Figure 5)

2. Sample identification is marked on the tablet with pencil or on paper affixed to the tablet. (lower right in image, Figure 5)

3. Embedding. The liquid resin is poured up to about one-half of the depth of the capsule mold. The tablet is then slid into the mold (Figure 6). Air bubbles are minimized by having what is the eventual sample face at the bottom of the mold. The resin is preferably room temperature cure-type, clear, low viscosity, and non-shrinking. Solvent effects from resin or hardener are to be avoided. Out-gassing is minimized by at least a one-hour pot life. A warming oven may be used to promote cure, but temperature should be below 55°C. (Figure 7 shows de-molded sample)

4. Mill end and face after de-molding. This step, using a milling machine or lathe, can greatly expedite polishing. This is an added benefit made possible by the parallel sides of the capsule. (Figure 8, sample in chuck of small machinist's lathe)

5. Place in holder. Place the holder on a glass surface, and insert the capsules in face down. The glass can be used for reference, or shims (such as cover glass or tape) under the ring can be used to expose a little extra material. If the capsules are oriented in the same manner (I normally position them radially), they will all appear in the same axis under the microscope (Figure 9).

6. Polish the samples. Since abrasive size can begin at 30 micron or less, grinding steps are unnecessary. Therefore, only polishing is done, which greatly shortens the preparation time. In addition to time savings, there is less potential damage to the samples. Non-aromatic aliphatic hydrocarbon (typically, Stoddard Solvent) is used as the lubricant. (Figure 10 shows motorized polisher; sample holder is hand-held)

7. Documentation and analysis. The samples are now ready for rapid examination and documentation. Samples removed from the holder can be examined in the SEM as well. (Figure 11, ring bonded to slide allows holder to rotate on stage)

B. Disk mold

Essentially, there are only four steps to preparation by this method.

1. Mount the sample. The sample is tacked in place with cyanoacrylate as above. Identification can be written in pencil in the cove below the tip of the sample (Figure 12, detail of Figure 14).

2. Embed the sample. Pour a small pool, no deeper than ¼ the mold, into the full disk mold. Lower the mounted sample into the mold. Place on a level surface, and in a warming oven if desired (Figure 13).

Figure 5. Upper left, tablet in profile; upper right, tablet with sample mounted; lower right, labeled tablet.
Figure 6. Mounted sample on tablet being placed into capsule mold well (partially filled with epoxy resin).

Figure 7. De-molded sample after cure.

Figure 8. Optional preparation step: milling cured epoxy capsule speeds polishing.

Figure 9. Epoxy capsules placed face down into holder; glass surface used as reference.

Figure 10. Four samples in holder polished on motorized platen.

Figure 11. Holder fits on microscope stage, making rapid examination very simple.
3. Polish de-molded sample. A file may be used to remove sharp edges (use it for this purpose only). Polishing film or papers can be used, starting with 600 grit; aliphatic hydrocarbon (such as Stoddard Solvent) is used as lubricant. After establishing a flat plane, working quickly will yield the best results. (Figure 15)

4. Documentation and Analysis. Again, since the samples are approximately the same height, the sample plane will be nearly the same. Thin samples or edges can also be examined in transmitted light. (Figure 16)

Figure 12. Mounting the sample on pre-cast epoxy half disk.

Figure 13. Half disk with sample placed in partially filled mold well.

Figure 14. Top, embedded sample from mold; bottom, sample mounted on half disk.

Figure 15. Polishing by hand using aluminum oxide film on glass plate.

Figure 16. Examination of embedded sample. Disk fits on standard microscope slide, which also allows storage in standard containers.
SUMMARY

Using either of the two methods described, small samples can be routinely prepared, even in high volume, with no sacrifice to quality. Both methods described are more efficient than those currently in use for historic materials. The disk method in particular improves hand preparation, and at low start-up cost.

While designed to improve sample preparation of particularly small and valuable museum and art objects, there is a broader applicability. Small, fragile, solvent sensitive, mixed hardness, porous samples of materials science or forensic materials are good candidates for these methods. Excellent results will be achieved in almost all cases, whether high-volume or "one-off."
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