

EFFICIENT NEW METHODS FOR EMBEDDING PAINT AND VARNISH SAMPLES FOR MICROSCOPY

MELVIN J. WACHOWIAK JR.

ABSTRACT—This article outlines several procedures for embedding, including selecting an appropriate resin, establishing uniform formats for the embedding matrix, and increasing the efficiency of preparation. While the methods for embedding samples in use for more than half a century do indeed work, this article suggests improvements in format efficiency and materials that achieve the same high-quality results. Two new embedding procedures are described as well as a holder for simultaneous preparation of multiple samples. The article also describes the use of an industrial epoxy resin (Tra-Bond 2113, made by Tra-Con) that works well for a variety of sample types prepared by milling and abrasive polishing.

TITRE—Nouvelles méthodes efficaces pour encastrier des échantillons de peinture et de vernis pour la microscopie. **RÉSUMÉ**—Cet article décrit plusieurs modes opératoires pour encastrier des échantillons, c'est-à-dire comment choisir une résine appropriée, établir des formats uniformes pour la matrice d'encastrement, et rendre plus efficace le processus de préparation des échantillons. Bien que les méthodes utilisées depuis plus d'un demi-siècle pour encastrier des échantillons aient fait leurs preuves, cet article suggère des améliorations concernant les moules et matériaux qui permettent d'obtenir des résultats d'aussi haute qualité. Deux nouvelles méthodes d'encastrement sont décrites, ainsi qu'un support pour la préparation simultanée d'échantillons multiples. L'article décrit également l'utilisation d'une résine époxyde industrielle (Tra-Bond 2113, fabriquée par Tra-Con) qui fonctionne bien pour une variété de types d'échantillons préparés par fraisage et polissage abrasif.

TÍTULO—Nuevos métodos eficaces para montar muestras de pintura y barniz para microscopía. **RESUMEN**—Este artículo delinea varios procedimientos para montar muestras, incluyendo la selección de la resina apropiada, el establecimiento de formatos uniformes para la matriz y el incremento en la eficacia de la preparación. Si bien los métodos que

se han usado por más de medio siglo para montar muestras realmente funcionan, este artículo sugiere mejoras en el formato, la eficacia y los materiales con los que obtienen los mismos resultados de alta calidad. Se describen dos nuevos procedimientos para montar muestras, como así también un soporte para la preparación simultánea de múltiples muestras. El artículo también describe el uso de una resina epoxi industrial (Tra-Bond 2113, manufacturada por Tra-Con) que funciona bien para una variedad de muestras preparadas por medio de molido y pulido abrasivo.

TÍTULO—Novos métodos eficazes para impregnação de amostras de tinta e de verniz para a microscopia. **RESUMO**—Este artigo descreve diversos procedimentos para a impregnação de amostras, incluindo a seleção de uma resina apropriada, estabelecendo formatos uniformes para a matriz impregnada e aumentando a eficiência da preparação. Embora os métodos utilizados há mais de meio século para a impregnação de amostras se mantêm eficazes, este artigo sugere melhorias na eficiência do formato e materiais que alcancem os mesmos resultados de alta qualidade. São descritos dois novos métodos de impregnação, assim como um recipiente para a preparação simultânea de múltiplas amostras. O artigo também descreve o uso de uma resina industrial epoxi (*Tra-Bond 2113*, fabricada pela *Tra-Con*), que funciona adequadamente para uma variedade de tipos de amostras preparadas por moagem e polimento abrasivo.

1. INTRODUCTION

Beginning more than 150 years ago with natural resins such as Canada balsam, scientists have refined procedures for microscopical preparation of various materials. Paint and other coating materials have been embedded for at least a century, in the beginning with natural resins and waxes (Laurie 1914). The shortcomings of these natural plastic media were partially overcome with mid-20th-century use of

MELVIN J. WACHOWIAK JR.

catalyzed polyester and methacrylate resins. Several resins, including a number of acrylics, polyesters, and epoxies, have been empirically tested as researchers looked for the best qualities and working methods. The most popular resin seems to be the unsaturated polyester resin in styrene monomer, catalyzed by methyl ethyl ketone peroxide. But this resin has not been without problems, either initially or over time. For instance, Derrick et al. (1994) note the continued curing of polyester over a month, as well as a crumbling of samples. A recent article by Khandekar (2003) is a good historical review of the materials and practice in easel painting conservation studies. Waentig (1993) tested some commercial polyesters, acrylics, and epoxies and suggested the use of three polyesters and an epoxy for embedding.

Successful embedding of paint and varnish samples can be defined as implementing appropriate and efficient means to preserve and reveal the microstructure of the area of interest sampled. Selecting the appropriate combination of materials and techniques for unknown samples requires experience on the part of the analyst. This situation is different from an industrial or academic perspective, in which materials are often well known beforehand. At the outset of conservation studies, the physical and chemical properties of the materials in the cross

section are often unknown. The embedding process and preparation procedures should therefore be well defined and reproducible and work in practice for most materials. Bousfield (1992) is an excellent source of guidance on this point. In common practice, optical microscopy may be followed by electron microscopy. All preparation outlined below is to the standard of electron microscopy: the microstructure is preserved at high magnification, and the sample is all in the same optical plane. Using the methods and materials described below, samples can be prepared to this standard at the rate of two minutes per sample (fig. 1). The analyst must determine the relative value, but the cost of new materials is low compared to the time saved. For example, the first holders for four samples were produced for under \$100. Many benefits derived from the development of the high-volume procedure were applied to a simple system for hand preparation of small numbers of samples, described in appendix 1 (fig. 2).

For the balance of this article, it will be assumed that (as is most often the case with coatings) the nature of the sample dictates embedding. Samples are often very small, porous, and friable, and their outer edges need to be retained for microscopical observation. A sample or area of interest can certainly be investigated without embedding. For instance, a cross

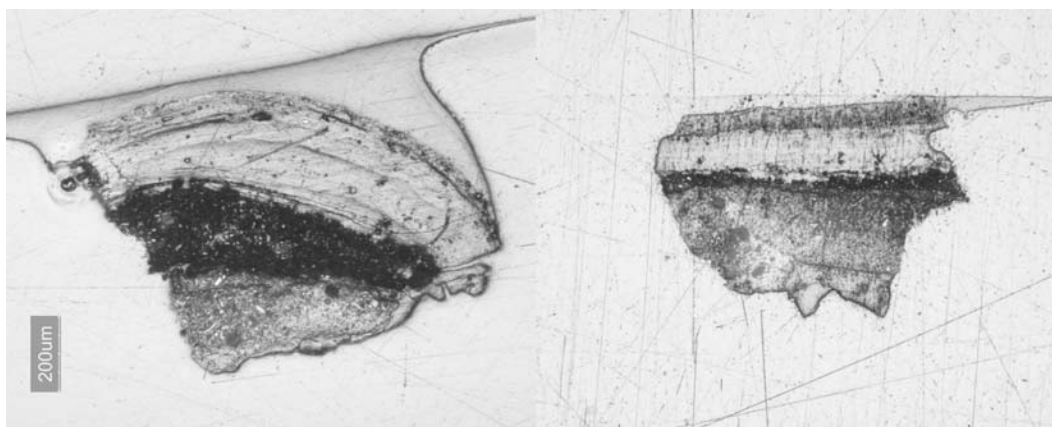


Fig. 1. Two samples from one object (18th-century English japanning): varnish over paint and ground, prepared by hand (left) for five minutes and by machine (right) for two minutes. Both are at the same magnification and lighting conditions. Bright-field shows scratches and other possible defects as well as planarity of samples. Hand-prepared samples typically show more relief than machine-prepared samples. Larger scratches from early polishing stages are visible in machine-prepared sample. For this test, 12 samples were prepared by machine in the same time as 4 samples prepared by hand.

EFFICIENT NEW METHODS FOR EMBEDDING PAINT AND VARNISH SAMPLES FOR MICROSCOPY



Fig. 2. Epoxy disc (left) and tablet (right) embedding forms, with holder for preparation and examination of four tablets. Preparation time is greatly reduced by mass production, but benefits are found in the application of principles to single samples (left).

section can be created with a few simple razor cuts, and the sample temporarily mounted on a stub for viewing under the microscope. However, the embedding process impregnates and supports the sample. Embedding can also achieve a uniform molded shape, which makes preparation, handling, and storage much more efficient.

Mounting materials, methods, and preparation techniques should not limit analysis of paints and varnishes. The choice should be guided by the need to exploit the optical microscopy contrast techniques available. Singly or in combination, bright-field, dark-field (with and without cover glass), fluorescence, and transmitted light can be very revealing in our studies. In fact, methods and materials should only enhance our analysis and increase our efficiency, if possible.

Some industrial epoxy resins not specifically formulated for microtomy or even polishing seem well suited to our task. The epoxy resin Tra-Bond 2113, made by Tra-Con, meets the selection criteria for conservation studies and criteria of Waentig (1993) and Derrick et al. (1994) (although not tested for microtomy). It is available in efficient, premeasured packaging that promotes reliability and reproducibility. The preparation techniques described below allow for systematic production of samples that can be easily monitored during the process and can be used in all optical contrast techniques.

2. DEVELOPMENT OF A STANDARD PROCEDURE

Modifying methods of Tsang and Cunningham (1991) and Martin (1992), and referring to earlier histological embedding, microtomy, and materials science procedures, produced a format for rapidly handling and processing small coatings samples. The methods described below have a number of advantages for those interested in preparing a few samples per month, or by the dozen. The holder used for the machine polishing can be held by hand; it is used to simultaneously prepare four samples, which can make the method up to four times faster than preparing single samples. Multiple holders fitted in semiautomatic preparation equipment further increase throughput.

The first conceptual step in development of the procedure was to consider the nature of paint and varnish coating materials. Coatings are generally described as plastic materials, and paint can be regarded as a filled or reinforced plastic. Therefore, the ideal embedding resin should have physical properties similar to this class of plastics. Since coatings are rarely composed of a single material, the model of a composite is appropriate. Another concept from microscopy of composites should be considered: preparation of only targeted areas. Sometimes there is a great disparity in the properties of individual components in a coating matrix, which can lead to preferential abrasion of a phase or layer. In this case, our target layers or zones will dictate the preparation scheme, and areas other than these will not be as well prepared. While the stated ideal is to produce a single flat plane containing all phases, it is not always required.

The next consideration was the form of the embedding matrix. The ideal would be relatively small, to negate possible heat and shrinkage effects. Second, a uniform shape would increase preparation efficiency. The embedding form is based on commercially available, rectangular, tablet-shaped microtomy molds. Initially, as many off-the-shelf components were used as possible to reduce cost and take advantage of established standards. Molds varying in shape and material and various resins and polishing methods have been tested. Section 2.2 discusses some of the advantages of rectangular-

section, tapered-tip forms. Empirical work has been directed in part by industrial and other research standards. The modifications described result in a systematic, efficient way to embed and process the samples in quantity. While polishing can be done by hand, further benefits can be realized from semiautomatic machine preparation. Holders were created to clamp four samples during preparation (in one machine, 12 or more can be processed simultaneously). While normally polished with fine abrasives, samples in this embedding form can also be clamped in standard equipment for microtomy and ultramilling. The final development has been to refine the methods and materials during routine work and microscopy courses.

Most of the capsule or tablet-shaped embedding methods in use can be traced to the decades-old gelatin capsule practices in histology and materials science. After embedding in a resin, the gelatin is washed off, leaving a 5–10 mm diameter shape that can be sectioned in the microtome. This method had no means to position the sample; it is simply allowed to settle in the bottom of the capsule. The cyanoacrylate method for mounting the sample described by Tsang and Cunningham (1991) is modified here. The “slotted capsule block technique” and “shelf” described by Martin (1992) also offered the advantage of orienting the sample to quickly arrive at areas of interest. Martin’s techniques utilized a hand-cut slot to cradle the sample and serve as a vessel for the temporary or permanent mounting medium. Once embedded, the sample could either be microtomed or polished. A version of the shelf, referred to as a half-tablet, is part of the method described here.

The first step was to mass-produce custom half-tablets with a step or shelf, created by milling away slightly more than one-half the thickness of the final tablet form. Several were milled at once, and the resulting model was cast in room temperature vulcanizing (RTV) silicone rubber (Plat-Sil 71-35). The stepped tablets were then cast in the rubber mold using epoxy resin (fig. 3). Mounting the sample at the edge of the middle of the half-tablet tip centered it in the final form and standardized location among the embedded samples (figs. 4 [center], 5). This procedure greatly speeded up embedding, handling, and analysis. Samples up to 5 mm

wide could be accommodated. By polishing only the tapered tip of the tablet, approximately 2 x 5 mm, preparation was greatly speeded up. The holder was needed to fix the orientation of the tablet tip with the sample, and it also maintained an optically flat surface.

2.1 MOUNTING

For both the tablet and disc formats, the basic steps are mounting the sample, embedding, polishing, analysis and documentation, and storage.

It is best to label the sample tablet with identification information before mounting. Two-line (8

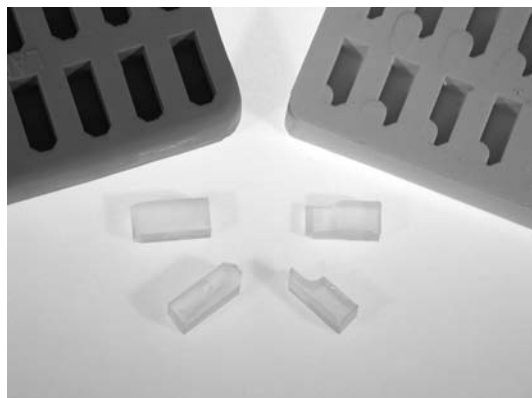


Figure 3. Epoxy tablet with shelf (lower right, custom-made mold above); final tablet form (left, commercial mold above)



Fig. 4. Essential materials for embedding, with tablet with sample on shelf at center. Clockwise from top center: samples in vial, labeled half-tablets ready to mount samples, adhesive-backed label paper, needle holders with fishing-line probes, tweezers, commercial mold for final casting

EFFICIENT NEW METHODS FOR EMBEDDING PAINT AND VARNISH SAMPLES FOR MICROSCOPY

mm or 2/6 in.) correction tape, cut into 5 mm pieces, works very well. A pencil with 0.5 mm lead has proven the best marker, as it will never bleed or fade (as do many “permanent” markers). The paper label becomes saturated with epoxy during embedding and is therefore quite durable. Pencil is also used to mark an abbreviated sample number near the tapered area at the tip of the tablet. This number is easy to find while samples are in a holder on the microscope stage. While miniature computer-generated printed labels do look elegant, they do not seem to offer any other advantage.

Mounting may be the preparation step that requires the most skill, patience, and time. As much as 15 minutes per sample may be needed for examination under the stereomicroscope, dividing the sample, and actual mounting. The sample is bonded to the precast epoxy resin half-tablet with cyanoacrylate to secure it in the desired location.

Cyanoacrylate adhesive with a 15–30 second open time seems best for most work. Open time is the period when all properties, such as viscosity, are still in evidence. Common cyanoacrylates (instant bonding Krazy Glue, etc.) have an open time of 10 seconds or less, simply too short a time to position the sample. Loctite 495 or Aron Alpha 202 have longer open times and are typically used. Gelled cyanoacrylates (such as Loctite 454) can be used and do sometimes lessen the

penetration of embedding resin (Chang et al. 2002). In any case, the cyanoacrylate will bond or consolidate depending on the amount applied, its viscosity, and the porosity of the sample. All that is normally needed is a small spot applied to an edge or from behind the sample. While consolidation or excessive amounts of cyanoacrylate will act as an embedding medium and are noticeable in the microscope, little difference is observed in the properties compared to the epoxy resin. Those mentioned above have virtually the same hardness and refractive index as the Tra-Bond epoxy resin, judging by optical properties and abrasion rate. They are not affected by milling, polishing, or the lubricants used. Cyanoacrylates are thermoplastic, but these are hard enough to compare favorably with the epoxy resin during polishing, and they fluoresce very little (similar to the epoxy matrix). The best way to apply small amounts of the resin is to transfer a droplet on the end of a needle tool, in the form of the beveled end of a 2–3 cm length of nylon fishing line (15 lb. test), clamped in a needle holder. The tip of the nylon line is dipped in a drop of cyanoacrylate and moved to the sample area on the half-tablet, or the drop of cyanoacrylate is wicked under the edge of the sample. The sample can be moved about with another probe made of fishing line (6–10 lb. test). These simple tools offer the right stiffness without the spring of wire needles or glass filament. They also can be cleaned and shaped and are readily available.

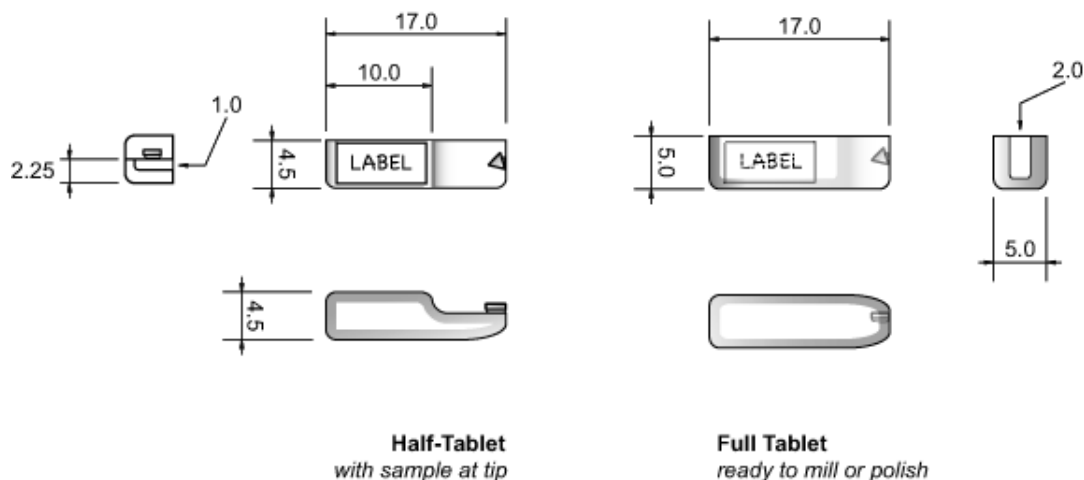


Fig. 5. Diagram of half- and full tablet

MELVIN J. WACHOWIAK JR.

Since the open time of the cyanoacrylate is fairly long, orientation is usually quite simple. One drop from the tube can be enough to mount more than 20 samples, and it will not become viscous for about an hour.

The sample is positioned near the tip of the half-tablet so that it requires little time or effort to polish it. Samples are typically triangular or rectangular in shape, and the broadest edge is usually mounted parallel to the tablet edge. The samples tend to be several times thinner than wide, so either the ground side (substrate) or paint face (exterior) is bonded to the surface. There is no established convention, and so the face bonded can be chosen from the following criteria. If one surface would be better preserved with consolidation, it should be saturated with the cyanoacrylate. For example, a very porous layer will be stabilized with consolidation. If the upper edges (the object's visible surface) are of great interest, the cyanoacrylate should be used on the opposite face. Finally, all other factors being equal, bonding the flat face of the sample to the half-tablet is easiest and fastest.

Minor revisions to the mounting procedure will enable the analyst to produce parallel, angled, wedge, and parallel serial sections. Angled faces of the half-tablet can be used, or the desired angle otherwise produced.

There are instances when poor edge retention, excessive relief, porosity, or erosion of the sample is observed during polishing. The cyanoacrylate is very effective as a consolidant even in these later stages. Capillary action is usually sufficient to fill the voids or saturate the sample. After cure, the sample is quickly repolished. UV-cure adhesives will also work, but only as thin films.

2.2 EMBEDDING

Consideration of embedding resins included a review of industrial and research selection criteria, and was based in part upon conservation needs. For a resin to be a true improvement, performance characteristics should increase both the quality and quantity of work. Table 1 compares some idealized selection criteria to the stated and observed performance of Tra-Bond 2113. For microtomy and micro-Fourier transform infrared (FTIR) analysis criteria, the reader should

consult Derrick et al. (1994) and Chang et al. (2002).

The main types of commercially available resins are in two groups: cold systems (such as room-temperature curing epoxy) and hot systems (such as phenolic resin powder cured in a high-pressure device at 140–200°C). Since coatings from the museum context are often heat-sensitive, it is best to eliminate hot systems. Chemically, the resins can be grouped as the thermoset polymers (epoxy, polyester) and thermoplastic polymers (Canada balsam, catalyzed acrylic, cyanoacrylate). The thermosets are generally better for our purposes, since they are more abrasion-, solvent-, and temperature-resistant. It is generally true for the cold systems that the faster the cure, or the larger the resin volume, the greater the cure temperature. Of the cold system thermoset resins, epoxies may be best for our purposes. Several authors discuss the relative merits, including Waentig (1993), who concluded that both polyester and epoxy would be useful. Derrick et al. (1994) discuss methods to improve polyester embedding for microtomy and binder analysis. Bousfield (1992) supplies very detailed descriptions of both preparation method and materials selection, to the point that he defines auditable laboratory procedures. Because of general performance qualities, Bousfield indicates that epoxy is preferable over polyester in materials science.

While acrylic and polyester resin fit some of the criteria, they fall short in important categories. Most important, both shrink considerably, and in larger volumes can cure in a comparatively high exothermic condition. In addition, typical polyesters used are softer than materials science epoxies, and they exhibit solvent effects (see Derrick et al. [1994]). For milling hard materials, both acrylic and polyester may be inadequate, since chip formation is very poor.

Several suppliers of embedding resins can be found, including microscopy supply companies and suppliers of materials science preparation equipment and consumables. Two major international companies, Beuhler Ltd. and Struers Inc., distribute several resins, including epoxies. These are available in 1 l kits and are reliable, general-purpose embedding resins. These are best described as potting resins, since they are meant to be cast into a vessel of some sort (typically 1 in. or 30 mm in diameter at the smallest). The Tra-Bond 2113 is

EFFICIENT NEW METHODS FOR EMBEDDING PAINT AND VARNISH SAMPLES FOR MICROSCOPY

designed as a general optical bonding adhesive. As such, it will cure in small amounts, particularly in laminates. It is exposed to light of various wavelengths throughout the service life of associated products (fiber optics, lasers, touch screens, etc.). These characteristics indicate potential application to embedding in very small volumes. Tra-Bond 2113 is one of a family of resins, selected for the closest match of product data to selection criteria listed above. This resin is cost-competitive, but at about 25¢ per sample, the resin cost is not a major factor. There is essentially no waste associated with the smaller packaging. Any resin left from casting is used to cast half-tablets to be used in the future. The bulk of the Struers and Beuhler 11 kits tend to remain past the six-month shelf life. The Tra-Bond 2113 resin and hardener are premeasured in precise proportion, and the dated, sealed packaging containing silica gel preserves the quality of the resin (fig. 6). There is no need for labor-intensive preparation, weighing with a laboratory balance, or specialized mixing. Together, these factors contribute considerable savings in time and money (fig. 7). The packaging also makes use in the field quite conceivable.

Silicone molds are used to produce half-tablets and the final embedding with the sample. A variety of hard plastic molds (BEEM, MicroMold, and others) have been used and have been lacking in one or more ways. For instance, the molds must be cut to remove the cured sample, which can be difficult to do safely. The unit cost per casting from plastic or rubber molds is essentially the same. But more important, all plastic mold shapes seem to prevent the sample from being embedded at the tip.



Fig. 6. Tra-Bond 2113 epoxy packaging. Epoxy is sealed in dated foil pouch (open at right), with silica gel packet.

This simply means more work is needed to expose the sample. For this reason, silicone rubber molds producing tablet shapes have been more useful (approximately 14 mm long x 5 mm wide x 6 mm deep, commercial or custom-designed). Using a tablet shape rather than cylinders offers several advantages. The tablet sits flat during mounting, and so requires no clamping to secure it. The rectangular cross section is self-jigging in holders. The tablet mold is milled precisely enough to produce a flat bottom face that is parallel to the tip. This feature will allow any sample to stand unsupported on the microscope stage for examination, should that be desired. The sample plane will be in focus across the entire field of view (fig. 8).



Fig. 7. Epoxy is mixed by sliding off center clamp (at left: catalyst in top portion, resin below); two-piece sliding clamp at center; corner cut open for pouring after mixing (small hole functions as dropper); unused resin allowed to harden prior to disposal.

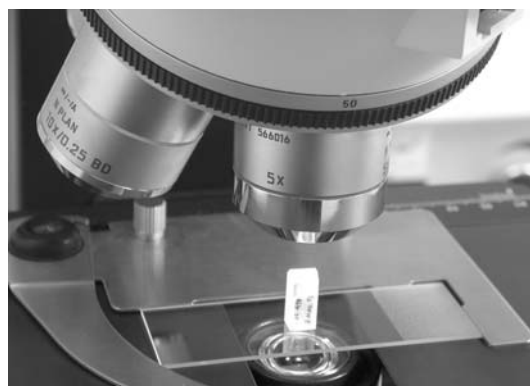


Fig. 8. Polished sample in tablet can be placed directly on stage for examination.

MELVIN J. WACHOWIAK JR.

Table 1. Explanation of Selection Criteria for Embedding Resins, Literature Values for Tra-Bond 2113, and Author's Comments

Primary Features		TRA-BOND 2113 (Tra-Con literature QAF-1057 Rev B)	Author's Comment
Supports and impregnates the sample	This property implies that the resin will conform to the outer surface of the sample—or even penetrate porous samples. This is a function of low initial viscosity, pot life, and good adhesion to a variety of materials. Epoxies are often noted for their exceptional bonding to the sample surface. Short pot life can lead to entrained air bubbles from outgassing sample; long pot life can cause overpenetration (all air spaces filled in cases of low binder concentration; see Derrick et al. [1994] for discussion).	Excellent flowability and wetting characteristics and adheres strongly to glass, ceramics, most metals, and many plastics. Lap shear, aluminum to aluminum, 2200 psi (2 hours at 65°C) Viscosity, 300 centipoise (after mixing, at 25°C) Pot life 30 minutes	Epoxies are often noted for their exceptional bonding to the sample surface. This quality will help retain edges of sample surfaces. Approximate viscosity of light oil; less than one-third that of glycerin; about one-half that of other epoxies tested. Good balance between outgas time and overpenetration (infiltration of porous samples).
Abrasion rate matches material	This characteristic will help produce an optically flat surface in the matrix. This is essential for microscopy. It is in part a measure of hardness of resin and sample phases. If the hardness is known for the resin, the sample and its phases can be compared.	Hardness, Shore D 88 Glass transition (T _g), 77°C	Hard or harder than other microscopy resins. Pigments and fillers tend to be harder, binders tend to be slightly softer.
Clarity	This property is very useful, especially when viewing the sample in transmitted light. A turbid or cloudy appearance can limit observations.	Clear (most Tra-Con epoxies of this type have near 100% transmission from 320 to 900 nm)	Clear
Colorless	In addition to being clear, the resin should be colorless.	Transparent	Slightly yellow in bulk (8 mm thick), but colorless under microscope.
Known refractive index	This quality gives another comparative tool to the analyst. If the refractive index is known, then the sample's phases can be compared to it.	1.58 (Becke line test by the author)	If the refractive index is known, then the sample's phases can be compared to it. Useful in transmitted light.
100% reactive	This percentage refers to formulations that do not include fillers or solvents. Only resin and hardener, in properly balanced amounts, are included. Fillers may be needed in some types to limit shrinkage (e.g., aluminum oxide, polymers).	100:11, resin to hardener, parts by weight	Only resin and hardener (no fillers). This ratio is the lowest of all the resins tested.
Room temperature cure	The resin should cure within 24 hours at or near room temperature. Many coating materials are heat-sensitive from the outset or over time. The rule of thumb adopted is the softening temperature range of beeswax. That is, warming temperature below 60°C is usually safe. While the typical epoxy resins will cure at room temperature within 24 hours, placing them in warming oven at 45 to 50°C for three hours produces the best cure within safe range.	24 hours at 25°C or 4 hours at 65°C	Hard and ready to polish after three hours in warming oven at 45–50°C, but best after additional 12 hours.
No shrinkage	Little or no physical interaction, which implies that the resin conforms to outer surface of sample and that it should not shrink. Similarly, low thermal expansion coefficient is expected. Shrinkage damage is seen in both a crushing of the sample and gap left by expansion differential.	Coefficient of expansion, cm/cm°C 0.00006	Little or no physical interaction (tensile or compressive forces) has been observed.

EFFICIENT NEW METHODS FOR EMBEDDING PAINT AND VARNISH SAMPLES FOR MICROSCOPY

No solvent effect	This type of chemical interaction is difficult to predict, but there is generally less with epoxies than polyester or acrylic resins. The benzoyl peroxide catalyst and monomeric styrene is one probable source in the polyester resin, and the dichlorobenzoyl peroxide catalyst in the acrylic system. Since many samples are of unknown composition and condition, solvent effects will be seen from time to time.	Resin: phenol,4,4,-(1-methylidene)bis-polymer with (chloromethyl) oxirane; hardener: diethylene triamine	Low catalyst proportion and short pot life may be predictor of low solvent effect.
Not exothermic	Temperatures above 60°C are risky for the reasons outlined under cure temperatures above. Some resins cure in exothermic reactions. Higher temperatures will often result in greater than normal shrinkage.	(no information; dependent on volume)	Volume of resin may affect temperature. Epoxies have negligible exotherm; this application uses about 0.5 ml.
Does not fluoresce	Fluorescence microscopy is an important technique in the characterization of coatings. While none of the resins commonly used is without some fluorescence, preference should be given to those with consistent and minimal effect. Known resin fluorescence may be potentially useful. In some cases, dyes are added in materials failure analysis to indicate voids and cracks.	(no information)	This resin is pale blue in UV (Leitz A), pale green in UV-blue (Leitz D).

Secondary Features			
Safety	Resins should be selected for low toxicity, including skin contact.	Hardener can cause severe eye irritation or burns, respiratory tract irritant. Prolonged breathing of resin vapors can cause irritation; skin irritant.	Packaging encloses both resin and hardener until ready to cast, greatly reducing exposure. Curing in container further reduces exposure.
Ease of use	Complex formulation of more than two components is unlikely to be favored. Imprecise weighing and mixing procedures will also lead to errors and inconsistent results.	A rigid plastic tube separated by a leak-proof clamp. Eliminate mix ratio errors with premeasured resin and catalyst components. Remove the clamp and mix right inside the BIPAX. Available in sizes from 2 to 200 grams.	Premeasured. This feature saves time and eliminates weighing errors and inconsistencies. Seven-gram size (BB-2113) is of greatest utility.
Long shelf life	Stability at room temperature is the ideal. For example, polyester is best kept at or below 15°C, which is seldom done in conservation studios. Epoxies, on the other hand, are stable without refrigeration for about six months from date of manufacture.	Prepared and dated for six-month shelf life at time of shipment.	Can be used successfully for up to a year; viscosity increase may be noted after one year.
No waste	The premeasured epoxy generates no toxic waste. In general, unused resin components are considered toxic waste. The best disposal method is to mix unwanted or expired resin in small amounts. Cured, these can be disposed of as solid, nontoxic waste.	Cured, remaining resin can be disposed of as solid, nontoxic waste.	Less waste generated due to smaller amounts ordered or stored beyond shelf life.
Long-term stability	The samples from objects are usually small and unique. The embedded sample should be considered part of the archival record. In this way, they will be available to future researchers, preserving the integrity of the object. Particularly poor storage characteristics have been noted for polyester embeddings done a decade ago. These include extreme yellowing, softening, and strong odor.	"...excellent resistance to weather, galvanic action, salt solutions, and many other chemicals, including mild acid and alkalis, petroleum solvents, lubricating oils, jet fuels and gasoline, and alcohol."	Good chemical stability and resistance to aliphatic hydrocarbons used during preparation. Samples 10 years old have no observable defects.

MELVIN J. WACHOWIAK JR.

There are several reasons for arriving at the small tablet shapes, compared to commercially available molds or the cocktail ice-cube tray. It is important to keep the mold size as small as practical to mitigate temperature and shrinkage effects. The rule of thumb is to have the mold only slightly larger than the sample. However, weighing such small amounts of resin precisely is critical. Since the total volume of 10 samples is less than 5 ml, precision is important. This requirement is resolved nicely by the premeasured Tra-Bond 2113 epoxy in BIPAX format. As an additional benefit, the small castings can be grouped in a holder for simultaneous polishing, saving considerable time. Uniform castings mean that several can be examined rapidly on the microscope stage. Groups of samples can be quickly compared, and the common format and material is a reference point.

The liquid epoxy resin is poured up to about one-third of the depth of the tablet mold. The half-tablet is then slid into the mold. Having what is the eventual sample face at the middle of the mold minimizes the chance of air bubbles appearing in the casting. A warming oven may be used to promote cure, but the temperature should be below 55°C (beeswax softening range is 60–65°C). The typical cure schedule is about 3 hours at 50°C, or about 24 hours at room temperature. Curing in the oven immediately after pouring resin often results in entrained air bubbles. Outgassing of the sample will naturally occur, but it will be virtually eliminated by a one-hour stage at room temperature. Empirical testing has shown this procedure gives the best combination of hard, bubble-free castings ready for polish within 24 hours.

One problem noted with silicone rubber is that new molds sometimes have uncured silicone on the surface (an oily surface is sometimes felt). One supplier suggested in a personal communication that new molds should be baked at 70°C for one hour, a procedure that seems to eliminate the problem (Electron Microscopy Sciences 1999). Before this procedure became routine, the sputtered carbon coating for scanning electron microscope (SEM) peeled from several epoxy castings from a new mold. As a matter of routine, all new molds are baked after receipt or fabrication, and the problem has not recurred.

Covering the mold during curing greatly reduces

loss of hardener and leaves a well-cured casting with no softness at the surface. Aluminum foil is adequate, but reusable food storage containers seem to work very well and are not affected by the uncured epoxy over time. These containers also virtually eliminate odors of the resin during cure.

Appendix 2 describes a number of modifications to the general procedure, including vacuum or pressurized impregnation and methods to lower the resin viscosity.

3. POLISHING

Since the now-embedded samples are near the very tip of the tablet, little preparation is required. In addition to time savings, there is less potential damage to the samples. Theoretically (as well as practically), grinding steps are not used, because abrasive grain size is 30 μm or less. Strictly defined, the preparation sequence is one of polishing steps only, not grinding.

An aluminum holder, designed by the author, holds the tablets throughout preparation and analysis of the samples (fig. 9). Up to four samples are clamped in the holder, which serves as a means both to position the samples for polishing and to expedite sample observation and documentation. Initially, the holder helps produce a flat optical plane during hand or machine preparation. Several of the holders can be used for large numbers of samples to shorten processing time. Stainless steel setscrews clamp each tablet in place. The 30 mm diameter holder is designed to fit in the most popular types of semiautomatic preparation equipment (manufactured by Struers Inc. and Beuhler Ltd.).

The holder has an expendable 40 mm stabilizing ring attached to the surface. The ring (cast of the same embedding epoxy or of milled plastic [fig. 10]) serves as the leveling device for the cross sections. It will abrade at the same rate as the epoxy castings. Since the upper surface of the ring is parallel to the bottom of the holder, the cross sections in each holder will be flat as well as parfocal. This planarity has been measurable to within a few microns over a 40 mm diameter, even after several polishing sequences. Planarity is maintained because of the small abrasive particle size used and consequent low

EFFICIENT NEW METHODS FOR EMBEDDING PAINT AND VARNISH SAMPLES FOR MICROSCOPY

material removal. It is actually difficult to distort a holder into a wedge shape, especially with such fine abrasives. The ring can be milled true, or replaced, if it does become out-of-parallel.

The holder, without ring, will also fit into a calibrated fixture made by Struers (fig. 11). This device is designed to remove specified amounts of material and is accurate in 10 μm steps. Stepwise removal of material in this fashion results in measurable serial sections. This procedure can be especially interesting

during “deconstruction,” or targeted removal of coating layers, either in cross or parallel sectioning.

While a simple holder for single samples has been designed and successfully used, it has limited utility beyond the classroom. It is made from a 1 in. aluminum SEM stub, bored out and tapped for the setscrew. A ring of extruded polycarbonate tubing, bonded to the surface of the stub, acts as a stabilizer.

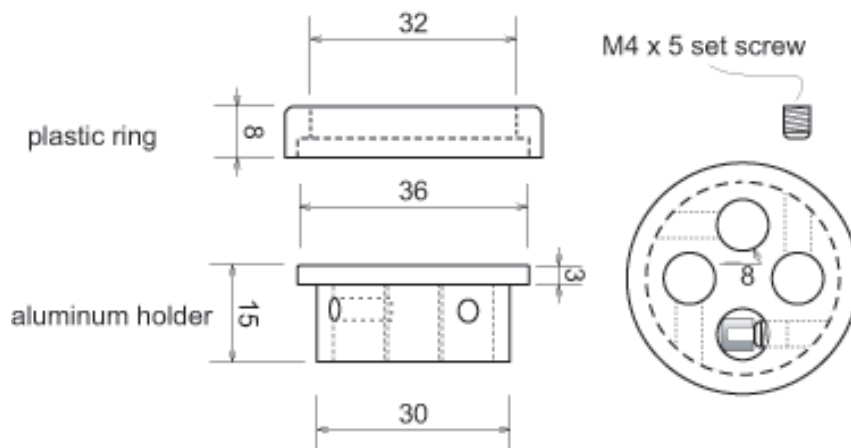


Fig. 9. Schematic drawing of the multipurpose holder.



Fig. 10. Holder with four epoxy tablets clamped in place (top) and replacement plastic ring



Fig. 11. Holder, without ring, can be used in Struers Accustop. This calibrated fixture (20 μm divisions) allows removal of measured amounts of material.

3.1 PLACING SAMPLE TABLETS IN HOLDER

To fix embedded samples in the holder, one inverts the holder on a glass surface and inserts the tablets tapered end down (fig. 12). A glass surface can be used for reference, or shims (such as cover glass, plastic film, or tape [fig. 13]) under the ring can be used to expose a little extra material. If the samples in each tablet are oriented on the same axis, they will all appear in the same orientation under the microscope. For example, each is clamped on the tangential axis (fig. 14). The rectangular tablet shape has another advantage over the cylindrical form: alignment of the tablet is simple, and cross sections in the holder are self-jigging. The upper mold face is also the clamping face, leaving the square cast edges to register on the walls of the holder. The “tripod” configuration ensures solid clamping, which will maintain consistent orientation. In this case, it also results in the long axis of the samples having the same orientation in the holder.

The preparation steps are monitored using reflected bright-field, which is particularly suitable for quality control. The sample holder simplifies this operation, since after each stage of preparation, it can be placed on the microscope’s stage and the samples examined.

There are a number of additional advantages to the holder and the uniform matrix. There are instances when one or more samples are not exposed sufficiently in early stages of polishing. With the holder inverted over tape spacers, the screws can be loosened and the samples advanced ahead of the others. Once documentation and analysis are complete, samples can be advanced as mentioned and repolished, creating serial optical sections. Embedded samples can be returned to the holder at a later date for another polishing and examination, such as after coating and SEM examination.

3.2 POLISHING MATERIALS

There are a number of factors to be considered when setting up a systematic preparation scheme. Abrasive type, grain size, whether fixed or loose grains, pressure, speed, rotation, lubricant, and time are the most important.



Fig. 12. Holder inverted on glass plate for registration of embedded samples. Adhesive tape on glass advances tip beyond ring edge prior to polishing (here, approximately 30 μm).

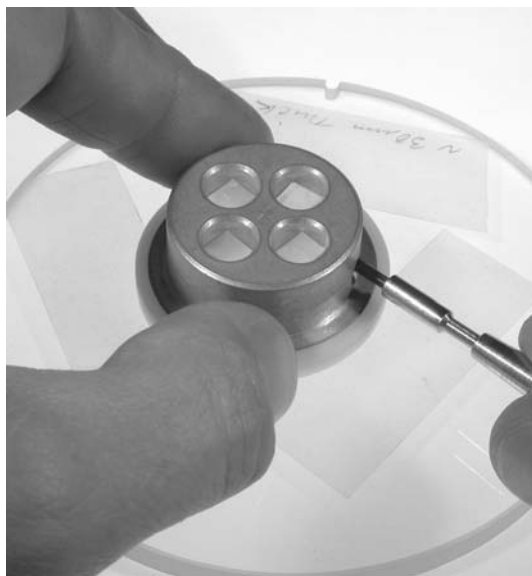


Fig. 13. Stainless steel setscrews clamp embedded sample in place. Samples are self-jigging with little effort. Tape serves as shim (approximately 30 μm in this case).

EFFICIENT NEW METHODS FOR EMBEDDING PAINT AND VARNISH SAMPLES FOR MICROSCOPY

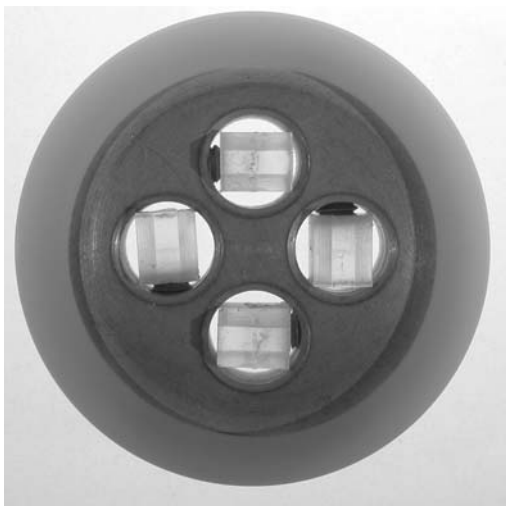


Fig. 14. Holder and samples viewed from above. Note tangential orientation of tablet.

3.2.1 Choice of Abrasive

Aluminum oxide bonded to polyester film is the abrasive of choice. Diamond is unnecessarily hard, incorrectly shaped for this purpose, and expensive. Silicon carbide particles are hard and sharp but break down far too quickly, and therefore particle geometry and size changes, and abrasive grains are often impressed into the surface (Bousfield 1992). The lower cost of silicon carbide is appealing, but the results are poor by comparison to aluminum oxide. The “blocky” diamond particle geometry is not ideal for plastic materials and in practice has given poor results. Fixed abrasive grains bonded to hard backing result in a flat plane with good edge retention of paint and varnish samples. This quality is important where phases or layers are of differing properties. Rubber or other soft backing should be avoided, as it causes unnecessary relief in the sample. Soft backings such as that of Micro-mesh silicon carbide sheets (Micro-Surface Finishing Products) were designed to remove scratches in aircraft windshields, and can polish beautifully, but they often give negative relief and poor edge retention.

Loose abrasive grains, even though pressed into a backing during polishing, are often caught in pores. The exception in practice is found in the final stages of sample polishing. At the 0.5 μm stage, loose abrasive on nonwoven backing outperforms that bonded to a film.

The abrasive film should be backed by a glass or plastic sheet (at least 6 mm thick) to maintain a flat plane. A light mist of water or Stoddard solvent will hold the glass or plastic sheet to the tabletop and the film to the sheet (or glass platen in the case of machine polishing).

3.2.2 Lubricant

A lubricant is generally advised in polishing, since heat and material buildup both cause problems. While the procedure can be done “dry” if needed, the results are typically not as good. It is important to remove epoxy and sample fragments cut by the abrasives. Without the lubricant, this swarf will build up and possibly adhere to the sample and abrasive surfaces. In addition, the swarf will quickly clog the abrasive film and shorten its useful life. However, there are instances where solubility problems cannot be avoided, so preparation is done without lubricant. Success in this case often depends on fresh abrasives and low pressure applied to the sample.

Water, detergent, oil, or glycerin is preferred in industrial applications, but most historic materials are sensitive to these lubricants. For historic coating materials, water or oil are not appropriate. Many materials are dissolved or swollen by water, and oils cannot be easily cleaned from the samples. Nonaromatic aliphatic hydrocarbon (typically Stoddard solvent, petroleum benzine, or equivalent) is usually the best lubricant. There is ample evidence in the literature of the beneficial use of these liquids. Proper procedures for extraction of the vapor must be followed during preparation. Most art materials are insoluble in the hydrocarbon, which is quite volatile. This volatility may be ideal, since if the lubricant is volatile in minutes, there is no need for a cleaning step, with its attendant dangers. Simple experiments, replacing the normally used hydrocarbon with higher boiling aliphatics such as tetradecane (as well

MELVIN J. WACHOWIAK JR.

as waxes, 1–5% in Stoddard solvent) work well, but the residues and cleaning are problematic. The observed oily-appearing residue of tetradecane may remain on the surface for hours and even days. It should be assumed that it would also wick into and remain in porous samples. In practice, the mixed aliphatics, such as Stoddard, work well in virtually every situation. More volatile solvents do not lubricate the work under standard conditions. In those few cases where phases or layers are removed, water or no lubricant may be a better option.

Where elemental analysis is carried out, the analyst should be aware of contamination (redistribution of particles) from polishing. This contamination is another reason to use a lubricant. In some cases where aluminum oxide may compromise elemental analysis, another abrasive such as cerium oxide might be chosen. Ultramilling, microtomy, and mounting a bulk specimen are other alternatives.

3.3 POLISHING BY HAND

Polishing by hand using the holder begins at 20 μm , progressing to 12, 5, 1, and finally 0.5 μm . This last is loose abrasive charged on a hard nonwoven fabric platen (ShaneyPol Medium, South Bay Technology). This sequence fits with general practice, which suggests that no more material than three times the grit size should be removed at each stage, a precaution that will help prevent deformation of the sample.

No more hydrocarbon is dropped on the surface than is necessary to easily move the holder. About five drops is sufficient per two minutes polishing. A figure-8 pattern over the abrasive surface (fig. 15) will give the most easily reproduced random pattern. This action will keep the ring and the sample surface flat and parallel to the bottom of the holder. It also allows more of the abrasive surface to be used. With the use of light pressure, each stage should be finished in about five minutes. Neoprene or other resistant gloves should be worn.

The holder and samples should be carefully cleaned between stages to avoid transfer of abrasive. Cotton swabs or tissue paper can be used. The best material by far is a cotton lithographer's pad, which is soft and leaves behind very little lint. Paper tissues can

scratch the highly polished surface. Washing the surface with hydrocarbon, or ultrasonic cleaning, has yielded no noticeable benefits. In fact, sonication can actually cause material to be lost. Final cleaning of the surface is best done with a foam swab, which will remove fine particulate and lint.

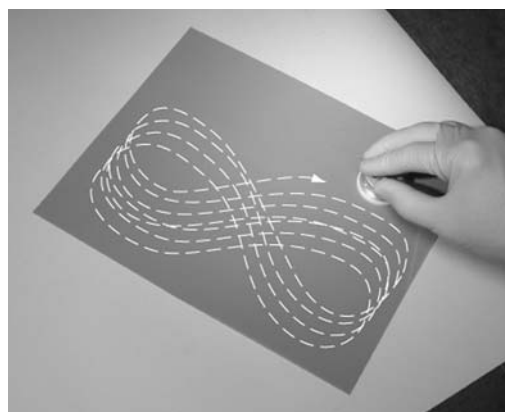


Fig. 15. Figure-8 motion used to randomize polish and maximize surface of polyester-backed aluminum oxide film

3.4 MECHANICAL PREPARATION

3.4.1 Milling the Samples

As mentioned earlier, when using the flat tablet molds, the sample is at the tip as cast in the mold. This means that the milling is not required if a little more time is spent polishing. However, milling the casting after cure using a vertical mill or lathe not only expedites polishing, it eliminates guesswork. Milling can—in seconds—remove microns of material to reveal the sample's region of interest. Monitoring the process using a stereomicroscope facilitates the targeted removal. Milling, made possible because of the parallel sides of the cast tablet, can even be a final step in some cases. When faced with dozens and dozens of samples to prepare, any reliable means to expedite the work should be exploited.

A small mill or lathe can be used for this and other purposes as well (Sherline, lathe approximately

EFFICIENT NEW METHODS FOR EMBEDDING PAINT AND VARNISH SAMPLES FOR MICROSCOPY

\$600, or mill, \$1,000). Leica manufactures the only precision trimmer made for milling prior to ultramicrotomy. The EM Trim (\$9,000) (fig. 16) is equipped with a prealigned stereomicroscope. This milling step, though apparently an additional operation, has led to the elimination of the first polishing step. The milled surface from the diamond bit is essentially polished. Therefore, the 12 μm polishing is the first stage in mechanical preparation. Milling on the EM Trim takes little more than a minute, and the microscope allows monitoring the controlled removal of material from even the smallest samples. The key idea, whatever the means used, is that the entire process can be monitored as microns are removed at each pass.

3.4.2 Polishing Wheel

Very good results, with much less effort than hand preparation, can be achieved through the use of a motorized polishing wheel (beginning at about \$1,000, Model 900, South Bay Technology). Essentially, the materials are unchanged from the hand polishing, but more samples can be processed in the same amount of time. Glass platens are available, and the abrasive film can be held in place with water as before. Cleaning between stages is as outlined for hand polishing. The motor is enclosed in the housing, so is shielded from the small amount of hydrocarbon. A dropper bottle can be mounted above the disc to dispense the lubricant, or a peristaltic pump can be used to ensure well-regulated flow. Extraction of vapor from above the platen is essential, and the bowl drain should be plugged with a stopper.

3.4.3 Semiautomatic Polishers

The principal advantage over hand polishing and the motorized wheel is the reproducible setup and economy of scale. Once an appropriate method is determined, it can be applied with low risk to a large group of samples. Time, pressure, and rotational speed can all be set with the microprocessor control. Custom-made abrasive films and other accessories are available from the manufacturer.

Whole groups of samples can be processed at once, limited only by the number of holders. Several manu-

facturers of material preparation equipment offer models that may be suitably modified. The custom-made sample holder has been designed in a standard diameter for industrial sample, to fit a number of machines. The Struers RotoPol-15, RotoForce-1, and Lupo dispenser are currently being used (fig. 17). A sample mover platen with three positions will carry up to three holders. Therefore, up to 12 samples may be processed simultaneously (fig. 18). The polishing stage may be repeated with additional holders. However, even a single holder may be used, since the sample mover adjusts the pressure for the number of holders present. The typical sequence of conditions for semi-automatic preparation for paints and varnish samples is found in table 2. Tests conducted to arrive at this sequence included varying abrasive grain size, speed, force, lubricant volume, and time for each stage. Interestingly enough, more time than noted does not produce any apparent advantages. This result is probably due to the fact that after a flat plane is established, subsequent steps only reduce the scratch size. Damage to microstructure is most likely to occur in the first



Fig. 16. Leica EM Trim used for ultramilling. This machine allows rapid exposure of the sample with great control.

steps, but samples can also be damaged by overlong preparation (Bousfield 1992).

Since the samples remain in the holders from polishing through the analysis and documentation steps—and can be checked at any stage—it is a well-controlled, efficient procedure. Although the Struers equipment is not designed specifically for this purpose, after two simple modifications, it has performed very well. The built-in water lines for lubrication and waste removal are not used. The Lupo unit is meant to dispense abrasive suspensions, but it will deliver the hydrocarbon lubricant with a Teflon tape added to better seal the bottle to its gasket. One to two drops per second are delivered. Again, the drain line should be plugged and vapors extracted from above the platen. The machine currently in use has been installed in a fume hood.

4. ANALYSIS AND DOCUMENTATION

After polishing, the samples are ready for efficient examination, analysis, and documentation. Samples removed from the holder can be examined in the SEM as well.

A ring bonded to a glass slide (fig. 19) is used to position the holder on the microscope stage. The ring



Fig. 17. Semiautomatic polisher (based on Struers RotoPol-15) for processing up to 12 samples in three holders simultaneously

has a 30 mm inner diameter, thus allowing rotation of the holder. Each sample tablet surface is in the same plane, so even short working distance objectives can be swung in and out of place, and the holder can be safely rotated as well (fig. 20). Since each sample has been positioned near the center of the tablet, each can be found—in focus—at or very near the previous field of view. This speeds up not only examination, but digital imaging or film documentation as well.

5. STORAGE

When the documentation is finished, the embedded samples are removed from the holder and stored in plastic boxes. Polystyrene matchboxes (Electron Microscopy Sciences) (fig. 21) will hold 10 tablets.

Archival storage, in the true sense, would require more controlled conditions. The polystyrene plastic box may affect the embedded samples over time. The epoxy resin is intended for electronics and optical bonding applications where the use is constant but

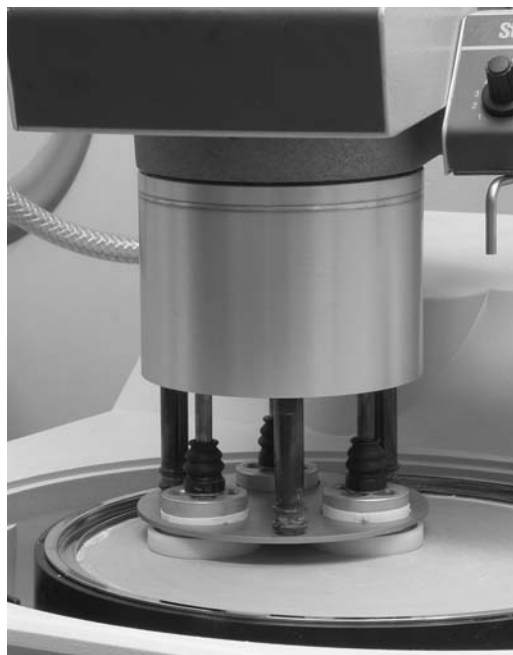


Fig. 18. Detail of Rotoforce-1 motorized sample mover. Pressure is controlled by adjustable air pressure pads over each holder. This head rotates at 60 rpm, while large platen rotates at adjustable speeds.

EFFICIENT NEW METHODS FOR EMBEDDING PAINT AND VARNISH SAMPLES FOR MICROSCOPY

the service life is unknown. However, the resin must remain clear and colorless during use, and our examination period is quite short by comparison. More important, the exposed samples will have to be held in desiccated storage conditions. Metal leaf and paint can corrode in just a matter of days, and some materials are so hygroscopic that they form salts on the surface of the prepared section. Desiccant cabinets for all samples are certainly not practical, but the specialized need is worth anticipating.

6. SUMMARY: TOWARD A STANDARD FOR EMBEDDING?

The most time-intensive step of analysis of paint and varnish cross sections may be revealing the microstructure of the coatings and substrates. In conservation, samples are typically prepared by abrasive polishing, and often in nonuniformly shaped matrices such as truncated pyramids from cocktail ice cube trays. These difficult-to-hold shapes must be prepared one at a time. The variable shapes often force the analyst to adjust the prepared sample's orientation along three axes, supported in clay, which prevents the use of transmitted light. A standard form or matrix for embedding would allow efficient preparation, examination, and documentation of many samples. Although sample preparation and protocols exist in biology and materials science, they are not immediately applicable. While the methods for embedding samples in use for over a half-century do indeed work, this article describes improvements in format efficiency, materials, and production of high-quality results.

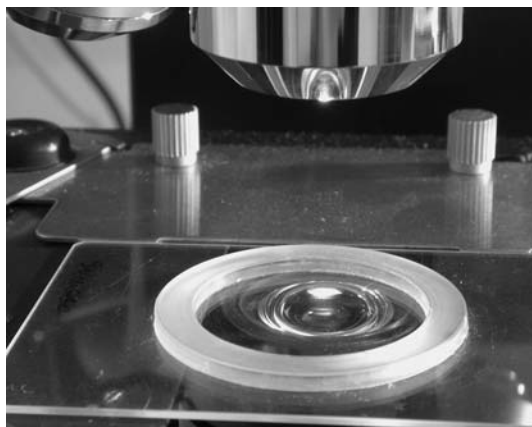


Fig. 19. Milled epoxy ring serves as guide for rotation of holder with samples. The ring is bonded to a 50 x 75 mm glass slide.



Fig. 20. Holder under 10x bright-field/dark-field objective. Since the sample surface and ring are parallel to the base of the holder, all samples are flat and in the same plane. The holder can be rotated to the next sample without losing focus or damaging objectives during rotation.

Table 2. Polishing Sequence Using Struers Polisher

Polishing Step	Time (minutes)	Force (Newton)	Speed rpm	Lupo setting
12 μ film	5	10	300	6
5 μ film	5	5	120	5
1 μ film	5	5	60	4
0.5 μ ShaneyPol Med.	5	5	60	4

This article also describes a new resin and two embedding procedures, as well as a holder for simultaneous preparation of multiple samples. Fixtures in the form of aluminum holders or mold-formed discs produce a surface that is parallel to the optical axis of the microscope as well as flat across the surface within microns. This feature allows the analyst to confidently prepare large numbers of very small samples and to concentrate on interpretation and documentation of structure. The methods are also easily learned and are applicable to machine or hand polishing. The methods and materials are effective for preparation of cross sections of coating samples, but broader application to other materials should be apparent. Minor revisions to the mounting procedure will enable the analyst to produce parallel, angled, wedge, and serial sections. Applications can include other materials that are degraded or solvent sensitive, water-soluble materials, and samples such as films, yarns, and fibers.

Even though samples embedded nearly 10 years ago show no noticeable flaws, this assurance is not enough. In the future, long-term and accelerated aging of Tra-Bond should be undertaken. Not all epoxies age well. Microtomy of Tra-Bond 2113 has not been attempted. Other epoxies have interesting properties, such as the related Tra-Bond 2112 and lower-viscosity F113. Other volatile lubricants could be tested, or cleaning procedures could be developed to remove polishing residues.

A case can be made for evaluation of these and other current methods, as well as previous methods and materials, to determine whether a standard

method is desirable. Standardization should also increase the utility of samples, which can then be confidently reexamined in the future or even exchanged among analysts as part of a large study. While a general method may work most of the time, there is certainly a place in conservation practice for several techniques. In some cases, a wax embedding may be more suitable than epoxy resin. Grinding and polishing work well for many types of material, but the microtome has advantages in the preparation of new or modern varnishes. Control of our methods and materials will enable better description of the sample, repeatable success, and the ability to trace a fault to its source. The methods described above have worked well with a wide range of material, from ethnographic to fine art and architecture, ancient and modern, and using a variety of optical techniques.

APPENDIX 1. MODIFIED METHOD FOR HAND PREPARATION: DA DISC

While the technique and materials described above have many positive features, can they benefit the practitioner with only occasional needs? A system with most of the features in a simplified form was created for use in the training of coatings microscopy. A conscious effort was made to retain the most important features and eliminate only those important for mass production. The result was a disc form that was produced in two parts and would be prepared by hand. This form produces high-quality results, sacrificing only high throughput possible with the holders. The low start-up costs are also appealing to those who only occasionally need to prepare samples. The new system was dubbed "DA Disc" and it is based on the same resin system and a similar mounting technique.

The ultimate form is a 20 mm diameter disc, about 6 mm thick (fig. 22). The disc has a center portion similar to the tablet, as it encapsulates the sample. The perimeter of the disc is a raised ring that keeps the sample in plane and minimizes the material removed in polishing. Since little material needs to be removed, the samples are discs that are about the same height (thickness) after polishing. A custom-

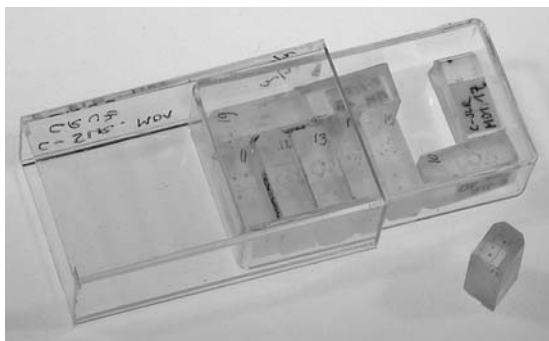


Fig. 21. Commercially available plastic matchbox is an ideal inexpensive storage solution for 10 tablets.

EFFICIENT NEW METHODS FOR EMBEDDING PAINT AND VARNISH SAMPLES FOR MICROSCOPY

made mold is used to form the platform for the sample, and another for the final disc shape (fig. 23). The first portion is a precast for mounting the sample. If the final casting is done carefully on a level surface, parallel planes are ensured. The polishing can be accomplished using the same methods and materials described above. The disc can be hand-held during polishing (while wearing appropriate gloves). Although this method is not intended for high-volume work, excellent results can be obtained at low cost (for more detail and a comparison of methods, see Wachowiak 2002).

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 "table."
2. Embed the sample. Pour a small pool, no deeper than half the mold, into the full disc mold. Lower the mounted sample into the mold. Place on a level surface, and in a warming oven if desired.
3. Polish the demolded 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. Working quickly will yield the best results.



Fig. 22. Polished disc for easy production of single high-quality embedded samples.

4. Document and analyze. Again, since the samples are approximately the same height, the sample plane will be nearly the same.

APPENDIX 2. MODIFYING EMBEDDING PROCEDURES

Under some circumstances, it is useful to modify normal procedures. Experiments were conducted to see if any would be useful in the processing of typical coatings samples. Benefits and deficiencies for several procedures are listed below.



Fig. 23. Silicone rubber mold produces half- and full-disk forms.



Fig. 24. Kitchen fruit and vegetable spinner serves as an inexpensive, highly effective centrifuge.

1. Centrifuging

Experiments have shown that centrifuging to remove bubbles of the mixed epoxy gives excellent results. While an expensive laboratory centrifuge will work, it can actually separate the phases of the epoxy if too much speed or time is used. Approximately one minute in a “salad spinner” (\$15) (fig. 24) works at least as well as in the laboratory centrifuge. The closed plastic envelope of the Tra-Bond BIPAX makes this a clean, easy step. The spinner is used as part of our normal procedure (after mixing the resin and hardener) rather than vacuum or other methods described below.

2. Heat

Warming epoxy resins for a few minutes after mixing (about 50°C for 10 minutes) will lower the viscosity. This result will help resin infiltration and allow small bubbles to rise and break before pouring. However, the pot life can be shortened to 15 minutes or less, depending on the resin mass.

3. Excess Hardener

Adding more hardener (10% or so) will also drop the viscosity. This step will lead to solvent effects in some cases. There may also be shortened cure, increased hardness, shrinkage, and yellowing of the cured resin. Separately packaged systems can be easily modified, but it is impractical with the prepackaged Tra-Bond BIPAX.

4. Vacuum

For more robust but porous samples, the vacuum method may produce good results. Placing the sample in the mixed resin in a partial vacuum can help remove bubbles and increase infiltration. The vacuum is created and then released several times to replace air with resin. But because the hardener is now more easily volatilized, an additional 10% or more must be added to ensure proper curing. As a result, solvent effects may be seen. Care must be exercised to prevent overheating the resin by creating too

strong a vacuum, volatilizing hardener, or entraining air bubbles during accelerated cure.

5. Pressure

Like a vacuum, increasing the pressure in a container can promote infiltration and decrease bubbles. Conceptually, this method works by compressing the volume of resin, thereby displacing air. Tests using castings without samples were indeed very clear. In practice, porous samples still produced more than the acceptable number of bubbles trapped in the embedding. Since the cure is accelerated, bubbles from outgassing samples are evidently trapped in the epoxy.

6. Solvent Ethanol

Ethanol is miscible with the resin and hardener, so it has been suggested as an additive to lower viscosity. Generally speaking, coatings samples are affected by exposure to polar solvents such as ethanol. Therefore, this additive is not normally used. For samples such as fibers or charcoal, added ethanol may be beneficial.

7. Extended Time

If the sample is particularly porous, the best method appears to be to exploit a longer pot life. In this case, the sample (as well as the resin) is allowed to outgas. This process can happen over several hours when curing at room temperature.

REFERENCES

- Bousfield, B. 1992. *Surface preparation and microscopy of materials*. New York: Wiley.
- Chang, W.T., T.-H. Chen, C.-C. Yu, and J.-Y. Kau. 2002. Comparison of embedding methods used in examining cross-sections of automotive paints with micro-Fourier transform infrared spectroscopy. *Forensic Science Journal* 1(1):55–60.

EFFICIENT NEW METHODS FOR EMBEDDING PAINT AND VARNISH SAMPLES FOR MICROSCOPY

Derrick, M., L. Souza, T. Kieslich, H. Florsheim, and D. Stulik. 1994. Embedding paint cross-section samples in polyester resins: Problems and solutions. *Journal of the American Institute for Conservation* 33(3):227-45.

Electron Microscopy Sciences. 1999. Personal communication. Product technical support. Fort Washington, Pa.

Khandekar, N. 2003. Preparation of cross sections from easel paintings. *Reviews in Conservation* 4:52-64.

Laurie, A. P. 1914. *The pigments and mediums of the old masters*. London: Macmillan.

Martin, J. S. 1992. Observations on cross-section examination of painted and coated surfaces. *American Institute for Conservation Paintings Specialty Group post-prints*. American Institute for Conservation 19th Annual Meeting, Albuquerque. Washington, D.C.: AIC. 39-42.

Tra-Con. 2003. Product literature QAF-1057 Rev B. Bedford, Mass.

Tsang, J.-S., and R. H. Cunningham. 1991. Some improvements in the study of cross sections. *Journal of the American Institute for Conservation* 30(2):163-77.

Wachowiak, M.J. 2002. Routine and high-volume preparation of embedded cross sections. *Microscope* 50(4):147-53.

Waentig, F. 1993. Gießharzsysteme zum Einbetten von Proben. *Restaura* 3:195-99.

SOURCES OF MATERIALS

The Smithsonian Institution has licensed McCrone Microscopes and Accessories (850 Pasquinelli Dr., Westmont, Ill. 60559; [800] 622-8122) as the supplier of holders and molds designed by the author, as well as resins, abrasives, and other supplies. Licensing fees support Smithsonian Institution programs and research.

Tra-Bond 2113 epoxy resin

Tra-Con Inc.
45 Wiggins Ave.
Bedford, Mass. 01730
(800) 872-2661
(781) 275-6363

Loctite 495, Loctite 454 adhesives

Techni-Tool
1500 N. Trooper Rd.
Worcester, Pa. 19490-1117
(610) 941-2400

Aron Alpha 202 adhesive (industrial Krazy Glue)

Ted Pella, Inc.
P.O. Box 492477
Redding, Calif. 96049-2477
(530) 243-2200

Plat-Sil 71-35 silicone rubber molds

Polytek Development Corp.
55 Hilton St.
Easton, Pa. 18042
(610) 559-8620

Small machinist lathe and vertical mill

Sherline Products Inc.
3235 Executive Ridge
Vista, Calif.
(800) 541-0735

MELVIN J. WACHOWIAK JR.

Leica EM Trim

Leica Microsystems Inc.
Specimen Preparation
2345 Waukeegan Rd.
Bannockburn, Ill. 60015
(856) 866-0820

Aluminum oxide abrasive bonded to polyester film,
ShaneyPol Medium, Model 900 polisher

South Bay Technology
1120 Via Callejon
San Clemente, Calif. 92673
(800) 728-2233

Plastic matchbox, cat. 64335

Electron Microscopy Sciences
P.O. Box 251
321 Morris Rd.
Fort Washington, Pa.
(215) 646-1566

Stoddard solvent, warming oven; Fisher 500 Series
Isotemp gravity-convected oven, Model 506G

Fisher Scientific
600 Business Center Dr.
Pittsburgh, Pa. 15205
(800) 926-0505

Struers RotoPol-15, Rotoforce-1, Lupo dispenser

Struers Inc.
24766 Detroit Rd.
Westlake, Ohio 44145
(888) 787-8377

MELVIN J. WACHOWIAK JR. is a senior furniture conservator at the Smithsonian Center for Materials Research and Education (SCMRE). He joined the Smithsonian Institution in 1989 after graduating with an M.S. from the Winterthur/University of Delaware Art Conservation Program. He was director of the Furniture Conservation Training Program at SCMRE from 1996 to 2002. His current work is research on the materials and technology transfer of painted Spanish and Native sculpture in colonial California and northern Mexico.

Received for review on July 30, 2003. Revised manuscript received May 3, 2004. Accepted for publication May 24, 2004.

Reprinted from the Journal of the American Institute for Conservation
with the permission of the American Institute for Conservation of
Historic & Artistic Works, 1717 K St., NW, Suite 200, Washington, D.C. □
20006; info@aic-faic.org; www.aic-faic.org. □

□
□