## A LOW-TECH EMBEDDING TECHNIQUE FOR COATINGS SAMPLES

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One sultry July morning, after spending several hours cutting away excess polyester casting resin from paint samples taken and embedded the previous afternoon, grinding, polishing, and examining the cross-sections, only to discover that the chosen samples were not adequate to “tell the story” of the succession of coatings this particular chair had received, we decided that there *had* to be an simpler and less time-consuming method of embedding and exposing cross-sectional paint samples. We discussed and eliminated many possibilities, but kept returning to Jamie Martin’s work with embedding samples in the small end of bullet-shaped forms of polyester which had been poured into beam-capsules. Mr. Martin would notch the small end of the form, place the paint sample into the notch, and affix it to the form with a drop of either Bioplast or hard dental wax, depending upon whether he wanted to be able to retrieve the sample and replace it on the painting.

Not having hard dental wax on hand, we decided to try the technique with the hardest wax the lab did have, a block of pure carnauba wax. We discussed what form we might want to cast the polyester holder into to facilitate polishing, the most obvious being the 1 1/4″ disc used in the Buehler polishing machine, but being impatient, we wanted to test the wax before proceeding. The solubility of the wax in stoddard solvent, which was commonly used under the cover slip, was a primary concern, so a shallow 3/16″ diameter hole was drilled into a piece of scrap 1/4″ plexiglass, a knife was heated, and wax was melted into the bottom of the hole. At first we attempted to set a flake of paint into the still-molten wax, but discovered that the molten state was very temporary; the wax cooled faster than one person could move the sample. The knife was heated and plunged into the wax, making a vertical slit in which the sample could be balanced, with approximately half of the sample protruding above the plane of the plastic sheet. Additional wax was dripped over the sample, the knife was heated and inserted alternately on either side of the sample to eliminate air bubbles next to the sample, and all was allowed to cool. within two minutes we were able to shave the bubble of wax/sample almost flush with the plexiglass with a scalpel blade, and begin grinding and polishing. Using water as a lubricant, in another few minutes we were able to progress through 400 grit, 600 grit, and 1.5 micron cerium oxide mylar, and put the sample under the microscope, with water under the coverslip. Due to the flatness of the broad sheet of plexiglass, it had been necessary to use the coarser grits only to remove the wax which was standing proud of it.

Under magnification, the paint sample was eminently readable; the wax was naturally colored a dingy tan, but a fair amount of translucence is apparent under magnification. The color and opacity of the wax doesn’t interfere with the clarity of the image, nor is the transparency of polyester casting resin missed. The cover slip was removed, and the topography of the wax fill was examined under reflected light, to see what effect the grinding may have had on it and the sample, since we were concerned that the wax might have been mushed over atop the sample. At 1OOX, reflected light showed that the scratches from the sandpaper progressed unbroken from the plexiglass across the wax, and the wax was still slightly proud of the plastic; no rolling of the wax onto the sample was evident.

Pleased with preliminary results, we tested various solvents on a chunk of the wax, and examined the spots under 30X magnification; stoddard solvent, our chief concern, had no discernable effect. Nor did ethanol, isopropanol, or acetone; xylene slightly polished the wax. Toluene was not tested because of its known effect on plexiglass. Obviously, one would not use these under a cover slip simply to clarify the image, but might want to perform micro-solvency tests on the sample; if so, impregnation of the sample with dissolved wax would be undesirable, as would dissolution of the wax matrix by the solvents in fluorescent dyes.

We next fabricated a sample holder which would hold more than one sample; the spring-loaded arm on the stage of our microscope (An Olympus BH-2 polarizing light microscope) would open wide enough to accept a 2″ square of plexiglas, so a grid of 3/16″ diameter holes was laid out on it on 3/8″ centers, and drilled with a twist bit, to a depth of 1/8″. For our microscope and stage, this gave us sixteen holes for samples on a single sample holder. We experimented with preheating the wax and the plexiglas holder on a variable hotplate, and partially pre-filling the holes by dripping the wax into the heated plexiglas, but results were less than satisfactory, due to variability in one's aim when delivering the drop of molten wax, and the fact that the molten wax tended to crawl up and over the sides of the hole in the heated plastic; this necessitated cleaning the surface of the holder with tissue or cloth, some strands of which invariably ended up in the hole, and which could distractingly reappear when examining the sample under ultraviolet. We were also concerned with the possibility of the sample floating in the molten wax.

We also experimented with scribing numbers under the holes with a diamond-point stylus, and filling the inscriptions with permanent india ink, thinking that they should still be legible after grinding and polishing. When practice samples were embedded and examined, the following practical shortcomings of the prototype became immediately obvious:

1. The stage on our microscope would not traverse far enough to allow examination of a sample placed any closer to the edge of the holder than 1/4 of an inch.

2. Holes one-eighth of an inch deep and partially filled with wax were too deep, since the process of melting the wax around the sample could, and sometimes did, melt the wax below it, forcing the sample below the level of plastic. This resulted in a sample dimly seen beneath a layer of wax, and would have necessitated grinding the entire sample holder down to expose it, at no savings in time over the cast polyester system. On the next holder, the holes were drilled half as deep, and with a brad-point bit, to eliminate the deep "V" from the twist bit. The center-spur hole from a brad-point bit also allows one to stand a long sample up on end in it, and the flat bottom of the hole lets one lay the sample on its side. Some pre-filling of the hole with wax is still necessary, to be able to vary the depth of the hole in the wax for the size of each individual sample.

3. The inscriptions in the plexiglas did not survive a series of embeddings and grindings on the same holder; sufficient material was removed from the face of the holder to largely obliterate them. If all samples were embedded before grinding, rather than one at a time, and the holder was polished only once, they might survive, but it was far simpler to write the numbers under each hole (in reverse) on the obverse of the holder, and read them from the front. Accession number and object description is most easily written on the face of the holder after polishing is complete.

4. When attempting to use a fluorescent reactive dye on a sample, it was discovered that the wax matrix had shrunk as it cooled, creating cracks in it and around the sample; the cracks were not visible until the Rhodamine B penetrated them, which then surrounded the sample. While this in no way changed the ability of the sample to absorb the dye, the dye surrounding the sample is distracting when one attempts to read the sample, or photograph it. Other embedding matrices should be investigated, preferably ones with quick-setting characteristics and high viscosities, to preserve the time and manipulability advantages inherent in the wax system.

The following advantages of this system were also noted;

1. Speed—The time from removal (of a single sample, once the holder is fabricated) to viewing is not more than ten minutes; this makes immediate resampling possible, to obtain the sample which tells you what you want to know—no waiting half a day for polyester to cure between sets of samples, and no lag in memory about what you were looking for when you took a specific sample. This latter has reduced the number of samples required to give one the desired information; if the previous sample was inadequate, another series of samples which will be unviewable for half a day is not necessary—one just takes one more from a more appropriate location, and looks at it.

2. Convenience, multiple samples—All of the samples from one object can be on one or two sample holders, and all samples, even when more than one holder is used, are in the same plane, allowing easy movement from one sample to another, without having to rack the microscope up or down. Additionally, careful layout of the holes allows one cover slip to cover four holes at once, and it is possible to cover-slip all samples on one holder, allowing simplified and immediate comparison of samples.

3. Manipulability—It is possible to remove a sample which was poorly positioned and re-embed it, but the success of this maneuver is largely dependent upon one's motor skills. One operation which was very successful, however, was the addition of the lower strata (including the wooden substrate) to a previously embedded and polished upper layers of a paint sample which had cleaved during sampling between paint layers, at a friable clear coating. We were able to carve a hole in the wax, and position the lower half of the sample next to the upper layers in their correct orientation to each other.

Cautions, unknowns:

Longevity—The long-term storage of samples embedded in a hard wax, carnauba or other, is uncertain; intuitively, one would expect a sample in a carnauba matrix which is stored in a dust-free and room-temperature environment to last forever, but the purity and aging characteristics (if any) of carnauba is unknown to us. It is known, however, that polyester resin yellows with age, and can degrade.

Along the same lines, while it is theoretically possible to remove a sample from a wax matrix and return it to the object, or embed it in polyester resin, the complete removal of pure carnauba from the sample becomes a problem, given the wax's relative insolubility. The solvents necessary to remove the wax from the paint would very probably be damaging to the sample.

Observations:

Obviously, this system of embedding would be worthless if one were searching for the presence of original wax coatings.

Polish the edges of the sample holders before you layout and drill the holes; polishing the edges allows one to see the depth of the holes being drilled from the side of the sheet of plastic.

Since this paper was written, some hard dental casting wax, dark blue in color, was acquired and tested; while it provided a striking background for the paint sample tested in it, it was readily eroded by the stoddard solvent used under the cover slip. There is no reason to assume that the ivory color would react any differently, but some has been ordered to determine if a mixture of it and carnauba would retain the hardness of the latter, while reducing the tendency to crack due to shrinkage.

It is well understood that individuals have preferences for their own systems for embedding, for their own very valid reasons; this paper is not intended to convert them, or supplant such systems. It is offered as a simple and quick alternative to polyester casting resin, and requires no more expense than some scrap plexiglass, and some hard wax (ivory dental casting wax in stick form is made by Kerr, available from Henry Schein Co., Port Washington, N.Y., and carnauba is listed as available from Wood Finishing Supply, Milwaukee, Wisconsin).

## SOME 1993 POSTSCRIPTS

Since Chris and I started experimenting with this wax system in 1992, I have found it to be so handy that I have virtually abandoned the polyester resin method (the only time I reverted to it was to check for the possible presence of an original wax coating). Not surprisingly, some more efficient methods have evolved.

1) The alcohol lamp used to heat the knife, while satisfying and lends an arcane feeling of importance, is slow, sooty, and too variable a heat source. An inexpensive soldering pencil was purchased, the original tip thrown away, and a longer one was made using copper rod which was filed and polished to a long point in a drill press. The addition of a rheostat allowed precise control of temperature.

2) It was found that the face of the plexiglas holder could be pre-numbered, permanently, with 3/32 steel stamps. Obviously, care must be taken when striking the stamp, since too strong a blow will shatter the plastic, but the impressed figure will survive several grindings.

3) The owner of Conservation Materials has suggested that the addition of not more than 10% by weight of Polywax 2000 to the pure carnauba will solve the problem of shrinkage cracking. This has not yet been tested.

4) A two-part mold was manufactured, using Dow RTV silicone cast around three sections of 1/4″ dowel, into which the molten wax can be poured to give rods of wax which are more easily manipulated than the chunks in which it was purchased.

5) If one attempts to reassemble, before embedding, samples of paint or finish which have cleaved during sampling, beware of excessive heat, which can soften and distort the paint layers. Heat is generally not a problem when one is just embedding an unbroken sample into a small pool of just-melted wax in the holder.

—Clint Fountain