

Tricks-of-the-Trade

Every profession has little "tricks-of-the-trade" to make the pursuit of that profession run just a little more smoothly. Carpenters wet nails in their mouths to prevent wood from splitting; plumbers tighten pipes in their unions before loosening the pipes; painters line up screens in a row before spray painting; woodworkers make their own wood filler by mixing clear glue with fine sawdust from the wood they are working with. The profession of metallography is no exception. Experienced metallographers (those who have been in the trade for a number of years), have developed little "tricks-of-the-trade" that have become second nature to them. Sometimes, they are willing to pass these techniques along to junior members of the group, but sometimes they guard them very jealously because of a misguided sense of job security. Fortunately, dedicated metallographers belong to the former group.

The following tips have been around for many years and have been used quite frequently. It is hoped that among them is one that will answer a particular problem that is being experienced in your laboratory.

Cleaning Stains from As-Polished Specimens

From the time a specimen is dried to the time it is put on a microscope, many stains can develop. The stains can be water or alcohol droplets that have dried randomly on the as-polished surface; they can be condensed from the air in a hot, humid atmosphere; from the crevices created by edge separation; or from oil or water in in-house air lines. The latter is notorious for this. However they occur, they are a nuisance, and they always seem to occur at the place of interest or where a photo-micrograph is to be taken. Fresh stains can be removed very effectively by rubbing the as-polished surface over a clean, medium nap cloth (such as Lecloth) several times in one direction. Best results are obtained by placing the specimen on the cloth and pulling toward the operator, lifting the specimen off the cloth and repeating. More stubborn stains or stains that have been allowed to remain for several days can be removed in the same manner by huffing breath on them prior to rubbing over the polishing cloth. Needless to say, the cloth should be clean and dry. Cloths are usually kept by the side of a microscope or metallograph for easy use. Some, but not all etched specimens can be treated in the

same manner. Specimens that have been stained-etched (4% Picral for instance) should never be treated with this method.

More often than not, staining can be eliminated at the source. For instance, if an air hose connected to an in-house air line is used for blowing specimens dry, placing cotton in the nozzle/air line union will trap oil and moisture droplets. Flushing the specimen surface with ethyl alcohol, as opposed to methyl alcohol, will also eliminate staining. Methanol (methyl alcohol) contains water and when dried, the alcohol evaporates first, leaving water behind that manifests itself in the form of stains. Isopropyl alcohol should never be used because of the high water content.

Grinding/Polishing Thin Gauge Material

Grinding and polishing thin gauge material on one of the flat sides posed no problem with the following technique. Select a small block of wood that is slightly larger than the flat surface of the material, and obtain flat surfaces by grinding on a belt grinder or by some other means of grinding (vertical, horizontal, etc.). Wrap double-stick masking tape several times around the wood block. The masking tape can be any width, but the tape should be wrapped with a slight edge overlap in such a manner that the tape is covering a larger area than the material to be prepared. Avoid wrapping the tape in any one position. If double-stick masking tape is not available, single-stick tape can be used just as effectively by tearing off a small length, reversing it, and reattaching it to the roll. The sticky side of the small strip is attached to the wood block, and wrapping is accomplished in the same manner as described. Masking tape should be used, not plastic tapes. Once the block of wood is wrapped with the tape, position the thin gauge material on the tape, invert and lay on a tabletop, and press with the ball of the hand to firmly attach the coupon to the tape. Another coupon of material can be attached to the opposite side, and two coupons can be prepared sequentially.

Grinding is accomplished using the normal procedures, taking care that the material does not become too warm and cause the adhesive on the tape to melt—grinding in the presence of water will usually eliminate the frictional heat build-up. With two coupons attached to the tape-covered wood, they can be alternated during the various grinding steps. When grinding is completed, remove the coupons and tape from the wood block, clean and dry the coupons, and retape the wood block in the above manner.

Repeat for the final polishing step. The secret for a good preparation is to use fresh tape at each of the three stages—grinding, intermediate polishing, and final polishing—to avoid contamination of the polishing wheels. A variation of the above technique is to use small double-sticky pads with release paper (the type that is frequently used to attach mirror tiles to vertical surfaces) instead of masking tape. Either method is very effective.

Mounting Thin Gauge Material on Edge

Perhaps the most popular technique for mounting thin gauge material on edge is with the use of spring clips or a segment of a plastic spiral that is used as a binder for notebooks. These techniques are very effective and are usually the first choice; however, every laboratory may not have spring clips or wish to remove the plastic spiral binder from notebooks. The following technique is equally effective and has several decided advantages.

A small segment of cured mounting media is slotted by any means—cut-off, hand-held hack saw, etc.—to obtain a series of slots separated by small fingers of the mount; in other words, a comb effect. The thin gauge material is slipped into the slots, making sure the material fits snugly. If the slots are too wide, small thin shims of the mounting media can be used as wedges. There should be no play when the material is in the slots, and the material should be in an as-perpendicular position as possible, otherwise the thin edge could be prepared on a taper. Mounting is accomplished by conventional methods; either compression mounting or castables.

Advantages to this technique include that when several pieces are being mounted, they are separated, and electrolytic etching can be accomplished on individual specimens. If they were mounted touching each other, adjacent pieces would be overetched, as they tend to act as one solid specimen. The mounting media will have better adherence to the separated pieces than it would if the specimens were mounted touching each other. When more than three or four thin specimens are mounted together with spring clips, the mounting media does not flow into the very narrow crevices, and these crevices become traps for grinding and polishing debris. When faced with this problem, see *Met-Tip #2*.

Mounting Thin Wires on End

There are several techniques used for mounting thin wires in a transverse direction. Using spring clips as for thin gauge material (however, the wires sometimes become tilted, and a true cross-section is not obtained); or mounting first in a longitudinal direction, sectioning off the ends, and remounting. The latter technique is quite effective when one is trying to obtain a longitudinal as well as a transverse direction. The sectioned-off end of the mount can be aligned with the remaining longitudinal pieces and remounted together. The second mount will contain both directions, and the wires lying in the longitudinal direction will match those in the transverse direction. It is a time-consuming technique, but the end justifies the means.

A better technique for mounting thin wires on end is to take a small segment of cured mounting media, drill holes completely through it, insert wires, and mount by conventional methods. The holes should be drilled on a drill press to ensure perpendicularity. If bakelite or diallyl phthalate mounting media is used, they should be sieved to obtain fines that are sprinkled liberally over and around the holes before adding the normal mounting media. The fines have better flow characteristics and will fill in the holes nicely. This technique is also used when mounting normal samples that have cracks or pores that are desired to be filled with mounting media. If a castable mounting media is used, a vacuum should be pulled in the mount to remove bubbles.

This technique is also very good when plated pins are to be examined in a transverse direction so plating thicknesses can be measured.

Mounting Fine Powders

The preferred mounting media for the metallographic preparation of fine particulate matter is a castable media which uses a resin plus a catalyst to promote hardening at room temperature. If compression mounting media is used, only a monolayer of material is usually achieved, and one can quickly grind through it. With fragile powders, the pressure can crush or fragment them, and any type of quantitative analysis is useless.

If there is a substantial amount of powder available, mounting can be accomplished in the following manner. Have a mold ready; mix castable media according to directions; and stir very gently to avoid excess bubbles from being introduced. Pour a very small amount of the mixed castable into a second container, add the powder, and stir very gently, making sure all the powder is wetted. Pour into the mold, then add the remaining castable from the first container. Cure in the normal manner.

When the amount of powder is quite limited (there must be at least 300 particles on a polished plane for a valid statistical quantitative analysis,) the following method of mounting is very effective. It is called the resin inset method. Blank mounts are made from a castable mounting media in sizes that are easily handled; for example, a parallelepiped $1" \times \frac{3}{4}" \times \frac{3}{8}"$. After the blanks are cured and removed from their mold, they are ground on one of the large surfaces to obtain flatness. A conical depression is drilled into the ground flat surface of the blank using a drill press and drills ranging from $\frac{1}{8}$ to $\frac{1}{4}$ -inch in diameter. The depth of the conical depression in the blank should be approximately one-third to one-half the diameter of the drill used. The powder to be mounted is placed into the conical depression, and liquid castable-catalyst is added drop-by-drop to the conical depression and stirred with a fine rigid rod, such as a needle or a straightened paper clip.

The prepared inset can be cured at room temperature in the normal process. Grinding and polishing are accomplished in the normal manner.

When there are multiple samples of powders to be mounted, it is evident that a suite of conical holes can be drilled side-by-side, and the powders mounted in the manner described above for comparative purposes.

As little as 5 milligrams of powder has been successfully prepared by the technique described above—with 300 to 500 particles on the polished surface.

Inexpensive Molds for Castable Mounting Media

When oversized specimens are to be mounted in castable mounting media, there is usually a search to find a suitable mold—with paper cups and tin cans being the first choice. While these choices of molds are effective, they do have drawbacks—the paper cups are usually tapered so a tapered mount is made; or the castable dissolves the cup, creating a mess. Cured mounts are difficult to remove from tin cans without cutting the tin can with snips and using pliers to pull the tin away from the mount.

The following method of making oversize molds is quick, easy, and inexpensive; no special equipment is required. Moreover, the molds are easily removed from the cured mount, and the mounts are uniform in morphology. All that is required is heavy-duty aluminum foil, blocks of wood, and masking tape.

The blocks of wood can be any size; however, the surfaces should be smooth. Two inches cut off the end of a 2x4 will have the morphology of a parallelepiped having three different sizes that can serve for making three different size molds. Grinding the wood on a belt grinder will produce flat surfaces and true corners.

Cut a piece of aluminum foil from the roll. If the foil has wrinkles, they can be removed by laying the foil on a flat surface and smoothing with the fingers. Position the surface of the wood block that is to be used as a mold pattern in the middle of the foil and hold between the fingers and thumb of one hand. Trim foil with scissors so the foil will not extend above the wood when folded. Wrap foil around one end of the wood block, making creased edges at the corners, and fold in (as if wrapping a package). Do the same at the opposite end. Wrap masking tape completely around the sides several times and then across the bottom to ensure rigidity. At this point, the foil is molded around the block of wood and should be to within about $\frac{1}{4}$ -inch from the top of the wood block. Rubbing the bottom of the mold across a flat surface will ensure flatness. The block of wood is "walked" out of the foil mold by a gentle zig-zag pulling motion. If the bottom of the foil mold is pulled up during removal of the wood, the smaller surface of the wood can be used to press and smooth the bottom.

Specimens are placed in the mold, and the castable-catalyst mounting media is poured to the desired height. When the mount is cured, the aluminum foil can be easily stripped off with the fingers. With some castables (epoxy for example), it has been found that aluminum foil is more easily removed when the shiny side of the foil is facing inward in the mold.

Metallographic laboratories using the above technique usually have a large variety of different sizes of wood blocks to draw upon for making molds.

Bakelite, acrylic, glass, or aluminum rings can be used when small samples are to be mounted in a castable mounting media. Smear a small amount of Stop-Cock grease, vacuum grease, or vaseline on a flat surface (such as a plate of glass). Position the ring and twist to ensure a seal. Place specimen(s) to be mounted inside the ring and add the mounting media. If a vacuum is to be pulled on the poured mounts, the casting surface needs to be small enough to get into the vacuum chamber.

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