No. 4

LECO MET-TIPS Ideas for Metallographic Procedures

What Type of Mounting Media to Use?

At first glance, the subject of this article may have a tendency to come across as a ho-hum subject and the reader may well say, "Mounting media is mounting media, so what's the big deal"? The big deal is that selecting the proper mounting medium can make the difference between a mediocre and a well-prepared metallographic specimen.

The reasons for mounting are many—it makes it possible to prepare very small specimens that ordinarily would be impossible to prepare; it produces flat surfaces necessary for microscopic examination; it allows multiple specimen preparation; it retains edges of specimens during the preparation stages; it prolongs polishing cloth life; the list can go on and on. Most automatic polishing equipment is designed for mounted specimens—mounted samples will allow microhardnesses to be taken closer to the edges. Perhaps the main reason for mounting is it makes samples easier to handle—it is much easier to process a 1-inch long by 1/8-inch wide specimen on a narrow dimension when it is mounted than it would be if the same size specimen were not mounted. Moreover, the quality of preparation is superior.

Mounting media falls into two categories—castable and compression mounts. Castables are cast as a liquid using a catalyst to promote hardening at room temperature. Compression mounts are those that are molded with heat and pressure using mounting presses.

Castables

There is a large variety of mounting media available that falls into the castable category. Most castables use a two-part system, one part is the resin, and the other a catalyst that produces a chemical reaction causing the resin to cure and become solid. Some castables use a powder and a liquid, other castables use a three-part liquid. Castables can be polyesters, epoxies, or acrylics and each have their own merits and shortcomings. Some will set up in a few minutes and others may require as long as 24 hours. Some have low shrinkage properties and others have large shrinkage properties; however, most are impervious to acids and etching reagents. Some advantages to using castables—over-large specimens can be prepared; small, fragile specimens that would be deformed in a compression mount can be safely mounted and prepared; some alloys with low melting temperatures or alloys with low temperature microstructural changes can be mounted and prepared without danger of altering the microstructure. Castables can also be poured into any size mold and the mold can be made of any material that will retain its shape during the setting up time. Many molds can be reused—flexible silicone molds, bakelite, acrylic, or glass rings that are greased inside for easy removal of the cured mount.

Castables can be removed from their molds as soon as they have set up. All castables give off heat during the setting up time because of the chemical reaction taking place. The higher the exotherm, the faster the curing; the faster the mount is cured, the more risk there is in cracking the mount and increasing the shrinkage properties.

The mixing ratio of the two-part castables should follow the manufacturers' recommended procedures; however, when mixing in larger quantities, cutting back on the amount of catalyst or accelerator will greatly reduce the risks of cracking and edge separation. For example, if the ratio is 1:10 for catalyst-to-resin for 100 ml, increasing the ratio to approximately 1:12 for quantities over 200 ml will ensure a more properly cured mount. If possible, freshly poured castables should have a vacuum pulled on them to suck the liquid into small crevices and to pull out air bubbles introduced by the mixing operation. Slow stirring will minimize the amount of air being trapped in the castable. Placing a freshly cast mount into a shallow basin and filling with water to just below the top of the mount will dissipate the heat and allow the mount to cool slower, thus reducing the risk of cracking and edge separation.

Most castables have a tendency to be brittle when cured, and will fracture when dropped. Placing the cured mount into a laboratory oven for approximately 15 to 20 minutes at 180 degrees Fahrenheit will temper the mount and reduce the brittleness.

The curing time for castables can be hastened by placing the freshly cast mount into a laboratory oven for 15 to 20 minutes at approximately 180 degrees Fahrenheit, but this is a critical procedure and must be monitored closely. The mounts should be removed from the oven when in a "rubbery" stage and allowed to cool naturally in air. This does not seem to increase shrinkage as much as letting the mount cure in air without dissipating the exotherm.

Compression Mounts

Available mold sizes for compression mounting include 1, 1¼, 1½, or 2-inch diameters, and the mounting media used for compression mounts can be divided into two types—thermosetting and thermoplastic. Both types are processed under the same conditions using a mounting press; however, some prefer to use lower pressures for the thermoplastic types during the heating cycle, then increase the pressure to the recommended psi after the curing temperature is reached.

The biggest difference between thermosetting and thermoplastic mounting media is in the way they are cooled after reaching the curing temperature of around 300 degrees Fahrenheit. The thermosetting types, such as bakelite and diallylphthalates, go through a liquid and a resolidification stage before reaching the curing temperature and, if necessary, can be ejected from the mold at this temperature. On the other hand, the thermoplastics, Lucites, transoptics, and formvars are more-or-less a fusing together of the particles and they remain a liquid at the curing temperature—albeit a very viscous liquid—and they must be cooled to approximately 150 degrees Fahrenheit before ejecting from the mounting press. Ejection at higher temperatures will cause the mount to swell as soon as pressure is released and the ejection cycle will be jerky because pressure will be built up and the mount has a tendency to "grab" the inside surface of the cylinder. Both the thermosetting and thermoplastic mounting media must be held under pressure during the curing, or soaking, time.

If compression mounting media has been exposed to the atmosphere for a long period of time it has a tendency to absorb moisture. When heated, the moisture changes to steam and can cause bursts, or the mount will bulge out on the back surface. Preheating loose powder in a laboratory oven at about 100 degrees Fahrenheit will dry out the moisture, or a degassing step can be used with the mounting press. Degassing is relieving the pressure for a few seconds then applying full pressure again after the heater has been on for approximately 1 to 1½ minutes, or a temperature of approximately 150 degrees Fahrenheit is reached.

Preforms of bakelite mounting media are available in multiple sizes. Preforms are made from compacted bakelite powder; no heat is involved in the process of compaction, however, preforms are also subject to moisture absorption if left open to the atmosphere for extended periods of time. Pre-heating is recommended. The use of preforms takes the guesswork out of how much mounting media to use and they are good for specimens of uniform size. They are not recommended when mounting thin specimens, wires on end, or specimens with intricate morphologies. Preforms do not seem to flow as easily as powders during the liquid stage.

Even though thermosetting mounting media can be ejected after soaking for several minutes at the curing temperature, there are several reasons why they should not be. Obviously, 310 degrees Fahrenheit is slightly uncomfortable to handle with the hands, but more importantly, there is a difference in the cooling rate between the part being mounted and the mounting medium, and separation at the specimen-mount interface will occur which can cause problems later during preparation stages. When this does occur for any reason, see Met-Tip #2 for solutions.

Certainly the choice of mounting media to use plays an important role in the quality of specimen preparation. Specimens that are prone to deformation caused by pressure or microstructural changes that occur at temperatures around 300 degrees Fahrenheit or lower would not be mounted in compression mounts, but rather would be mounted in a castable media. Besides the former, there are other considerations to be taken into account when choosing a mounting media.

A good rule to follow when in doubt as to which type of media to use is to select one that has a comparable abrasion rate (not hardness) as the material being mounted. The mount should grind away at about the same rate as the specimen. If very hard materials, such as metal carbides, metal borides, ceramics, or cermets are mounted in soft mounting media, the mount will grind away more rapidly and leave a slightly domed mount, and it would be almost impossible to examine the edges of the specimen. Conversely, soft materials such as bismuth, lead, or noble metals should not be mounted in a hard mounting media because these materials would abrade faster than the mount, resulting in the specimens being slightly recessed below the surface of the mount. When this occurs, it is very difficult to final polish the mounts because the fibers of the polishing cloth cannot reach the specimen, and again edges would be difficult to examine.

Thermosetting mounting media will have comparable abrasion rates to those of ferrous and nonferrous alloys; thermoplastic types do not and should only be used when mounting softer materials. Some of the castables have comparable abrasion rates to that of ferrous and nonferrous material, but many do not. However, the abrasive rates can be equated by the addition of pelletized alumina that is available in three grades—soft, medium, and hard. Soft grades to be used for very soft material, medium grades for ferrous and nonferrous alloys, and the harder grades for extremely hard materials such as metal carbides, metal borides, or ceramic specimens.

Thermosetting mounting media is impervious to acids and the normal etching reagents typically used in the laboratory, however, they will break down in boiling solutions of $K_3Fe(CN)_6$-KOH and $KMnO_4$.

Thermoplastic mounting media (Lucite, transoptic) will dissolve in acetone and methyl-ethyl-keytone. Uncured Lucite dissolves in ethyl alcohol. Also, prolonged time in alcohol-based etchants or just plain ethyl alcohol will "craze" or cause cracking in cured Lucite mounts.

When grinding thermoplastic mounts such as Lucite and transoptic (formvar is seldom used these days), it is necessary to have a copious water flow. If not, the specimen becomes hot and causes the thermoplastic to soften, or actually melt around the specimen surface and pull away from the specimen surface.

While it seems there is an unusual number of decisions to be made in selecting the correct mounting media to use, the options available will make the difference between a mediocre or a well prepared mount, and a metallographic laboratory should have a variety of mounting media to call upon, both compression and castables, to ensure that a proper mounting media can be selected for the different materials that are processed through the laboratory.

Castable Mounts

	Acrylic	*Polyester*	*Quick Cure Epoxy*	*Long Cure Epoxy*
Cure Time (hh:mm)	~00:10	00:10–08:00	00:30–00:60	04:00–08:00
Peak Cure Temp (°F)	~210	~210	~200	~175
Clarity After Cure	Excellent	None—Excellent	Excellent	Excellent
Edge Retention	Fair	Good	Excellent	Excellent
Hardness (HRR)	110–120	90–125	110–120	120–130
Abrasion Rate	Excellent	Excellent	Excellent	Excellent

Compression Mounts

	Bakelite	*Epoxy*	*Diallyl Phthalate*	*Lucite (Acrylic)*
Cost	Low	Moderate	Moderate	Low
Variety of Colors	Excellent	Fair	Poor	Poor
Clarity After Cure	None	None	None	Excellent
Edge Retention	Good	Good–Excellent	Excellent	Fiar
Hardness (HRR)	120–130	120–130	120–130	115–125
Abrasion Rate	Good	Excellent	Excellent	Poor

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