Specimen Mounting

4.0 SPECIMEN MOUNTING

The primary reasons for specimen mounting are to better hold the part to be ground and polished, and to provide protection to the edges of the specimen. Secondarily, mounted specimens are easier to fixture into automated machines or to hold manually. The orientation of the specimen can also be more easily controlled by fixturing it and then setting it in place via mounting. Metallographic mounting is accomplished by casting the specimen into a castable plastic material or by compression mounting the plastic under pressure and temperature.

4.1 CASTABLE MOUNTING

Castable resins are monomer resins which utilize a catalyst or hardener for polymerization. Polymerization results in cross-linking of the polymer to form a relatively hard mount. Castable resins also have the advantage of simultaneously mounting multiple samples at one time for increased throughput. A number of resin systems (Figure 4-1) are used for metallographic mounting and include:

- Epoxy resins
- Acrylic (castable) resins
- Polyester (clear) resins

Figure 4-1 (a) 2-part liquid epoxy resins (b) 1-part powder, 1-part liquid castable acrylic resins (c) 2-part liquid polyester resins.

Table VI lists the common properties of epoxy, acrylic and polyester resins.

TABLE I. Castable Mounting Properties

PROPERTY	EPOXY	ACRYLIC	POLYESTER RESIN
Peak Temperature	100-375°F	150°F	100°F
Shore D Hardness	82	80	76
Cure Time	30 minutes to 8 hours	5-8 minutes	6-8 hours
Comments	Moderate hardness, low shrinkage, transparent	Very fast cure, translucent, some shrinkage	Transparent, water clear

4.1.1 Epoxy Resins

The most common and best performing castable resins are epoxy based (Figure 4-2). Epoxy resins are typically two-part systems consisting of a resin and a catalyst (hardener). Mixing ratio's vary from ten parts resin with one part hardener to five parts resin with one part by weight of hardener. The advantages of mounting with epoxy resins include:

- Low shrinkage
- Relatively clear
- Relatively low exotherms
- Excellent adhesion
- Excellent chemical resistance
- Good hardness
- Relatively inexpensive

Figure 4-2 Epoxy resins are available in standard, low viscosity, or fast curing systems.

Epoxy curing times are dependent upon a number of variables including:
- Volume of mounting resin (larger mounts cure faster).
- Thermal mass of specimen (larger specimens absorb heat and therefore require longer curing time).
- Specimen material properties.
- Initial resin temperature (higher temperatures cure faster).
- Ambient temperature (higher temperatures cure faster).
- Relative humidity and shelf life (absorption of water degrades resin and shortens shelf life).
- Mounting mold (plastic, phenolic rings, rubber absorb heat differently).

As a general rule, curing times can vary from 30-45 minutes for fast curing epoxies up to 24 hours for slower curing epoxies. For metallographic epoxies to grind properly, the hardness needs to be at least a Shore D80. Note that epoxy resins typically will continue to harden over a longer period of time (maximum hardness, Shore D90).

In some cases, the curing time and temperature may need to be controlled to compensate for the above variables. For example, an 8-hour resin system can be cured in 30-45 minutes by preheating the resin to approximately 120°F (50°C) prior to mixing and then curing at room temperature. This procedure initiates the catalytic reaction sooner; however, this may also increase the maximum exotherm temperature.

TIP: Preheat the specimen to initiate the epoxy resin at the surface of the mount and thus have the epoxy shrink towards the sample for better edge retention.

Conversely, the resin curing cycle can be slowed or reduced by decreasing the curing temperature by forcing air over the curing mounts (fume hood or fan), placing the mounts into a water bath, or curing in a refrigerator. In these cases, care must be taken to not stop the reaction; however if this does occur or the resin is too soft after curing, heating it to 100-120°F for several hours should push the reaction to completion and the mount should be hard after cooling to room temperature.

Table VII lists the relative properties for several metallographic epoxy resin systems.

TABLE II. Castable Epoxy Mounting Properties

EPOXY	Standard Epoxy	Quick Mounting Epoxy	Low Viscosity Epoxy
Characteristics	Lower cost	Fast cure	Lower viscosity, clearer, lower exotherm, low shrinkage
Cure Time	1-5 hours	30 minutes	2 hours
Mixing ratio (weight) (resin : hardener)	5:1	10:1	10:1
Peak Exotherm 20 grams 30 grams	 300° F 300° F	 350° F 375° F	 120° F 150° F
Color	Clear to slight yellow tint	Slight yellow tint	Clear
Viscosity	Medium	Medium	Low
Shrinkage	Moderate	High	Low

4.1.2 Acrylic Castable Resins

Castable acrylics are easy to use and are very robust (Figure 4-3). The main advantage of mounting with castable acrylics is the fast curing time. Depending upon the mixing ratio, castable acrylic mounts are typically ready to use within 8-15 minutes. Also unlike epoxy resins, the ratio of the various acrylic parts (powder to liquid) can be altered by up to 25% with no adverse effect to the final properties of the mount. This is because both the liquid and powder are acrylics with various additives and curing agents. By varying the ratio of the liquid to powder, the curing time and viscosity can be altered. **Note:** the powder contains a catalyst that reacts with the liquid hardener to start the curing process. Fillers are added to increase hardness and to reduce shrinkage.

Figure 4-3 Castable mounting acrylics include powder and liquid components.

Characteristics of Castable Acrylics (see Table VIII) includes:
- Rapid mounting
- Very repeatable and consistent mounts
- Moderate shrinkage
- Good hardness
- Semi-transparent
- High odor

TIP: Acrylics can be submerged into a water bath during curing. This reduces the exotherm heat and thus reduces the shrinkage of the mount at the specimen interface. A secondary advantage is that the water absorbs the odor.

TABLE III. Castable Acrylic Mounting Properties

ACRYLICS	CASTAMOUNT	PREMIUM	SUPERMOUNT
Characteristics	Fast, semi-clear, l ow shrinkage	Fast, clear, low shrinkage	Fast, hard, opaque
Cure Time	10 minutes	8 minutes	10 minutes
Mixing ratio (volume) Powder: Liquid	3:2	3:2	3:2
Color	Semi-clear	Semi-clear	Gray to black

4.1.3 Polyester Castable Resins

Polyesters are typically used when a very clear mount is required. Polyester resins are also useful for mounting parts for display. In this case, the part appears suspended in the plastic. The procedure for molding samples for display is to first determine the mixing ratio of the resin to hardener (catalyst). This ratio is variable depending upon the mass of the casting (Table IX).

TABLE IV. Polyester Catalyst Mixing Ratios
(Proportions are based on ambient and resin temperature of 70°F)

Single Layer Casting (metallographic)		**Multiple Layer Casting (display)**	
Layer Thickness	Drops of catalyst per ounce of resin	Layer	Drops of catalyst per ounce of resin
1/8"	15	1st layer	5
1/4"	8	2nd layer	4
1/2"	6	3rd layer	3
3/4"	5	4th layer	2
1" - 1-1/2"	4	5th layer or more	2

For larger volumes, the amount of hardener needs to be reduced significantly. The procedure for suspending the sample in the mount is to pour an initial layer and allow it to pot or gel (do not let it fully cure). The object or specimen is then placed on the initial rubbery polyester layer and another layer of the liquid polyester is poured. Multiple layers can be poured in this fashion if required.

Characteristics of Polyester include:
- Very clear (water clear)
- High odor
- Best resin system for making large castings

Polyester resins are similar to acrylics and can be submerged into water during the curing cycle in order to reduce the exotherm temperature and shrinkage.

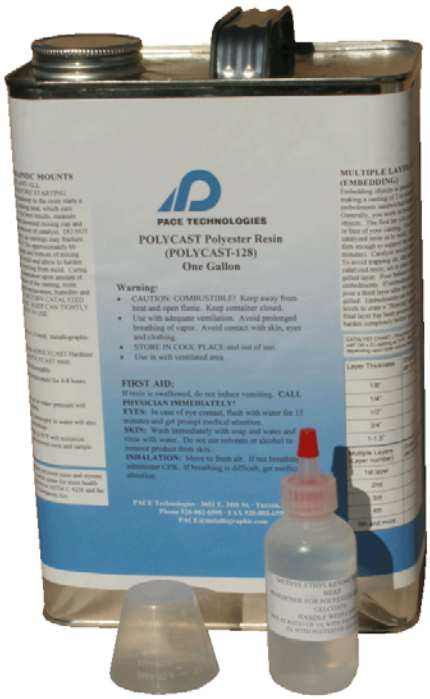

Figure 4-4 Clear castable mounting polyesters.

4.2 CASTABLE MOUNTING PROCEDURES

- Clean and thoroughly dry specimens to remove cutting and handling residues.
- Remove debris from molding cups.
- Apply thin coat of mold release compound to molding cup.
- Center specimen in molding cup.
- Accurately measure resin and hardener.
- Mix thoroughly (gentle mixing to avoid producing excessive air bubbles).
- To reduce air bubbles, pull a vacuum on the specimen before pouring the resin. After pouring the resin over the specimen, cure at room pressure or apply pressure in an autoclave chamber.

TIP: Before mixing, preheat resin, hardener and specimen to 30°C (85°F) to expedite curing cycle **Note:** this will also increase maximum exotherm temperature.

4.2.1 Vacuum/Pressure Mounting

Vacuum impregnation is a very useful technique used to fill in pores or voids prior to specimen preparation. It is highly useful for thermal spray coatings and other porous samples.

The most effective technique is to pour the resin under vacuum and/or apply pressure during the curing cycle (advantages - better infiltration of pores and cracks, more transparent mounts, and fewer air bubbles) (see Figure 4-5).

Figure 4-5 Vacuum impregnation device.

For porous or cracked specimens, the resin can aid in supporting these features. Filling these voids can be difficult depending upon their size, with the smaller voids being much more difficult to impregnate than larger voids. This arises mainly because of the compressibility and volume of air within the void. By applying a vacuum to the specimen and pouring while under vacuum the total pressure of this air can be reduced significantly. Subsequent curing at increased pressures will force (or push) the resin into the voids. Note that the vacuum time on both the resin and specimen should be kept to a minimum in order to minimize degassing of the resin.

PV = nRT (gas law)
P - Pressure
V - Volume
T - Temperature

$$V(\text{bubble size}) = \frac{nRT}{P}$$

Thus in order to decrease the air bubble size, impregnate at low pressures and cure at higher pressures.

Recommended Procedure:
1. Place mold and sample into impregnation chamber
2. Mix castable mounting resin
3. Place cover on chamber and pull vacuum
4. Pour resin into mount
5. Slowly increase the pressure
6. Allow the mount to cure at room pressure or apply an external pressure.

TIP: Do not pull vacuum for more than 60 seconds. Extended vacuum causes the dissolved gases in the liquid resin to degass and bubble (similar to opening up a carbonated beverage bottle).

TIP: To reduce the curing time, preheat resin, hardener and specimen to 30°C (85°F). **Note:** this will also increase maximum exotherm.

TIP: Slight preheating of the epoxy will also reduce the viscosity of the resin and allow it to flow better.

4.3 CASTABLE MOUNTING MISCELLANEOUS

Figures 4-6 to 4-8 show a variety of accessories used with castable mounting, ranging from mounting molds and mounting clips to mixing cups and storage containers. Table X provides a description of each..

Figure 4-6 Castable mounting molds
(clockwise: silicon rubber, 2-piece plastic, ring forms, disposable).

Figure 4-7 Castable mounting clips (plastic, metal).

Figure 4-8 Plastic mixing cups, measuring cups and storage containers.

TABLE V. Castable Mounting Accessories

ACCESSORY	APPLICATION
Silicon rubber molds	Reusable molds
Ring forms	Molds used to maintain the planarity of the specimen mounts Very useful for upright microscopes
2-piece Reusable molds	Reusable molds
Mounting cups	Disposable mounting molds
Silicon mold release	To aid in releasing the mount from the mold
Plastic clips	Used for holding or orienting thin specimens perpendicular to the examination plane
Metal clips	Used for holding or orienting thin specimens perpendicular to the examination plane
Plastic mixing cups	For mixing acrylic resins which absorb into paper cups
Measuring cups	For measuring the volume of the castable mounting material
Storage cups	To protect and archive the specimens

4.4 CASTABLE MOUNTING TROUBLESHOOTING

In general, acrylics are the easiest and most robust castable mounting materials to use. Epoxies are very useful; however, complete mixing and the proper resin-to-hardener ratio is very important. Polyesters, especially for larger casting, may require some trial and error testing prior to mounting one-of-a-kind samples.

TABLE VI. Castable Mounting Troubleshooting

SYMPTOM	CAUSE	ACTION
Lack of or partial curing of resin	Improper or insufficient mixing	-Remount taking care to sufficiently mix epoxy resin and hardener -Check expiration date on catalyst (typically 1-year life)
Soft or gummy resins (grinding produces a matted finish)	Insufficient curing of resin – primarily by too low an exotherm	-Heat mount in an oven at 90-100°F (30-40°C) for 1-2 hours and let cool. -Resin should harden upon cooling
Bubbling, cracking, or yellowing of resin	Exotherm too high	-Mount at room temperatures below 85°F (30°C) -Decrease volume or volume percentage of hardener
Curing time takes too long	Improper resin mixture, old hardener, or mounting temperature to cold	-Replace old hardener -Mount at room temperature 70-80°F (30°C) -Preheat resin, but cure at room temperature
Bubbles in resin	Improper mixing or degassing of specimen	-Mix with a slow folding motion -Pour resin under vacuum and/or cure under higher pressures -Clean specimen prior to mounting

4.5 CASTABLE MOUNTING CONSUMABLES

TABLE VII-a. CASTAMOUNT ACRYLIC

Description	Quantity	Catalog Number	
CASTAMOUNT Acrylic resin kit (includes mixing cups, stirrer and several molds)	1 lb resin 12 oz hardener	AK-5000	
CASTMOUNT Acrylic resin powder	1lb (454 gms) 5 lbs (2.2 kg) 25 lbs 100 lbs	AR-5000-P1 AR-5000-P5 AR-5000-P25 AR-5000-P100	
CASTAMOUNT Acrylic hardener (liquid)	12 oz (0.36 l) 32 oz 1/2 gallon 1 gallon 5 gallons	AH-5000-H12 AH-5000-H32 AH-5000-H64 AH-5000-H128 AH-5000-H640	

TABLE VII-b. CASTAMOUNT ACRYLIC

Description	Quantity	Catalog Number	
PREMIUM Acrylic resin kit (includes mixing cups, stirrer and several molds)	1 lb resin 12 oz hardener	PAK-5000	
PREMIUM Acrylic resin powder	1lb (454 gms) 5 lbs (2.2 kg) 25 lbs 100 lbs	PAR-5000-P1 PAR-5000-P5 PAR-5000-P25 PAR-5000-P100	
PREMIUM Acrylic hardener (liquid)	12 oz (0.36 l) 32 oz 1/2 gallon 1 gallon 5 gallons	PAH-5000-H12 PAH-5000-H32 PAH-5000-H64 PAH-5000-H128 PAH-5000-H640	

TABLE VII-c. SUPERMOUNT ACRYLIC

Description	Quantity	Catalog Number	
Glass Filled Acrylic resin kit (black) (includes mixing cups, stirrer and several molds)	1 lb resin 12 oz hardener	GKAK-5000	
Glass Filled Acrylic resin powder	1lb (454 gms) 5 lbs (2.2 kg) 25 lbs 100 lbs	GRAR-5000-P1 GRAR-5000-P5 GRAR-5000-P25 GRAR-5000-P100	
Glass Filled Acrylic hardener (liquid)	12 oz (0.36 l) 32 oz 1/2 gallon 1 gallon 5 gallons	GRAH-5000-H12 GRAH-5000-H32 GRAH-5000-H64 GRAH-5000-H128 GRAH-5000-H640	

TABLE VIII-a. STANDARD CURING EPOXY

Description	Quantity	Catalog Number	
Epoxy resin	32 oz (0.95l) 1 gallon (3.8l)	EP-3000-32 EP-3000-128	
Epoxy hardener	8 oz (0.24l) 32 oz (0.95l)	EH-3000-08 EH-3000-32	
1-gallon pump dispenser	each	ED-3000-01	

TABLE VIII-b. FAST CURING

Description	Quantity	Catalog Number	
QUICMOUNT 2 fast epoxy resin	32 oz (0.95l) 1 gallon (3.8l)	EPF-3000-32 EPF-3000-128	
QUICMOUNT 2 fast hardener	8 oz (0.24l) 32 oz (0.95l)	EHF-3000-08 EHF-3000-32	

TABLE VIII-c. LOW VISCOSITY CURING EPOXY

Description	Quantity	Catalog Number	
ULTRATHIN 2 low viscosity epoxy resin	32 oz (0.95l) 1 gallon (3.8l)	ULTRA-3000-32 ULTRA-3000-128	
ULTRATHIN 2 low viscosity hardener	8 oz (0.24l) 32 oz (0.95l)	ULTRA-3000-08 ULTRA-3000-32	

TABLE IX. POLYESTER CLEAR RESIN

Description	Quantity	Catalog Number	
Polyester Resin	1 gallon (3.8l)	POLYCAST-128	
Polyester hardener	2 oz	POLYHARD-02	
Polyester dye kit	3 colors/pkg	EYE-1000	

TABLE X. MOUNTING MOLDS

Description	Quantity	Catalog Number	
Reuseable two piece plastic molds 1.25-inch molds 1.5-inch molds	12/pkg	METPREP-0125 METPREP-0150	
Disposable plastic mounting molds 1-inch molds 1.25-inch molds 1.5-inch molds	50/pkg	MOUNT-0100 MOUNT-0125 MOUNT-0150	
Reuseable Silicon rubber molds 1-inch molds 1.25-inch molds 1.5-inch molds 2-inch molds 3-in x 1.5-inc x 7/8-inch rectangular mold	3/pkg 3/pkg 3/pkg 3/pkg each	RMOUNT-0100 RMOUNT-0125 RMOUNT-0150 RMOUNT-0200 RMOUNT-3050R	
Phenolic Ring forms 1-inch molds 1.25-inch molds 1.5-inch molds	100/pkg	RF-0100 RF-0125 RF-0150	

TABLE XI. MOUNTING MISCELLANEOUS

Description	Quantity	Catalog Number	
Mold release	8 oz (0.24l) 165 oz (0.47l)	MR-1000-08 MR-1000-16	
Stirring sticks (6-inch depressors)	100/pkg	SS-1000-100	
Paper mixing cups (3 oz graduated cups)	100/pkg	MCUPS-1000-50	
Large paper mixing cups (7 oz graduated cups)	50/pkg	MCUPS-0700-50	
Plastic mixing cups (9 oz)	50/pkg	PCUPS-050	
Plastic specimen mounting clips	100/pkg	KLIP-0100	
Metal specimen mounting clips	100/pkg	MKLIP-0100	
1.25-inch specimen storage containers	25/pkg	STORE-0125