

**University of Saskatchewan  
Department of Mechanical Engineering  
Standard Operating Procedure # Mat0004**

**Procedure Title: Mounting Metallographic Specimens**

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**1. Version History**

Version #:           .1            
Supersedes:           n/a          

Handwritten amendments to the official procedures can be made by a single line through the text, along with the date, and initialed by the authorized individual making the correction. Changes are to be noted below. Formal changes to this SOP are made on the date of revision or sooner, where required.

Section	Changes made to official copy	Date	Initials

Reference Only

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**2. Introduction**

This SOP provides general instructions to:

1. Cold mount metallographic specimens using two part acrylic resins.
2. Hot mount metallographic specimens using heat and pressure activated resins.
3. Temporary mount thin specimens to a stub for polishing purposes.

All general lab safety practices must be followed in addition to those cited in this SOP. This SOP applies to all parties conducting this type of work.

**3. Definition**

SOP: Standard Operating Procedure  
MSDS: Material Safety Data Sheet  
WHMIS: Workplace Hazardous Materials Information System  
PPE: Personal Protective Equipment  
WSEP: Workplace Safety and Environmental Protection

**4. Personnel**

Persons authorized to perform this SOP:

By signing this form I acknowledge that I have read and understand this SOP, as well as the applicable MSDS's and that I will conduct myself in accordance with this SOP and the general laboratory rules.

**NOTE: ALL SIGNATURES MUST BE PRESENT ON THE SOP LOCATED IN THE YELLOW BINDER IN ROOM 2C26 – Mechanical Engineering Materials Lab, digital copies of SOP's are made available for reference and convenience only. Printed SOP's are valid for 24 hours only, after that time their accuracy must be verified with the OFFICIAL HARDCOPY VERSION.**

Name (Print)	NSID	Dep't	Signature	Date



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**5. Safety**

**NOTE: NONE OF THE GLOVES USED IN THE LABORATORY ARE INTENDED FOR SUBMERSION USE, THEY ARE FOR SPLASH/SPILL PROTECTION ONLY.**

**Cold Mounting:**

- Acrylic resins have different toxicological properties, all of the liquid portions are highly flammable and toxic, and most are also hazardous to the environment. These resins are often particularly harmful to pregnant females and their unborn child.
- The applicable MSDS's should be consulted for the particular resin being used.
- Compressed air is used to pressurize the Lecomat pressure vessel, safety glasses should be worn when venting the chamber and removing the lid.
- Any waste resin should be combined as per the instructions below and allowed to harden, then placed in a normal garbage bin. Do not place uncombined or unhardened liquid or powder in the garbage.

**Hot Mounting:**

- The resins used in hot mounting have different toxicological properties and the applicable MSDS's should be consulted prior to use for the material being used.
- Avoid creation of dust.
- Do not breath any dust created by the mounting media, ensure that the work area is clean after mounting and collect any spilled media as indicated.
- The heater for the hot mount press is hot enough to burn, only touch the handle of the heater.
- The hot mount press is a pressurized vessel and if the instructions are not followed properly the top plunger can be ejected from the machine potentially causing injury.
- Do not eject specimens while they are still hot as they could cause a burn.

**Temporary Mounting:**

- Some temporary mounting procedures involve the use of a hot plate and melted wax or a heat activated adhesive. Tongs should be used to handle any hot components and care must be taken to avoid touching a hot hot-plate or hot glue or wax.
- Acetone is used to clean the metal stubs and possibly the samples of the Crystal Bond, the MSDS for Acetone can be found on the Mechanical Engineering Safety page.
  - Acetone MUST be used in a fume hood, with Latex gloves, NOTE: Nitrile gloves are not compatible with Acetone.
  - All general lab procedures for chemical handling, labeling and disposal MUST be followed for the Acetone and Crystal Bond used.

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**6. Procedure**

6.1 Cold Mounting

Different cold mounting resins are available for use, if the resin is not listed here, follow the general instructions below as well as the packaging for the mixing ratio and safety precautions.

**Table 1: Cold Mounting Resins**

<b>Vendor</b>	<b>Product</b>	<b>Mixing Ratio Liquid:Powder</b>
Struers	ClaroCit	6:10 (weight)
Struers	VersoCit-2	1:2 (volume)
Buehler	Sampl-Kwick	See packaging

**Stage 1: Setting Up**

1. Ensure that the fume hood to be used is operational and empty of other chemicals.
2. Place samples, the Liquid and Powder into the fume hood.
3. Retrieve an appropriate number of paper cups, molds, measuring spoons and mixing sticks and place in the fume hood.

**Stage 2: Procedure**

4. Place the specimen(s) with the face to be examined down in the mold.
5. Mix the resin as per the instructions on the packaging, adding the powder into the paper cup followed by the liquid. Use a different measuring spoon for liquid and powder.
  - a. It is easiest to put the powder spoon into the powder container to avoid having to clean it every time, this will result in your gloves becoming contaminated, but they are easier to dispose of as per Mat0001 and Mat0002.
  - b. The liquid spoon can be left in the fume hood for the liquid resin to evaporate if it will evaporate, if it will not then it must be triple rinsed (with the waste rinse collected in a properly labeled waste container as per Mat0001 and mat0002).
6. Using a wooden stir stick mix the liquid and powder to homogenize the mixture.

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7. Pour the resin over the specimen(s) and tap the sides of the mold to remove air bubbles.
8. Place the mold(s) containing the specimen(s) into the Lecomat pressure vessel and seal the lid.
9. Turn on the compressed air valves.
10. Flip the toggle switch to pressurize the Lecomat.
11. Wait for a minimum of 20 minutes.
12. Close the compressed air valves.
13. Flip the toggle switch to depressurize the Lecomat.
14. Remove the lid of the Lecomat.
15. Remove the specimens.
16. The specimens can now be removed from the molds.

**Stage 3: Clean-up**

17. Allow any left-over resin to harden and dispose in the regular garbage.
18. If any powder spills in the fume hood just add some of the liquid and allow it to cure, then dispose of the cured resin in the regular garbage.
19. If any liquid spills in the fume hood add some powder and allow it to cure, then dispose of the regular garbage.
20. If the gloves become contaminated (very likely with the powder) then they must be disposed of as hazardous waste, as per Mat0001 and Mat0002. A labeled sealable plastic bag should be used and then they can be placed in the 2ndary containment bin for disposal at a later time. Ensure that the outside of the plastic bag does not become contaminated.
21. Return the supplies to the cupboard and close the door.
22. Ensure that the fume hood contains no resin either uncured or cured and that all materials have been disposed of properly.

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6.2 Hot Mounting

Different hot mounting resins are available for use, if the resin is not listed here, follow the general instructions below as well as the packaging.

**Table 2: Hot Mounting Resins**

<b>Vendor</b>	<b>Product</b>	<b>Special Properties</b>
Struers	MultiFast Red	General Mount
Struers	PolyFast	Conductive Mount for SEM
Buehler	ProbeMet	Conductive Mount for SEM

**Stage 1: Setting Up**

1. Select the appropriate mounting media (only use the conductive mounts if SEM work is being conducted as they are much more expensive, and pose a greater health risk).
2. Open the compressed air line to the mounting press (two valves, one yellow handle, and one silver).
3. The top closure of the press should be in its storage location and not inserted into the press, if it is in the press, contact the departmental assistant.

**Stage 2: Procedure**

4. Rotate the ram to the "UP" position
5. Position specimen on the mold base (with the part to be examined facing down onto the mold base).
6. Rotate the ram control handle to the "DOWN" position.
7. Add approximately 1 scoop of mounting medium to the mold chamber.
8. Place the top closure over the mold chamber with the "OPEN" label facing the front of the press.
9. Lower the top closure into the mold chamber and once it has been lowered sufficiently so that the locking ears will go under the locking tangs on the mold cylinder, rotate 90° clockwise to the stop position. The "LOCK" label should not be facing the front of the press, and the top closure should not be able to be pulled out.
10. Place the heater into position.
11. Push the top closure handle towards the back of the press to an angle of 45°.
12. Rotate the ram control to the "UP" position; ensure that the top closure is secured by the locking ears being under the tangs on the mold cylinder.
13. Ensure that the appropriate pressure is being applied – the pressure gauge has 3 red dots corresponding to 3 different mold



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diameters, the most common being 1 inch, the largest available in the lab is 1.25 inches. Ensure that the pressure reading is on the appropriate red dot for the mold size being used.

14. Turn on the heater.
15. Set the timer for 25 minutes.
16. After 25 minutes, turn off the heater and remove it from the mounting chamber, returning it to the storage location.
17. Place the cooling fin apparatus on the mounting chamber and wait a minimum of 20 minutes.
18. Rotate the ram to the "NEUTRAL" position and wait until the mold pressure indicates zero.
19. Pull the top closure handle forward 135° (until it stops).
20. Rotate the top closure 90° counter clockwise, so that the "OPEN" label is toward the front of the press and that the ears are no longer under the tangs of the mold cylinder.
21. Raise the top closure handle to the vertical position.
22. Rotate the ram control to the "UP" position, ensuring that the ears clear the mold cylinder tangs and the top closure raises smoothly.
  - a. If the top closure gets stuck in anyway immediately rotate the ram control to the "DOWN" position to avoid a dangerous increase in cylinder pressure with only a partially secured top closure.
23. Remove the top closure and place in its storage location.
24. Remove the cooling ring and place in its storage location.
25. Remove specimen.
26. Rotate the ram to the "NEUTRAL" position.
27. Close the two (2) compressed air valves.

**Stage 3: Clean-up**

28. If the gloves become contaminated (very likely with the powder) then they must be disposed of as hazardous waste, as per Mat0001 and Mat0002. A labeled sealable plastic bag should be used and then they can be placed in the 2ndary containment bin for disposal at a later time. Ensure that the outside of the plastic bag does not become contaminated.
29. Return the supplies to the cupboard and close the door.
30. Ensure that the counter space around the hot mount press contains no mounting medium.
31. To clean up spilled powder, it is best to use a damp paper towel and dab the powder so that it sticks to the paper towel, then wipe the surface clean with another paper towel. Collect the paper

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towels in a labeled sealable plastic bag and place in the waste disposal 2ndary containment area.

### 6.3 Temporary Mounting with Crystal Bond or Wax

The procedure for mounting with Crystal Bond or Wax is the same, with Wax having a lower melting point and providing less holding power. In addition it will be possible to scrape the wax off of the sample back and the mounting stub with a blade instead of using acetone.

Crystal Bond 509 Amber:

- MSDS is available on the Mechanical Engineering Safety Page.
  - Softening Point: 71°C
  - Flow Point: 121°C
  - Soluble in Acetone
- 1) Ensure that your specimen is clean, dry and free of grease or oils.
  - 2) Place a mounting stub onto the hot plate and turn the hot plate to an appropriate temperature (>121°C).
  - 3) Allow the metal stub to heat up, you can check the temperature of the top surface with a radiation thermometer (laser pointer kind).
  - 4) Once the top surface is a suitable temperature put a small amount of Crystal bond on the surface and wait until it flows (It will not flow like water, more like a thick oil).
  - 5) Place your sample on the Crystal Bond using tweezers and push down gently to secure the specimen – ensure that you do not touch the region that you wish to indent on.
  - 6) Remove the metal stub from the hot plate using metal tongs and place on a metal or heat resistant surface to allow air cooling.
  - 7) Turn off the hot plate.
  - 8) Wait for the specimen to cool to room temperature before polishing.

Specimen Removal Procedure:

After testing the stubs must be cleaned to be 100% free of Crystal Bond.

- 1) Re-heat the stub using the hotplate, with the sample attached until the sample can be removed (using tweezers).
- 2) Turn off the hot plate and place the metal stub on a heat resistant surface and allow it too cool to room temperature (handle the hot components with tongs or tweezers).

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- 3) Once the metal stub is cool, place it in a beaker containing Acetone (in a fume hood, while wearing Latex gloves and using tongs), allow the Acetone to dissolve the crystal bond.
- 4) Remove the Metal Stub, rinse with ethanol and dry.
- 5) Collect the Acetone and ethanol waste in an appropriate labeled waste container.
- 6) Wash the beaker and utensils used and collect the waste according to the Laboratory Procedures provided in Mat0001 and Mat0002.

#### 6.4 Temporary Mounting with Foam Mounting Tape

While permanent mounting provides the best stability for the finest finish, crystal bond provides a very stable mount for polishing as well. If neither of these alternatives are viable for a sample then the use of foam mounting tape is a suitable option.

Tape: Scotch Exterior Mounting Tape (it is red, comes in rolls about 1 inch wide). The tape is water proof and double sided.

1. Using a holding stub use the double sided tape to stick your specimen onto the stub.
2. Polish using normal polishing procedures.

Note: If your specimen comes unstuck it is best to replace the tape with a new piece for maximum adhesion.

- For “normal” polishing (i.e. using water based suspensions and Si-C) the tape can go in the general garbage.
- For alcohol based suspensions the tape must be collected as hazardous waste in an appropriately labeled bag.

#### 7. Equipment or Materials Required

The following Personal Protective Equipment (PPE) is required for this procedure:

- Fume hood
- Gloves (latex or Nitrile) (Disposable)
- This SOP
- Lab Coat
- Closed Toed Shoes
- Safety Glasses
- Long Pants
- This SOP
- MSDS Information

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- Operational Eye Wash Station

This procedure may also require the following:

- Lecomat pressure vessel.
- Plastic, rubber or silicon molds.
- Paper or plastic mixing cup.
- Wooden mixing stick.
- Plastic measuring cups.
- Hot plate.
- Metal tongs.
- Mounting medium.
- Infrared thermometer.

### **8. Highlights / Critical Control Points**

Fume hoods, counter tops and work areas must be kept clean at all times, any debris must be cleaned up according to the applicable SOPs and standard laboratory practices.

### **9. Regulatory / Standards**

ME0001, Mat0001 and Mat0002 must be followed for all laboratory use.

### **10. Trouble Shooting**

If any of the equipment is not functioning correctly contact the Departmental Assistant.

### **11. References**

Mat0001 – Materials Lab Requirements

Mat0002 – General Chemical Usage Guidelines

University of Saskatchewan DHSE Documents:

Laboratory Safety Manual:

[http://www.usask.ca/dhse/file\\_view/download.php/Laboratory\\_Safety\\_Manual.pdf?id=32&view=1](http://www.usask.ca/dhse/file_view/download.php/Laboratory_Safety_Manual.pdf?id=32&view=1)