OFFICE OF THE CURATOR
SAMPLE PROCESSING PROCEDURE

DATE: December 12, 2017 SPP 60

PREPARATION OF STANDARD 30 MICRON PETROGRAPHIC THIN SECTIONS OF LUNAR, METEORITE AND TERRESTRIAL SAMPLES

1. INTRODUCTION

This procedure defines steps to be taken in the production of standard 30 micron thin sections of lunar, meteorite and terrestrial samples in the Lunar and Meteorite Curatorial Thin Section Laboratory (TSL/MTS). Unless specially requested, all meteorite and lunar thin sections will be double polished and single mounted to produce a high quality section. A cover glass is not used because sections are studied by electron and ion beam bombardment (Appendix A.). This procedure can also be used to produce polished thick sections (Ref. Step 6.2.11).

The procedure for making thin sections has been used at the JSC since about 1970. Great care is taken to prepare sections with minimum sample loss. Sample numbering and orientation must be maintained and documented.

Lunar and meteorite samples are shock damaged silicate materials with numerous cracks and loose grains. Because lunar and meteorite samples contain metallic grains that could rust and important salts that would dissolve if water were used, alcohol (200 proof or 180 proof) is generally used instead of water in the preparation of these thin sections.

Note: Variations in this procedure can be used as necessary. Thin section preparation is an art, requiring judgment by skilled personnel, based on the nature of the sample.

2. RESPONSIBILITY

2.1 It is the responsibility of the laboratory personnel to:

2.1.1 Verify that all thin section requests and related work are authorized by a Curatorial Order (CO) before processing begins. Curatorial Orders can be found in the Data Center, J31N-1110.

2.1.2 Prevent any cross contamination of lunar, meteorite and terrestrial materials.

2.1.3 Not use water on lunar and meteorite samples unless specified.

2.1.4 Always use silica glass slides to produce lunar sections.

2.1.5 Notify the appropriate curator of the TSL/MTS prior to any variation of polishing compounds, lubricants or lapping films mentioned in this procedure, so they can be properly tested prior to use. There are some polishing compounds and lapping abrasives that could contaminate or even damage certain specimens.

2.1.6 Comply with operating (Section 6) and safety procedures (Section 3).

*Revised, relative to version dated May 7, 2015, to specify 180 and 200 proof ethyl alcohol and 400 grit instead of 15 micron.

2.1.7 Comply with provisions of JSC-03225, current version, (JSC Plan for Safeguarding Lunar Samples) when handling lunar samples.

2.1.8 Document all operations on an F-6 form in a sample data pack.

2.1.9 An important part of this procedure involves epoxy impregnation of sample to prevent loss of grains and scratching during grinding and polishing.

2.2 The curators of lunar and meteorite samples are responsible for:

2.2.1 Insuring that laboratory operation is consistent with the requirements of this document.

2.2.2 Initiating CO and establishing priorities.

2.2.3 Signing or approving F-6's for lunar and meteorite processing.

3. SAFETY

3.1 This procedure requires the use of 180 and 200 proof ethyl alcohol as a cleaning agent. Alcohol is very flammable. Keep all flames or other ignition sources away from the general work area. Use this material in fume hood when possible.

3.2 Epoxy mixtures and mounting procedures should be accomplished within the fume hoods located in the meteorite laboratory, Room 1026, and the lunar lab, room 1022. Both vent hoods shall be re-certified yearly and the vent hood windows shall remain at the 100 feet per minute arrow, as noted on the certification tag. For re-certification call Occupational Health Services/SD3229 (x36726).

3.3 Nitrile gloves are required to be used when handling materials containing epoxy since there is a potential to develop allergic sensitization.

3.4 Refer to Safety Data Sheet notebook regarding properties of the epoxy, alcohol, diamond paste, and other materials that are used. Notify the ARES Safety Representative of any need for further information.

3.5 Safety precautions for using high speed Ingram saws and grinders are listed below.

3.5.1 Evaluation of mineral oil mist by Occupational Health Services suggests the use of disposable respirators with the use of the Ingram grinders. Disposable respirators (3M, model 8240, R95) will be available and utilized during the grinding process. Disposable masks shall be replaced at least once a week.

3.5.2 Sparks can occur when the Ingram saws and grinder motors are turned on. Alcohol or other flammable solvents shall not be used near saws or motors.

3.5.3 Make certain the vacuum pump switch, used to hold the specimen on the vacuum chuck, and the coolant switch are turned on before cutting or grinding begins.

3.5.4 Plastic safety shields located on the front of the Ingram saws and grinders shall be kept closed during grinding and cutting procedures.

3.5.5 Safety glasses shall be worn to protect against glass slides or PBs that can be thrown from the machines during grinding and cutting procedures.

3.5.6 Always feed samples into the blades and grinding wheels very slowly. If samples are fed too fast, they can be thrown from the vacuum chuck and become airborne.

3.5.7 Long hair and loose clothing items shall be secured so that they will not get entangled in the pulley belts or rotating equipment located on the saws and grinders.

4. DEFINITIONS

AEP	- Aminoethyl piperazine (Hardener for Araldite 506)
Almag Oil	- Mineral oil used as coolant in Ingram saws & grinders
Araldite 506	- Epoxy resin (base compound)
CO	- Curatorial Order
F6	- Weight Check Sheet
Hg	- Mercury
Interference Color Chart	- Helps determine thickness of minerals by order of interference colors in conjunction with the petrographic microscope
MDA	- Diamino-p-Menthane (Catalyst for Araldite 506)
MTS	- Meteorite Thin Section Laboratory
Orientation of chip	- Lunar orientation
PB	- Potted butt
Plucking	- The loss of small grains from section during grinding, polishing or handling
PM	- Probe mount, circular thin section
Polarized light microscope	- Transmitted light microscope used to check thickness of minerals
Polishing Oil	- Light mineral oil used for polishing
Polishing Paper	- 100% cotton fiber bond paper used for polishing
Reflected light microscope	- Microscope used to check polished surface
SDS	- Safety Data Sheet
Thick Section	- Single-mounted polished section thicker than 30 microns
TS	- Thin section
TSL	- Thin Section Laboratory
Wedging	- Sample thickness becomes uneven during final polishing process

5. REQUIRED EQUIPMENT

5.1 Chemicals

Araldite 506 resin
MDA - Catalyst for 506 epoxy resin
AEP -Hardener for 506 epoxy resin
Buehler Epoxicure
Buehler Epothin
200 proof ethyl alcohol
180 proof ethyl alcohol
Almag oil
Diamond paste - 6, 3, and 1 micron
.05 alumina oxide
Buehler polishing oil
Superglue (used only for special purposes)

5.2 Equipment

Oven
Hot plate
Vacuum chamber
Ingram cut-off saw with Almag oil coolant system
Ingram grinder with Almag oil coolant system
Vacuum pump used to pump vacuum to vacuum chuck
Polarized light microscope
Reflected light microscope
Ultrasonic cleaner
Polishing table with 8" polishing wheel attached
Final polishing table with drain, 8" polishing wheel attached
Lapping machine, sander, with 8" wheel attached
Diamond impregnated blades (5" x .015" x 5/8"
Diamond impregnated silicon carbide grinding wheel
100% cotton fiber bond finish polishing paper
Plastic molds for impregnating (1/2" to 3" around)
Polishing wheel ring-metal ring used to hold polishing paper
Top loading balance (0-100 grams)
Micrometer - inches and microns
Sandpaper, silicon carbide, 240 - 320 grit
3M Model 8240 R95 mask
Felt polishing pads, 8"
Interference color chart for minerals
Glass slides - round and rectangular (1" and 1x2")
Silica slides - round (1")
Teflon stir sticks
Plastic mixing cups, 50 and 100 ml.
Buehler fine cut-off saws with blades
Logitech polishing machine
Diamond Scribe
Nitrile gloves

6. PROCEDURE

6.1 Impregnating Samples

Araldite 506 is used for making Lunar thin sections.

Buehler Expoxicure is used for potting meteorite chips and Buehler Epothin is used for mounting the potted butts to the slides.

This procedure presents a widely applicable method of immobilizing small pieces of rock in epoxy resin. The impregnation portion of this procedure is very critical in order to insure minimal amount of sample plucking during the cutting and grinding procedures.

Sample impregnation is usually performed at the end of the workday, so the epoxy can remain in the oven for 10-12 hour cure. All hot plates and ovens operate at 125° F.

6.1.1 Choose the proper size plastic impregnating mold so that the sample will be completely submerged, allowing approximately 1/8" layer of epoxy on all sides including the top.

6.1.2 Inscribe the sample number on the bottom of the plastic mold and the orientation of the sample, if required.

6.1.3 Place the sample into the mold, orientate and, place the mold on the hot plate at 125°F while the epoxy is being mixed.

6.1.4 Weigh required amounts of epoxy components into a beaker using a top loading balance, 0-100 grams (Ref. Step 3.2) and stir for 2 to 3 minutes.

Standard mixing ratio for the Araldite is:
7.5 g Araldite 506
1.2 g AEP (hardener)
0.8 g MDA (catalyst)

Mixing ratios for the Buehler Epoxicure or Epothin are written on the resin and hardener containers. Double check when new containers are opened as the mixture ratios are subject to change. Mixing ratios may be adjusted if necessary.

The amount of epoxy to mix depends on both the size of the sample and the mold. Follow mixing ratios carefully, since adding excess hardener or catalyst can cause shrinkage and overheating, resulting in damage to the specimen.

6.1.5 Pour pre-mixed resin into the mold until the chip is completely covered (Ref. 6.1.1).

6.1.6 Place the mold into the vacuum chamber and slowly pull a vacuum to 29 inches of mercury to avoid forming air bubbles. Hold vacuum for 5 to 10 minutes; then release.

6.1.7 Remove the mold from the vacuum chamber, making certain the orientation of the chip is correct. Check photos supplied with chips to verify orientation. Place the mold back in the oven at 125°F for 10-12 hour cure.

6.1.8 Following a 10-12 hour cure, remove the mold from the oven and inscribe sample number and orientation on the top surface of the hardened epoxy. Pry the impregnated sample (PB) out of the plastic mold.

6.2 Primary Mounting Procedure

6.2.1 Flatten bottom of epoxy potted butt sample (PB) using lapping machine with 320 grit sandpaper until desired surface for thin sectioning is obtained. Fit check the PB to the glass slide to make certain it doesn't overlap the glass slide. If so, grind off excess epoxy.

Use 180 or 200 proof ethyl alcohol as directed as a coolant and cleaning agent for all lunar, meteorite and terrestrial samples.

6.2.2 Sonicate PB, sample side facing up, for 1 minute, using 200 proof alcohol.

6.2.3 Fix 400 grit silicon carbide disc to polishing wheel and flatten PB, using 180 proof ethanol as a coolant.

400 grit discs are used to remove 320 grit scratches and damage generated from Step 6.2.1.

6.2.4 Remove 400 grit disc, clean wheel and sonicate PB.

6.2.5 Fix cotton bond polishing paper to wheel, saturate with 6 micron diamond paste and polish PB using polishing oil. A high gloss polish, to the naked eye, is sufficient. Surface does not have to be scratch free.

6.2.6 Sonicate PB with 200 proof alcohol. Place it on the hot plate, sample surface down, for approximately 20 - 30 minutes to dry.

6.2.7 Inscribe the sample number and orientation on the glass or silica slide using a diamond scribe, clean with alcohol, and place on the hot plate next to PB to dry for 5 minutes.

6.2.8 Using fume hood, mix epoxy (Section 3.2) and stir for 1 minute.

Araldite 506 or Buehler mixture ratios are outlined in Section 6.1.4.

6.2.9 Place epoxy mixture onto the hot plate for approximately 5 minutes, until bubbles dissipate.

After 5 minutes all bubbles should have disappeared. The epoxy should be at its lowest viscosity and ready for mounting procedure. Araldite 506 starts to thicken about 30 minutes after it has been mixed, which allows 20 - 25 minutes of application time.

6.2.10 Turn the PB over onto the hot plate so that the sample surface faces up. Spread 2 or 3 drops of epoxy over the surface using a Teflon stir stick. Let set for 1 minute.

6.2.11 Place PB into the vacuum chamber at -29 inches Hg for five minutes to impregnate exposed sample surface with epoxy. Remove the PB and place it on the hot plate with sample side facing up.

6.2.12 Mount the slide onto the PB using the surface opposite the scribed numbers. Being careful not to touch the mounting surface, place the edge of the slide down first, slowly laying it onto the sample surface, in order to force any trapped bubbles out the other side. Gently apply pressure on the back of the slide with your finger and press any excess epoxy out. Turn the PB over, slide side down onto the hotplate, for an 8 - 10 hour cure. Wear Nitrile gloves to avoid getting epoxy on fingers.

Single mounted polished thick section requests can range anywhere from 100 microns to 1 mm final thickness. Following the cutting and grinding procedures, Steps 6.3.1 and 6.3.2, allow approximately 100 - 125 microns above the requested thickness in order to complete final polishing procedure Steps 6.5.1 through 6.5.9.

6.3 Cutting and Grinding Procedure

Refer to Step 3.4 of the safety procedures using Ingram saws and grinders .

6.3.1 Using the Ingram cut-off saw with a diamond blade attached, cut the excess PB off leaving approximately .020" (500 microns) of sample thickness on the glass slide. The kerf of the diamond blade is .015".

Use the micrometer located on the feed control of the Ingram saw to determine the thickness of the cut.

Note: See Section 6.7 for use of alternate cut-off saw.

6.3.2 Using the Ingram grinder with a 320 grit diamond impregnated wheel attached, grind the section down to 250 - 275 microns. Sonicate in 200 proof ethyl alcohol.

A standard hand-held micrometer can be used to check the sample thickness. Use a razorblade to cut away a small portion of the epoxy surrounding the sample in order to get an accurate measurement of the glass slide. Measure the thickness of the glass including the sample and subtract the two values.

6.3.3 If necessary attach 8" polishing wheel to the polishing table and clamp a 400 grit silicon carbide disc to the wheel. Using polishing oil as a coolant, flatten the 250 - 275 micron thick section until it is approximately 200 microns thick. To produce a double mounted 30 micron thin section, continue to section 6.4 (Final Mounting Procedure) at this point. To make a single mounted 30 micron thin section continue lapping until a thickness of approximately 100 microns is achieved and now proceed to Section 6.5 (Final Polish Procedure).

400 grit silicon carbide discs not only conserve diamond paste, but also provide a quicker way of delicately removing sample during the thinning process.

6.4 Final Mounting Procedure (for making double mounted sections only)

6.4.1 Attach a sheet of 100% cotton bond polishing paper to the wheel and saturate with 6 micron diamond paste. Polish a 200 micron thick section until it is approximately 150 microns thick.

6.4.2 Sonicate the 6 micron polished, 150 micron thick section and place it on the hot plate to dry (20 -30 min.).

6.4.3 Clean the glass slide with alcohol, wipe with lint free wipe and place it on the hot plate next to the thick section to dry (5 min.).

6.4.4 Mix mounting mixture (Ref. 6.2.7). Place mixture on the hot plate for 5 minutes.

6.4.5 Remount thick section using same technique as primary mount (Ref.6.2.10). Let mounted sample cure on the hot plate for 8 - 10 hours.

6.4.6 Remove the double mounted section from the hot plate, scrape hardened epoxy off both glass slide surfaces with a razorblade, and scribe the sample number onto the new glass slide.

Scribe an "X" on the primary glass slide to indicate the slide to be removed during the grinding procedure.

6.4.7 Place the double mounted section onto the Ingram grinder chuck with the slide marked "X" facing out. Slowly grind the primary slide off. The sample thickness cannot be measured until the slide has been completely ground away. Final sample thickness should be approximately 100 - 125 microns (Reference rates from Step 6.3.2).

6.5 Final Polish Procedure

100% fiber cotton bond polishing paper, along with polishing oil as a lubricant, may be used throughout 6, 3, and 1 micron diamond polishing procedure. Thin sections are held by hand, rotating sample frequently. Samples are sonicated in ethyl alcohol to remove grit between steps (extremely important).

6.5.1 Attach wheel to polishing table. Attach polishing paper to wheel and saturate with 6 micron diamond paste. Polish until thickness is approximately 50 microns.

Frequently check the thickness of the minerals by using a polarized light microscope along with an interference color chart for minerals, located in lunar and meteorite polishing rooms (Rooms 1022 and 1024). Desired thickness is approximately 50 microns. Any wedging <u>must</u> be corrected during the 6 and 3 micron polishing phase.

6.5.2 Remove 6 micron polishing paper, clean wheel, and sonicate sections in alcohol.

6.5.3 Secure polishing paper to wheel, saturate with 3 micron diamond paste, polish sections until they are 30 - 35 microns thick.

Final thickness, 30 - 35 microns, should be accomplished during the 3 micron diamond polishing procedure. It is <u>very important</u> to check the sample thickness frequently, using the transmitted light microscope.

CAUTION: Wedging can occur very rapidly!

6.5.4 Remove 3 micron polishing paper, clean wheel, and sonicate thin sections.

6.5.5. The final two steps of this procedure are 1 micron diamond polish and .05 aluminum oxide final polish. For lunar and meteorite sections a mixture of .05 aluminum oxide and 200 proof alcohol are used for final polish. For terrestrial samples filtered water can be used in place of alcohol. These two steps do not remove much material. They are used strictly for final polishing.

a) Attach sheet of polishing paper to the wheel, saturate with 1 micron diamond paste. Polish sections until the majority of 6 and 3 micron scratches disappear.

b) Using reflected light microscope with 25/0.50 P lens, scan the sections from side to side checking for scratches. If scratches persist remove the 1 micron polishing paper, sonicate and repeat Step 6.5.5.

c) Sonicate sections.

d) Attach wheel to final polish table. Secure felt polishing pad to polishing wheel. Saturate felt pad with .05 aluminum oxide. Polish sections until all scratches are gone. If scratches persist, sonicate thin sections, flush felt pad with 200 proof alcohol and repeat .05 aluminum oxide polishing procedure.

6.5.6 Consumable materials should not be shared among samples of different types to prevent cross contamination

6.6 Documentation

6.6.1 Document all sample processing as described in SPP-4, Data Procedures for the Thin Section Laboratory.

6.6.2 Scribe "SG" on slides mounted with superglue.

6.6.3 Call for inspection by the initiator of the CO and obtain signature on F6.

6.7 Alternate Procedures (See Introduction)

6.7.1 When a small sample must be conserved, use a fine cut-off saw (Buehler) with a thin blade (Reference 6.3.1).

6.7.2 When large area sections are polished, use the automated polishing machine (Logitech). (Meteorite only).

6.7.3 Superglue may be substituted for Araldite during final mounting procedure (Ref. 6.4), when it is determined the requirements of the investigator will be to detach the thin section for special experiments.

6.7.4 Use 3M plastic lapping paper to prepare Boron-free sections.

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This procedure replaces SPP 60 dated May 7, 2015.

The procedure takes effect on the date of the last signature.

The term of the procedure is indefinite.

Double-mount, double-polish procedure (JSC~1996)