THIN SECTION PREPARATION PROCEDURE

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THIN SECTON PREPARATION PROCEDURE

1. PURPOSE

The purpose of this procedure is to document the process for the preparation of thin sections from rock samples. Thin sections are used for geological analysis on the microprobe, various microscopes, and for other geological analysis.

2. SCOPE

This procedure covers any geologic thin sections prepared in ESS Division for the Yucca Mountain Project (YMP). For the purpose of this procedure, the term thin section will refer to standard petrographic thin sections, as well as grain mounts and fluid inclusion slides.

3. PROCEDURE

3.1 Inital Preparation

3.1.1 There are no unusual environmental conditions.

3.1.2 The requestor submits samples for thin section preparation by completing the thin section request form (Attachment 1). The request is entered into the log book.

3.1.3 Set samples (pre-numbered in ink) on the table in rows.

3.1.4 Mark the sample number in an aluminum dish and on a bag. Use extreme care to mark the sample ID correctly. Set the bag under the rock and the aluminum dish upside down on top of the rock. Do this for every rock sitting on the table.

3.1.5 At this point you need to decide if the sample needs to be impregnated to aid in its slabbing. This is a subjective decision made by the technician. If not, go on to 3.1.5.

- 3.1.5.1 If the sample needs to be stabilized by impregnation, place it on top of a cookie sheet covered with aluminum foil.
- 3.1.5.2 Paint the surface of the rock completely with epon 815-diethylene triamine.
- 3.1.5.3 Make sure the sample I.D. is still clearly legible. If not, write the I.D. in lead pencil on a piece of paper and attach the paper to the epoxy surface.
- 3.1.5.4 Allow the epoxy to cure before proceeding (24 hours at room temperature, 30-45 minutes in the oven).

3.1.6 Use appropriate slabbing saw to cut a slab 3/8 to 1/2 in. thick. Set the slab on top of the corresponding aluminum dish and put the excess material in the bag that has been pre-numbered.

3.1.7 At this point, refer to the worksheet to determine whether to core each sample with the diamond core or cut them to chip size with the diamond saw.

3.1.8 Use a round or rectangular slide to find the best area on the sample for the thin section. Trace around the pattern with a marking pen. If coring is to be done, continue with step 3.1.8. If cutting is to be done, skip to step 3.1.9.

3.1.9 Coring using the Drill Press

3.1.9.1 Put the coring adapter into the drill press.

3.1.9.2 Refer to the worksheet to decide what size bit you will need.

3.1.9.3 Be sure that the threads on the bit and adapter are clean and slightly lubricated with WD 40.

3.1.9.4 Screw the bit on to the adapter.

3.1.9.5 Check to make sure that there is water in the Coolant Slump.

3.1.9.6 Place the rock holding vice on the drill press table under the core bit.

3.1.9.7 Secure one rock slab firmly in the jaws of the vice.

3.1.9.8 Plug in the water pump.

3.1.9.9 Adjust the water flow at the bit so that it is flowing light to moderate.

3.1.9.10 Turn on the drill press (red button in the front of the drill press).

3.1.9.11 Use the handle on the side of the drill press to slowly lower the core bit until it lightly touches the surface of the rock.

3.1.9.12 Slowly increase the pressure until the diamond core bit cuts into the rock sample and maintain that pressure until it cuts through the sample.

CAUTION: Do not apply excessive pressure as this may ruin the bit!

3.1.9.13 Use the handle to raise the core bit out of the rock and place the cored plug in its aluminum dish.

3.1.9.14 Place the remainder of the slab in its respective sample bag.

3.1.9.15 Core each of the remaining slabs one at a time, placing cores in aluminum dishes and remaining samples in sample bags.

3.1.9.16 Turn off the drill press and unplug the water coolant.

3.1.9.17 Go to step 3.10.

3.1.10 Cutting using the 8" or 10" saw and high speed lapping with the 100 mesh metal bonded diamond plate. The goal is to shape the slab into a circle or rectangle.

3.1.10.1 Using the 8" or 10" saw, cut each slab one at a time, slightly smaller than the pattern traced on it.

3.1.10.2 Place the unneeded cuttings in each respective sample bag and each trimmed slab in its aluminum dish before going on to the next slab.

3.1.10.3 After finishing the trimming, turn on the power on the high speed lap and adjust it to a comfortable speed.

3.1.10.4 Turn on the water so it is slowly flowing.

3.1.10.5 Hold the chip firmly and grind to produce a sample with parallel faces, while being careful not to let your fingers touch the plate.

3.1.10.6 Dry the sample with the air blower and put it back in its aluminum dish.

3.1.11 Put the aluminum dishes containing the sized slabs on a cookie sheet and set them in the oven for one hour, or until dry, at 150-250°F (approximately 90°C).

3.1.12 Take the cookie sheet out of the oven and place them on the table and cool to room temperature.

3.1.13 The sample number is placed on the top of the slab by painting a white stripe on it with typewriter correction fluid and then writing the number on the stripe with a lead pencil (an epoxy coating permanently retains the sample number on the top). Use extreme care to mark the sample ID correctly.

3.1.14 To stabilize the sample slab it is either dipped in epoxy or if requested, vacuum impregnated with epoxy.

3.1.15 Stabilization by epoxy dip.

3.1.15.1 Reheat the samples for 10-15 minutes.

3.1.15.2 Mix up the Shell epoxy - 10 parts resin to 1 part hardener - by weight, not volume. (50 grams resin + 5 grams hardener will fit into an 8 ounce paper cup).

3.1.15.3 Use the forceps and dip the rock chip in the epoxy and put it back in the aluminum dish number-side down. Set the aluminum dishes back on the cookie sheet.

3.1.16 Stabilization by vacuum impregnation.

3.1.16.1 Place samples in aluminum dish, sample number down.

3.1.16.2 Place dish into vacuum chamber.

3.1.16.3 Mix Epoxy per 3.1.14.2 and pour into beaker in the vacuum chamber.

3.1.16.4 Place vacuum chamber lid onto the vacuum chamber.

3.1.16.5 Start vacuum pump and pull a vacuum for 15-20 minutes.

3.1.16.6 While still under vacuum align each sample by rotating it under the beaker of epoxy and pour epoxy over the top of the sample.

3.1.16.7 After all the samples have been covered, close the vacuum valve and open the bleed valve slowly and allow the chamber to return to normal atmospheric pressure.

3.1.16.8 Remove vacuum chamber lid and place samples on cookie sheet and put into the oven at 150° to 250°F to cure, approximately 30 minutes.

3.1.17 After all of the chips have been stabilized, return the cookie sheet to the oven for appproximately 30 minutes or until the epoxy has cured.

3.1.18 Remove the cookie sheet from the oven with pliers and set the sheet on the table. Let the samples cool for about 10 minutes.

3.1.19 Remove a sample from its aluminum dish and after ensuring that the number is legible, throw the old aluminum dish away and put the sample in a box. Do the same for all the samples.

3.2 Exposure and Flattening of Rock Chips

3.2.1 Use the high speed lap and the 100 mesh plate for the following steps:

3.2.1.1 Turn on the power and adjust it to a comfortable speed.

3.2.1.2 Turn on the water so it is slowly flowing.

3.2.1.3 Using the lap remove the sharp edge of epoxy from the side of the rock chip.

3.2.1.4 Remove the epoxy from the rock chip surface that is to be mounted to the glass section by holding the chip surface parallel to the lap. Do not go too deep into the rock chip or you will lose the benefit of impregnation.

3.2.1.5 Surface Impregnation - If grinding breaks through the initial epoxy impregnation and sample begins to pluck, a second impregnation may be necessary. Place sample on 250°F hot plate until dry, approximately 15 minutes, proceed to 3.3.2.2 through 3.3.2.6 - Preparing of Epoxy.

3.2.1.6 Apply small amount of epoxy to the exposed surface with a glass stir rod to impregnate surface of sample. Repeat this step until the sample no longer absorbs epoxy. Leave on hot plate until epoxy cures, then proceed to 3.2.2.

3.2.2 Put the 45 micron plate on the low speed lap - use this lap only long enough to remove the deep scratches caused by the 100 mesh lap. Be sure the final surface has no wedges on it.

3.2.3 Lapping on the Logitech

3.2.3.1 Put the exposed and roughly flattened rock chips into the rings on the Logitech (LP 30). Be sure the rock chips fit tightly in the rings and the exposed face is down. If the chips do not fit tightly, proceed to step 3.2.4.

3.2.3.2 Put a piece of 1/4 inch rubber on top of the rock chips and on top of the rubber, add a 1 inch thick piece of foam. Be sure these two items are cut in a form that fits tightly in the lapping rings.

3.2.3.3 Add the weights used for lapping.

3.2.3.4 If near empty, fill the LP 30 drum with Pella oil to the mark on the side of it, and add 50 to 60 ml of silicone carbide (600 grit).

3.2.3.5 Run the machine for 20 minutes using a slurry mixture of 600 grit silicone carbide and Pella oil.

3.2.3.6 After the rock chips are removed from the machine, clean them in the ultrasonic with isopropyl or ethyl 190 alcohol and set them on a hot plate (180°F, 82°C) for at least 4 hours or until dry and oil free.

3.2.4 Lapping on the glass plates

3.2.4.1 The samples that would not fit into the ring of the Logitech (LP 30) or that are too soft and friable will have to be lapped by hand.

3.2.4.2 Pour a small amount of silicone carbide (600 grit) and kerosene on the glass plate.

3.2.4.3 Move your sample around in the mixture in figure 8's and circles.

3.2.4.4 Rinse the sample off in alcohol and hold it up to the light to see if it is smooth. A smooth sample will reflect light. If it does not, repeat step 3.2.4.3 until it does.

3.2.4.5 Pour a small amount of aluminum oxide (1200 grit) and kerosene on another glass plate.

3.2.4.6 Move your sample around in the mixture in figure 8's and circles.

3.2.4.7 Rinse the sample off in alcohol and hold it up to the light to see if it is smooth. Repeat step 3.2.4.6 if necessary.

3.2.4.8 When the remaining chips are lapped, clean them in the ultrasonic with isopropyl alcohol and set them on a hot plate (180°F) for at least 4 hours or until dry and oil free.

3.3 MOUNTING

3.3.1 Preparing the Slides

3.3.1.1 Pre-grade the slides with a dial-caliper. Use only slides with less than a 10 micron taper from end to end.
3.3.1.2 Clean the slides in the ultrasonic with alcohol for 30 seconds.
3.3.1.3 Remove the slides, one at a time, with the tongs and dry them off with a clean wipe.
3.3.1.4 Set the slides on the hot plate.

3.3.2 Preparing the Epoxy

3.3.2.1 A fairly thin, low-temperature, slow curing epoxy is needed to use with the mounting press. The Buehler (resin 20-8130-032, hardener 20-8132-008) epoxy is a very good choice of epoxies. The index of refraction is 1.58 and the shelf life is at least one year if stored at room temperature.
3.3.2.2 Use the Dial-O-Gram balance with the disposo weigh boat.
3.3.2.3 Adjust the scale to "0" with the disposo dish on the scale.
3.3.2.4 Using a 5 to 1 ratio by weight, mix up a very small amount of epoxy (e.g.: 1-1/4 grams of resin to 1/4 gram of hardener on the scale).
3.3.2.5 Stir the epoxy with a glass stir rod for approximately 30 seconds or until the resin and hardener are thoroughly combined.
3.3.2.6 Be careful not to stir the epoxy too vigorously or you will create bubbles.
3.3.2.7 For hand mounting, continue with step 3.3.3. To use the mounting press, skip to step 3.3.4.

3.3.3 Hand Mounting

3.3.3.1 Blow off the slide and sample with the airblower.
3.3.3.2 With the sample I.D. facing down, set the sample on the low temperature hot plate.
3.3.3.3 Put a thin line of epoxy down the center of the rock chip (on the exposed and flattened side) with the glass stir rod.
3.3.3.4 Set the glass slide on top of the rock chip.
3.3.3.5 Press the eraser-end of a pencil onto the glass to work out all of the air bubbles. Note: Pressing out too much of the epoxy will result in a poor mount.
3.3.3.6 Turn the sample and slide over (be careful not to let the sample slip off of the glass slide) and set them on the hot plate (150°F, 65°C) for 30 minutes (or until the epoxy has cured). Use a pick to test the epoxy on the sides of the slide to check its hardness.
3.3.3.7 Repeat steps 3.3.3.1 through 3.3.3.6 for the remaining samples.
3.3.3.8 Hand mounting is now complete. After the epoxy has cured, skip to step 3.4 for labeling and trimming.

3.4 LABELING AND TRIMMING

3.4.1 Labeling

3.4.1.1 Remove all the excess epoxy from the bottom of the slide with a razor blade.
3.4.1.2 Only work with one sample at a time when labeling.
3.4.1.3 After the epoxy has cured, the samples are identified by transferring the number from the top of each sample to the back of each slide using an electric engraver. Use extreme care to transfer the sample ID correctly.
3.4.1.4 Always write the number near the edge of the slide in a semicircle fashion on round slides and straight on rectangular slides.

3.4.2 Trimming Using the Ingram Thin-Section Cutoff Saw

3.4.2.1 Turn on the vacuum by flipping on the toggle switch near your feet.
3.4.2.2 The vacuums of the Ingram Saw and the Ingram Grinder are connected by a Y-shaped hose. To use the Ingram Saw, the vacuum of the Ingram Grinder must be sealed off with a glass slide.
3.4.2.3 While pushing in the brass vacuum seal knob, set a slide with the rock chip mounted outwards over the vacuum holes. Now release the vacuum seal knob.
3.4.2.4 Watch the pressure gauge behind the saw. If it does not indicate 15 or more there is a leak in the seal and you will have to remove the slide and clean both the slide and vacuum chuck.
3.4.2.5 Slowly move the slide towards the saw with the handle.
3.4.2.6 The goal is to make the distance between the side of the saw blade and the side of the glass slide as small as possible without touching the two. To adjust the position, turn the knurled knob on the right side of the Ingram in a clockwise direction.
3.4.2.7 Now take note of the number on the dial indicator, also on the right side of the Ingram. Turn the knurled knob counter-clockwise back 15 thousanths from this original mark.
3.4.2.8 There are three toggle switches on the front of the Ingram and they are from left to right: Motor, Vacuum, Coolant. Turn on the Motor Switch.
3.4.2.9 Close the plastic covering over the saw.
3.4.2.10 Turn on the Coolant toggle switch on the front of the Ingram.
3.4.2.11 Use the handle to slowly push the slide back towards the saw.
3.4.2.12 As you are cutting, occasionally pull the saw away from the rock to keep the blade clean.
3.4.2.13 As you near complete cutting, apply less pressure to the rock to avoid a break in the chip.
3.4.2.14 When the rock has been cut all the way, bring the slide back to its original position, turn off the Coolant, and open the plastic covering.
3.4.2.15 Remove the slide by pushing in the brass vacuum seal knob.
3.4.2.16 Rinse the slide in alcohol and dry it with a clean wipe.
3.4.2.17 Continue this process for the remaining slides while being sure to check the pressure gauge each time.
3.4.2.18 When all the slides are complete, turn off the motor and coolant toggle switches, and turn off the vacuum.

3.4.2.19 Use the large tweezers to remove the rock samples from the Ingram. Rinse the rock samples in alcohol and place in a plastic bag with the scientist's name on the bag.

3.5 GRINDING AND LAPPING TO FINAL THICKNESS

3.5.1 Grinding on the Ingram Grinder

3.5.1.1 Turn on the vacuum by flipping on the toggle switch near your feet.

3.5.1.2 The vacuums of the Ingram Saw and the Ingram Grinder are connected by a Y-shaped hose. To use the Ingram Grinder, the vacuum of the Ingram Saw must be sealed-off with a glass slide.

3.5.1.3 While pushing in the brass vacuum seal knob, set your slide over the vacuum holes with the 15 thousanths thick sample pointing outwards. Now release the vacuum seal knob.

3.5.1.4 Watch the pressure gauge behind the saw. It should read between 15 and 20. If it does not, take your slide off and clean the slide and the vacuum holes. Also at this time make sure that the Ingram Saw is sealed off. Do not continue until the pressure gauge reads between 15 and 20.

3.5.1.5 Turn the big knurled knob on the front of the Ingram to the left until the slide completely clears the grinding wheel.

3.5.1.6 Once you are sure that the slide clears the grinder, bring the slide back to its original position. Now lower the plastic covering over the grinder and turn on the Coolant toggle switch. Make sure the oil is flowing before you continue with the next step.

3.5.1.7 There is a smaller silver knurled knob in front of the Ingram that allows you to feed the slide more slowly into the grinding wheel. From this point on, use this knob to advance the slide into the grinding wheel (never more than 1 thousanth at a time).

3.5.1.8 The goal here is to get the thickness of the slide down to about 5 thousanths of an inch. To do this you must check the slide constantly with the dial indicator to measure its thickness.

3.5.1.9 Petrographic experience is necessary in the final steps of grinding to assure proper thickness. You must look at the slide under the petrographic microscope to determine its thickness by its birefringence.

3.5.1.10 It is extremely important to be aware of the color changes that a particular crystal goes through as it is thinned.

3.5.1.11 Move the handle of the grinder back and forth as you are turning the silver knurled knob. Always be aware of the number on the dial indicator.

3.5.1.12 As you get closer to the final thickness on the grinder, you'll want to move the silver knurled knob only a 1/4 of a mark (thousanth) on the dial at a time so that you don't go too thin.

3.5.1.13 To remove your slide, bring the slide back to its original starting position, turn off the Coolant, and open the plastic covering. Release the vacuum seal and when the pressure gauge reads 5, remove your slide. Rinse the slide in alcohol and dry it with a clean wipe before measuring.

3.5.1.14 View the slide under the petrographic microscope to determine its thickness by birefringence.

3.5.1.15 Repeat steps 3.5.1.12 through 3.5.1.14 until the final grinding thickness is reached.

3.5.1.16 Follow steps 3.5.1.3 through 3.5.1.15 for the rest of the slides.

3.5.1.17 When all the slides are complete, turn off the motor and coolant toggle switches, and turn off the vacuum.

3.5.1.18 If Logitech LP-30 is to be used for lapping to final thickness, refer to Reference 5.1 Logitech Machine System Technology Manual.

3.5.2 Hand Lapping to Final Thickness

3.5.2.1 Pour a small amount of silicone carbide (600 grit) and kerosene on the "600" glass plate.

3.5.2.2 Move your sample around in the mixture in figure 8's and circles. circles. Be sure to check your sample frequently under the microscope to determine its thickness by its birefringence. (Rinse the sample off in alcohol before you check it under the microscope).

3.5.2.3 When your sample is almost to its final thinness, it will be time to work on the 1200 grit. At this point, refer to your worksheet to determine whether the sample needs to be stained, polished, or covered.

3.5.2.3.1 If it needs to be stained, go to step 3.6.

3.5.2.3.2 If it needs to be covered or polished, continue with step 3.5.2.4.

3.5.2.4 Pour a small amount of aluminum oxide (1200 grit) and kerosene on another glass plate.

3.5.2.5 Move your sample around in the mixture in figure 8's and circles. Be sure to check your sample frequently under the microscope to determine its thickness by its birefringence. (Rinse the sample off in alcohol before you check it under the microscope).

3.5.2.5.1 For a polished section, leave your sample relatively thick, 40-45 microns and go to step 3.6.4.

3.5.2.5.2 For a covered section, take your sample down to 30 microns and go to step 3.6.3.

3.6 SINGLE STAINING, DOUBLE STAINING, COVERING, AND POLISHING

At this point, refer to the worksheet to determine if single staining or double staining needs to be done. If single staining is requested, follow only subsection 3.6.1. If double staining is requested, follow both subsections 3.6.1 and 3.6.2. If no staining is to be done, go to step 3.6.3 for covered sections or step 3.6.4 for polished sections.

3.6.1 Staining--Potassium Feldspar Single Stain

3.6.1.1 Using a 30 or 40 micron smooth metal bonded diamond plate on the rotating lap wheel, grind the edges and corners of the glass slides until they are round.

3.6.1.2 Clean the sections in an ultrasonic cleaner and Isopropanol for 5 to 10 seconds. Caution: Do not leave sections in an ultrasonic bath longer than 30 seconds, as they may be severely damaged. Dry the sections.

3.6.1.3 Clean the following equipment: one pair of 12' forceps, one 40 ml beaker, two 600 ml beakers, one staining dish, and one rubber suction cup bent at 90°C.

3.6.1.4 Fill the 40 ml beaker with distilled water and add sodium-cobaltinitrite powder until the solution is saturated (there should be a thin layer of powder on the bottom of the beaker).

3.6.1.5 Carefully pour HF acid into the staining dish.

3.6.1.6 Fill one of the 600 ml beakers with tap water and the other 600 ml beaker with distilled water.

3.6.1.7 A rotational cycle is used when staining. In this example, three slides will be stained.

3.6.1.8 Take slide one and put it face down over the HF acid in the staining dish for 10 seconds. Use the suction cup to remove the slide and set it face up on a clean wipe.

3.6.1.9 Take slide two and put it face down over the HF acid in the staining dish for 10 seconds. Use the suction cup to remove the slide and set if face up on a clean wipe.

3.6.1.10 Take slide one with the forceps and submerge it in the sodium-cobaltinitrite solution for 10 seconds.

3.6.1.11 Using the forceps, agitate slide one in the solution and then remove the slide. Rinse slide off first in tap water and then in distilled water. Now hold slide one in one hand.

3.6.1.12 Take slide two with the forceps and submerge it in the sodium-cobaltinitrite solution for 10 seconds.

3.6.1.13 Take slide three and put it face down over the HF acid in the staining dish for 10 seconds.

3.6.1.14 Take slide one and blow it dry with the air blower. Set it on a clean wipe face up.

3.6.1.15 Use the suction cup to remove slide three from the staining dish and set it face up on a clean wipe.

3.6.1.16 Using the forceps, agitate slide two in the solution and then remove the slide. Rinse slide two in tap water first and then in the distilled water and hold it in one hand.

3.6.1.17 Take slide three with the forceps and submerge it in the solution for 10 seconds.

3.6.1.18 Take slide two and blow it dry with the air blower. Set it on a clean wipe face up.

3.6.1.19 Using the forceps, agitate slide three in the solution and then remove the slide. Rinse slide three first in the tap water and then in the distilled water and dry it with the air blower. Set slide three on a clean wipe face up.

3.6.1.20 At this point, if double staining has been requested, continue with subsection 3.6.2. If the sample is to be covered, skip to step 3.6.3.3.

3.6.1.21 If sample is not to be covered, place in a section box and log the sample as completed on the log sheet (Figure 3).

3.6.2 Staining--Plagioclase Double Stain

3.6.2.1 The example for double staining will only include one slide.

3.6.2.2 Clean the following equipment: one pair of 12' forceps, one 40 ml beaker, two 600 ml beakers, and two 150 ml beakers.

3.6.2.3 Pour a barium chloride solution in a 150 ml beaker.

3.6.2.4 Pour MeOH in a 150 ml beaker.

3.6.2.5 Fill two 600 ml beakers with distilled water.

3.6.2.6 Fill a 40 ml beaker with distilled water and add the Amaranth powder until the solution is saturated (there should be a thin layer of powder on the bottom of the beaker).

3.6.2.7 Take the slide and put it face down over the HF acid in the staining dish for approximately 5 seconds. Use the suction cup to remove the slide and set it face up on a clean wipe for 2 minutes.

3.6.2.8 Take the slide with the forceps and submerge it in the barium chloride solution for 5 seconds with agitation.

3.6.2.9 Using the forceps, remove the slide. Give the slide a couple of quick dips in the first beaker of distilled water. Drain the slide briefly by putting its edge against a clean wipe.

3.6.2.10 Take the slide with the forceps and submerge it in the Amaranth solution for 10 seconds with agitation.

3.6.2.11 Using the forceps, remove the slide. Give the slide a couple of quick dips in the second beaker of distilled water. Touch the slide against the beaker rim to drain, and quickly rinse the slide in the MeOH solution.

3.6.2.12 Drain the slide briefly by putting its edge against a clean wipe. check the results under a microscope.

3.6.2.13 Dry the slide with the air blower. If covering is requested, skip to step 3.6.3.3.

3.6.2.14 If sample is not to be covered, place in a section box and log the sample as completed on the log sheet (Figure 3).

3.6.3 Covering

3.6.3.1 Using a 30 or 40 micron smooth metal bonded diamond plate on the rotating lap wheel, grind the edges and corners of the glass slides until they are round.

3.6.3.2 Clean the sections in an ultrasonic cleaner and Isopropanol for 5 to 10 seconds. Caution: Do not leave sections in an ultrasonic bath longer than 30 seconds, as they may be severely damaged. Dry the sections.

3.6.3.3 Place the samples on the low temperature (150°F) hot plate, rock-side-up, for 15 minutes.

3.6.3.4 Place the back drop mounting plate on the high temperature (250°F) hot plate.

3.6.3.5 Mix up the Buehler epoxy (see step 3.3.2).

3.6.3.6 Take a sample from the hot plate and blow the dust off of it with the dust chaser.

3.6.3.7 Set the sample on the back drop mounting plate rock-side-up.

3.6.3.8 Using a glass stir rod, spread a line of epoxy over the center of the slide.

3.6.3.9 Take a cover slip (microscope cover glass) and using dust chaser, remove the dust from both sides.

3.6.3.10 Place a cover slip on the sample. Use the eraser-end of a pencil to remove the bubbles.

3.6.3.11 Place the sample on the low temperature hot plate and let it cure for 30 minutes.

3.6.3.12 Place covered samples in section boxes and log them as complete on the log sheet (Figure 3).

3.6.4 Polishing

3.6.4.1 Using a 30 or 40 micron smooth metal bonded diamond plate on the rotating lap wheel, grind the edges and corners of the glass slides until they are round.

3.6.4.2 Clean the sections in an ultrasonic cleaner and Isopropanol for 5 to 10 seconds. Caution: Do not leave sections in an ultrasonic bath longer than 30 seconds, as they may be severely damaged. Dry the sections.

3.6.4.3 Check the lap plates on the machines, and if the Texmets and/or silk are reusable, clean them with alcohol and clean wipe while the lap plate is rotating clockwise, and go to step 3.6.4.5. If they are not reusable, continue with step 3.6.4.4.

3.6.4.4 Prepare the lap plates as follows (assuming that usable plates aren't already on the machines):

3.6.4.4.1 Clean any old glue from the surface of each lap plate using toluene.

3.6.4.4.2 Apply Texmet or PAN-W to 2 of the 3 lap plates being careful not to allow bubbles to form under the cloth (these will be used for 6 and 3 micron). Check the numbers scribed on the plates; they should be 6 and 3 microns for the Texmet.

3.6.4.4.3 Apply the silk cloth to the 3rd lap plate (this will be used for 1 micron). Check the number scribed on the plate; it should be 1 micron for the silk cloth.

3.6.4.4.4 Place the lap plates on the machines.

3.6.4.5 Work with the 6 micron lap plate first. Squeeze out about a 1/2 an inch of 6 micron diamond paste onto your finger and dab it on the plate. Now rub in the paste on the plate. The lap plates are scribed as either 6, 3, or 1 microns. Be sure that you are using the proper plate with the proper diamond paste (i.e., 6 micron plate with 6 micron diamond paste).

3.6.4.6 Lightly spray the plate with 6 micron diamond spray.

3.6.4.7 Start the lapping machine and make sure that it is going counter-clockwise. Set variable speed knob to between 45% and 55%, this will give an rpm of between 125 and 150. Caution: Higher speeds may cause over heating of the thin section.

3.6.4.8 Fill the coolant bottle with Diamond Polishing Extender (mixture of propolenegycol and isopropanol) and open the drip feed knob until a drip rate of approximately 1 drop per second is obtained.

3.6.4.9 Clean each of the section holders with alcohol and a clean wipe, and place a drop of two of Extender on the section face of each (this will serve to hold the section in place as it is being placed in the auto fotating system). Place the slide in the holder so that the rock side is facing out.

3.6.4.10 Start the Pdm Force and one at a time, place the section holders into the five openings provided in the carier in the following manner:

3.6.4.10.1 Carefully turn the holder upside-down in one hand.

3.6.4.10.2 In the other hand, lift up the weights.

3.6.4.10.3 Place the inverted holder in the opening under the weights and slowly lower the weights on top of the holder (the rock face should be facing down so that it will be against the cloth lap).

3.6.4.10.4 Repeat steps 3.6.4.10.1 through 3.6.4.10.3 for the remaining samples.

3.6.4.11 Set the timer. The actual time used for polishing in each of the 3 steps (6, 3, and 1 microns) is totally dependent on the hardness and type of sample being polished. A general time guide is as follows:

3.6.4.11.1 Very hard samples: 30 to 40 minutes on 6 micron, 30 minutes on 3 micron, and 5 minutes on 1 micron.

3.6.4.11.2 Medium hard samples: 20 to 30 minutes on 6 micron, 15 to 20 minutes on 3 micron, and 3 to 4 minutes on 1 micron.

3.6.4.11.3 Soft samples: 10 to 15 minutes on 6 micron, 15 minutes on 3 micron, and 2 to 3 minutes on 1 micron.

3.6.4.11.4 Very soft samples: 10 minutes on 6 micron, 5 to 7 minutes on 3 micron, and 1 to 2 minutes on 1 micron.

3.6.4.12 The above times are only guidelines and the samples should be checked periodically during the polishing by the operator to determine when each step is complete.

3.6.4.13 When the 6 micron polishing is thought to be complete, clean the slide with alcohol and a clean wipe. Now, check the slide under the microscope paying particular attention to thickness and pitted crystals. Our goal for polishing is to have a clear, unpitted section.

3.6.4.14 Repeat steps 3.6.4.4 through 3.6.4.13 for 3 microns.

3.6.4.15 Now, depending on the thickness of the slide and the pits in the crystals, you need to decide one of three steps to take:

3.6.4.15.1 If the thickness is uneven and you still have pits, hand polish the sample using 3 micron diamond paste on 40# draft paper. Frequently check the slide for thickness and pits and when you are satisfied with its uniformity and clarity, skip to step 8.4.15.3 for 1 micron lapping.

3.6.4.15.2 If the sample is still fairly thick and there are a lot of pits, repeat steps 3.6.4.4 through 3.6.4.15 using 3 microns again.

3.6.4.15.3 If the sample is uniformly thin and all of the pits are gone, repeat steps 3.6.4.4 through 3.6.4.13 using 1 micron and the silk cloth. Never go more than five minutes on the lapping machine using 1 micron, regardless of the sample hardness. The sample must be pit-free before going to 1 micron. Lapping on a 1 micron plate removes the scratches from the 3 micron lapping, but it won't remove any pitting. It is the final buff.

3.6.4.15.4 Clean the samples.

3.6.4.15.5 Place the slides in a section box and log the work as complete on the log sheet (Figure 3).

3.7 Fluid Inclusion Slides

Fluid inclusion slides are prepared in the same manner as is described above, with the following exceptions.

3.7.1 A low temperature hot plate (130°F - 150°F) is used to dry the samples (step 3.1.11).

3.7.2 Follow steps 3.3.31 through 3.3.35 using Duro Super Glue. Super glue does not require heat to cure, so place the mounted samples on the counter to cure.

3.7.3 The final thickness of fluid inclusion slides is between 100 - 250 microns.

3.8 Grain Mounts

Grain mounts are prepared in the same manner as described above, with the following exceptions.

3.8.1 Write the sample ID in an aluminum dish and place on a hot plate (between 240°F and 260°F).

3.8.2 Mix a five to one ratio of epoxy to hardener and apply a small amount along the edge of a 1-in.-round ring mold. Place the mold in the aluminum dish with the epoxy coated end down. Allow 15 to 20 minutes for the epoxy to cure.

3.8.3 Pour the grains or loose sample material into the prenumbered mold until the mold is about 1/4 full.

3.8.4 Allow the material to dry for 15 to 20 minutes.

3.8.5 Mix a ten-to-one ratio of epoxy to hardener and pour just enough mixture into the mold to cover the material.

3.8.6 Move the samples to the low temperature hot plate (140°F - 170°F) and allow them to cure for about one hour.

3.8.7 Mix a ten-to-one ratio of epoxy to hardener and fill the mold to within 1/8 in. of the top. Allow to cure on the low temperature hot plate.

3.8.8 Using White-Out correction fluid, paint the top surface inside the mold, and with a lead pencil transfer the sample ID into the mold and coat with epoxy.

3.8.9 After the sample is cured, peel the mold from the aluminum dish.

3.8.10 Proceed with the standard operating procedure for making thin sections (TWS-ESS-DPO-04), beginning with the section on exposure and flattening of rock chips (Step 3.2).

4. QUALITY ASSURANCE

4.1 Personnel

Thin sections are made by qualified Los Alamos National Laboratory technicians. Documentation of training will be accomplished in accordance with QP-02.1.

On-the-job-training is necessary to learn thin section preparation. Thin section preparation personnel-in-training may perform operations using YMP samples. The only critical steps in this procedure, which will result in the eventual production of inaccurate data if improperly performed, are sample identification transfer. The importance of this step must be stressed to trainees.

4.2 Calibration

No calibration is necessary for operations outlined in this procedure. Commercial-grade calipers, dials, gauges, and thermometers are utilized to produce the highest quality thin sections possible.

4.3 Documentation

Sample preparation is documented on the thin section form which is retained in the files. Any procedural deviations from this procedure shall be noted on this form and/or in the investigator's notebook.

4.4 Responsibilities

The requester completes the thin section request form. The thin section lab supervisor is responsible to see that this procedure is followed correctly. This person is also responsible for the proper care and use of the equipment as well as to see that any necessary calibrations are performed and documented. The thin section lab supervisor may delegate these responsibilities to a YMP certified person.

4.5 Accept/Reject Criteria

The requester determines if the thin section is suitable for his/her end use.

4.6 Storage

When samples are not in use, they are to be stored in a locked cabinet. They are to be kept separate form all other samples. Upon completion of this procedure, the thin sections rock chips and other original sample material are to be returned to the investigator.

There are no storage requirements for the equipment used in this procedure.

4.7 Sample Traceability

Samples are tracked by use of the thin section logbook and thin section request form.

5. REFERENCES

5.1 Logitech Machine System's Technology Manual for Thin Rock Section Production, 20 p.

5.2 David Mann's Thin Section Manual.

6. ATTACHMENTS

Attachment 1 Thin Section Request Form.

THIN SECTION REQUEST FORM

NAME: _____ PRIORITY (check one)

PHONE: _____ GROUP: _____ _____ 1 DAY

DATE SUBMITTED: _____ _____ 3 DAYS

NUMBER OF SAMPLES: _____ _____ 2 WEEKS

PROGRAM CODE: _____ _____ SPACE AVAILABLE

NNWSI SAMPLE: _____

I. TYPE OF MOUNT (check one)

_____ Glass Rectangular _____ Glass Round _____ Epoxy Casting

_____ Silica Rectangular _____ Silica Round _____ Probe Standard

II. THICKNESS REQUIRED (check one)

_____ Unlimited _____ 150 μ _____ 30 μ

III. POLISHING REQUIRED (check one)

_____ None, _____ Polished on Bottom Side, _____ Polished on Top Side, _____ Polished on Both Sides

IV. SPECIAL INSTRUCTIONS

_____

_____

V. SAMPLE NUMBER

_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

SAMPLE LOG SHEET

Samples received in Sample Prep Lab _____ _____
Signature Date

Samples distributed to _____, _____ _____
Signature Date

NNWSI _____ QA Level _____