Standard operating procedure for preparation of grain mounts for SHRIMP analysis

Mineral Separation Laboratory

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Executive Summary

This Standard Operating Procedure (SOP) documents the process and methodology used by the Geoscience Australia (GA) Mineral Separation Laboratory to produce high quality epoxy resin grain mounts for analysis on a Sensitive High Resolution Ion Micro Probe (SHRIMP). All aspects of producing epoxy resin mounts for isotopic analysis are detailed (including the materials required, steps for sample preparation through to mount production, preparation for imaging of epoxy mounts, and mount cleaning procedures for gold coating and subsequent analysis), assuming that heavy mineral concentrates have previously been prepared via conventional density and magnetic separation techniques (e.g. Chisholm et al., 2014).

Making a mount is a precise and time consuming process and contamination risks are high. Procedures to mitigate the risks include thorough cleaning before and during the process, accurate sample labelling, and maintaining tidy, well organised workspaces.

Final mineral concentrates for analysis are achieved through handpicking individual grains using a stereo binocular microscope. Grains are placed in Petri dishes with ethanol, providing a liquid medium that emphasises variation between grains, and helps prevent grains flicking out of the dish during handpicking. Up to 500 grains are selected for mounting. Grain selection is determined by analytical requirements, which can vary greatly between samples. For example, detrital and igneous zircon samples are treated differently: the former are generally handpicked without bias to establish as representative a population spectrum as possible, whereas handpicking of the latter is typically biased towards 'best quality' (i.e. euhedral, transparent, and fracture- and inclusion-free grains) to determine a single magmatic age.

Grains are transferred from the Petri dish to a prepared mount plate using a disposable syringe; grains are placed on a razor blade and then transferred from the razor blade into a prepared row template. Once the grains are placed, the template is moved to the next row for the next sample. When all samples are mounted, epoxy resin is poured and the mount is left to cure. A standard SHRIMP mount at GA is manufactured to a diameter of 25 mm, whereas a ‘mega-mount’ has a diameter of 35 mm. The procedure for making standard mounts and mega-mounts is the same; only the sizes of the moulds and collets used in their manufacture are different.

Once cured, the back of the mount is cut (parallel to the face of the mount), producing a mount 6 mm thick. The face of the mount is polished with decreasing grades of abrasive to expose equatorial sections through the grains, remove scratches, and produce a flat surface for analysis. The polished mount is imaged via reflected light and transmitted light microscopy, cleaned, and coated with a 2nm thickness of gold for cathodoluminescence (CL) and/or back-scattered electron (BSE) imaging via scanning electron microscope (SEM). Following imaging, the mount is cleaned with solvents and ultrapure water to remove environmental lead and other surface contaminants, and dried. The mount is then coated with a 15 nm thickness of high-purity gold, and is ready for analysis.

This document provides a step-by-step guide to this mount preparation process. Each step is accompanied by a list of materials, a process breakdown, and explanations, images and cautions where necessary.
1 Introduction

1.1 Preamble

This document is to formalise a Standard Operating Procedure (SOP) for the process and methodology used by the Geoscience Australia (GA) Mineral Separation Laboratory to produce high quality epoxy resin grain mounts for analysis on a Sensitive High Resolution Ion Micro Probe (SHRIMP). This document was originally developed for ‘internal’ use as a laboratory guide, but has since been updated and modified for external publication, in response to significant interest from the international SHRIMP community in the detail of the mount preparation procedures used by GA.

The aim of this document is to detail procedures, equipment and consumables in order to make the GA methodology transparent, in the hope that other laboratories similarly tasked will find it useful, and to encourage those laboratories to publish their own versions of a similar document. Ideally, inter-laboratory exchange of information and ideas will foster innovation and overall process improvement as the best ideas and methods are combined.

This document presupposes the preparation of heavy mineral concentrates of the samples targeted for mounting, via conventional density and magnetic separation techniques. Such concentrates should be purified to the point where the target mineral(s) can be handpicked without that process being unduly laborious: the separation and purification procedures employed by GA were documented by Chisholm et al. (2014). From that point, this guide documents the remaining preparatory procedures, culminating in a conductively-coated SHRIMP mount ready for insertion into the ion probe.

Finally, it should be noted that the procedures are described primarily in the context of preparing samples of zircon (or other accessory minerals useful for U–Pb geochronology) for SHRIMP analysis. However, many of the these techniques are more broadly applicable to other micro-analytical methods where the target sample is amenable to polishing and conductive coating, and is capable of withstanding very high vacuums (1–10 × 10⁻⁸ torr). ‘Non-mineral’ materials prepared by GA using these procedures include strands of wire and hair (oriented both parallel and perpendicular to their cross-sections), as well as fragments of glass.

1.2 Materials

The following is an outline of the materials (consumables, tools and equipment) required for the production of SHRIMP Grain Mounts.

1.2.1 Consumables

- Block eraser, 60 × 20 × 12 mm (with 25 mm slot cut out of main face, used to protect mount surface while engraving back)
- Cylindrical polypropylene (PP) or polytetrafluoroethylene (PTFE) moulds (25 mm diameter for standard mounts; 35 mm for mega mounts)
- Compressed-air/carbon dioxide (CO₂) duster and CO₂ canisters (16 g)
- Detergent, glassware and laboratory equipment (RBS35, 10% dilution in deionised water (DI H₂O))
- Detergent, general purpose (Dobatex, 5% dilution in DI H₂O)
• Diamond suspensions for polishing (Struers Dia-pro Largo 9 µm, Dia-pro Dac 3 µm, Dia-pro Nap 1 µm, Dia-pro Susp ¼ µm, Dia-pro Lubricant Green)
• Disposable pipette (5 mL)
• Disposable graduated mixing container (20–25 mL)
• Double sided tape – 48 mm wide (Dupont Kapton HPP-ST)
• Epoxy resin (WEST System 105 resin and 206 slow hardener)
• Felt-tipped pen (0.35 mm tip, black ink: e.g. Artline)
• Felt-tipped pen (0.35 mm tip, red ink: e.g. Artline)
• Glass beakers – Quartz (50 mL, qty. 4)
• Glass beaker (100 mL, qty. 1)
• Glass microscope slide (75 mm × 25 mm)
• Glass mount plate (70 mm × 70 mm × 4 mm)
• ‘Hollow-square’ template (acrylic, 70 mm × 70 mm, prepared as described in Section 4.2.2)
• Latex and/or nitrile gloves
• Lens tissues
• Lint-free disposable wipes for delicate tasks (e.g. Bemcot PS-2)
• Lint-free disposable wipes, general purpose (e.g. Kim-wipes)
• Lint-free cleaning cloth for benches
• Marker pen, semi-permanent (for Petri dishes)
• Paper points (#10, #40, #80; available through dental equipment suppliers)
• Petri dishes (glass, minimum qty. of 7)
• Polishing pads (Struers MD-Largo, MD-Dac, MD-Nap)
• Polyethylene cutting board (100 x 150 mm)
• Potassium iodide solution (8 g Potassium iodide, 3 g iodine and 8 ml H2O; Appendix A)
• Razor blade (single edge)
• Row-mask template material (X-film Montex DX-1)
• Safety glasses
• Squeeze bottle containing standard ethanol (99.5%) for cleaning
• Squeeze bottle containing absolute-grade ethanol (99.999%) for handpicking
• Squeeze bottle containing Isopropanol
• Squeeze bottle containing petroleum ether
• Squeeze bottle with DI H2O
• Syringes (1 mL without needle)
• Syringe tips (pipette tips, disposable)
• Wet/dry silicon carbide (SiC) paper (#240, #400, #800 & #2000 grit)

1.2.2 Tools

Figure 1.1 shows a typical toolkit in a layout tray. The numbered items are as follows:

1. Small Spatula
2. Scalpel (with no.11 blade)
3. Tweezers no.5 Biologie (Dumostar, nonmagnetic)
4. Tweezers no.4 Biologie (Dumostar, nonmagnetic)
5. Tweezers no.3 Biologie (Dumostar, nonmagnetic)
6. Needle probe tool
7. Flattening tool (optional)
8. Fine tip probe tool (tip should be 100 µm diameter and blunt at the end)
9. Handpicking tool (350 µm diameter wire, bent 3.5 mm from its end; see Step 3 and Figure 3.2)
10. Tweezers no.1
11. Syringe 1 mL
12. Paper points
13. Wire tool (for bubble removal) sharpened to <100 µm point
14. Eraser with cut out (used for supporting mount while engraving)
15. Razor blade and glass slide
16. Cryo-vial holder
17. Large tweezers

Additional tools (unlabelled in Figure 1.1, or not shown) include:

- Sample forceps (25 mm)
- Forceps (25 mm SEM Stub)
- Steel ruler (150 mm long)
- Layout tray (obtainable from dental suppliers)
- Curved forceps
- Scalpel blade, retracting
1.2.3 Equipment

- Automated polishing apparatus (Struers Tegra-Pol 15, Tegra-Force-1, Tegra-Doser-5)
- Digital weighing scales (range 0.01–30 g)
- Engraver pen fitted with small diamond tip
- High quality compressed air (air supply with attached coalescing filter and carbon filter, and with small nozzle attached to hose)
- Hot plate (capable of 40–50°C)
- Incubator oven (temperature range 20–70°C)
- Infrared thermometer
- Micro-lathe (Sherline) or Diamond saw
- Microscope, Binocular, incidental light, 5x and 10x objective
- Microscope, Petrographic with 2.5x and 10x objective, digital camera fitted
- Microscope, Stereo (10x ocular, 0.63 objective, minimum 8:1 zoom) with large work area stand (Figure 2.1) light source and fitted camera (optional)
- Milli-Q Ultrapure laboratory (Type 1) water filter system
- Scanning Electron Microscope (SEM)
- Small oven (Temperature range 35–70°C)
- Ultrasonic bath (low strength)
2 Work Area Preparation

2.1 Material

- Standard-grade ethanol
- Cleaning cloth
- A4 and A3 sheets of paper (clean)
- Tape to secure paper

2.2 Preparing the work area

1. Thoroughly clean the work area using standard-grade ethanol. Ensure the microscope and surrounding equipment are clear of foreign particles such as dust and mineral grains remaining from the previous mount to avoid cross-sample contamination.

2. Place the A4 sheet over the microscope base and the A3 sheet alongside the microscope as an area to place Petri dishes. Secure the sheets of paper with tape.

Keep the workspace clean, organised, and ensure that tools and equipment are within reach. Figure 2.1 shows a standard mount-making workstation set-up.

Figure 2.1 Standard mount making work space.
3 Sample Preparation

Step 3 details preparation for final handpicking and grain selection.

3.1 Material

- Petri dishes (one per sample)
- Marker pen, semi-permanent
- Squeeze bottle containing absolute-grade ethanol (99.999%) for handpicking (standard-grade ethanol is unsuitable because ethanol is hygroscopic, and moisture absorbed by ethanol adversely affects the surface of mineral grains immersed in it, by reducing epoxy adhesion)
- Compressed air duster
- Handpicking tool
- Previously prepared heavy mineral concentrates for all target samples
- Microscope, Stereo (10x ocular, 0.63 objective, minimum 8:1 zoom) with large work area stand (Figure 2.1), light source and fitted camera (optional)

3.2 Placing the sample in a Petri dish

1. Inspect each Petri dish for cleanliness. Replace any which are unclean or heavily scratched. Blow each dish out with compressed air.
2. Label each Petri dish with a sample number that corresponds to a sample to be mounted (top and bottom).
3. Place each mineral separate into the dry Petri dish with the corresponding sample number.
4. When ready to handpick, add absolute ethanol to half fill the target dish.
5. Using the handpicking tool, gather the mineral grains together in the centre of the dish.

3.3 Recording of sample and handpicked grains

Develop a complete record of the sample both pre- and post-handpicking, to accompany the composite images produced for the analyst (Step 11). Photograph the sample in the Petri dish using a stereo microscope with an attached camera (Figure 3.1, left). Completed handpicks will be imaged in the same manner (Figure 3.1, right).

3.4 Grain selection and storage

General criteria for grain selection should be determined in consultation with the analysts prior to handpicking, and will depend on the nature of the target mineral(s). In the case of zircon, grain selection is influenced by the sample rock type—igneous, metamorphic or sedimentary—and analytical requirements.
1. Determine the quantity of grains to be mounted per sample: depending on the size and number of grains in the final concentrate, up to 500 grains may fit in a 7 x 0.35 mm row. A handpicking tool made from 0.35 mm diameter wire, bent 3.5 mm from its tip (Figure 3.2) provides a gauge for how many grains will fit into a mount row, as a row is equal to the width of the wire and double the length from tip to bend.

2. Image the handpicked component of the sample (Figure 3.1. right).

3. Place Petri dishes with the handpicked samples and reference materials aside until ready to make the mount.

Note that ethanol is hygroscopic. Any absorbed moisture will affect mineral grains immersed in it, reducing epoxy adhesion, so the time mineral grains spend immersed in ethanol should be minimised. Concentrates (and reference materials) awaiting handpicking should be stored dry. Ethanol should only be added for the purpose of handpicking, and it should be drained immediately afterward.

Figure 3.1 Mineral concentrate for handpicking (left) and handpicked grains ready to mount (right).

Figure 3.2 Handpicking tool; close-up of end. The diameter of the wire is 0.35 mm, and the distance from the tip of the wire to the bend is 3.5 mm.
When handpicking zircon concentrates, grain selection utilises the optical qualities of the grains, such as crystal integrity, grain clarity, cracks, crazing, inclusions (gas bubbles, other minerals), staining, transparency, metamictisation, and core–rim relationships. For example; if inheritance characterisation is required in an igneous or metamorphic sample, it may be necessary to select grains that are not best quality and/or appear detrital (pitted, crazed etc.) and/or have obvious cores. Conversely, sedimentary samples usually require as little bias as possible in grain selection.

For igneous samples, best quality grains are selected to determine a magmatic age unless advised otherwise by the analyst. Grains should be euhedral, transparent, free from cracks, inclusions and pitting, and have clean surfaces. Grains containing cores may be selected depending on analytical requirements; metamorphic grain selection takes grain clarity and crystal integrity into consideration in conjunction with cores and rims, as metamorphic grains are frequently selected to examine these relationships.

Sedimentary samples may contain grains from numerous sources of various ages. Therefore, a random assortment of detrital grains should be selected ensuring all populations present in the sample are captured, unless advised otherwise by the analyst. During the mineral separation process using the Frantz magnetic barrier separator, it is essential that multiple populations of zircons are not being biased and, therefore, being stripped in the increasing Frantz magnetic fractions. This is done by noting the different populations (colour, morphology, etc.) in the whole sample prior to magnetic separation and ensuring that one particular population is not being stripped away through multiple varying magnetic strength fractions; therefore biasing the sample. In this way multiple populations are being retained. The same holds for when handpicking; grains that are too heavily included are discarded, but the rest of the concentrate is subjected random selection in an effort to best represent all population variances of the concentrate. The purpose of random selection is not to bias the grain size distribution or population(s).

3.5 Planning the mount

Once samples have been handpicked and are ready for mounting, a mount plan is developed to design the layout of the mount and determine the number of required rows (Figure 3.3).

The ‘optimal analytical area’ on a standard (25 mm diameter) SHRIMP mount lies within a circle of radius 5 mm in the centre of the mount face. This region is exploited by mounting samples in rows 7 mm long by 0.35 mm wide, with 1 mm spacing between centres of rows; this allows for a total of eight rows within the optimal analytical area. Typically, two central rows are dedicated to reference materials, leaving up to six rows per mount for ‘unknown’ samples.

At least one row (often one containing multiple reference materials) should be designed with deliberate ‘west-to-east’ asymmetry in plan view, to ensure samples are identifiable relative to each other; if all rows are designed symmetrically, it can be difficult to distinguish ‘north’ from ‘south’ on the mount-plan, which may result in the mount being misoriented when it is loaded into the ion probe. For example, in Figure 3.3 the row containing reference materials (OG1, FC1, QGNG, 91500 and M127) is broken into numerous ‘part-rows’ and individual grains, with four full rows above and three full rows below. Asymmetry can also be generated using samples in cases where there are not enough grains for a full row.
Figure 3.3 Mount plan. Note that this map shows the grain-rows as placed on the tape surface and viewed from above. The mount surface corresponds to this layout viewed from below, in which case ‘west’ and ‘east’ are reversed when ‘north’ and ‘south’ are matched.
4 Mount Plate Preparation

Step 4 details the preparation of the mount plate and mounting tape.

4.1 Material
- Mount plate (glass, 70 × 70 × 4 mm)
- ‘Hollow-square’ template (acrylic, 70 × 70 mm, prepared as described in Section 4.2.2)
- Double-sided tape – 48 mm (Dupont Kapton HPP-ST: Silicon Polyimide or similar)
- Scalpel (with a sharpened blade)
- Felt-tipped pen (0.35 mm tip, black ink: e.g. Artline)
- Steel ruler (150 mm)
- Petri dish lid
- Microscope, Stereo (10x ocular, 0.63 objective, minimum 8:1 zoom) with large work area stand (Figure 2.1), light source and fitted camera (optional)

4.2 Preparing the mount plate

The mount plate is a 70 × 70 × 4 mm glass plate used as a base for making the mount. Accurate preparation, including tape alignment and adhesive contact across the base, is crucial for centring the mounted grains within the mould and avoiding undulations on the mount surface.

For SHRIMP analysis, the rows of grains should not extend beyond a circle of radius 5 mm in the centre of the mount. This distance allows for a maximum of eight rows of 7 mm long spaced approximately 1 mm apart from the centre line (Figure 3.3).

To ensure correct alignment of the tape on the mount plate, etch alignment marks on the back of the mount plate: horizontal and vertical centre-lines defining a cross hair, with four tick-marks on this cross hair corresponding to a distance of 12.5 mm from the centre in each case (Figure 4.1). Draw additional horizontal lines parallel to (and spaced 24 mm) from the horizontal centre-line, and on each of these additional horizontal lines, add two tick-marks each spaced 24 mm from the vertical centre-line; this allows for the alignment of the tape on the plate. Additionally, the cross hairs allow for the alignment of the acrylic template and subsequent alignment of the mould.

4.2.1 Preparing the mount plate with double-sided tape

1. Clean the mount plate surface well with standard-grade ethanol and a lint-free cloth or wipe.
2. Blow the surface well using compressed air to remove any lint remnants.
3. Cut a piece of double sided tape and lay it on the glass (without removing the coversheet of the tape), using the etched lines as a guide to centre the tape. While laying down the tape, use a flat spreader (such as the back of a razor blade) to smooth down the tape, removing any bubbles. It is important that no air bubbles are trapped under the tape as this will affect the mount surface resulting in an undulating surface or a ‘crater-and-dome’ effect. Trim the tape with a razor blade, exposing the marks on the sides of the mount plate (Figure 4.3).
4. Inspect the mount plate under a microscope to see if there are any air bubbles or foreign particles trapped between the tape and the glass. If they are present, remove the tape and repeat the procedure.

5. Mark the mount ID number on the mount plate with a marker pen.

![Mount plate with alignment marks](image)

*Figure 4.1 Mount plate with alignment marks 12.5 mm and 24 mm from the centre, for the mount mould and the double-sided tape, respectively.*

### 4.2.2 Cutting out mount surface and preparing row markers

This step describes how to cut out the inner area of the double sided tape and the creation of the markers that will be used to align the row mask template to the corresponding rows.

An acrylic template is employed to cut out a surface on the double sided tape, and to cut out the row mask template. It consists of a piece of acrylic 2.5 mm thick, cut to a square with outer edge length 70 mm, but with a concentric square (with inner edge length 40 mm) excised from the inside. Included are marked central crosshairs and notches on the inside edge, corresponding to the crosshairs (Figure 4.2).

1. Place the acrylic template on top of the tape coversheet on the mount plate.
2. Align the crosshairs of the acrylic template to the crosshairs on the mount plate and using the scalpel mark the four cross hair marker notches that are on the inside cut out of the acrylic template.
3. Cut out the 40 mm centre square of the acrylic template, cutting as cleanly as possible into the coversheet of the double-sided tape as this will be removed from the tape in Step 5.4.
4. Remove the acrylic template and set aside in a clean area for Step 5.2. Using the black 0.35 mm Artline marker pen mark over the ‘north’ and ‘south’ cuts made with the scalpel. Ensure these marks continue on the vertical axis across the outer square (Figure 4.2). The total number of sample + reference material rows to be mounted (determined in Step 3.5) will determine how the ‘east’ and ‘west’ scalpel-cuts are treated.
5. Using the steel ruler (and under the stereo microscope required for accuracy), select one vertical side of the hollow square (e.g. the one containing the ‘west’ scalpel-cut), and consider the total number of rows to be mounted. If it is an odd number, place the steel ruler so that the ‘west’ scalpel-cut is aligned with an integer millimetre-mark, and make an equal number of scalpel-cuts at 1 mm intervals along the edge of the hollow square, using the integer millimetre-marks either side of the ‘west’ scalpel cut (for example, if there are seven rows to be mounted, the ‘west’ scalpel-cut will be used as the marker for row 4, with three rows above and three below). If the total number of rows to be mounted is an even number, place the steel ruler so that the ‘west’ scalpel-cut is aligned with a half-millimetre mark, and make an equal number of scalpel-cuts at 1 mm intervals along the edge of the hollow square, using the integer millimetre-marks either side of the ‘west’ scalpel-cut (for example, if there are eight rows to be mounted, rows 4 and 5 will each be 0.5 mm distant from the ‘west’ scalpel-cut, with additional rows at 1 mm intervals above and below).

6. Draw over the row markers using the marker pen to make them more visible; the ink should bleed into the cut to highlight the marks (Figure 4.3). Remember that the central ‘west’ and ‘east’ scalpel cuts are not row-markers if the total number of rows to be mounted is an even number.

7. Repeat steps 6 and 7 on the opposite vertical side of the hollow square (i.e. the one containing the ‘east’ scalpel-cut) so that there are matching alignment marks on both sides.

8. Leave the cut-out coversheet in place on the double-sided tape and cover the template with a clean Petri dish lid to protect from dust and fibres while preparing the row mask template in Step 5.

There are two essential factors to consider in the selection of double-sided tape.

Firstly, the glue layer of the tape must be consistent in thickness and the tackiness of the glue must be neither too adhesive nor lacking sufficient adhesive. The best tape will allow the grains to be fixed lightly on top of the tape. If a glue layer is too thick, the grains will sink in too far and not present enough surface area for epoxy adhesion; possibly resulting in them ‘plucking-out’ when removing the finished mould from the tape. Conversely, tape with insufficient glue will result in grains not fixing properly to the tape, causing them lift off the tape during epoxy pouring.

Secondly, many types of tape can out-gas when introduced to the epoxy, producing bubbles on the mount surface which can disrupt analysis, especially if the bubbles are trapped around the mineral grains. It is prudent to do extensive research on various types of tape until one is found that performs well and monitor the performance of the tape over time. Typically, rolls of tape dedicated to the preparation of ion-probe mounts are not rapidly consumed, so it is important to monitor tape surfaces for long-term degradation and/or the development of dry patches. Tape life can be extended by keeping it in a sealed plastic bag, away from sun light. The tape used at GA is Dupont Kapton HPP-ST, a double-sided Silicon Polyimide tape.
Figure 4.2 ‘Hollow square’ acrylic template.

Figure 4.3 Mount plate with 48 mm wide double sided tape in place, showing horizontal alignment marks at 1 mm intervals either side of the ‘west’ and ‘east’ cross hairs. Note that because there is an even number of rows to be mounted (eight), the horizontal centre line does not define a row-marker: instead, the marks for rows 4 and 5 are evenly spaced 0.5 mm either side of the horizontal centre line.
5 Row Mask Template Preparation and Transfer

Step 5 describes preparation of the ‘row mask template’ (which is used to align the rows of grains on the mount face), and the transfer of the row mask to the mount plate with the mounting tape prepared in Step 5.4.

5.1 Material

- ‘Hollow-square’ template (acrylic, 70 × 70 mm, prepared as described in Section 4.2.2)
- Cutting board (Figure 5.1)
- Row-mask template material (X-film Montex DX-1)
- Scalpel (with a sharpened blade)
- Felt-tipped pen (0.35 mm tip, black ink: e.g. Artline)
- Felt-tipped pen (0.35 mm tip, red ink: e.g. Artline)
- Steel ruler (150 mm)
- Microscope, Stereo (10x ocular, 0.63 objective, minimum 8:1 zoom) with large work area stand (Figure 2.1), light source and fitted camera (optional)
- Petri dish lid
- Lint-free disposable wipes, general purpose (e.g. Kim-wipes)
- Tweezers

5.2 Preparing a scalpel with a sharpened blade

The row mask template used is made from a solid polypropylene plastic cover sheet supplied on a double sided tape media (which is discarded after the mask is made). Scalpel blades straight from the packet are not sharp enough to cut it without ripping and creating fibres that can get caught on the tape.

Make a sharpening tool by adhering #2000 grit wet/dry silicon carbide paper to a spare mount plate with double sided tape. This can be used under a microscope to produce a sharp edge on the scalpel blade. Hone the blade several times across the paper until a well sharpened edge is observed under the microscope; usually 10 – 20 passes per side are sufficient.

5.3 Preparing the row mask template

As mentioned above, the row mask template is made from the fibre-free solid polypropylene plastic cover sheet on some types of double-sided tape. The tape itself is not part of the template: it simply provides a convenient substrate for template preparation.

Alternative possibilities for row mask template materials include the backing paper from double-sided tape, or good quality baking paper: however, both are inferior as they produce fibres as the material degrades through use, requiring removal of fibres from the mount. If these media are used, line markings are made in pencil, as ink may bleed through the fibres.
1. Cut a piece of the masking material approximately 50 x 50 mm square.
2. Using the ruler and the red 0.35 mm Artline marker pen, measure and mark the vertical and horizontal centre lines on the cover sheet.
3. Place the mask material on the cutting board under the microscope.
4. Using the ruler and the black 0.35 mm Artline marker pen, mark a horizontal line 7 mm long, coincident with the red horizontal centre-line, and centred on the red vertical centre-line. Using the well-sharpened scalpel and the ruler, cut out the marked black line, so that the excised area measures 7 x 0.35 mm (Figure 5.2).
5. Use the handpicking tool to gauge the 0.35 mm width of the cut out. Extend the cuts defining the shorter sides of the rectangle to create flaps. Note: the extended flap enables the mask to be placed in rows below previously mounted grains, allowing some flex of the masking material.
6. Overlay the acrylic template on the masking material, aligning the crosshair marks. Cut out the central square of the acrylic template.
7. Cover the cut-out with a clean Petri dish lid and place the cutting board aside in a clean area.

The cover layer with the cut-out 7 x 3.5 mm is the row mask for positioning the rows of grains. The next stage is to transfer the row mask to the mount plate.

*Figure 5.1 Acrylic template and cutting board.*
5.4 Transferring the row mask template to the mount plate

The horizontal red centre-line on the row mask template prepared above will be aligned to the various pairs of horizontal row-markers on the mount plate template prepared in Step 4 (Figure 5.2). Transferring the row mask template is done as follows:

1. Remove the Petri dish lid from the mount plate and place in a clean area.
2. Using tweezers, remove the coversheet from the double-sided tape on the mount plate. Ensure that the tape adhesive is clean and fibre free.
3. Cover the now-exposed tape adhesive with the clean Petri dish lid and set aside.
4. Remove the Petri dish lid from the cutting board, and place the lid in a clean area.
5. Using tweezers carefully separate the polypropylene row mask template from the adhesive media of the masking material, avoiding creases and ripping of the sheet. Place the template on the cutting board under the microscope.
6. Use a small piece of the lint free wipe, soaked in ethanol and, holding it in tweezers, gently swab the area around the cut out on both sides of the template. This step is to remove any traces of ink from the template cut out (which can bleed onto the tape surface).
7. Place the cutting board aside in a clean area.
8. Ensure the row mask template is clean (a gentle flick to the side will achieve this, or alternatively use the compressed air duster).
9. While holding the row mask template, retrieve the mount plate and remove its Petri dish lid.
10. Align the vertical centre-line on the row mask template with the vertical centre-line on the mount plate and align the horizontal centre-line of the row mask template with the 'northernmost' pair of...
horizontal row-markers on the mount plate. Use the microscope to check that both alignments are accurate (Figure 5.3).

11. Use a flattening tool to ensure that the template is firmly adhered to the tape, particularly around the cut out area. By adjusting the microscope light source wands to be more parallel to the glass plate, the tape/template contact will be more visible.

12. Cover the mount plate with the clean Petri dish lid and set aside.

The mount plate is now ready for the first row of sample grains to be laid (Figure 5.4). Laying grains on the mount tape is described in Step 6. The process of moving the row mask after completing one row and preparing to lay the next row is described in Step 6.3.

Figure 5.3 Row mask template (left, defined by red ink) aligned with horizontal row-marks defined by scalpel cuts in the doubled-sided tape on the mount plate.
Figure 5.4 Completed mount plate with row mask template in place, ready for the laying of the first (northernmost) row of grains.
6 Sample Transfer and Mounting

Step 6 is in three parts, and each part must be repeated for each sample placed on the mount.

Step 6.1 describes the preparation of sample grains for mounting in a row, transferring the grains from the Petri dish to a razor blade, in preparation for mounting. Step 6.2 describes the transfer of the grains from the razor blade to the row mask on the mount plate for mounting the sample. Step 6.3 describes moving the row mask to mount the next row of grains.

6.1 Preparing grains for transfer to the row mask template

6.1.1 Material

- Syringes (1 mL, one per sample)
- Syringe tips (pipette, one per sample)
- Paper Points (#10, #40, #80)
- Razor Blade (It is recommended that two marks 7 mm apart be made on the edge of the razor blade: this mitigates the risk of overloading the edge of the razor blade with target grains, as all grains placed between the two marks will fit within the confines of the row mask template when transferred to the tape.)
- Glass microscope slide
- Handpicking tool
- Tweezers no.1
- Compressed air duster
- Kim-wipes
- Squeeze bottle containing standard ethanol (99.5%) for cleaning
- Microscope, Stereo (10x ocular, 0.63 objective, minimum 8:1 zoom) with large work area stand (Figure 2.1), light source and fitted camera (optional)

6.1.2 Transferring grains from Petri dish to razor blade

1. Prepare a pre-marked razor blade and a glass microscope slide for the grains to be laid on:
   a. Using a Kim-wipe saturated with ethanol, wipe over both the razor blade and microscope slide.
   b. Blow both with the air duster to remove lint.
   c. Lay the razor blade perpendicular to the microscope slide long axis. Use a small ball of Blu-Tack on the back of the razor blade to keep it from moving around on the microscope slide. (The purpose of the glass slide is purely to enable easy movement of the razor blade)
   d. Place the slide and razor blade beside the microscope base.
2. Take the Petri dish with the first sample to be mounted (according to the plan established at Step 3) and gather the grains to be mounted into a pile with the handpicking tool.
3. Place the Petri dish under the microscope and focus on the mineral grains at a low magnification.
4. Affix a new pipette tip to a new syringe (1 mL).
5. Use the syringe to carefully suck the mineral grains into the pipette tip. Avoid filling the syringe body; only draw up the required grains, and as little ethanol as possible.

6. Lift the syringe from the Petri dish gently and slowly, so that grains do not spill from the pipette tip (quick or violent movements increase the risk of spillage). Hold the syringe directly over the dish.

7. While holding the syringe still, remove the Petri dish with the other hand and place the microscope slide with razor blade directly under the syringe.

8. Carefully place the end of the pipette tip onto the razor blade between the row markers and very gently lay the grains onto the surface (Figure 6.1). Grains should flow out of the pipette tip with the ethanol. Inspect the pipette tip to ensure that all grains have been transferred.

9. Using the handpicking tool, carefully align the grains to the edge of the razor blade, attempting to create a row in between the marks at 7 mm spacing (Figure 6.2). Keep the line of grains as narrow as possible, approximately 350 µm (0.35 mm) wide (use the diameter of the handpicking tool wire as a guide; another good guide is the very edge of the blade). Once this is completed, the grains should be neatly aligned on the edge of the razor blade as shown in Figure 6.3. Do not push the grains and ethanol over the edge of the razor blade: grains will spill onto the microscope slide and are difficult to retrieve.

10. Absorb excess ethanol using a paper point held with tweezers (Figure 6.4); avoid touching grains with the paper point as they will adhere. Depending on the amount of ethanol to be absorbed, use a #80 point (large), #40 (medium) or #10 (small) paper point.

11. Allow any remaining ethanol to evaporate completely from the surface. Observe this process under the microscope.

12. Discard pipette tip.

The grains are now ready to be laid onto the double sided tape. The procedure for mounting the grains is described in Step 6.2.
Figure 6.1 Placement of grains on razor blade using a pipette.

Figure 6.2 Arranging grains on the edge of the razor blade, using the handpicking tool. Note the ‘row width’ marks etched onto the razor blade 7 mm apart (near the left and right edges of the photo), oriented perpendicular to the blade edge.
Figure 6.3 Final alignments of grains on blade edge.

Figure 6.4 Absorption of excess ethanol with paper point.
6.2 Mounting grains on the tape

Step 6.2 describes the process of mounting the grains; i.e. transferring grains from the razor blade onto the mount plate, placing grains into the row mask and adhering the grains to the tape.

There are two methods for laying mineral grains within the row mask. The first (6.2.2) involves bulk placement, with all grains placed in the row mask at once. The second (6.2.3) involves single grain placement where each grain is positioned individually within the row mask. The bulk placement method has the advantage of laying multiple grains quickly, but can result in grains overlaying each other, and is best suited for rounded detrital grains. The single grain placement method has the advantage of being able to place individual grains with ample space between grains, however, it takes more time to manipulate each grain. Both methods are detailed below.

Ensure that all tools are cleaned and inspected for grains that might have adhered to them, both before use and between samples.

6.2.1 Material
- Handpicking tool
- Tweezers no.5 Biologie
- Probe tool
- Microscope, Stereo (10x ocular, 0.63 objective, minimum 8:1 zoom) with large work area stand (see Figure 2.1), light source and fitted camera (optional)
- CO₂ duster

6.2.2 Method 1—Bulk placement of grains

1. Bring the prepared mount plate close to the microscope slide/razor blade.
2. Remove the Petri dish lid from the mount plate and place aside in a clean area.
3. Carefully lift the razor blade off the microscope slide and move the mount plate beneath the lifted razor blade, being careful to not disturb the grains on the razor blade.
4. Gently lay the razor blade onto the mount plate, aligning the edge to the cut-out row mask in the template (Figure 6.5).
5. Refocus the microscope so that the image is clear.
6. Bring the razor blade to the very edge of the row mask.
7. Keeping the length of the sharp edge of the razor blade in contact with the row mask template, lift the opposite edge until the plane of the blade is tilted to an angle of 60–70°.
8. Use the handpicking tool to carefully move the grains from the edge of the blade onto the tape surface in the row mask (Figure 6.6).
9. When all grains have been transferred onto the tape, use the handpicking tool (or fine tip probe tool) to gently touch the grains, ensuring that they are well fastened to the tape but not pushed in. Avoid excessive manipulation and rolling of the grains as this will cause the glue to adhere to the sides of the grains, resulting in ‘moating’ when the epoxy is poured.
10. Grains that fall outside the template outline will need to be placed manually using the fine tip probe tool or tweezers (no.5 Biologie) as at Method 2 (6.2.3).
11. Inspect the row to ensure that grains are not stacked or overlapping as this will cause problems when the epoxy is poured, such as the entrapment of air bubbles between grains.
12. Inspect the entire template and mount plate for stray grains. Individual grains that fell from the razor blade onto the polypropylene template can be picked up with the probe and placed within the row mask.

13. Move the template for the next row, as described in Step 6.3.

Figure 6.5 Pre-placement of grains into row adjacent to row mask.

Figure 6.6 Transferring grains from razor blade into row mask using the handpicking tool.
6.2.3 Method 2—Individual grain placement

The ability to lay grains individually can increase the analytical capability of the material, and inevitably produces a better mount. Although individual grain placement is time consuming, benefits include better grain spacing, the option of placing two (or more) low-yielding samples in a single row, and optimised grain orientation (e.g. by crystallographic long axis or by fracture surface) to maximise the area of the equatorial section produced during polishing.

Individual grain placement is a useful technique for mounting reference materials, which are usually too valuable to waste by mounting more than is needed for analysis, and which must not take up mount-space that might be needed for unknown samples. Such considerations may require the mounting of up to three or four different reference materials in a single row.

It is possible to pick up an individual grain using tweezers; however, it may flick out, and a friable grain can be crushed. A better option is to use a (blunted) needle-probe, which provides more control. Utilising ‘static cling’, a grain can be held on the end of the probe, and placed on the tape (Figure 6.7). The size of the probe pictured here has been shaped to be 100 \( \mu \text{m} \) at the tip and blunted. Avoid using a sharp point needle, as grains tend to ride up the side of the probe, which makes placement very difficult.

As shown in Figure 6.7, it is best to adhere the grain to the tip of the probe during transfer. Sometimes, a grain simply will not stick to the probe: in this situation, the stickiness of the probe tip can be enhanced either by a quick touch to either the side of some tape (which will deposit a small amount of glue), or alternatively the side of your nose: the oil will help the grains to stick.

Grains designated for individual placement can be picked either directly from a dry Petri dish, or from the edge of a razor blade using the system described in Step 6.1.2 and Step 6.2.2 (up to item 6), with the exception that if the grains are being picked directly from a dry Petri dish, they can be placed onto the polypropylene surface adjacent to the row mask. Grains can then be picked individually from the razor blade or mask surface, and placed in the row mask.

To pick grains directly from a Petri dish (useful for reference materials or low-yield samples):

1. Place the Petri dish under the microscope and carefully lift a grain with the probe or tweezers. Keep the probe hand steady as you lift it above the dish.
2. With the other hand, slide the dish backwards, place the glass slide under the microscope, and refocus the microscope.
3. Carefully lower the grain to the intended location and place it onto the tape as shown in Figure 6.7 and Figure 6.8; all movements should be slow and gentle as erratic movement will cause the grain to drop from the probe. Avoid manipulating or rolling the grains once they are on the tape.
4. Continue this process until all grains in the row have been positioned.
5. Move the template for the next row, as described in Step 6.3.
6.3 Moving the row mask for the next row

Step 6.3 describes moving the row mask template when one row is completed and the next row is ready to be placed.

1. When all grains in a row are positioned inspect the entire template and mount plate for any stray grains.
2. Stray grains can be picked up with the probe or tweezers and placed on the tape within the row mask.
3. Remove the row mask template with tweezers by gently lifting vertically from a corner while observing under the microscope; ensure that no grains are loosened near the row mask edges in the process.
4. Inspect the grains on the tape for any that may have moved outside the row mask area.
5. Cover the mount plate with a clean Petri dish.
6. Move the row mask template away from the mounting area.
7. Use the CO$_2$ duster to blow the template thoroughly to remove any missed grains or fibres.

8. Remove Petri dish. Align the template on the next set of horizontal markers, covering the previous row/s (Figure 5.3). Check the positioning of the template under the microscope. As described in item 11 of Step 5.4, use the light wands of the microscope to check adherence of mask to the tape and its alignment with other rows. Often, the template mask will not be correctly aligned to the corresponding marks so it is prudent to check the correct placement. Continue through Steps 6.1, 6.2 (using either or both Methods 1 and 2) and 6.3 for each row until all rows have been mounted.
7 Preparing the Mount Plate for Epoxy

Step 7 describes the preparation of the mount plate and mounted grains for the pouring of epoxy in Step 8.

7.1 Material

- Tweezers no.4 Biologie
- Tweezers no.5 Biologie
- Flattening tool
- Lens tissue
- 25 mm diameter or 35 mm diameter mount mould (with a 10 mm mark on the outside of the mould measured perpendicular to the mould face. This mark is to define the depth to which the epoxy should be poured.)
- Microscope, Stereo (10x ocular, 0.63 objective, minimum 8:1 zoom) with large work area stand (Figure 2.1), light source and fitted camera (optional)

7.2 Preparing the mount plate

Once all of the rows have been placed on the tape, the mount plate is prepared for pouring the epoxy. Remove the row mask template from the mount plate and fully inspect the mount surface. Ensure that all the grains are within their rows. (If grains are regularly found outside their rows, it is possible that the row mask template has not been held firmly against the tape on the lower edge of the row.) Remove any stray grains with tweezers, and discard them as an anti-contamination precaution.

Wipe the tweezers between each grain removal with a piece of lens tissue and inspect the tip to ensure that no grains or fibres are stuck to the end before touching the tape surface again. Lint and fibres may fall onto the mount tape despite this. These will cause surface imperfections on the epoxy and must be removed before the epoxy can be poured. It can be difficult to see the fibres and lint on the tape surface; an easy way to make them visible is to use only one of the light source wands situated so it is almost parallel to the surface, illuminating the fibres. Remove each fibre with a pair of tweezers (Figure 7.1) wiping the tweezers each time with the lens tissue. The glue may lift when a fibre is removed, causing a surface defect; these should be repaired wherever possible, using a flattening tool (an angled probe tool that has been flattened on the bottom and rounded on the tip) to press the glue back down to a smooth surface (Figure 7.2).

Once all stray grains and fibres have been removed and affected glue flattened, ensure the epoxy mould has a mark at a height of 10 mm above the mould base (measured parallel to the cylindrical axis of the mould). Place the epoxy mould on the tape (Figure 7.3), centring the mould on the mounted rows by using the 12.5 mm marks on the horizontal and vertical centre lines etched on the glass plate (as shown in Figure 4.3). Press the mould firmly against the tape. Place a clean Petri dish lid over the top of the mould to prevent any further dust or fibres settling on the tape while the epoxy is being prepared.
Figure 7.1 Removing a fibre from the tape surface using tweezers.

Figure 7.2 Using a flattening tool to repair the tape surface after the fibre has been removed.
Figure 7.3 Completed mount plate with mould placed, ready for epoxy to be poured.
8 Preparing Epoxy and Pouring the Mount

Step 8 details the process of preparing the epoxy for pouring and the method used to pour the epoxy into the mount mould without creating bubbles.

Correct epoxy selection is determined by multiple factors: hardness, shrinkage, resilience to high vacuum and resistance to an electron beam.

- Hardness: Epoxy hardness is specified as ‘Shore D Hardness’ which refers to a surface test on fully cured epoxy with a durometer. It is supplied by manufacturers as a numerical value. It is essential that the epoxy used has a maximum possible shore hardness value which facilitates the polishing of the mount surface.
- Shrinkage: It is essential that the epoxy used has minimal shrinkage as this will affect the contact of the epoxy to mineral grains encapsulated within.
- Resilience to high vacuum: It is imperative that the epoxy used can withstand ultra-high vacuum analytical instruments such as the SHRIMP (1–10 x 10⁻⁸ torr). This means that the epoxy does not continue to outgas or expand when exposed to ultra-high vacuum.
- Resistance to electron beam: It is necessary that the epoxy used is stable under an electron beam generated by scanning electron microscopes. Some epoxies may readily ‘raster’ under an electron beam, thereby damaging the surface directly around mineral grains which can cause analytical problems.

8.1 Material

- Small spatula
- Disposable pipette (5 mL)
- Hot plate
- Disposable graduated mixing container (20–25 mL)
- Epoxy resin and hardener
- Digital scale
- Incubator oven
- Small oven
- Infrared thermometer
- Wire tool (for bubble removal) sharpened to <100 µm point
- Curved forceps
- Microscope, Stereo (10x ocular, 0.63 objective, minimum 8:1 zoom) with large work area stand (Figure 2.1), light source and fitted camera (optional)
8.2 Preparing the epoxy

To achieve a satisfactory epoxy composition with a good shore hardness rating and a uniform cure throughout, the hardener and epoxy should be measured out in a ratio of 1:5; where 1 part hardener is added to 5 parts resin. The best method to ensure that the correct ratio is achieved is to measure the epoxy and hardener by cumulative weight: the procedure is documented below, and an example given in Table 8.1.

- Place an empty mixing container on a scale and record its weight (W_c).
- Add epoxy to the cup, ensuring there is at least 12 g of epoxy for each batch. This can be done by either adding the epoxy to a mixing container whilst on a balance or by filling to approximately the 10–15 mL mark (if present) on the side of the container. A smaller amount will not be sufficient to achieve the correct hardener/epoxy ratio and fill the mould to the required level. (Note that 12–15 g of epoxy is sufficient to fill either a 25 mm-diameter or a 35 mm-diameter mould.)
- Place the container with epoxy back on the scale, and record the total weight of the container and resin (W_{c+e}). Subtract the weight of the cup (W_c) from this figure to determine the net weight of the resin (W_e).
- Divide the net weight of the resin by 5 to determine the net weight of hardener you will need (W_h = W_e / 5).
- Determine the target cumulative weight of the container, epoxy and hardener (W_{c+e+h} = W_c + W_e + W_h). This is the reading on the scale that must be achieved in order to combine the epoxy and hardener in the correct proportions. (Do not add the hardener to the container at this stage! The epoxy must be properly warmed first: see below.)

Table 8.1 Calculations for 2.5 g mixing cup with 15 g of epoxy poured in

<table>
<thead>
<tr>
<th>Step</th>
<th>Calculation</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Weigh an empty mixing cup:</td>
<td>W_c = 2.5 g</td>
</tr>
<tr>
<td>2</td>
<td>Add some epoxy to cup. Total weight of mixing cup + epoxy:</td>
<td>W_{c+e} = 17.5 g</td>
</tr>
<tr>
<td>3</td>
<td>Subtract cup weight from total weight to determine net weight of epoxy:</td>
<td>W_{c+e} - W_c = W_e = 17.5 g - 2.5 g = 15.0 g of epoxy</td>
</tr>
<tr>
<td>4</td>
<td>Divide net weight of epoxy by 5 to determine net weight of hardener needed:</td>
<td>W_e / 5 = W_h = 3.0 g of hardener</td>
</tr>
<tr>
<td>5</td>
<td>Determine the target cumulative weight of the cup, epoxy and hardener:</td>
<td>W_c + W_e + W_h = W_{c+e+h} = 2.5 g + 15.0 g + 3.0 g = 20.5 g</td>
</tr>
</tbody>
</table>

Before the hardener is added, the epoxy must be warmed, as epoxy at room temperature is too viscous to pour evenly over the grains: typically, air bubbles become trapped between the grains. Note that the process of warming the epoxy, adding the required weight of hardener, mixing the epoxy and hardener and pouring the mount typically takes about 90 minutes, and must not be interrupted. Ensure that the adequate time is set aside for this procedure before commencing it.

Cover the cup containing the epoxy to prevent dust and fibres entering the resin. Place the container on a hotplate set at 40–45°C for at least one hour. The correct viscosity is achieved when the epoxy drips from a spatula quickly and in even droplets, like water, and does not adhere to the spatula or form elongated droplets.

When ready to mix the hardener into the epoxy, place the prepared mount plate on the hotplate so that it can warm up to the temperature of the epoxy. Once the resin has reached the correct
temperature (40–45°C), place the cup containing the resin back on the scale, and carefully add hardener (using a disposable 5 mL pipette) until the target cumulative weight is achieved, taking care not to ‘over-add’ hardener by more than 0.1–0.2 g, as this will dramatically shorten the ‘pot life’ of the epoxy (i.e. the time before reaction starts and the mixture hardens).

Use the spatula to mix the epoxy and hardener for 4 minutes, keeping the mixing container in contact with the hotplate. Ensure that the epoxy is mixed evenly and relatively slowly (approximately one to two circles per second), as mixing too fast will generate air bubbles within the mixture. Once the epoxy and hardener is thoroughly mixed, it is ready to pour. Be aware that the ‘pot life’ of the epoxy is greatly diminished when it is warm. There is a 5–8 minute window before the epoxy is unusable, so be ready to pour once mixing is complete.

8.3 Pouring the epoxy

Epoxy cures through an exothermic reaction, pouring too much resin risks spiking the curing temperature above 100°C, causing the epoxy to boil and allowing air to be sucked in under the mould ring at the tape surface. The result will be mount surface bubbles which will affect the polishing process. For this reason the resin is poured into the mould to a depth of 10 mm only.

1. Place the glass mount plate under the microscope and remove the Petri dish cover.
2. Tilt the mount plate at a slight angle, approximately 10–15 degrees towards the side from which the epoxy is being poured.
3. Slowly pour the epoxy from the side and simultaneously lower the mount plate slowly. This will allow the resin to flow over the grains evenly. Pour only enough for the resin to make it to the other side of the mould in a thin layer (~ 2 mm). Do not allow the resin to flood into the mould, but do not pour too little. The ‘rolling front’ of the resin (Figure 8.1) should flow evenly and smoothly, infilling the spaces between the grains through capillary action.
4. Inspect the resin under the microscope for air bubbles in the vicinity of the grains, or along the inside edge of the mould where it is attached to the tape, or any fibres that may have been entrapped. A fine wire sharpened to a point (less than 100 µm diameter) can be used to ‘pluck’ bubbles away: insert the tool into the epoxy, and very carefully nudge the bubble away from the grains (or the edge of the mould). The bubble will then rise upwards, away from the mount surface. Sometimes fibres can be trapped in the resin; these can be removed with curved forceps.
5. Pour the remaining resin into the mould until a 10 mm depth has been achieved, using the pre-marked line on the outside of the mould as guidance. Once again, check that there are no air bubbles or fibres trapped in the resin. Remove them at this time, within the pot life of the resin.
8.4 Curing the epoxy

When no trapped bubbles remain in the resin, place the mount plate on the side of a metal sink basin with some cold water beneath it as a heat-sink. (Alternatively, the mount plate can be placed in a shallow tray with water filled to a depth of 2–3 mm). As mentioned above, the reaction responsible for resin curing is exothermic, and it is vital that the temperature does not rise too high. Providing a heat-sink assists in maintaining an even resin temperature as it cures.

The temperature of the resin can be monitored during curing using an infra-red thermometer. Epoxy setting temperatures of 60–70°C are normal. If the temperature continues to rise, increase the size of the heat-sink (e.g. using a shallow tray containing 2–3 mm of cold water). Let stand until the resin hardens, which usually takes about one hour. When the epoxy temperature drops to 25–30°C, remove the mount plate from the sink, dry the base, and place it in an oven set at 60°C for 12–16 hours to begin curing.

After the mount plate has been in the oven for 12–16 hours, remove it and let it cool to room temperature. Once cool, remove the mount mould and epoxy puck from the mount plate by twisting the mould and epoxy cast while holding the mount plate still. To facilitate easy removal of the mount, lightly warm the mount plate on a hotplate for a few minutes to soften the glue, being careful not to overheat the mount to the point where the twisting motion can distort the puck.

Remove the tape from the glass plate. Clean the mount plate with ethanol and set it aside. Clean excess adhesive off the mount surface by dabbing it with another piece of tape until all traces of glue have been removed. (Using Silicon Polyimide tape does not leave a residue on the mount surface). Slide the mount out of the mould, place the mount on the cleaned mount plate with the surface containing the grains facing upwards, and return the mount and mount plate to a 60°C oven for a further eight hours to fully cure. After removing the mount from the oven, let it stand for another 24 hours. Do not attempt to trim or polish until the resin has had at least 48 hours in total to cure; this is to ensure that the resin has achieved its full shore hardness.

Figure 8.1 Pouring epoxy into mould (note the rolling front of epoxy from left to right).
9 Trimming and Labelling the Mount

Step 9 explains the process of trimming the mount using a lathe, and labelling the mount with the mount name and sample numbers. This process only details the preparation of 25 mm diameter mounts.

9.1 Material

- Lathe with vacuum extraction (or Diamond trim saw)
- Engraving pen
- Block eraser, 60 x 20 x 12 mm (with 25 mm slot cut out of main face, used to protect mount surface while engraving back)
- Microscope, Stereo (10x ocular, 0.63 objective, minimum 8:1 zoom) with large work area stand (Figure 2.1), light source and fitted camera (optional)
- Wet/dry SiC paper (#240, #400 & #800 grit)
- Sample forceps (25 mm)

9.2 Preparation before trimming the mount

Once the mount has fully cured, use the pen engraver to mark the ID number of the mount on the side of the resin cylinder. Make sure the text is confined to the area within 6 mm of the grain-bearing surface, as this will be the final thickness of the puck.

Making the back and face of a mount as parallel as possible maximises the probability of the mount sitting ‘flush’ in the steel mount-holder used in the SHRIMP. Either a diamond trim saw or a lathe (Figure 9.1) can be used to trim the mount to the correct thickness. The latter is preferred at GA as it achieves better parallelism of the mount back and face. Furthermore, the lathe can be used to create a light bevel on the edge of the mount face, which helps position the mount properly in the SHRIMP mount holder.

To enable the mount to be held precisely in the lathe, a pot collet is employed. The collet is machined with an internal diameter of 25 mm and an internal depth of 4.7 mm (Figure 9.2). (A custom made holder is used for 35 mm mounts to be trimmed in the lathe; this process is not described in this document, but the technique is the same.) Fitted to the lathe is a tungsten carbide tip instead of a standard high speed steel tip (Figure 9.3). The tungsten carbide tip permits multi-directional cutting of the surface, and the edge of the tip can be used for bevelling. Figure 9.4 shows the basic layout of a lathe, and defines the X and Y axes.

Multiple cutting steps are employed to cut the mount to the desired thickness (Figure 9.5). These steps are outlined below.
Figure 9.1 High precision mini lathe used to trim mounts.

Figure 9.2 Left: Pot collet, machined with an internal diameter of 25 mm and an internal depth of 4.7 mm. Right: SHRIMP mount (back side) after being trimmed on the lathe; note the circular striations from the tungsten carbide tip.
Figure 9.3 Tungsten carbide lathe tip used to trim mounts.

Figure 9.4 Diagrammatic representation of a typical small lathe.
Figure 9.5 Diagram depicting mount cut steps (lines depicted as cuts may not be actual number of cuts required).
9.3 Trimming the mount with the lathe

9.3.1 Remove the mount face edge flaring to fit the pot collet (Step 1)

Rub the back of the mount on #240 wet/dry paper to remove the worst of the uneven meniscus.

Fit the mount into the collet with the grain-bearing surface (mount face) facing outwards, and tighten the collet. The mount will not sit perfectly as the back of the mount will still be slightly uneven, but it should be seated as best it can be (Figure 9.6, right). Turn on the vacuum extraction to the lathe. Turn on the lathe.

This purpose of this step is to remove the small flaring from the front of the mount created by the mould to tape interface. If this flaring is not removed, then the face of the mount will not fit into the collet.

- Set the RPM to 3600.
- Advance the Y axis in until the cutting tip point is past the edge of the mount, presenting the side of the tip to the edge of the mount face.
- Using the side of the tip, advance the X axis in, and trim the edge back to a minor bevel not advancing more than 0.5 mm from first contact (Figure 9.6, left).
- Retract the X axis out, and retract the Y axis out.

Turn off the lathe. Remove the mount from the collet for stage 9.3.2.

Figure 9.6 Mount inserted into collet showing flaring (left) and flaring removed and bevelling edge (right).
9.3.2 Remove the meniscus and machine the mount back parallel (Step 2)

Refit the mount to the collet so the grain-bearing surface is facing inwards and the grain surface is flush with the base of the collet. Tighten the collet, and turn on the lathe.

- Advance the Y axis, presenting the tip to the edge of the meniscus; advance the Y axis 0.3–0.5 mm. Remove the material by winding the X axis in until all available material is cut; then wind the X axis out to the original starting position.
- After each cut, advance the Y axis 0.3–0.5 mm. (Attempting to cut more than 0.6 mm in a single cut may result in the mount flicking out of the collet and being damaged.)
- Continue cutting across the X axis until the tool tip cuts across the full radius of the mount back. Take note of the cutting tip when it reaches the centre of the mount back (this will become apparent when the last bit of material being cut is removed), re-zero the X axis digital readout at this point, and then withdraw the X axis. Advance the Y axis by another 0.2–0.3 mm and perform one final cut to the mount again.

By the end of Step 9.3.2, the back of the mount should be parallel to the mount face, but the total thickness of resin removed should not have exceeded about 1.5 mm (Figure 9.7).

Turn off the lathe. Remove the mount from the collet for stage 9.3.3.

Figure 9.7 Removing meniscus from back of mount and paralleling surface.

9.3.3 Complete the edge bevel on the mount face (Step 3)

This is a repeat of Step 9.3.1 where a light bevel was created on the edge of the mount face, however, the mount face and back are now parallel and an accurate bevel on the mount face can be produced.

- Fit the mount into the collet with the grain-bearing surface (mount face) facing outwards, and tighten the collet.
- Turn on the lathe.
• Advance the Y axis in until the cutting tip point is past the edge of the mount, presenting the side of the tip to the edge of the mount face.

• Using the side of the tip, advance the X axis in, and trim the edge back to a minor bevel not advancing more than 0.5 mm from first contact (Figure 9.6).

• Retract the X axis out, and retract the Y axis out.

Turn off the lathe. Remove the mount from the collect for stage 9.3.4.

9.3.4 Cut the mount to the preferred thickness (Step 4)

GA’s preferred thickness for a SHRIMP mount is 6 mm. This preference is due to multiple factors. If mounts are less than 6 mm thick, they are susceptible to warping, and they also present problems for the polishing apparatus. Mounts less than 5 mm thick are capable of ‘slipping’ under the mount holder of the polishing machine. In addition, the travel of the feet of the polishing machine extends no closer than 5.5 mm from the surface of the polishing pad, and unless a spacer is used (Figure 10.3), the force applied to the mount during polishing will be insufficient. Conversely, mounts must not be made too thick. As mentioned at Step 8.3, the epoxy can only be poured to a maximum depth of 10 mm as excess epoxy will create too much heat during the curing process. Furthermore, mounts that are significantly thicker than 6 mm will not fit into the mount holder of the Scanning Electron Microscope, and can also be problematic in the mount holder of the SHRIMP.

With these considerations in mind, each mount is cut to a thickness of 6.1 ± 0.1 mm (with the additional 0.1 mm removed at Step 9.4). As mentioned earlier, the pot collet used at GA has an internal depth of 4.7 mm, so to achieve the preferred thickness of 6 mm; a mount thickness of 1.4 mm must remain outside the collet once cutting is completed.

Insert the mount into the collet with the grain surface facing inwards and tighten the collet. With the lathe off:

• Advance the Y axis to the shoulder of the collet until the tip of the blade just touches and zero the digital readout for the Y axis.

• Retract Y and X axis out.

Turn on the lathe.

• Advance the Y axis until the cutting tip just touches the mount. The Y axis position on the digital readout should read significantly more than 1.4 mm (>3.0 mm). Retract the Y axis away from the mount surface.

• Retract the X axis beyond the edge of the mount and then advance the Y axis by 0.4–0.5 mm.

• Advance in the X axis in and remove material, until the 0 position (on the X axis readout) is reached. Then retract X axis out beyond the mount edge.

• Advance the Y axis another 0.4–0.5 mm.

Repeat the previous four steps until 2.4 mm remains on the Y axis (as per digital readout of the Y axis). These cuts can be done quickly.

• Advance the Y axis 0.5 mm to a value of 1.9 mm and slowly advance the X-axis in to cut to the centre of the mount, just passing the 0 position (on the X-axis readout) to -0.05 mm. Then withdraw the X axis completely. The purpose of this step is to remove a small amount of material before the final cut, because the previous quick cuts of the epoxy material induce a degree of
micro-fracturing, and these micro-fractures will be visible in the finished surface if this step is ignored.

To make the final cut:

- Advance the Y axis to 1.42–1.43 mm. Advance the X axis slowly until past the 0 position of the mount and then slowly draw it out to edge of the mount.
- Retract out both the Y and X axis.
- Turn off the lathe at motor switch.
- Clean lathe with vacuum.

Turn off the lathe at the wall socket. Turn off the vacuum extraction. Remove the mount from the collet with a pair of 25 mm diameter sample forceps.

9.4 Finishing the mount back

Finishing the mount back by grinding the surface with progressive grades of silicon carbide papers (#240, #400 and #800 grit) removes the remaining 0.1 mm from the lathing process to produce a mount thickness of 6 mm. The aim is to produce a lightly sanded finish rather than a reflective polished surface. This surface will allow transmitted light to diffuse through the mount evenly for light microscopy imaging unlike a polished surface which will cause a flare in the imaging.

Adhering the silicon carbide papers to a glass (or steel) plate with double-sided tape prevents buckling of the papers during rubbing and provides a flat, true surface.

Using the #240 grit paper, rub the back of the mount in a figure-eight motion until all prominent lathe marks are removed. This process usually takes only 30–40 seconds. Continue with the #400 grit paper to remove prominent marks generated by the #240 grit, and finish with the #800 grit paper to achieve a lightly scratched surface.

9.5 Labelling the mount

Place the mount under the microscope, and engrave the sample numbers contained in the mount along the side edge. If some sample numbers share a common prefix, then inscribe the first sample number complete, followed by the last digits that alter for the remaining samples (e.g. 2132573, 574, 575 and 2134621, 622, 627).

Place the mount face-down on the eraser, which will protect the mount face while the mount-back is labelled. Keeping the mount face-down, rotate the mount until the ‘northernmost’ of the grain-rows (as defined by the mount plan; Figure 3.3) is in the northernmost position. Taking care to stay outside the analytical area (defined by the extents of the grain-rows on the mount face), mark the back of the mount with its ID number in normal lettering across the top, and in reverse lettering across the bottom (Figure 9.8). To one side of the grain-rows, inscribe a directional arrow indicating ‘north’ (Figure 9.8), as defined by the mount plan. Ensure that none of the inscriptions on the mount-back encroach on the analytical area (defined by the extents of the grain-rows on the mount face) as having the inscriptions in the field of view of transmitted-light images of the zircons will cause distortion.

The mount is now trimmed to the correct thickness, marked with the appropriate identifications and is ready for polishing.
Figure 9.8 Labelled mount.
10 Polishing the Mount

The mount must be polished to achieve the best possible optical surface (with minimal surface relief) if high-quality SHRIMP analyses are to be obtained. Analytical problems arising from poor mount surfaces include low sensitivity, fractionation of the secondary ion beam, and poor surface conductivity.

The aim is to polish grains to a depth that exposes an equatorial section. In practice, this cannot be achieved for all grains on the mount owing to varied diameters within and between samples; therefore, the depth of polishing is usually minimised, in order to expose equatorial sections of the smallest grains. Step 10 details the process involved in polishing the mount and cleaning the polishing equipment.

10.1 Material

- Struers polishing equipment (or similar)
- Polishing pads
- Diamond suspension (9, 3, 1 & ¼ µm)
- Microscope, Binocular, incidental light, 5x and 10x objective
- Detergent, general purpose (Dobatex, 5% dilution in DI H₂O)
- Squeeze bottle with DI H₂O
- Squeeze bottle with DP Lubricant Green
- Mount spacer discs (qty. 4) (Figure 10.3)

10.2 Polishing equipment, and key points to remember

The polishing equipment used in the GA Mineral Separation Laboratory is a Struers polishing system that consists of a TegraPol-15, TegraForce-1 and TegraDoser-5 (Figure 10.1), with a range of polishing pads and compatible diamond suspensions. The specific pads and suspensions are listed in Table 10.1 with predefined polishing program. The programs have been formulated through trial and error at GA to achieve the best polishing results. The program depicted in Table 10.1 defines the program used for zircon grain polishing; other minerals or media may require minute changes in the polishing times but the sequential polishing abrasives will remain the same.

Key points to remember when polishing:

1. Always use latex gloves when handling the pads. Touching the pads directly with fingers will deposit oil on them.
2. Inspect pads regularly for wear or damage and replace them if necessary.
3. Always mark the bottom of the polishing pad for the designated media to be used (e.g. 9 µm) and the date of replacement.
4. Always ensure the pad is matched to the suspension media (this should be written on the back of the disc as at 10.2).
5. Take care not to cross contaminate the pads with the wrong diamond media as this will make the pad unusable.

6. Ensure that the method step defined by the polishing program is matched to the pad designated for that step.

7. Ensure that the sample holder of the polishing apparatus (Figure 10.3) does not cross the centre of the pad (at least 15 mm from centre).

8. Rinse the pads thoroughly under fast running water after each use, without rubbing them.

9. Thoroughly clean the sample holder after each step to remove all media from the previous step.

10. Clean the sample with detergent/water mix and running water after each polishing step.

11. Examine the sample under the microscope after each polishing step.

Figure 10.1 Polishing equipment with 25 mm-diameter mount holder plate fitted. There are different plates for mounts of different diameters.
Table 10.1 Programmed polishing methods by steps, Zircon.

<table>
<thead>
<tr>
<th>Step</th>
<th>Surface</th>
<th>Suspension</th>
<th>Dose</th>
<th>Time</th>
<th>Force</th>
<th>Disc/Sample</th>
<th>RPM</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>User Type (MD-Largo)</td>
<td>DiaPro 9 mm</td>
<td>5_5</td>
<td>1 min</td>
<td>10N</td>
<td>100/100</td>
<td></td>
<td>Hand polishing</td>
</tr>
<tr>
<td>2</td>
<td>MD-Largo</td>
<td>DiaPro 9 mm</td>
<td>7_8</td>
<td>4 min</td>
<td>30N</td>
<td>100/100</td>
<td></td>
<td>Planar grinding</td>
</tr>
<tr>
<td>3</td>
<td>MD-Dac</td>
<td>DiaPro 3 mm</td>
<td>5_7</td>
<td>2 min</td>
<td>10N</td>
<td>100/100</td>
<td></td>
<td>Fine polishing</td>
</tr>
<tr>
<td>4</td>
<td>MD-Dac</td>
<td>DiaPro 1 mm</td>
<td>7_8</td>
<td>30 sec</td>
<td>10N</td>
<td>100/100</td>
<td></td>
<td>Fine polishing</td>
</tr>
<tr>
<td>5</td>
<td>MD-Nap</td>
<td>DP-Susp ½ mm</td>
<td>8_10</td>
<td>2 min</td>
<td>10N</td>
<td>100/100</td>
<td></td>
<td>Final buff</td>
</tr>
<tr>
<td></td>
<td></td>
<td>DP-Lubr Green</td>
<td>8_10</td>
<td>45 sec</td>
<td>10N</td>
<td>100/100</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

10.3 Polishing procedures

Do not over-polish the mount. The objective of planar grinding (Step 10.3.2) is to remove surface material and establish the flatness of the surface; fine polishing #1 and #2 (Steps 10.3.3 and 10.3.4 respectively) removes the defects created from planar grinding. Although scratches may remain from planar grinding, repeating fine polishing results in excessive relief on the mount that can cause analytical issues (Figure 10.2). Relief is defined as a varying height profile between the mounted grains and the mounting media and also as the rounding or doming of the grain surface. The nature of the Nap pad will also lend itself to creation of sample relief. The principle for polishing is ‘less is better’.

![Figure 10.2 Excessive relief due to over-polishing. The surfaces of the mineral grains are domed, rather than flat and even the edges of the domes stand proud from the low-lying epoxy surface.](image)
10.3.1 Hand grinding

The polishing process begins with hand grinding, which involves only nominal pressure and is aimed at removing gross surface relief from the completely unpolished grain-surfaces that were originally in contact with the double-sided tape. ‘Smoothing down’ the grains by removing sharp edges and gross surface irregularities during an initial, low-pressure polish helps prevents grains from being plucked out of the mount surface, especially during subsequent steps where force is applied to the mount.

A blank epoxy disc is used as a ‘decoy’ disc in the Tegra-Force head holder while the mount is hand-polished on the pad.

   a. Switch on the machine (located on left hand side at back of machine).
   b. Select the correct programmed method for zircon—Ensure that Step 1 method is highlighted on the polisher screen.
   c. Place a MD-Largo pad designated for 9 µm diamond suspension upon the disc. The disc is magnetic and the pad will adhere to it; ensure that it is centred.
   d. Engage the Tegra-Force head.
   e. Place a blank epoxy disc into the sample holder.
   f. Start the polisher.
   g. Using DP-Lubricant-Green in a squeeze bottle, lightly spray some onto the pad as the doser is delivering the diamond suspension to pre-lubricate the pad.
   h. When the disc achieves the higher RPM (the machine spins the disc at a lower RPM whilst the diamond suspension is being added to the disc, and then speeds up to the pre-set RPM value), begin hand-polishing the mount on the disc, away from the sample holder. Use only very light pressure, and polish the mount in a circular (counter clockwise) motion, occasionally rotating the mount on its axis. Do not cross the centre of the pad; stay at least 15 mm away. This method provides relative uniform surface removal.
   i. Clean the mount with detergent and water, and inspect it under the microscope in order to verify how much material is removed and condition of the surface (even material removed across the whole surface). Leave the MD-Largo pad on for Step 10.3.2.

10.3.2 Planar grinding

Planar grinding is aimed at polishing the grains to their equators and removing any moats that are present. Bear in mind that when there are multiple samples mounted, the grain size average will vary from one sample to the next. Always polish to the smallest grain size.

   a. From 10.3.1, keep the Tegra-Force head down and remove the blank disc.
   b. Insert the mount into the holder, grains side to the pad, and use a backing epoxy disc as a spacer. Spacers can be made using a 30 mm diameter epoxy blank, machined flat on one side with a 25 mm diameter cut out recessed to 1 mm on the other side to place the mount in (Figure 10.3). Using a spacer ensures that the feet of the polisher do not extend too far, keeping even pressure upon the mount. It is prudent to have several spacers prepared for each diamond media.
   c. Ensure the ‘Step 2’ method is highlighted on the polisher.
   d. Start the machine and allow it to run for 30 seconds.
   e. Stop the machine (red button) and lift the Tegra-Force head.
   f. Remove the mount and rinse it with a detergent/water mix under fast running water.
g. Blow the surface of the mount with compressed air.

h. Inspect the mount surface under the microscope in reflected light. The surface should show large scratches (from the diamond) and the polished depth of the grains to see if the equators of the grains have been reached (exposed surface of the grains in relation to the outline of grains sub-epoxy).

i. If the equators of the smallest grains have not been reached, lower the Tegra-Force head, return the mount to the holder and run the polisher for another 30 seconds. Remove, clean and inspect. Repeat this process until the desired polishing depth has been reached. If the program times out and more planar grinding is required, back-step through the program menu to reselect ‘Step 2’.

j. When the desired polishing depth is obtained: clean the mount and spacer thoroughly to remove any residual media. Remove the pad and rinse well under fast running water. Place the pad back in the storage holder.

k. Clean the sample holder plate using water in a wash bottle and tissue papers; removing all traces of diamond media (using white tissues will aid this, as the media is grey).

![Figure 10.3 Mount spacer and mount (left) and spacer and mount fitted into polisher sample holder (right).](image)

### 10.3.3 Fine polishing #1

Fine polishing #1 is designed to remove the scratches arising from planar grinding.

a. Select the 3 µm MD-Dac pad (check that 3 µm is written on the back) and place the pad on the disc.

b. Lay a clean tissue on the disc under the Tegra-Force head to catch any dried media left from 10.3.2, preventing it from falling onto the pad. Lower the Tegra-Force head, and remove and discard the tissue.

c. Ensure the ‘Step 3’ method is highlighted on the polisher. If the previous program is selected, 9 µm diamond will be deposited on the disc, contaminating it: the disc will need to be discarded and replaced with a new one.
d. Insert the mount with the 3 µm spacer into the holder and start the machine. Allow the process to time out.
e. Remove the mount, clean it thoroughly with detergent mix and running water; and blow dry with compressed air.
f. Inspect the mount under the microscope; the surface should show minor scratches with some imperfections removed.
g. Remove the pad and rinse well under fast running water. Place the pad back into the storage holder.
h. Thoroughly clean the sample holder plate as in Step 10.3.2 k.

10.3.4 Fine polishing #2

a. Select the 1 µm MD-Dac pad (check that is has 1 µm written on the back).
b. Lay a clean tissue on the disc under the Tegra-Force head to catch any dried media left from 10.3.2, preventing it from falling onto the pad. Lower the Tegra-Force head, and remove and discard the tissue.
c. Ensure that ‘Step 4’ method is highlighted on the polisher.
d. Insert the mount and 1 µm spacer into the holder and start the machine. Allow the process to time out.
e. Remove the mount, clean it thoroughly with detergent mix and running water; and blow dry with compressed air.
f. Inspect the mount under the microscope; the surface should show only minor scratches and minimal relief.
g. Remove the pad and rinse well under fast running water. Place the pad back into the storage holder.
h. Thoroughly clean the sample holder plate as in Step 10.3.2 k.

10.3.5 Final buff

a. Select the ¼ µm MD-Nap pad (check that ¼ µm is written on the back) and place the pad on the disc.
b. Lay a clean tissue on the disc under the Tegra-Force head to catch any dried media left from 10.3.2, preventing it from falling onto the pad. Lower the head, and remove and discard the tissue.
c. Ensure that ‘Step 5’ method is highlighted on the polisher.
d. Insert the mount and ¼ µm spacer into the holder and start the machine. Allow the process to time out.
e. Remove the sample and clean thoroughly. Do not use tissues as this will scratch the final surface. Rinse with ethanol and immediately dry with the CO₂ duster or high quality compressed air nozzle.
f. Inspect the mount under the microscope. The surface should be almost scratch-free, and it should have an optical finish (i.e. a flat surface characterised by a single focal plane when examined under a microscope).
g. Place the finished mount into a clean Petri dish in preparation for imaging.
h. Remove the pad and rinse well under fast running water. Place the pad back into the storage holder.

i. Thoroughly clean the sample holder plate as in Step 10.3.2 k.

j. Rinse the rotating disc with water from a wash bottle and wipe it dry with a paper towel.

k. Thoroughly rinse out the bowl of the polisher. Wipe down entire machine with paper towels.

l. Replace the cover over the disc and switch off the machine (switch on the back left corner of machine).
11 Imaging the Mount

Step 11 details the process for making a complete set of images for the mount. The mount is imaged in light microscopy in both incidental light (IL; also known as reflected light) with DIC (Differential Interference Contrast) and transmitted light (TL) and is then imaged using either cathodoluminescence (CL) or backscattered electron (BSE) detectors in a Scanning Electron Microscope (SEM). The images are compiled into a map to aid determination of analytical spot placement.

11.1 Material

- Delicate task wipes (Bemcot)
- Detergent, glassware and laboratory equipment (RBS35, 10% dilution in deionised water (DI H₂O))
- Squeeze bottle containing absolute-grade ethanol (99.999%)
- Air duster
- Mount press
- Glass slide
- Plasticine
- Microscope, Petrographic with 2.5x and 10x objective, digital camera fitted
- Carbon tape
- Scanning Electron Microscope

11.2 Imaging process

11.2.1 Preparation for light microscopy

Clean the mount:

1. Remove the mount from the Petri dish.
2. Thoroughly clean the mount in detergent mix (10% RBS35) using a wet Bemcot wipe to scrub the surface; do not use a dry cloth or tissue as this will cause surface scratches.
3. Rinse well with water and then rinse with absolute ethanol.
4. Dry the mount with high purity compressed air.

The mount is then prepared for placement on the microscope using a mount press, a glass slide and plasticine balls (Figure 11.1):

1. Place three small balls of the plasticine (approx. 2 mm in diameter each) in a triangular formation on glass slide.
2. Place the mount on the three balls, aligning the grain-rows parallel to the long edge of the slide.
3. Place slide onto the press and place a folded Bemcot wipe over the top of the mount.
4. Apply gentle pressure to the press to seat the mount; this allows the mount to be positioned at a true horizontal plane. Steps 1–4 are not essential when using a microscope with both an automated stage and predictive focus parameters.

5. Place the glass slide into a light microscope.

The mount is imaged in IL and TL. The methods used for this will vary depending upon the system used. Generally, the mount is imaged using the 2.5x objective in IL contrast for the production of a mount map and then with the 10x objective in both IL DIC and TL methods for row composites.

Figure 11.1 Grain mount fixed to microscope slide with plasticine ready for light microscopy imaging.

11.2.2 Preparation for Scanning Electron Microscopy

1. Re-clean the mount using the same process for light microscopy above in Step 11.2.1.

2. Place the cleaned mount in a clean Petri dish in preparation for gold coating and imaging in the SEM.

3. Coat the mount surface with a 2 nm thickness of gold. Carbon tape is fixed to the edge of the mount when placed into SEM sample holder. This is to create a conductive path for the electrons and prevent charging on the mount surface while imaging.

4. Return the coated mount to the clean Petri dish after imaging in the SEM.

The mount is imaged in the SEM using the method best suited to the samples (generally CL for zircon, BSE for most other accessory mineral phases).

11.2.3 Final image preparation

After the mount has been imaged using light microscopy and SEM, a complex set of images are produced:

- A mount map (from IL images): identifies the positions of each individual sample-row (and reference materials) placed on the mount; samples labelled accordingly.
- A set of composite images are produced from the IL, TL and CL/BSE images for each sample row (Figure 11.2).
The combination of a map and high magnification composite row images assist the analyst to identify and target desired grains to analyse and precise target areas of the mineral grains. These composite images are compiled using imaging software. At present, GA assembles the composite using Adobe Photoshop™.

Figure 11.2 Composite image of zircon row. From top to bottom: incident light (with DIC), transmitted light and cathodoluminescence.
12 Cleaning the Mount for SHRIMP

Step 12 details the mount cleaning procedure in preparation for the final gold coat prior to SHRIMP analysis.

12.1 Material

- Small oven
- Ultrasonic cleaner (low strength)
- Glass beakers – Quartz (50 mL, qty. 4 [2 for use with cleaning agents and 2 for use of Milli–Q water])
- Glass beaker, 100 mL
- Forceps (25 mm SEM stub)
- Squeeze bottle containing absolute-grade ethanol (99.999%)
- Squeeze bottle containing Isopropanol
- Squeeze bottle containing petroleum ether
- Potassium iodide solution (Appendix A)
- Detergent, glassware and laboratory equipment (RBS35, 10% dilution in deionised water (DI H₂O))
- Milli–Q Ultrapure water
- Delicate task wipes (Bemcot)
- Clean Petri dish
- High quality compressed air
- Latex or Nitrile gloves
- Safety glasses

12.2 Mount cleaning

After imaging is complete, the mount is cleaned in preparation for SHRIMP analysis; any external contamination that may affect analysis such as dust, grease, oil or any other foreign material must be removed from the mount. The most important of all is to remove any traces of common (environmental) lead, and to minimise subsequent accumulation of common Pb post-cleaning. It is very easy to contaminate the surface of a SHRIMP mount with common Pb. For instance, tobacco when lit produces non-negligible amounts of Pb greatly increasing its mobility. Therefore, smoking and mount cleaning should be separated by time and distance. Other factors to consider are the environmental sources of Pb, such as the presence of Pb in the air from the combustion of leaded petrol. Therefore, during the cleaning process and post-cleaning of the mount due care is taken to minimise the accumulation of Pb until the mount can be placed safely into a vacuum chamber where common Pb accumulation will stop. To achieve this, a rigorous cleaning method is employed to achieve the best results as follows:
12.2.1 Remove the 2 nm gold coat from the mount

The gold coat is removed from the mount surfaces and any scratches and crevices using Potassium Iodide Solution (Appendix A).

1. Don a laboratory coat, latex or nitrile gloves, and safety glasses.
2. Pour a small quantity of the pre-made Potassium Iodide solution into a 100 mL beaker. A depth of 8–10 mm is sufficient.
3. Hold the mount with a pair of 25 mm SEM stub forceps, dip the mount in the solution and agitate gently.
4. Rinse the mount thoroughly with water.
5. Rinse the mount again with ethanol.
6. Return the potassium iodide solution to its container and thoroughly rinse the beaker.
7. Remove gloves potentially contaminated with the potassium iodide solution.

12.2.2 Clean the mount with ethanol

From the completion of this stage on, do not touch the mount with un-gloved hands, and do not touch the mount grain surface at all.

1. Put on a new set of gloves.
2. Fill the first clean beaker (50 mL) with ethanol and place the mount into the beaker face up.
3. Place the beaker into a low strength ultrasonic cleaner bath filled with water to a depth of approximately 3 cm for 2 minutes.
4. Remove the beaker from the bath and touch the base of the beaker to a clean paper towel to dry it.
5. Discard the ethanol, using a gloved finger (but not touching the mount) to ensure that the mount stays in the beaker.
6. Remove the mount, either by using forceps or by tipping the beaker on its side and catching the mount with gloved hands as it falls out. The mount will always land with the grain-bearing face upwards.
7. Rinse the beaker with Milli–Q water.

12.2.3 Clean the mount with isopropanol

1. Fill the second clean beaker (50 mL) with isopropanol and place the mount into the beaker face up.
2. Place the beaker into a low strength ultrasonic cleaner bath filled with water to a depth of approximately 3 cm for 2 minutes.
3. Remove the beaker from the bath and touch the base of the beaker to a clean paper towel to dry it.
4. Discard the isopropanol, using a gloved finger (but not touching the mount) to ensure that the mount stays in the beaker.
5. Remove the mount, either by using forceps or by tipping the beaker on its side and catching the mount with gloved hands as it falls out. The mount will always land with the grain-bearing face upwards.
6. Rinse the beaker with Milli–Q water.
12.2.4 Clean the mount with petroleum ether

1. Fill the first beaker with petroleum ether and place the mount into the beaker face up.
2. Place the beaker into a low strength ultrasonic cleaner bath filled with water to a depth of approximately 3 cm for 2 minutes.
3. Remove the beaker from the bath and touch the base of the beaker to a clean paper towel to dry it.
4. Discard the petroleum ether into a storage container designed for chemical disposal, using a gloved finger (but not touching the mount) to ensure that the mount stays in the beaker.
5. Remove the mount, either by using forceps or by tipping the beaker on its side and catching the mount with gloved hands as it falls out. The mount will always land with the grain-bearing face upwards.
6. Rinse the beaker with Milli–Q water.

12.2.5 Clean the mount with RBS35 detergent

Only use a delicate-task wipe (e.g. Bemcot) for this task, as many other types of wipe (e.g. Kimwipes) contain binders in the fibres which will scratch the mount surface.

1. Wet a Bemcot wipe with the diluted RBS35 detergent and gently scrub in the following order: the surface, the side and then back of the mount to remove any residual particulates.
2. Fill the second clean beaker with diluted RBS35 and place the mount into the beaker face up.
3. Place the beaker into a low strength ultrasonic cleaner bath filled with water to a depth of approximately 3 cm for 2 minutes.
4. Remove the beaker from the bath and touch the base of the beaker to a clean paper towel to dry it.
5. Discard the RBS35 detergent, using a gloved finger (but not touching the mount) to ensure that the mount stays in the beaker.
6. Remove the mount, either by using forceps or by tipping the beaker on its side and catching the mount with gloved hands as it falls out. The mount will always land with the grain-bearing face upwards.
7. Rinse the beaker with Milli–Q water.

12.2.6 Clean the mount with Milli–Q ultrapure water

1. Fill the first beaker with Milli–Q water.
2. Rinse the mount under the stream of water from the Milli–Q system.
3. Place the mount into the beaker face up.
4. Place the beaker into a low strength ultrasonic cleaner bath filled with water to a depth of approximately 3 cm for 2 minutes.
5. Remove the beaker from the bath and touch the base of the beaker to a clean paper towel to dry it.
6. Discard the Milli–Q water, using a gloved finger (but not touching the mount) to ensure that the mount stays in the beaker.
7. Remove the mount, either by using forceps or by tipping the beaker on its side and catching the mount with gloved hands as it falls out. The mount will always land with the grain-bearing face upwards.
8. Repeat steps 1–7 a further four times, alternating beakers which are rinsed with Milli–Q water between cleaning the mount. A total of five cleans with Milli–Q water are required.
12.2.7 Rinse and blow-dry the mount

1. Rinse the mount under a stream of Milli–Q water.
2. Blow the surface of the mount clean with ultra-pure compressed air. This can be achieved by using a coalescing filter and a carbon filter to obtain high quality air free of particulates and oils.

12.2.8 Oven-dry the mount

1. Place the mount in a small oven set at 30–40°C for a minimum of two hours.
2. Using gloved hands, place a clean Petri dish (lid or inverted base) over the mount to prevent dust from settling on the surface. Do not touch the Petri dish with un-gloved hands as oil will transfer to the mount during handling.
3. When the mount is dry, use ultra-pure compressed air to blow out the Petri dish and the mount surface.
4. Place the mount into the Petri dish and mark the dish with the mount ID number.
5. Secure the Petri dish lid with tape for transport.

The mount is ready for the final 15–20 nm gold coating for SHRIMP analysis and subsequent storage in a vacuum chamber until ready for analysis.
13 Final Notes

When making the mount, work slowly and methodically. Rushing any stage of preparation may lead to errors, possible cross contamination and the production of a sub-standard mount.

Several aspects of the preparation process can affect mount production that must be taken into consideration:

13.1 Epoxy viscosity

It is vital to achieve the correct viscosity and temperature for the epoxy when pouring the mount.

If the viscosity is too low and the ambient temperature is too high, air bubbles may become trapped within the mount surface, adhering to the grains as the ‘rolling front’ of epoxy traverses them, and rendering these grains unviable for SHRIMP analysis.

If the viscosity is too high and the ambient temperature is too low, an ‘Amine Blush’ can form via the reaction of amines in the epoxy with the atmosphere (carbon dioxide and moisture) to produce ammonium carbonate. In worst case scenarios, this presents as a whitish skin on the surface. Amine Blush can cause a layering effect between the first pour and the second pour and should be avoided. It is prudent to perform several trials before attempting to pour a mount intended for production SHRIMP analysis.

13.2 Environmental factors

GA’s experience suggests that environmental conditions can affect mount production processes. In particular, variations in laboratory temperature and humidity can affect the behaviour of (1) resin as it cures, resulting in the post-pouring development of micro-bubbles within the resin particularly nucleating on the tape surface near grains, and (2) some tape glues, which become more pliable and allow grains to sink further into the glue layer.

Although it is has proven impossible to quantify the exact conditions and causes of these phenomena, GA has established that monitoring and controlling laboratory temperature and humidity improves overall mount quality. The following measures are recommended:

- Use a temperature/humidity data logger to monitor the environmental conditions within the laboratory (even if the laboratory is climate-controlled), and identify the times during which the highest and most stable temperatures and lowest humidity levels prevail. Temperatures of 21–25°C and 20–40% relative humidity represent ‘ideal conditions’ for handling epoxy.
- Mix and pour resin during ‘ideal conditions’ whenever possible.
- Store resin in a temperature-controlled environment, such as a low humidity incubator oven set at 25°C.
13.3 Polishing

The resin must be fully cured before attempting to polish. Full curing involves at least 12–24 hours of high temperature curing (within an oven set at 60°C) and another 24 hours afterwards. If the resin is not completely cured, complete polymerisation of the resin will not occur. This has serious implications for the surface characteristics of the mount post-polishing, as it will be susceptible to change, particularly when subjected to high vacuum. Adverse effects can include dimpling of the entire mount surface, development of minute troughs and waves within and between sample-rows, tilting of polished grain surfaces into non-horizontal orientations, and crazing of the surface. All of these features have the potential to significantly compromise SHRIMP analysis, and can make the mount unusable.

In addition, the mount must remain in its mould for the first 12–24 hours in the oven, and this must be followed by complete cooling to ambient temperature before attempting to remove the mould. This prevents distortion of the mount surface due to either swelling or contraction of incompletely cured resin (different types of epoxy resin behave differently).

13.4 Cleaning

Complete and diligent cleaning of the polished mount surface is essential for productive SHRIMP analysis, especially when analyses are aimed at the measurement of isotopic compositions of Pb. SHRIMP instruments are designed and optimised to facilitate such analyses, and even minor quantities of environmental Pb are easily detected. In many cases, no analytical mitigation will be possible: it will be necessary to remove the mount from the high-vacuum source chamber of the SHRIMP and re-clean and recoat the mount, prior to reinserting the mount in the SHRIMP to restart the pumping-down process. In the very best case scenario this process will take several hours; more often it will take 1–2 days, with disastrous consequences if the availability of SHRIMP analytical time is limited.
Acknowledgements

Method development is an iterative process. Methods in the GA Laboratory Microanalysis discipline have evolved over many years through the careful consideration and input of many excellent laboratory technicians. Particularly to be acknowledged for his dedicated contribution to the development of the SHRIMP mount making procedure is Chris Foudoulis (ret.), who set the high standard to which GA SHRIMP mounts are made and renowned for.

I would like to acknowledge Emma Chisholm for manuscript assistance, and Simon Bodorkos and Joanne Tubby for formal review of this Record.
Appendix A Potassium Iodide Solution

The reusable solution is made by mixing the following ingredients together in a light-proof (amber) glass bottle:

- 8 g Potassium Iodide
- 3 g Iodine and
- 8 mL Water (Deionised)

A.1 Synonyms

Lugol’s Solution, Lugol’s Iodine

A.2 Hazards

Avoid inhalation. Use in well ventilated areas. Where an inhalation risk exists, mechanical extraction ventilation is recommended. Avoid contact with skin. Avoid heat, sparks, open flames and other ignition sources.

A.3 Use

Use of safe work practices are recommended to avoid eye or skin contact and inhalation. Observe good personal hygiene, including washing hands before eating. Prohibit eating, drinking and smoking in contaminated areas.

A.4 Personal Protection Equipment

Table A.1 Required personal protection equipment.

<table>
<thead>
<tr>
<th>Eye/face</th>
<th>Wear splash-proof goggles.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hand</td>
<td>Wear viton (R), Nitrile, Latex or neoprene gloves.</td>
</tr>
<tr>
<td>Body</td>
<td>When using large quantities or where heavy contamination is likely, wear coveralls.</td>
</tr>
<tr>
<td>Respiratory</td>
<td>Where an inhalation risk exists, wear an Air-line respirator.</td>
</tr>
</tbody>
</table>

A.5 Storage

Store in a cool, dry, well ventilated area, removed from incompatible substances, heat or ignition sources and foodstuffs. Ensure containers are adequately labelled, protected from physical damage and sealed when not in use. Check regularly for leaks or spills.