1 Scope  This procedure is to be used for preparing a metallographic specimen of printed wiring products. The finished microsection is used for evaluating the quality of the laminate system and the plated-through holes (PTHs). The PTHs can be evaluated for characteristics of the copper foils, plating, and/or coatings to determine compliance with applicable specification requirements. The same basic procedures may be used for mounting and examination of other areas. Because manual metallographic sample preparation is regarded by many as essentially an art, this method describes those techniques that have been found to be generally acceptable. It does not attempt to be so specific as to allow no acceptable variations that can differentiate metallographers. Furthermore, the success of these techniques remains highly dependent upon the skill of the individual metallographer.

2 Applicable Documents

IPC-MS-810  Guidelines for High Volume Microsectioning
ASTM E 3  Standard Methods of Preparation of Metallographic Specimens

3 Test Specimens  Cut the required specimens from a printed board or test coupon. Allow sufficient clearance to prevent damage to the area to be examined. The recommended minimum clearance is 2.54 mm [0.0984 in]. Abrasive cut off wheels can cut closer to the area of examination without causing damage. Some commonly used methods include sawing using a jewelers saw, miniature band saw, or abrasive cut-off wheel; routing using a small milling machine; or punching using a sharp, hollow die (not recommended for brittle materials, i.e., polyimide and some modified epoxy resin systems) (see IPC-MS-810). It is recommended that a minimum of one microsection containing at least three of the smallest diameter PTHs shall be made for each specimen tested. When microsectioning multilayer production printed boards designed without nonfunctional lands on all layers, care needs to be exercised in choosing the test location such that internal lands are connected to the selected PTHs. This is so that a complete quality evaluation can be made.

4 Apparatus or Material

4.1 Sample removal method (see IPC-MS-810 for the best method to meet your needs).
4.2 Mount molds.
4.3 Smooth, flat mounting surface.
4.4 Release agent (optional).
4.5 Sample supports (optional).
4.6 Metallographic rotary grinding/polishing system.
4.7 Belt sander (optional).
4.8 Metallographic microscope capable of 100X to 200X magnification.
4.9 Vacuum pump and vacuum desiccator (optional).
4.10 Room temperature curing potting material (recommended maximum cure temperature 93 °C [200 °F]).
4.11 Abrasive paper USA CAMI Grade grit numbers 180, 240, 320, 400, and 600. See Figure 1 for conversion from American to European grit sizes.
4.12 Cloths for polishing wheels: a hard, low, or no nap cloth for rough and intermediate polishing and a soft, woven, or medium nap cloth for final polishing.
4.13 Oxide or colloidal silica polishing suspension (final polish, 0.3 µm to 0.04 µm [11.8 µin to 1.57 µin]).
4.14 Diamond polishing abrasive (6.0 µm to 0.1 µm [236 µin to 3.94 µin]).
4.15 Polishing lubricant.
4.16 Specimen etching solution (see 6.4).
4.17 Cotton balls and swabs for cleaning and etchant application.

4.18 Isopropyl alcohol, 25% methanol aqueous solution, or other suitable solvent (check for reaction with the encapsulation media and marking system).

4.19 Specimen marking system.

4.20 Ultrasonic cleaner (optional).

5 Procedure

5.1 Preparation of Specimen  Grind the sample sequentially on 180, 240, 320 grit wheel to within approximately 1.27 mm [0.05 in] of final polish depth. Deburr all edges prior to mounting.

5.2 Mounting Metallographic Sample

5.2.1 Clean mounting surface and dry thoroughly, then apply release agent to the plate and mounting rings.

5.2.2 Thoroughly clean the sample using a suitable solvent such as isopropyl or ethyl alcohol. This is especially important when microsectioning “thermally stressed” (solder floated) specimens. Residual flux may result in poor adhesion of the encapsulation media causing gaps between the specimen and the media. These gaps make proper metallographic sample preparation extremely difficult, if not impossible.

5.2.3 Stand specimen in mount ring, perpendicular to the base using sample supports, clips, or with the use of double-sided adhesive tape.

5.2.4 The surface to be examined should face the mounting surface.

5.2.5 Fill the mounting ring carefully with potting material, by pouring from one side to ensure complete PTH filling. Some potting materials may require dilution as recommended by the material manufacturer to reduce the viscosity in order to fill small diameter PTHs. Hand protection is recommended to prevent skin sensitization.

5.2.6 The sample must remain upright and the holes filled with encapsulating material.

5.2.7 Epoxy potting materials may require vacuum degassing in order to achieve complete hole filling.

5.2.8 Allow specimen to cure and remove hardened mount from ring. The minimum qualities the mount should exhibit are:

- No gaps between the potting material and the sample.
- The PTHs filled with material.
- No bubbles in the potting material.

The presence of these deficiencies will result in sample preparation difficulties, as noted in 5.2.2. Identify the specimen by a permanent method. The selected marking system should remain unaffected by solvent and lubricant exposure.

5.2.9 For finite plating thickness measurements, such as gold and nickel thickness on edge board contacts, the overplated specimen may be placed at a 30° angle. This will provide viewing at twice the actual thickness. The measured thickness is then divided by two to arrive at the true thickness. For a more thorough discussion of the techniques of taper sectioning, refer to the references in 6.5.

5.3 Grinding And Polishing

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Figure 1  Abrasive paper grit size (American vs. European)
5.3.1 Using the metallographic equipment, rough grind the mount on 180 grit abrasive paper no closer than to the edge of the PTH barrel walls.

**Note:** Copious water flow must be used to prevent overheating and damage to the specimen and removal of grinding debris.

5.3.2 Fine grind specimen, using copious water flow, to center of the PTHs utilizing 240, 320, 400, and 600 grit discs, in that order. The final paper (600 grit) should finish at the axial centerline of the PTHs. Wheel speeds of 200 to 300 rpm are generally used during fine grinding. Rotate the specimen 90° between each successive grit size and grind for twice to three times the time it takes to remove the scratches from the previous step. The scratch removal can be verified by microscopic inspection between steps. It is of great importance that the ground surface of the microsection is in a single plane. The purpose of rotating the microsection 90° between successive grit sizes is to facilitate inspection. If scratches are observed to be perpendicular to those made during the last step performed, it is a good indication that the surface is not flat and the microsection requires additional grinding. If the surface of the microsection is not flat upon completion of the grinding operations, it may not be possible to remove all of the grinding operations, and grinding scratches during rough polishing. The metallographer should recognize the fact that the coarser grit sizes (180, 240, and 320) induce a larger depth of deformed and fragmented material. Since the depth of deformation decreases sharply below a particle size of about 30.0 µm [1181 µin] (400 grit), it is better practice to spend longer times on 400 grit and especially 600 grit to achieve the final plane sectioning, rather than on the coarser grit sizes.

5.3.3 Rinse sample with running tap water and blow dry with filtered air. Ultrasonically clean, if desired, between each step.

**Note:** Ultrasonic cleaning is highly recommended, especially between the finer grinding steps, prior to rough polishing and between all polishing steps. It is the nature of printed board specimens, especially those with epoxy base material following thermal exposures, to contain voids that can trap grinding and polishing residues that are not removed during simple rinsing. Care needs to be exercised not to damage the specimen surface with excessive ultrasonic cleaning. Ultrasonic cleaning for as little as one minute can damage a polished surface.

5.3.4 Rough polish the specimen with 6.0 µm [236 µin] diamond abrasive on a hard, low, or no nap cloth. Following rough polishing, microscopically examine the specimen to verify removal of all 600 grit scratches. Ultrasonically clean the specimen, if desired. Continue polishing with 1.0 µm to 3.0 µm [39.4 µin to 118 µin] diamond abrasive again using a hard, low, or no nap cloth and microscopically examine the specimen to verify the removal of all the 6.0 µm [236 µin] diamond scratches. Ultrasonically clean the specimen, if desired. Generally, polishing a few minutes using medium pressure during the above steps is sufficient if the microsection has been ground correctly. Wheel speeds of 200 to 300 rpm are generally used during rough and intermediate polishing. Final polishing is accomplished using a soft, woven, or medium nap cloth using a 1.0 µm to 0.1 µm [39.4 µin to 3.94 µin] diamond, 0.05 µm [1.97 µin] alumina or other oxide, or a colloidal silica polishing suspension. This final step is only performed for 10-20 seconds using light to medium pressure when using oxide or silica polishing compounds. When using diamond compounds on soft woven cloths, final polishing may extend several minutes (see 5.3.5). Reduced wheel speeds of 100 to 150 rpm are generally used during final polishing due to increased drag on the microsection. Typically, 6.0 µm [236 µin] followed by 1.0 µm [39.4 µin] diamond and a 0.04 µm [1.57 µin] colloidal silica or 0.05 µm [1.97 µin] alumina have been used successfully. However, other variations such as 6.0 µm [236 µin], 3.0 µm [118 µin], and 0.25 µm [9.84 µin] diamond have also been used successfully. Some have even used 1.0 µm and 0.3 µm [39.4 µin and 11.8 µin] alumina on napless cloths followed by 0.05 µm [1.97 µin] alumina on a soft, medium napped cloth. This procedure can be used successfully, depending upon the skill of the metallographer, but will generally result in poorer edge retention and more relief effects than the diamond compounds (see 6.5, Reference 1).

5.3.5 **Warning** The use of napped cloths can result in poor edge retention (rounding) and relief between constituents since it exacerbates the varying rates of material removal (i.e., tin-lead alloy and the softer encapsulation media are removed at a faster rate than the copper or glass fibers in the base material). The higher the nap, the more the effect. The user needs to minimize the polishing time and use ample lubricant and light pressure during final polishing.

5.3.6 Rinse in mild soap and warm water or solvent and blow dry.
5.3.7 Examine and repolish, beginning with 6.0 µm [236 µin] diamond, if necessary, until:
1. There are no scratches larger than those induced by the final polishing abrasive.
2. The specimen is not higher or lower than the mounting material.
3. There is no smearing of the copper plating into the PTH or base material.
4. The plane of microsectioning is at the centerline of the hole as defined by the governing specification. If the grinding depth is insufficient, additional grinding and repolishing may be required.
5. There is little, if any, visible preparation induced damage to the glass fibers of the base material.

See IPC-MS-810 for photomicrographs illustrating some of the above qualities. When the required microsection quality has been achieved, examine the microsection of multilayer printed boards in the “as-polished” condition as specified in 5.4.1 to identify suspect areas of internal layer separation that appear as dark lines or partial dark lines. These areas should be verified after metallographic etching. There may not be a one-to-one correlation of all separations noted “as-polished” versus those noted after etching, when examined at the specified magnifications.

5.3.8 Swab specimen with suitable etching solution (see 6.4) typically applied for two to three seconds, repeat two to three second swabbings if necessary, to reveal the plating interfaces.

**Caution:** Over etching may totally obscure the demarcation line between the copper foil and electroplate copper, preventing accurate inspection.

5.3.9 Rinse in running tap or deionized water to remove etchant.

5.3.10 Rinse in solvent and blow dry.

5.4 Evaluation

5.4.1 Set the magnification at 100X and measure all characteristics required by the standard or specification using a metallograph set for bright field illumination. Referee at 200X, unless otherwise specified.

5.4.2 Measure the plating thickness in at least three PTHs. Total surface copper thickness can also be determined on the same specimen cross-section. Record the plating thickness determinations and quality of the plating. Plating thickness determinations should not be made at nodules, voids, or cracks.

5.4.3 Quality observations may include the following: blisters, laminate voids, cracks, resin recession, hole wall pull-away, plating uniformity, burrs and nodules, plating voids, and wicking. In addition, plating quality for multilayer printed boards may include: innerplane bond to PTH, resin smear, glass fiber protrusion, and resin etchback. Some of the plating conditions may be observed on the polished specimen prior to etching.

6 Notes

6.1 Overplating the specimen per ASTM E 3 with a layer of copper or other plating with a hardness similar to the specimen, prior to encapsulation, provides better edge retention, thereby providing more accurate thickness measurements.

6.2 For a more accurate evaluation of possible internal layer separations, the procedures covered in 6.2.1 and 6.2.2 are recommended.

6.2.1 Regrind Procedure

6.2.1.1 After polishing and examining with a metallographic microscope, turn power off at the final grinding wheel.

6.2.1.2 Gently regrind the specimen using copious amounts of water and 600 grit paper with the wheel in a stationary position parallel to the PTH barrels. Six to eight double strokes should be sufficient. This action will remove any copper metal smear that may have occurred over the interconnection separation during rotary polishing.

6.2.1.3 Rinse and dry specimen and repolish per 5.3.3 through 5.3.7, then reexamine under the metallograph to determine if interconnection separation exists.

6.2.1.4 After examination in the “as-polished” condition (and taking photomicrographs, if desired), etch the specimens with the mild etchant described in 6.4, and reexamine the specimen again for interconnection separation and all other characteristics. There may not be a one-to-one correlation of all separations noted “as polished.”

6.2.2 Mechanical/Chemical Preparation (Attack Polishing) Another useful technique is a simultaneous mechanical/
chemical polish at the final polishing step. Use a mixture of 95% colloidal silica and 5% by volume hydrogen peroxide (30% concentration) and polish on a chemically resistant cloth. This results in a simultaneous mechanical and chemical abrasion of the specimen. The metallographer must be careful to balance the mechanical abrasion with the chemical abrasion. Too much mechanical abrasion will result in fine scratches; too much chemical polishing will result in etching of the specimen. Neither of these conditions is desirable. Experimentation will be required to develop the optimal balance.

6.3 In order to develop more insight into detected interconnection separations, regrind and repolish the specimen in the horizontal plane (perpendicular to the original vertical plane), and examine the semicircumferential interface. This method has a low success rate when the separation affects less than 50% of the internal layer thickness (as noted on the vertical microsection).

6.4 The following is the recommended solution for specimen etching.

25 ml ammonium hydroxide (25-30%)
25 ml-35 ml of 3-5% by volume stabilized hydrogen peroxide

The addition of 25 ml of water (distilled or reverse osmosis) will dilute the solution, resulting in longer etching times, which may be desirable in certain situations.

Wait five minutes before using. Prepare fresh every few hours.

6.4.1 There are other etchant solutions that have been used or that may be developed for etching copper. Care must be exercised in their selection and use because of the sensitive nature of the electrolytic, electroless, and foil etching characteristics as well as possible galvanic effects in the presence of tin-lead (see 6.5, Reference 2 and IPC-MS-810).

6.4.2 When studying tin-lead solders, it is sometimes helpful to use etchants specifically designed to reveal those alloy's microstructures (see 6.5, Reference 2).

6.5 Additional references on metallographic laboratory practice.