TECHNotes

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Metallographic Preparation of Lead Free Solder

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Introduction

Solder joints are an important part of micro-electronic packages because they serve as mechanical as well as electrical interconnects. Historically, solder for joining electronic parts to copper terminations of a printed wiring board have been made with an alloy of tin and lead. Tin-lead (Sn-Pb) solders are less expensive and have low melting temperatures, which reduces thermal stress during manufacturing. However, due to concerns about safety and the environmental pollution caused by lead, European Union (EU) legislation passed the restriction of the use of lead in Electrical and Electronic Equipment (RoHS) directives in effect since July 2003. The RoHS limits the use of six hazardous substances, including Lead, Mercury, Cadmium, Hexavalent Chromium, Polybrominated Biphenyls, and Polybrominated Diphenyl Ether.

Since this time, many lead-free alloys have been proposed including Sn-Ag and Sn-Cu. Sn-Ag-Cu (SAC) has been identified as a potential candidate to replace Sn-Pb but it is relatively expensive, and many assemblers are opting for less costly options including tin-copper based solders with the addition of microalloy, for their wave, selective and dip tinning operations.

In many applications, especially automotive, solder interconnects experience degradation due to prolonged temperature and power cycling. This degradation behavior manifests itself as changes in the grain structure, primarily through grain coarsening. Because the magnitude of the change in grain structure is strongly dependent upon environmental stresses, quantifying microstructural changes provides an alternative approach to calculating an acceleration transform number or function. Accurate detailing of phase morphology allows for the use of grain growth models to potentially develop acceleration factors.

The difficulty of polishing these materials is very well known in the microelectronics industry and failure analysis laboratory. It usually takes a long time to attain a suitable polished surface. Lead free solder joint assessment and reliability testing involves extensive failure analysis and microstructural investigation, and so many samples prepared to a high finish can be needed. As the sample preparation involves the simultaneous processing of both hard and soft materials together it can be particularly challenging. The rate of material removal and the deformation layer from each stage are important factors. Polishing parameters such as type and size of grinding paper, type and size of polishing abrasive, lubricant used and many other settings must be carefully selected to control this effectively.

It is also typically necessary to etch the sample to reveal the grain structure or identify sub-grain boundaries – often an iterative process involving alternating polishing and etching stages to obtain the optimum results. Etchants are typically short-lived, and hazardous to handle, so there is a very significant advantage to reducing or eradicating this requirement in the analysis.

The purpose of this technical note is to develop an improved and efficient technique for the metallographic preparation and analysis of lead-free solder materials. Optical polarized-light microscopy and SEM analysis has been performed on four different solder alloys including SnCu, SN100C, SAC305 and Kester's new alloy K100LD (SnCu- micro-alloyed with Ni and Bi).

Metallographic Preparation

Solder materials, of course, are part of a composite of components, where the solder is intended to provide both a joint and a conductive path. The starting point in preparation is to extract the area of interest in such a way as to ensure that the area to be inspected is not going to be damaged. The larger component is most often a populated circuit board. In this case, we have components and the circuit board itself that will become part of the sample. The combination of harder, more brittle materials and softer, ductile materials make each stage of the process – and particularly sectioning – very important to efficient preparation.

Sectioning

It's useful to look first at the depth of damage that can be introduced into a circuit board with different sectioning techniques. Cutting damage can appear in many ways – thermal damage, mechanical deformation, brittle fracture and delamination are a few. In addition, sectioning damage can be internal as well as external, and is not always readily visible. Table 1 shows typical depths of damage we have previously measured on this type of sample. More aggressive sectioning techniques can do up to 100 times deeper damage on samples compared with recommended techniques. Recovering this typically involves grinding long distances into the sample using coarse SiC paper, which itself can cause deep damage in the sample. The ideal scenario is to section with the lowest damage possible, and close to the area of interest. This minimizes potential for sectioning damage but also minimizes the amount of planar grinding needed. This immediately improves efficiency, and the quality of the result.

Table 1: Potential damage depths from sectioning techniques on PCB

Method	Type of Damage	Potential Depth
Shearing	Deep mechanical damage	5 mm
Band/hacksaw lubricated not cooled	Moderate thermal and mechanical damage	2.5 mm
Dry abrasive cutting	Moderate thermal damage	1.5 mm
Wet abrasive cut off saw	Minimal thermal and reduced mechanical damage	250 µm
Diamond/precision saw	Minimal thermal and mechanical damage	50 µm

The recommended method for cutting lead free solder is with a diamond wafering blade. Buehler supply a wide range of wafering blades, but in most circumstances a high concentration blade with medium to small particle size is recommended – this would typically be the "15HC" type.

Diamond blades are metal bonded and will cut these materials easily without embedding in to the cut surface. Conversely, abrasive blades should be avoided as the blade smears solder and can embed easily into the sample as the blade wears. This smearing can cause excessive friction, slowing down cutting and generating heat. Small amounts of solder that smear onto the cutting edge of diamond blades can likewise be detrimental, and so regular dressing of the blade is recommended. Buehler supply dressing sticks with every blade, formulated to gently clean and refresh the surface of the blade. We would recommend doing this by cutting once into the dressing stick every 20-30 minutes of use.

When sectioning these materials, we would typically recommend the use of precision sectioning machines. In many cases, the Isomet 1000 (Figure 1) is ideal for this application when there is a low throughput of samples, as it can be supplied with a wafering table that allows the user to section boards to a manageable size. For higher sample throughput, we recommend the use of an automated cutter such as the Isomet High Speed (Figure 2) – configured with T-slot tables, so that the board can be clamped effectively. The Isomet High Speed has a cutting length of up to 7 inches, and can be positioned accurately on the board with the use of the laser alignment function. The ability to fully automate the cut not only saves operator time, but greatly improves both the speed and the reproducibility of the cut surface.

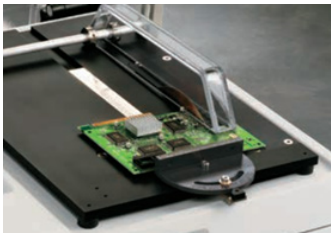


Figure 1: IsoMet™ 1000 with Table Saw Attachment

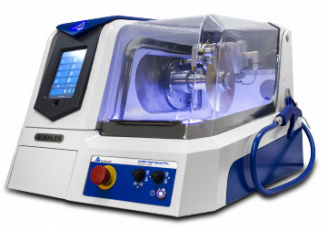


Figure 2: IsoMet™ High Speed with T-slots and laser alignment

Mounting

"Mounting" is a process of encapsulating the sample with a polymer such that it's supported and protected through the remainder of the preparation process. In some cases, it is preferable to mount the sample prior to sectioning, and then re-mount for grinding and polishing processes. Doing this ensures that sectioning damage is controlled and can be especially effective in failure analysis where the sample is particularly prone to being affected by the sectioning process. In most cases, however, mounting is done directly after the sample is sectioned. For solder specimens, the level of heat during the mounting process is critical.

Lead solder alloys are typically close to the eutectic point of the Sn-Pb phase diagram – around 63% Sn with a melting point of 361°F (183°C). A typical lead-free solder (96.5 Sn, 3.0 Ag, 0.5 Cu - commonly referred to as SAC 305) has a melting point of 422°F (217°C) to 428°F (220°C). The increase in melting temperature of lead free solder is a challenge to maintaining quality as well reworking in printed circuit board assembly manufacturing, as discussed earlier, and so melting point is always desired to remain low. Many mounting processes and materials involve relatively high temperatures and/or pressures. Hot compression mounting processes should never be used for these materials. Mounting should be done using castable mounting resins, with particular attention to the peak exothermic temperature the resin is expected to attain during curing. This normally means that acrylic resins such as SamplKwick and fast curing epoxies such as EpoKwick FC – commonly used in other areas of electronics preparation – are unsuitable for this application. A low temperature curing epoxy is vital to ensure the solder structure remains undisturbed. Low exotherm temperature usually requires curing time to be longer. Buehler's EpoThin 2 is ideal for these applications, as it combines a low exotherm with low viscosity, where encapsulation of complex sample shapes is needed. Where harder specimens are being encapsulated, faster speeds are required and higher viscosity is acceptable then EpoxiCure 2 may also be used. For best results, always ensure that the specimen is clean and dry prior to mounting.

Historic Sample Preparation Method

Table 2 shows an historic preparation method that has commonly been used for of Pb, Pb-Sn and Sn-based alloys. It involves a series of SiC grinding stages, followed by five intermediary polishing steps on a soft cloth with alumina prior to final polishing on a vibratory polisher with colloidal silica. This method is effective, but time consuming and it is not unusual for samples to have excessive rounding at the end.

Table 2: Historic approach to preparation of solder materials						
	Surface	Abrasive	Load (lb per 1.25"Ø sample)	Time (min:sec)	Base (rpm)	Relative Rotation
1	SiC paper	240 grit*	4	1:00	250	< >
2	SiC paper	320 grit*	4	1:00	250	< >
3	SiC paper	400 grit*	4	1:00	250	< >
4	MicroFloc	9.5µm Alumina	4	5:00	150	< >
5	MicroFloc	5.0µm Alumina	4	5:00	150	< >
6	MicroFloc	3.0µm Alumina	4	5:00	150	< >
7	MicroFloc	0.3µm Alumina	4	3:00	150	< >
8	MicroFloc	MasterPrep 0.05µm Alumina	6	3:00	120	< >
9	MicroFloc	MasterMet	400g holder, no weights	60-120	Vibratory 6-8 revolutions/min	

*Coated with paraffin wax and water is used as coolant during grinding. <<Complimentary <>Contra

Table 3: Modern approach to preparation of lead free solder						
	Surface	Abrasive	Load (lb per 1.25"Ø sample)	Time (min:sec)	Base (rpm)	Relative Rotation
1	CarbiMet SiC paper*	600 grit	5	Until plane	150	< <
2	TexMet-C	3µm Metadi Ultra Diamond paste MetaDi Fluid	5	5:00	150	< >
3	ChemoMet/ MasterTex	MasterPrep 0.05µm Alumina	5	2:00	130	< >
4	MasterTex?	MasterMet 0.06µm SiO_2	400g holder, no weights	30-60	Vibratory 6-8 revolutions/min	

*SiC paper wears out so change paper if sample is not planar after one minute of usage. <<Complimentary <>Contra

In contrast, the modern preparation method recommended in Table 3 requires far fewer steps, and significantly less time, as well as improving preparation flatness and quality. Detailed comments on the steps used for these samples are given below.

Grinding

The initial grinding step is a very important phase of sample preparation as it must remove cutting deformation without causing excessive damage itself. Coarse abrasive sizes, or use of the incorrect abrasive type, are common causes of problems at this stage. Diamond grinding discs are not recommended for these samples, as they can cause excessive deformation in soft materials. When sectioning is carried out close to the area of interest, using a diamond wafering blade, it is quite possible to use a 600 grit SiC paper for planar grinding. This is the most effective approach for quality and speed. For other sectioning methods, coarser paper may be used – however, this usually results in the need for additional fine grinding steps and longer preparation in the intermediary polishing stages in order to recover sample flatness. In all cases, SiC paper should be used for short times in order to maintain best performance. For coarser grit paper this may be a few minutes of use, but for 600 grit we recommend changing the paper every 1 minute of use.

After the grinding step, samples were cleaned in an Ultrasonic Cleaner for five minutes with 1% liquid soap solution. The samples were then inspected under 50x magnification to check for any embedding of SiC particles. If any SiC embedding is seen, then repeat the 600 grit grinding step, but apply a light coat of paraffin wax to the SiC paper prior to grinding.

Intermediate Polishing

For the polishing stage, we used a TexMet C polishing cloth with diamond paste. This non-woven pressed fiber cloth combines with the waxy carrier of the paste to retain a high concentration of abrasive at the surface, with a high degree of lubricity. These characteristics help to prevent embedding of particles in the solder during preparation, as well as ensuring a high material removal rate.

During this preparation stage, lubricate with Metadi Fluid as needed to keep the surface damp. Excessive lubricant will reduce removal rate and promote embedding, so it is recommended to add fluid "little and often".

After this stage, the samples should be washed thoroughly and again cleaned in an Ultrasonic bath with 1% liquid soap. Rinse thoroughly and then dry. It's recommended to inspect under a microscope in both Bright Field and Dark Field to confirm that this step is successively completed. The sample should have consistently sized scratches and excellent flatness (Figure 3) , with no bright points in Dark Field present, that would indicate embedded diamond.

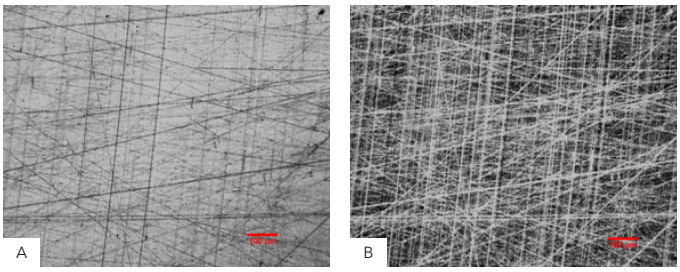


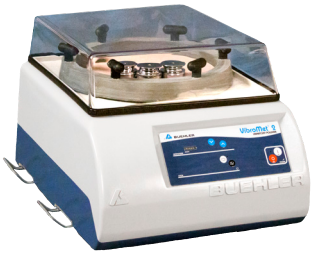
Figure 3:Typical sample in (A) bright field and (B) dark field, after preparation using 3µm diamond paste on TexMet C

Fine Polishing

The mechanical polishing stage can be done using either the ChemoMet polishing pad or MasterTex polishing cloth, depending on the solder being prepared. The ChemoMet is a nap free synthetic surface that provides exceptional flatness, but may leave fine scratches on extremely soft materials. The MasterTex is a dense, napped cloth suitable for the softest of materials, but will leave slightly more polishing relief.

The mechanical polishing step should be as short as possible, as longer times will tend to cause polishing relief and edge rounding. Polishing relief makes fine detail in microstructures more difficult to see, as well as causing visual artefacts if it is excessive.

Vibratory polishing



Vibratory polishing provides an excellent surface finish without deformation and with minimal difficulty. It requires no hazardous chemicals and is suited to any material or mix of materials. The Vibromet 2 polisher (Figure 4) generates a high frequency, variable amplitude vibrational motion, without vertical movement. The sample is polished without inducing stresses and, for very soft materials such as solder, vibratory polishing allows completely scratch free preparation without polishing relief and with sharply defined edges.

In this work, the samples were mounted in a specimen holder (400g mass) and no additional weights were added. A MasterTex cloth was used with MasterMet colloidal silica. It's important to use a slurry with a pH <11, so MasterMet 2 is not recommended. The surface of the polishing cloth should be dampened with water before use, and MasterMet added to completely cover the surface of the cloth. The samples are placed on the plate, and amplitude of vibration set such that the samples move around the surface of the cloth ~6 times per minute. It was found that 30-60 minutes typically gave the best surface for inspection. Figure 5 shows the improvement that vibratory polishing has on the appearance of the solder. Note the improvement in detail level and contrast, even though the mechanical polish is very good (no large scratches) there is still sub-surface deformation and small scratches that disguise structure, whereas the vibratory polished specimen provides perfect contrast and detail.

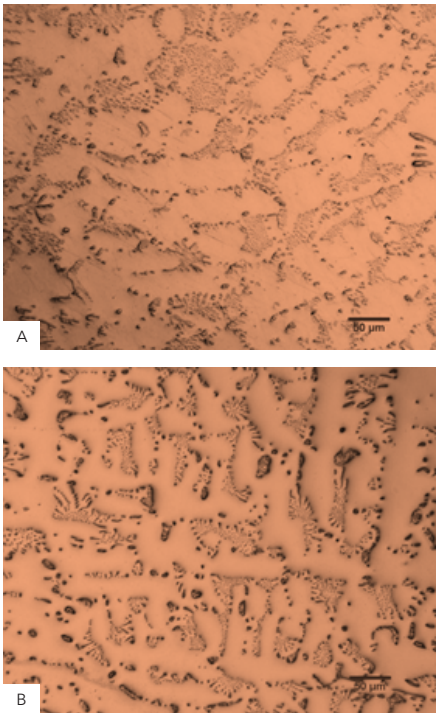


Figure 5: Solder structure after (A) mechanical polishing and (B) vibratory polishing.

Optical examination

Many of the features of a solder joint are best examined initially with optical microscopy, and it is also highly recommended to inspect samples during preparation itself. Bright field microscopy shows comparative reflectance of different areas and should be used for measuring sizes and shapes of features. Dark field microscopy is excellent for revealing cracks and edges, as well as scratches and embedded particles.

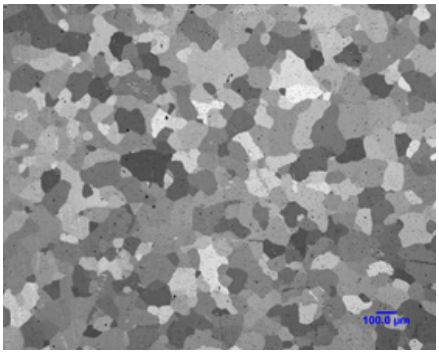


Figure 6: SN 100C with polarized light

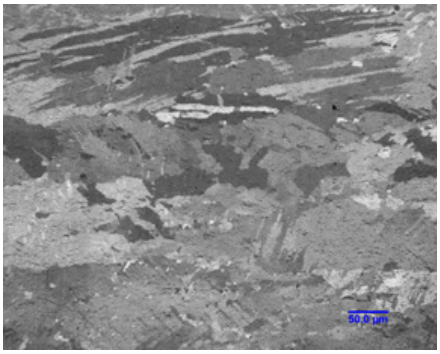


Figure 7: Sn-Cu with polarized light

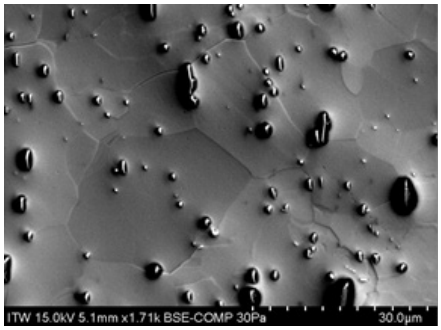


Figure 8: BSE image of Sn-Cu Solder grains structure

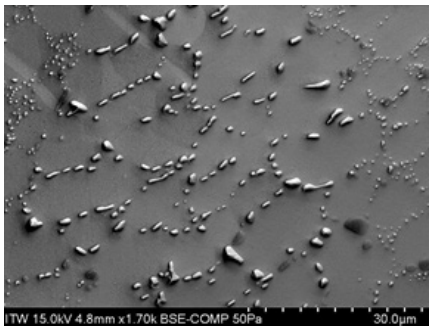


Figure 9: BSE image of SAC305 solder

For some materials (typically non-cubic crystalline structures), cross-polarized light can be used to show the grain structure of the material. An interference effect between the incident light beam and the sample surface, dependent on the orientation of the crystal structure at that point, results in a variation in contrast through the microscope. Using polarized light, we can quickly and accurately assess the grain structure of the solder without any need for etching. This greatly improves speed and safety of the process. Figures 6-7 show typical structures of the materials tested, under cross-polarized light.

Conclusions

High quality and efficient preparation of lead free solder materials requires the following critical steps:

- Using Precision Cutters and Isomet wafering blade to section with minimal damage
- Start grinding on the finest possible SiC grit paper
- Cloth and diamond selection for recovery of grinding deformation is critical
- Mechanical polishing times should be minimized to avoid polishing relief
- Vibratory polishing provides a deformation free surface for rapid and accurate inspection of grain structure

Using this method provides many immediate benefits, including:

- Fewer metallographic preparation steps, with more potential for automation
- Significant reduction in specimen preparation time and cost
- Removal of deformation artifacts commonly left by the traditional polishing route
- Improved reproducibility
- Removal of the need for etching – fewer steps, and fewer health and safety concern
- Electron microscope (SEM) imaging can be used to observe the grain structure without re-preparation

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