Solar Cell Microstructural Analysis

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Introduction
Photovoltaic (PV) cell development and commercialization continues at a rapid pace. Wafer cell and modular manufacturers are expected to produce highly reliable and long lasting panels. While the market demands long warranties greater than 25 years, all too often, product quality can degrade over time due to the continuous stresses of the metallic interconnects. Cross sectional microscopic analysis can play an important role toward increasing module/cell reliability from material inspection, and interconnect verification to coating analysis. This TechNote describes high quality cross section sample preparation techniques of crystalline silicon solar cells for identifying degradation mechanisms and soldering process quality.

Common Anomalies
One of the most predominant failure mechanisms in electronic assemblies is exposure to thermal cycling. Tab ribbon bonds must be robust, reliable and able to withstand repeated thermal stress. Microstructural analysis is performed to evaluate thermo-mechanical stress before and after accelerated aging on cell interconnects. As solder interconnects age, the constant expansion and contraction of the joint causes a solder to fatigue, become more brittle, and dissociate into larger grains. Modified or enlarged grains will change its mechanical properties. Cell research is performed to evaluate the effectiveness of existing and new bonding materials or during a transition from leaded solders to lead-free solders. Cross sectional analysis on microelectronic electric components, wafers and raw materials all benefit and reveal the following parameters:

- Impurities
- Poor solder wetting
- Joint cracks
- Solder porosity & microvoids
- Solder thickness and delamination
- Unacceptable solder, silicon, and copper microstructure
- Solder meniscus
- Intermetallic Phases, coarsening, etc.

Sampling Techniques
It is possible to extract a solder joint, bus bar (metalized lines) and copper ribbon from a cell and prepare micro-sections. Solar cells are connected on the front and back side in series (Figure 1) with copper solder coated ribbons that are soft soldered (lead based solder) to the bus bar material. This paste-like bus bar material (when applied) is mainly silver and glass particles; the silver component provides the cells electrical conductivity and solderability. The bus bar material is hardened in an oven to bond it to the cell prior to applying the solder-coated ribbons (Figure 2). High quality solder joints are critical to extended service life and with high electrical conductivity.

Sectioning is performed on polycrystalline or monocrystalline solar cells, often referred to as thick-films. It is performed to reduce a sample down to a manageable size for mounting, grinding and polishing. Preparing a PV cell interconnect for cross sectional analysis first requires sectioning the solder joint and wafer (Figure 4) with minimal damage to the cut surface. The preparation is demanding, since the materials are brittle and have a broad range of mechanical properties. To prevent cracking and chipping during sectioning, encapsulation is recommended with a quick drying resin called SampKwick®, which cures in 15-25 minutes. The resin is applied by a brush, simply painting the bus lines on both sides of the cell (Figure 3). When coating the bus lines with a resin, it may be necessary to remove the resin after cutting and before potting. Removing the acrylic resin will provide a stronger bond next to the sample when mounting it in epoxy.

Figure 1: Solar cells connected in series with tabbing ribbons.

Figure 2: Interconnector on a soldered solar cell sample that exhibits a poor quality cut (top) vs a good quality cut.
Once the acrylic resin covering the bus lines has fully cured, the entire cell, 156mm x 156mm x approximately 0.2mm in size, is placed in a solar cell clamping device (Buehler #11-2706 holder) used in the IsoMet® 4000 Linear Precision Saw (Figure 5). Note: an acrylic resin coating may not be necessary when using the solar cell holder. First, the entire bus bar is sectioned and then it is cross cut into a manageable size that can later be mounted. Approximately one cm² square samples are cut from the cell using an IsoMet® Diamond Wafering Blade LC-15 type, with a cutting speed of 3,750 rpm and a feed-rate of 5mm/minute. Proper blade selection will minimize any damage that will then have to be removed by additional grinding and polishing. The most common problems encountered during sectioning are bus bar detachment, silicon breakage and edge cracking. Broken edges or cracks (Figure 2) may indicate that the feed-rate is too high or the blade may be too coarse.

Sample Mounting

Once a sample has been sectioned from the PV solar cell, they are mounted in epoxy. A UniClip Support Clip (Figure 7) is used to keep the sample in a vertical position in the SampKup® mounting cup. Mounting begins with the PV solar cell sample cast in an epoxy resin EpoxiCure® (Figure 8). EpoxiCure® maintains good sample edge retention and cures in six hours, without a significant temperature rise in a 1.25” (32mm) size mounting cup. If additional edge support is required, the epoxy can be mixed with Flat Edge Filler (Figure 9). Flat Edge Filler is made up of globular ceramic oxide particles which will increase the mount hardness and show evidence of reduced shrinkage. Sample edge retention is important especially when viewing features at the edge or when performing coating thickness measurements. EpoxiCure® can also be used with a Conductive Filler for Scanning Electron Microscopy (SEM) or Energy Dispersive X-Ray Spectrometer (EDS) analysis.
Sample Grinding
Once cell samples have been mounted, they are mechanically ground and polished. Preparation begins with planar grinding, using CarbiMet® 2, 320 grit sized SiC grinding paper. Grinding will remove the sectioning damage and establish a flat surface. During this procedure, induced artifacts can occur so grinding time should be kept to a minimum. Soft, ductile metals such as copper or solder can trap the abrasives that break off during grinding, but generally, they will be removed during polishing. After grinding, and with every preparation step, it is recommended to thoroughly clean the samples and sample holder (used with semi-automated grinder polishers). The samples and machine parts are cleaned with tap water. If using an UltraMet® Ultrasonic Cleaner for the samples, distilled water is recommended. If any abrasive is carried to the next preparation step, it can generate scratches in the sample. Scratches can cause problems with some analytical techniques, such as fault analysis and electron backscatter diffraction (EBSD) microstructural analysis.

Sample Polishing
After grinding, samples are further refined by several polishing steps. With Si cells, four steps are done including polishing with diamond and alumina abrasives charged on a polishing cloth. Each step uses a finer abrasive lubricated with MetaDi® Fluid. For best results, hard polishing cloths are used due to the varied hardness of the cell material. For example, solder and copper are very soft, ductile metals vs. silicon which is hard. When these materials are next to each other in a sample, such as in a solar cell, the softer material is going to be removed at a higher rate, causing relief. Excessive relief will make it more difficult to analysis the sample and reduces the depth of field, due to excessive different heights in the materials.

Final Polishing
The last step of the standard method includes a final polish that uses exceptionally fine abrasive (0.02 μm) silica to remove any fine scratches remaining from the previous step. Very little material is removed, so any remaining damage may not be corrected. Final polish should take less than 2 minutes if the sample was properly prepared up to this step. An option at this point is to follow up with a final preparation vibratory polishing; it removes any smearing and shows clear delineation between materials and some alloy phases. Scratch removal, particularly for copper and solder alloys, can be very difficult. Vibratory polishing used with a low nap polishing cloth and a colloidal silica suspension will remove light scratches and remaining surface damage. Figures 10 and 11 show the results of vibratory polisher on an SAC 305 solder alloy. The complete sample preparation method and parameters are listed in Table 1.

Metallographic Examination
Quality assurance during solar modular production is maintained by the examination of soft solder joints. Solder interconnect quality is essential for extending the life of a solar cell. The individual cells are connected in series by flat copper solder coated ribbon which is soldered to the metalized bar traces. Interconnects must be properly soldered to maintain the life of a panel. A cross section of the ribbon interconnect is shown in figure 12. In figure 13, the solder encapsulated copper ribbon shows insufficient solder at each end. The other solder layers and bus bar can be measured for proper thickness.

Figure 12: Microstructure of Sn62-Pb36-Ag02 solder, unetched solar cell copper ribbon.
Soldering which joins the solder coated ribbon to the bus bar is an alloy process called wetting. Proper wetting is essential for strong stable connections. In electronics, a wetting angle can be measured and less than 40°, slightly concave, is considered acceptable. Solar cells use screen-printed pastes for the bus lines and achieving this angle can be difficult. Figure 14 shows an acceptable solder wetting angle between the bus bar and soldered coated copper ribbon. An improper wetting angle can be seen in figure 15, along with voids in the solder. A certain percentage (area) of voids is acceptable but may also depend on their location and size. Microvoiding (small voids along the interface), typically less than 1 mil in diameter, is usually associated with contamination or surface finish.

Cracks and delaminating can occur in cell and wafer manufacturing, over their lifespan, or during soldering due to the thermal stresses. It is important during sample preparation not to smear the soft metals that would hide any micro-cracks that may have developed. Figure 16 shows a crack in the silicon wafer and delaminating of the bus bar.

Intermetallic compounds can form when two dissimilar metals diffuse into one another. Many leaded and lead free solders will begin to develop and grow these compounds over the life of the cell. Such compounds are not desirable, since they may become hard and brittle and the electrical resistivity can be higher than the constituent metal. Figure 17 shows a bismuth containing a leadfree solder interconnect.

Accelerated aging of a solder interconnect is performed on a bismuth lead-free solder. The SEM images in figure 19 show intermetallic phase growth of the solder above the bus bar before and after the aging process. Various compounds formed can be analyzed with EDX marking the different materials and their elemental maps. The microstructure and their mechanical properties will constantly evolve when exposed to accelerated aging. Understanding this behavior and the effects on the solder joint will allow for a better understanding of extending the life of these interconnects.
Silicon Ingot Sample Preparation

Microstructural analysis on amorphous silicon ingots used for the production of the cell wafers is critical to maintaining greater performance. Material characterizing for quality purposes can reveal impurities, structure, grain size, annealing twins and provide a record of its process for future reference. Pure silicon or any pure metal is much more difficult to prepare and requires more time than alloys. The preparation process begins with material removal for the ingot. Sectioning is when most damage occurs, so great care should be made to use the proper sectioning blade. This will minimize any mechanical and thermal damage.

Once a sample is ready for mounting, and since it is not as delicate as the solar cell interconnects, compression mounting is suggested. Compression mounting is done by using a SimpliMet® 1000 automated Mounting Press. EpoMet® F Molding Compound is used for the mount media since it provides the greatest hardness and better edge support. Once the sample has been mounted, the sample preparation is relatively straightforward using a seven step preparation method, listed in table 2. Figures 21 and 22 show light optical images of high purity Si ingot material etched with NaOH.

A vibratory video is available for viewing at http://www.youtube.com/user/BuehlerMaterials#p/u/3/MenlojlSruw. Details on the preparation methods used to prepare these samples can be obtained at the web site: http://www.buehler.com.


Table 2: Preparation Method for Pure Silicon

<table>
<thead>
<tr>
<th>Step</th>
<th>Surface</th>
<th>Abrasive/Size</th>
<th>Time (mins.)</th>
<th>Force lb [N]</th>
<th>Base/Head rpm</th>
<th>Head Direction</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.</td>
<td>CarbiMet® 2 SiC Paper</td>
<td>Water Cooled</td>
<td>320 grit [P400] SiC</td>
<td>1</td>
<td>5lb [22]</td>
<td>300</td>
</tr>
<tr>
<td>4.</td>
<td>TriDent®</td>
<td>MetaDi® Fluid</td>
<td>3μm MetaDi®</td>
<td>5</td>
<td>5lb [22]</td>
<td>150</td>
</tr>
<tr>
<td>5.</td>
<td>TriDent®</td>
<td>MetaDi® Fluid</td>
<td>1μm MetaDi®</td>
<td>2</td>
<td>5lb [22]</td>
<td>150</td>
</tr>
<tr>
<td>6.</td>
<td>MicroCloth®</td>
<td>0.02μm MasterMet® 2 Colloidal Silica</td>
<td>3</td>
<td>5lb [22]</td>
<td>150</td>
<td>Contra</td>
</tr>
<tr>
<td>7.</td>
<td>MicroCloth®</td>
<td>0.02μm MasterMet® 2 Colloidal Silica</td>
<td>≥60</td>
<td>VibroMet® 2 Vibratory Polisher</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 19: SEM image showing the intermetallic phase growth after accelerated aging. From the top, Silicon cell, (a.) Bismuth containing solder on (b.) bus bar.

Figure 21: Polycrystalline silicon viewed with Nomarski DIC (etched: 100mL water, 75g NaOH).

Figure 22: Standard Sample Preparation of Pure Single-Crystal, Band Contrast 205.8.

Figure 23: Standard Sample Preparation Plus Vibratory Polishing of Pure Single-Crystal, Band Contrast 233.