In-situ study of electromigration-induced grain rotation in Pb-free solder joint by synchrotron microdiffraction

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ABSTRACT

The rotation of Sn grains in Pb-free flip chip solder joints hasn't been reported in literature so far although it has been observed in Sn strips. In this letter, we report the detailed study of the grain orientation evolution induced by electromigration by synchrotron based white beam X-ray microdiffraction. It is found that the grains in solder joint rotate more slowly than in Sn strip even under higher current density. On the other hand, based on our estimation, the reorientation of the grains in solder joints also results in the reduction of electric resistivity, similar to the case of Sn strip. We will also discuss the reason why the electric resistance decreases much more in strips than in the Sn-based solders, and the different driving force for the grain growth in solder joint and in thin film interconnect lines.

INTRODUCTION

Electromigration (EM) has been known to induce grain rotation in Al (T_m = 933 K) [1-3], Cu (T_m = 1356 K) [4], and white tin (β-Sn) (T_m = 505 K) [5-7] lines at enhanced temperature. However, since the melting points of these three metals are different, so are the diffusion mechanisms involved [8]. During Electromigration stressing in Al, the grains rotate more at the cathode end than at the anode end while there is no trend in grain rotation between the cathode and the anode in Cu lines [1-4]. The average rotation rate in these metal lines is slower than in Sn lines. The grains in Al and Cu lines rotate as a result of dislocation redistribution, while the grains don't grow significantly in this process. In Sn lines, grains increase in size as the rotation occurs even if the sample has been annealed at high temperature for long periods before the electromigration experiment. Since Sn has a body-centered tetragonal structure, the electric conductivity is anisotropic. It has been measured that the electric conductivity increases by a few percent in Sn lines due to the electromigration-induced grain rotation [9] and the a-direction of the crystal is realigned along the electron flow direction. However, there is no literature report about the conductivity increase coupled with Sn grain rotation induced by electromigration in Pb-free solder joints even though most of the industrial Pb-free solder joints contain over 90% of Sn. Here we report a detailed study of the orientation evolution of Sn crystals in Pb-free solder joints. The high orientation resolution (0.01 degree) and high spatial resolution (1 μm) provided by synchrotron radiation based X-ray polychromatic diffraction makes such study possible. The resistivity evolution is also estimated in this letter based on the precisely measured orientation of each individual grain and reasonable assumptions.

EXPERIMENTAL

A flip chip sample with Pb-free solder balls with composition Sn–0.7% (wt) Cu , was cut into four pieces and grinded by SiC sand papers, then polished by submicron-sized Al_2O_3 powders to produce a mirror-like smooth surface. The configuration of the flip chip sample has been described elsewhere [10]. The cross-sectioned sample was annealed at 150 ^oC for 2.5 hours to eliminate damages caused by polishing. Two solder joints were then stressed by electrical current at 75 ^oC. The average current density was kept constant at 1.25×10^4 A/cm^2 for 42.8 hours. The temperature and the applied current density were mild, compared to the accelerated EM experiments reported in the literature because we intended to track the orientation evolution of multiple grains during the process, and avoid growing them too much during the experiment.

The polychromatic X-ray microdiffraction measurements were performed on Beamline 12.3.2 at the Advanced Light Source (ALS) in Lawrence Berkeley National Laboratory. One of the polished solder joints on which the electric current was applied was continuously raster scanned under the focused beam before and during the electromigration test and a Laue pattern has been collected at each step. The energy range of the X-ray beam was about 5 keV to 24 keV. The beamline configuration and capabilities have been described elsewhere [11-12]. The flip chip sample was attached to a copper block containing a cartridge heater that sits on the diffractometer sample XY stage and the cross-section of the solder joints was kept at 45^o with respect to the incident beam. The temperature of the sample was set to 75 ^oC and controlled within ± 2 ^oC. The X-ray beam was focused by Kirkpatrick-Baez (K-B) mirrors and the beam size was about 1 μm × 1 μm at the sample. Since the X-ray beam size was smaller than the grain size in the Pb-free solder joint, each crystal grain could be regarded as a single crystal when the X-ray impinged on the sample surface. The raster scan step size was 3 μm, and the exposure time was 0.5 sec. Each raster scan lasted about 4 hours. A MAR133 X-ray CCD detector was mounted at about 8 cm above the sample and 90^o with respect to the incidence beam to record the Laue diffraction patterns.

RESULTS AND DISCUSSIONS

The Laue patterns were analyzed using the in-house XMAS software, to obtain the indexation of each grain and the orientation map for each scan. Figure 1 shows the orientation map of the solder joint before EM; the different colors show the out-of-plane orientation of each grain. The arrow indicates the electron flow direction, and the current crowding region is located at the upper left corner of the joint (indicated by a white ellipse). By comparing the orientation maps from each scan, it was found that the grains in this solder joint didn't grow significantly during the 45 hours period of EM test. The thirteen grains we tracked to study the orientation evolution are indicated by black numbers in the figure.

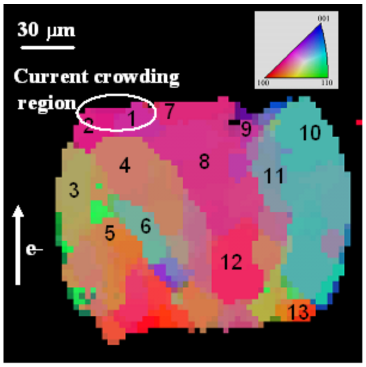

Figure 1. Out of plane grain orientation map of the solder joint and the electron flow direction

Previous observation by A. T. Wu et al [5] indicated that the a-axis realigned itself very significantly along the electric current direction in Sn strip. To study the grain orientation evolution, the coordinates of the center position of each grain were determined and the Laue patterns taken at this position in the different scans were carefully indexed. The a-, b-, and c-axes of the tin crystal after different period of EM test were represented by three vectors. It is reasonable to assume that the current direction was pointing toward the upper left corner of the colder joint, although the exact electric current path is unknown. Based on this assumption, it was possible to calculate the angle between the current flow direction and the a-axis of the tin crystal. In fact, since β-Sn has a tetragonal crystal structure, the a and b axis are equivalent so either a or b whichever is the closest to the current flow direction was chosen to calculate the angle. The evolution of the angles is shown in figure 2 (a). It was found that most of the grains in the solder joint didn't rotate during EM test. Only the two grains at the current crowding region rotated at almost constant rate as the electric current kept stressing them. These two grains rotated by 0.3-0.4º values which are more than an order of magnitude higher than the sensitivity of 0.01 provided by the technique. Another possible source of error that could influence orientation measurement of anisotropic tetragonal crystal is Joule heating, and thus thermal expansion, especially in the current crowding region. However, since what we calculated here is the relative angle of each individual grain after hours of electromigration test, thermal balance should have been reached within much shorter time than the process even if there was a temperature gradient from the top of the sample to the bottom [13-14]. The rotation rates of Grain 1 and Grain 2 were 8×10^{-3} degree / hr and 1.1×10^{-2} degree / hr, respectively. The angles were negative, which means the a-axes of these two grains were realigned closer to the electron flow direction. The most likely explanation of the reason why only the two grains in the current crowding region rotated is a much higher current density value in this region than the average value [15]. In other words, rather high current density was required to induce grain rotation in Sn solders. Unlike the cases in Al lines and Cu lines, the diffraction peaks of the Sn crystal were not significantly streaked (i.e: the material is plastically deformed) after EM stressing. This is because creep and recovery could happen much easier at 75 ^{o}C in Sn due to the relatively lower melting temperature and high diffusivity comparing to Al and Cu.

Based on the same assumption, the electric resistivity of individual grains in the electric current direction was calculated by applying equation 1.

$$\sigma = \sigma_1 \cos^2 \theta_1 + \sigma_2 \cos^2 \theta_2 + \sigma_3 \cos^2 \theta_3 \qquad (1)$$

Where σ is the conductivity for a given direction, σ_1, σ_2, and σ_3 the conductivity in a-, b- and c-directions respectively, θ_1, θ_2, and θ_3 the angles of the given direction with respect to the a-, b-and c-axes, respectively [16]. Since the vectors of a, b and c had been obtained by the indexation of the Laue patterns, and the center of each grain was carefully pinpointed from the orientation maps, θ_i could be calculated for each grain. For β-Sn, the resistivity is 13.25 μΩ cm along a- and b-axes and 20.27 μΩ cm along c-axis. From our calculation, we found that the resistivity of most of the grains ranges between 13.5 μΩ cm and 15.0 μΩ cm. In figure 2 (b), the change of resistivity of 3 grains was plotted as a function of EM time. It was found that the resistivity of Grain 1 and Grain 2 decrease gradually as EM was performed, while the resistivity didn't change in Grain 9, which didn't rotate within the EM test period. However, the resistivity drop in these two grains is less than 0.1%, which was much less what was observed in the Sn strips.

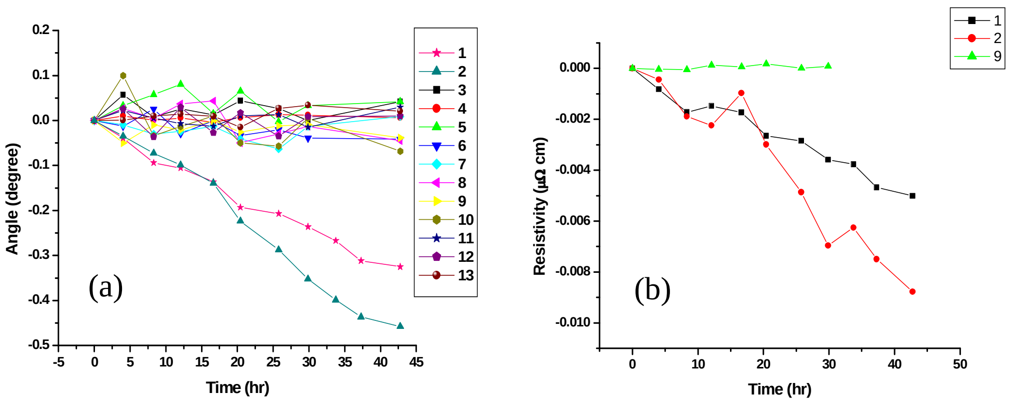

Figure 2. The evolution of (a) the angle between a-axis of the Sn unit cell with respect to the electron flow direction and (b) the calculated resistivity as a function of EM time

For comparison, another pair of solder joints was stressed with the same electric current density at 150 ^{o}C for about 27.5 hours. The solder joint was scanned by the X-ray microbeam before and after EM. Before the first scan of the solder joints, the flip chip sample was annealed at 150 ^{o}C for 2 hours as well. Under this condition, the grains in the solder joints grew much larger after EM as seen in the orientation maps (figure 3 (a) and (b)). Since some grains disappeared and the grain sizes changed greatly after EM, there is a difficulty to track the center of individual grains like in the previous case. For this case, the resistivity of some grains in the same position of the solder joint was calculated before and after EM, and the results were listed in table I. It was found that Grain 1, 2 and 3 disappeared after EM and Grain 1' occupied their positions, and the resistivity of Grain 1' was significantly lower than those three small grains. The positions of Grain 4 and 5 were occupied by Grain 2' after EM. The resistivity of Grain 4 was much higher than Grain 2', while Grain 5 was lower.

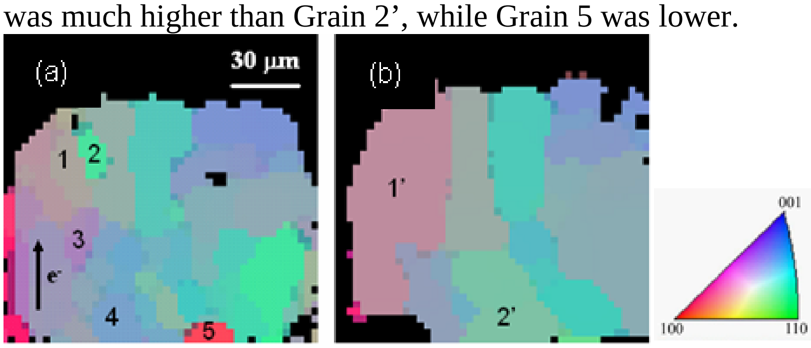

Figure 3. Out of plane orientation maps of a solder joint before and after EM test at 150 ^{o}C for 27.5 hr

An important issue we ignored when estimating the resistivity change was the electron scattering by grain boundaries. However, let's note that for thin film Sn strips the electric resistivity could decrease by 10% during EM, while for Sn solder joint no such change has ever been reported, even if grain growth and grain boundary elimination are observed at 150 ^{o}C. Resistance of Sn interconnect line has scattering contribution from both grain boundaries and lattice. In the case of thin narrow line, grain size is rather small so resistance is dominated by scattering at grain boundaries. In Sn-based solder joints grains are extremely large, so resistance is dominated by lattice scattering and grain boundary contribution can be ignored in the calculation of resistivity.

Table I The grain resistivity in the solder joint before and after EM

<table>
<tr><th colspan="2">Before EM</th><th colspan="2">After EM</th></tr>
<tr><th>Grain #</th><th>Resistivity (μΩ cm)</th><th>Grain #</th><th>Resistivity (μΩ cm)</th></tr>
<tr><td>1</td><td>14.192</td><td rowspan="3">1'</td><td rowspan="3">13.315</td></tr>
<tr><td>2</td><td>14.298</td></tr>
<tr><td>3</td><td>13.823</td></tr>
<tr><td>4</td><td>15.190</td><td rowspan="2">2'</td><td rowspan="2">14.268</td></tr>
<tr><td>5</td><td>13.703</td></tr>
</table>

The driving force of grain growth in Sn strips and Sn solders is also proposed to be quite different based on our observation. In Sn strips, grains growth was clearly observed after only 7 hours EM at 150 ^{o}C even if the sample has been annealed at 150 ^{o}C for 60 days [5]. In Sn solder joints, in contrast, most of the grains didn't grow after 42 hours EM at 75 ^{o}C with much higher current density. When higher temperature (150 ^{o}C) was applied, grain growth was observed after 28 hr. As a result, we can conclude that electric current played an important role for the grain growth in Sn strips, while in solder joint, temperature was the critical factor for ripening. Ripening happens faster at higher temperature mainly due to higher diffusivity.

CONCLUSION

In summary, by synchrotron radiation based polychromatic x-ray Laue microdiffraction, we studied the electromigration-induced microstructure evolution in tin solder joints at different temperatures. At 75 ^{o}C, only the grains in the current crowding region of the Pb-free Sn-Cu flip chip solder joints rotated slowly, where the current density could be about one order higher than the average, and no grains orientation evolution was detected in most of the grains beyond this region, with sufficiently high orientation resolution. The rotation rates were much slower than observed in Sn interconnect lines even though the current density was much higher. At 150 ^{o}C, grains grew much bigger after EM. The electric resistivity was estimated based on the anisotropy of the β-Sn structure and the orientation matrix of each individual grain. It was revealed that the electric resistivity of the grains in the current crowding region decreased almost linearly as they rotated. This provides strong evidence that the grains reoriented to reduce the resistivity. The driving force for grain growth in solder joints was discussed and compared with the case in Sn strips.

For future work, it is proposed to map the 3D distribution of orientation and strain in the solder joint, especially in the current crowding region, on the microdiffraction beamline by using triangulation method on similar sample, so that a better understanding of the grain rotation mechanism in such bulk material could be achieved.

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