

Characterization of SAC Solder Microstructure Evolution During Thermal Cycling with Statistical and In-situ Methods

Bite Zhou¹, Thomas R. Bieler¹, Guilin Wu², Stefan Zaefferer²,
Tae-kyu Lee³, Kuo-Chuan Liu³

¹Chemical Engineering and Materials Science,
Michigan State University, East Lansing, MI 48824-1226

²Max-Planck-Institut für Eisenforschung, Düsseldorf, Germany

³Cisco Systems, Inc., San Jose, CA

Abstract

The evolution of Sn grain microstructure during thermomechanical cycling occurs due to grain boundary movement, as some single crystal solder joints gradually transform to multicrystals. This process has been documented statistically using polarized light and EBSD mapping by counting the numbers of joints with different orientations and microstructures in packages with different aging and thermomechanical cycling histories. The process by which evolution occurs is examined assessed by comparing diffraction patterns and peak shifts obtained from in-situ measurements from the full volume of a solder ball at different stages of thermal cycling history using synchrotron radiation. Diffraction patterns were used to identify crystal orientations present in the joint, and peak shape and peak shifts were used to assess changes in internal stress in several joints.

Keywords: Lead-free solder, Orientation Imaging Microscopy (OIM), Cross Polarized Imaging, Synchrotron X-ray diffraction

Introduction

Lead-free solder joints tend to be single-crystal or multi-crystals due to the high extent of undercooling and rapid growth of a nucleus [1-2]. The dominant crystal orientations in lead-free solders also differ from one sample to another [3]. Because of the anisotropy of tin [4,5], mismatches of coefficient of thermal expansion (CTE) between solder and substrate (or package) introduce stresses that vary with crystal orientation during thermal cycling which could accelerate failure. Since crystal orientations are apparently randomly distributed, each solder ball has the unique microstructure and mechanical response under cyclic loading. Thus, identifying initial crystal orientations of each solder joint, and gaining understanding of how crystal orientations and microstructure evolves is crucial in understanding which deformation mechanisms become operative, and affect damage evolution.

Crossed Polarized Light Optical Microscopy (XPM) and Orientation Imaging Microscopy (OIM)

have been used to measure orientations and microstructure of an array of solder joints, which provides statistical information on changes of microstructures (single or multi-crystal) and crystal orientations during thermal cycling [6]. Thus the effect of thermal aging conditions and initial microstructure distribution can be analyzed and correlated with evolution of stress states that may affect microstructure evolution within the solder joints, as internal stresses have been shown to cause grain boundary migration, recovery, and recrystallization [7]. While this can be assessed statistically, further insight will be gained by studying the evolution of crystal orientation in the same joint throughout time, and to measure the strain state and microstructure evolution using in-situ non-destructive methods. Synchrotron X-ray diffraction can be utilized to obtain much of this information during the thermal cycling [8-12].

Experimental methods

A 15x15 mm plastic ball grid array (PBGA) with a 14x14 full array of solder joints (600 μm in diameter) with 1.0 mm pitch between each solder joint having a 5x5 mm silicon die attached were examined. The package side substrates had an electrolytic NiAu surface finish. Assembled parts were attached to high T_g FR4 printed circuit board with a thickness of 2.4 mm with an organic surface preservative (OSP) surface finish. Sn-3.0Ag-0.5Cu (wt%) (SAC305) solder paste was used for the reflow and a typical peak temperature of 240°C, with 60 seconds above the liquidus temperature reflow, was used for board assembly. To study the effect of aging conditions, samples were aged at 150°C for 1000 h in ambient air. For samples subjected to 2500 thermal cycles a 0-100°C thermal cycle with a ramp rate of 10°C per minute and a 10 min dwell time was used.

To permit accurate orientation measurements, the specimens were cold mounted and a z-axis cross section (bottom view sample preparation) was used to polish from the printed circuit board (PCB) side to the middle of the solder joint. Optical images using crossed polarized filters of each individual solder joint were taken in high resolution format for subsequent analysis. For OIM, the resin surrounding the balls and the package were carefully painted with carbon paint, to cover non-conducting surfaces, and a small portion of each ball was covered to insure a good conductive path. The OIM data were obtained in a CamScan 44FE electron microscope and then analyzed using EDAX TSL OIM Analysis software (ver. 5.2) to generate orientation maps. Maps were cleaned up using the Neighbor CI Correlation, to replace pixels that have had low confidence index (CI) with their neighbors having a high CI. Due to imperfect application of the carbon paint, some portion of each joint could not be measured. To assist in analysis, a Matlab code was developed to assist visualization of slip systems for a given orientation [13].

Samples prepared for synchrotron measurements consisted of a single row of solder



Figure 1. Single row specimen from the edge of a PBGA package. The solder balls have 1 mm pitch.

joints sliced from an assembled component, such as the edge row shown in Fig. 1. The sliced row of joint specimens were set on a copper pedestal placed on a Peltier stage at the eucentric center of a goniometer system in beam line 6-ID-D at the Advanced Photon Source at Argonne National Laboratory. The copper pedestal had heaters on it to be able to heat the specimen to 100°C, and the Peltier stage was able to cool the specimen to ~0°C. Measurements were made between February and April 2008, which allowed the samples to be thermally cycled as indicated above at Cisco Systems Inc. in San Jose, CA. The time-temperature profile used in the *in-situ* cycling experiments was longer, so that changes in strain could be resolved during longer hold times. Further details are found in [14].

Statistical approach

Lee and colleagues studied thermal-cycled SAC305 (Sn-3.0Ag-0.5Cu wt.%) lead free solder joint arrays in plastic ball grid array (PBGA) packages with 196 solder joints in each package [6]. The results illustrated effects of different pre-conditions on the distribution of crystal orientations and the variation of the overall fraction of single-grain solder joints using Cross-Polarized Light (XPM) microscopy. Six cases with different pre-conditions and cycling histories were investigated and the results showed that thermal cycling facilitates the transformation of single-grain joints into multi-grain joints so that the fraction of single-grained solder joints changes significantly. In contrast, the percentage of single-grained joints

Image	Category/type	Indicator
	Single Grain structure Solder joints with a single grain under the polarized filter	
	Near single grain structure Solder joints with a second grain orientation occupies less than 20% of the joint area	
	Two grain structure Solder joints with two grain orientation	
	Near three grain structure Solder joints with two dominant grain orientations and a minor third orientation	
	Three grain structure Solder joints with a clear beach ball structure or more than three grain orientations	

Figure 2. Criteria for grain structure category

was not strongly affected by isothermal aging.

XPM imaging is able to display different contrast for different crystal orientations [1], making it possible to identify joints that are not single crystals. However, two crystal orientations with symmetric orientations with respect to the polarized light source can give the appearance of a single crystal in XPM images. Similarly, a low angle boundary may provide a difference in contrast, and make a solder joint that is effectively a single crystal appear as a multi crystal. Thus, results from XPM imaging and OIM are compared using

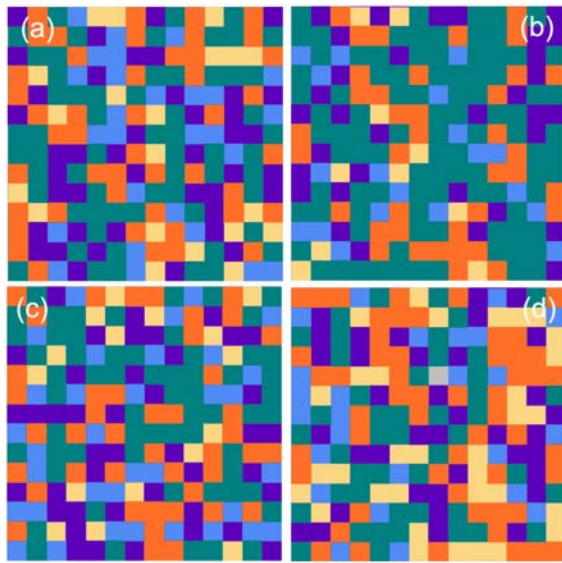


Figure 3. Color maps from XPM illustrating crystal structures under different pre-conditions: (a) As assembled (b) Aged 150°C for 1000hrs without thermal-cycling (c) Unaged sample after 2500 thermal cycles (d) Aged at 150°C for 1000hrs followed by 2500 thermal cycles.

the same statistical metrics as those used by Lee.

The statistical analysis was conducted based on specific criteria (Fig. 2) using different color/gray indicators to represent different types of grain structures observed in XPM images. Based on observation of XPM images, grain structures were collected and plotted in spatial maps (Fig. 3).

These XPM results are obtained by the author, which are not the same as those listed by Lee [6] (reasons for the differences are explained later). In comparison, results from OIM are shown in Fig. 4 with the same criteria (OIM data for some joints

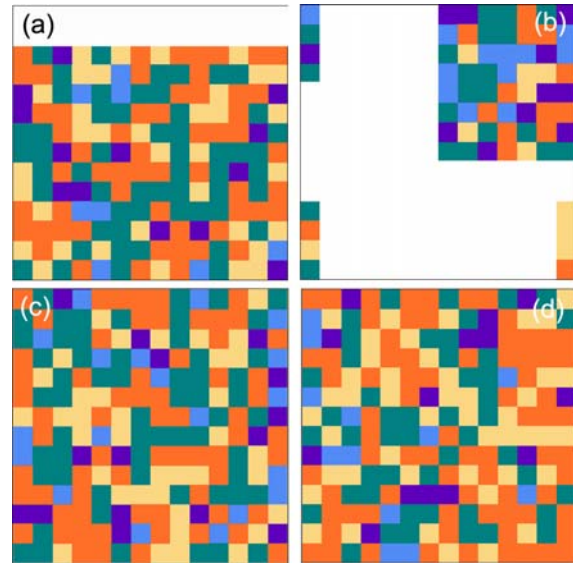


Figure 4. Color maps from OIM illustrating crystal structures under different pre-conditions: (a) As assembled (b) Aged 150°C for 1000hrs without thermal-cycling (c) Unaged sample after 2500 thermal cycles (d) Aged at 150°C for 1000hrs followed by 2500 thermal cycles.

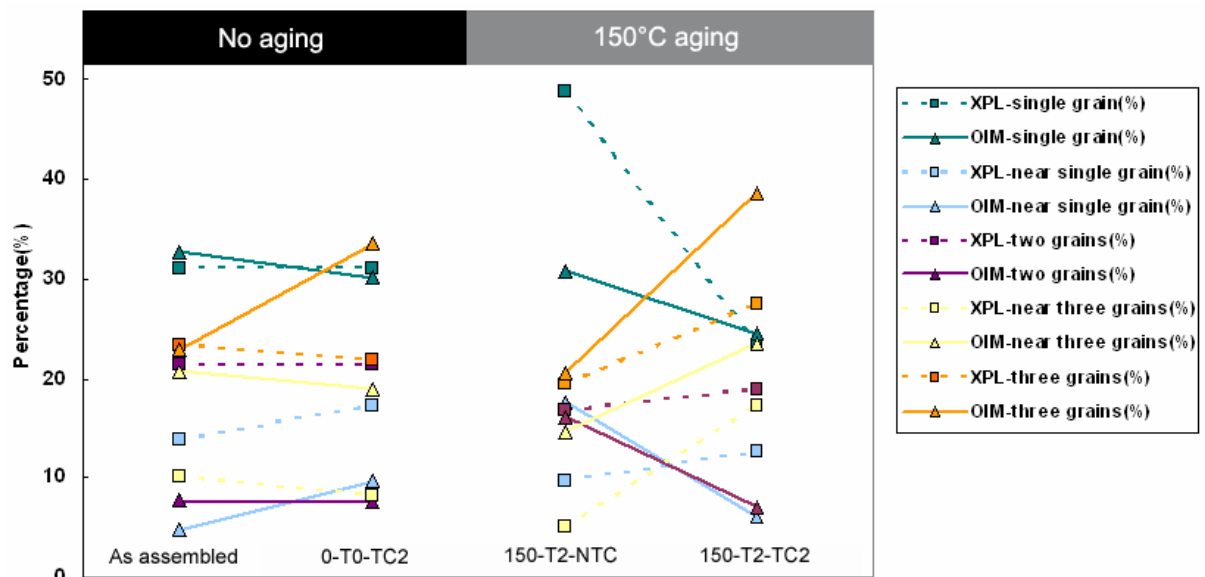


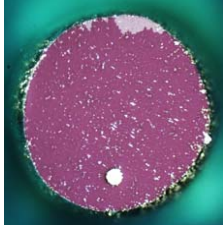
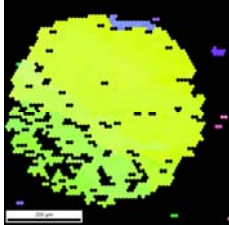
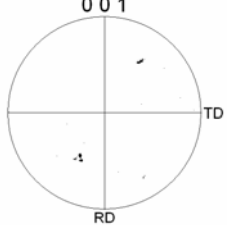
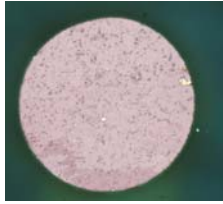
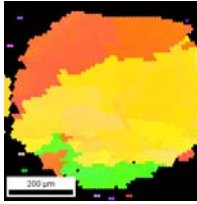
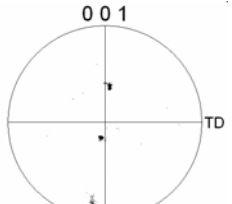
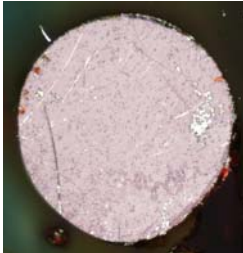
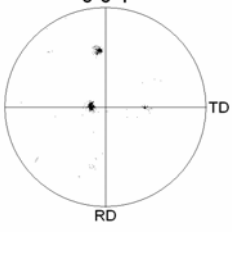
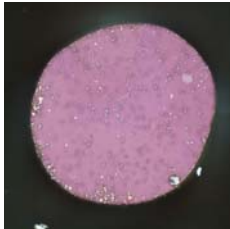
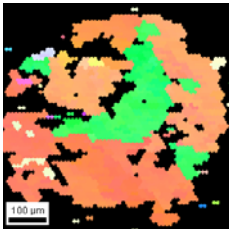
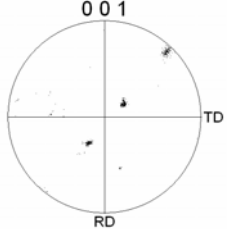
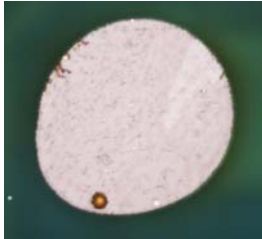
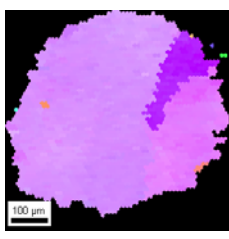
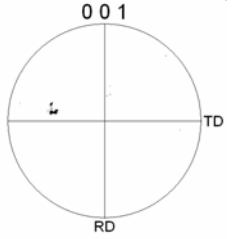
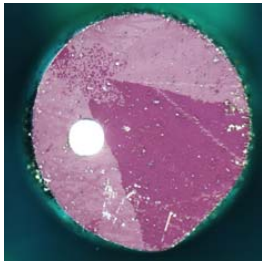
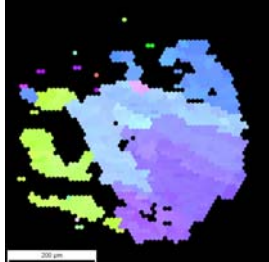
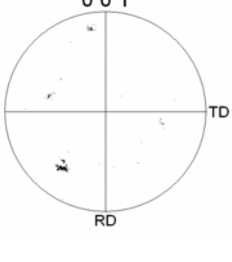
Figure 5. Fraction of different grain structures with various pre-conditions

were not collected).

XPM and OIM results both show a mixture of single-grain joints as well as multi-grain ones. Lee identified a decreasing trend of single-grain joints

after thermal cycling, and less of a change during the aging process. Fig. 5 shows how the fraction of different types of joints changed; dashed lines with square symbols represent XPM data, and solid

Table I Examples of disagreement between XPM and OIM interpretations on same joint

#	XPM Results	OIM Results	OIM Pole Figure
1. Near-single grain does has small secondary grain; the large grain has two low angle boundaries (split peaks)			
2. Apparent near-single grain is actually three grains			
3. Apparent single grain has two large grains and small third grain orientation			
4. Apparent single grain is actually three grains			
5. Near-single grain Single grain is actually a single grain with two low angle boundaries (split / double peak)			
6. Two apparent grains are actually three grains; and a very small fourth. One of the single grains has multiple low angle boundaries			

lines with triangles represent OIM data. As shown in Fig. 5, the fraction of three grain joints increased for both unaged and aged samples after thermal cycling. In the unaged sample there were only minor changes in the fraction of most other types of joints after thermal cycling, and this was evident in both the XPM and OIM data sets. However the actual fractions measured are not in agreement for the two methods, for any group except single crystals. In contrast, there are much more significant changes in aged joints after thermal cycling; a much larger increase in the fraction of three grain joints, and a corresponding decrease in single and bi-crystal joints. Thus, it is observed that single and bicrystal joints transformed into triple-grains. While similar trends are observed with both XPM and OIM data sets, the actual fractions are not usually the same for all types of samples (the largest differences are for the aged but not cycled data, but there was also a smaller fraction of samples in this OIM data set).

The fraction of each kind of grain structure derived from OIM differs significantly from XPM results, suggesting that several factors influence the judgment of the different grain quantities. Some detailed comparisons of exemplary joints are shown in Table 1 to illustrate reasons why these two methods provide different results. Based on Lee's study, the criteria used in XPM analysis could miss orientation differences if they don't display different contrast, so they could be counted as single-grain solder joints. Cases 1 to 4, show how two or three grain joints can be counted as single or near-single

grain joints. This can partially explain how the XPM statistics indicate a higher fraction of single-grain solder joints. But this cannot explain the difference in the extent of decrease of single-grain joints after thermal cycling observed in XPM results. One possible explanation is that those counted as near-single grain structures by XPM may actually belong to the single-grain category as shown in case 5 in Table I. There is an apparent contrast difference in the upper right quadrant, which could be interpreted as a different grain, however, the OIM map and pole figure shows that grain boundaries are all within 10° misorientation, so this joint is better described as a single-grain. Moreover, certain joints such as the one in case 6, appear as two grains in XPM, but are in fact, three or more grains in the OIM measurement.

In addition to the possibilities listed above, results could differ if XPM images are observed by different people. For instance, with knowledge of physical metallurgy, it is not likely that the upper right and lower left areas in case 6 could belong to the same grain even they show similar contrast in XPM images, as it is not thermodynamically likely for a two grain microstructure to have the shape shown in the XPM image. With experience and understanding of SAC microstructures, an observer could identify the XPM image of case 6 as a natural triple crystal with a 'beachball' microstructure with three grains rotated 60 degree around common [100] axis. Thus, deciding which grain structure category a particular joint belongs to (using Lee's

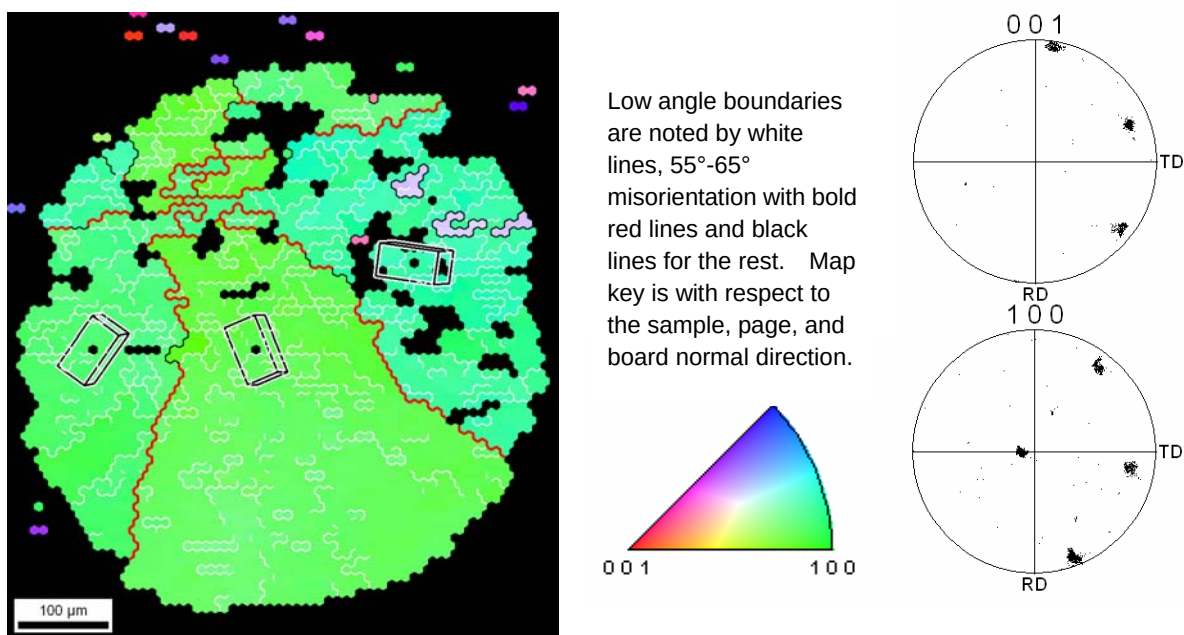


Figure 5 OIM map showing 'Beachball' twinning structure.

criteria) will be sensitive to the knowledge and experience of the observer. This is demonstrated by the fact that Lee and Zhou (as observers of the same XPM data set) obtained different results. Zhou, however, had the OIM data set available to clarify uncertainties in the XPM data, which Lee lacked. Nevertheless, XPM could still serve as a preliminary method to identify changes in microstructure, as the trends observed are similar even if the quantitative results are different.

Both methods are capable of being mis-read,

however. An improvement to the XPM approach can be obtained by rotating the specimen under polarized light, so that different grain boundaries can be made more or less visible [6]. Also, a single OIM map may not fully reveal grain boundaries, if grains rotated 60° about $[001]$ are imaged so that the common $[001]$ crystal direction is nearly parallel to the sample direction which is the basis for the OIM map, such as the sample 001 direction illustrated in Fig. 6. Hence, to gain a more accurate measure of grain structure, at least

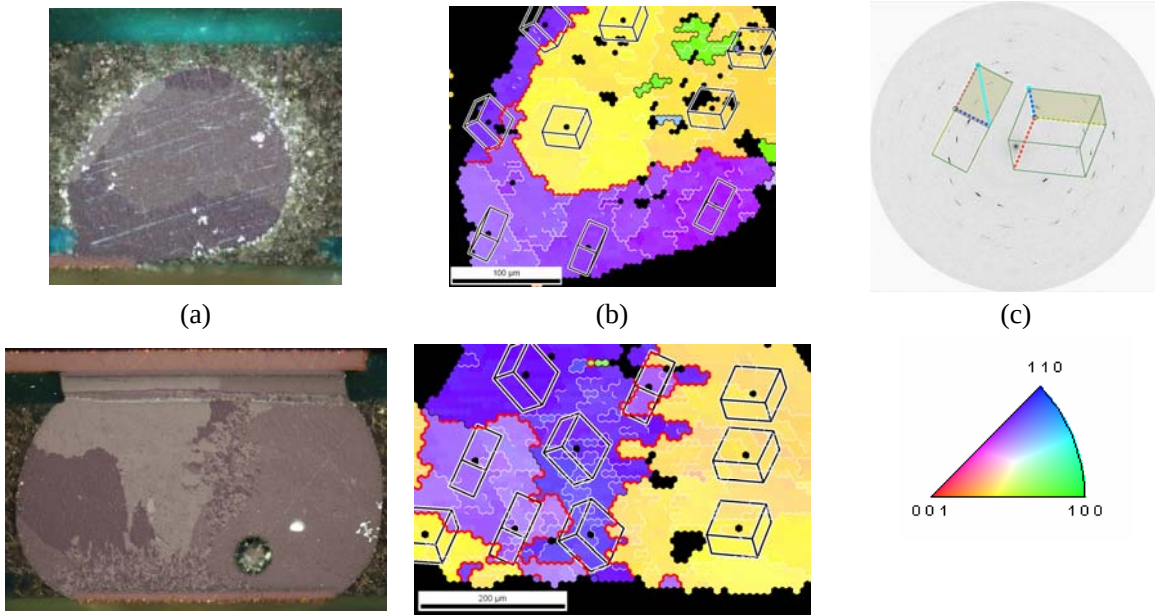


Figure 7. (a) XPM images of un-aged Ball-14 after two different serial sections; (b) corresponding OIM scans, (c) X-ray diffraction pattern showing two orientations present in the diffraction pattern that are in agreement with OIM results. The color maps are with respect to the board normal direction (vertical on map).

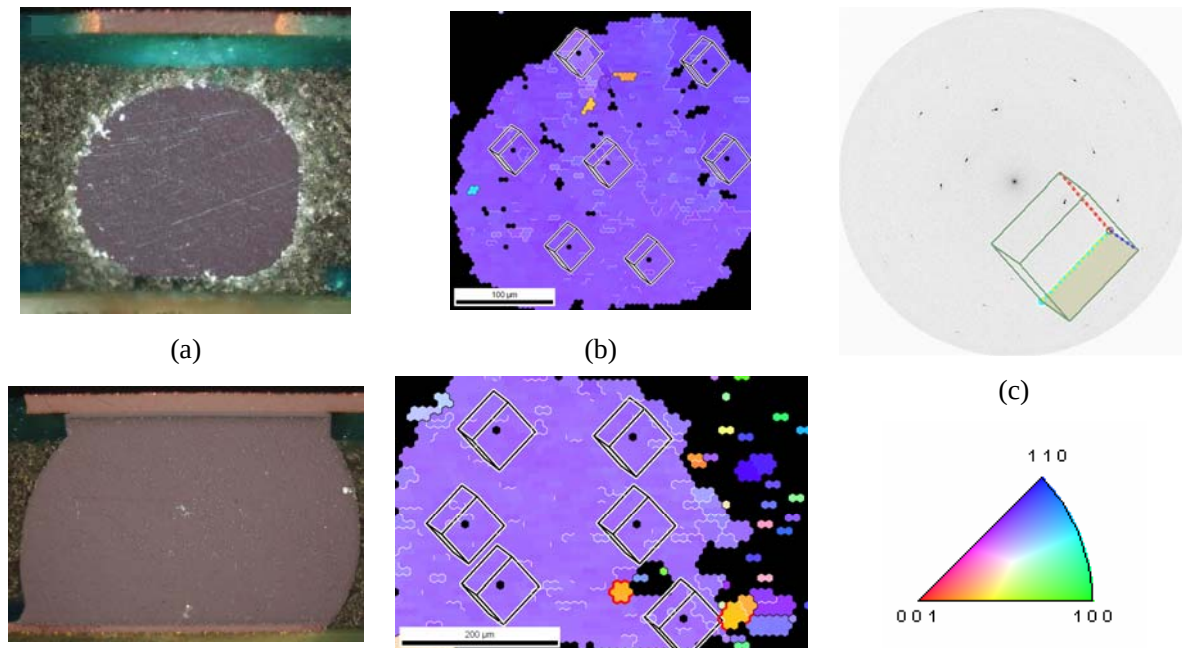


Figure 8. (a) XPM images of un-aged Ball-5 after serial sections; (b) corresponding OIM scans, (c) X-ray diffraction pattern, with one orientation present in the diffraction pattern in agreement with OIM results.

two images with different viewing perspectives are required to identify all of the boundaries. OIM has the advantage in that pole figures provide the most clear indication of the number of crystal orientations, because a (001) pole figure will identify all distinct grains, because there is only one 001 direction in a Sn crystal (but it has the disadvantage of requiring more careful specimen preparation and costlier microscope time). Clearly, both methods can be used to gain information about microstructure evolution that is meaningful, if interpreted carefully.

***In-situ* Synchrotron X-ray diffraction**

The methods described above to identify microstructure evolution in a solder joint, are statistical, and destructive. To gain non-destructive information about microstructure evolution in a thermal cycled joint so that history of deformation can be identified, it is possible to measure changes in crystal orientations and volume fraction using synchrotron x-ray diffraction [8-12]. Fig. 1 shows an image of a single slice of a PBGA package along the outer edge of the package used to study crystal orientation and internal strains.

The sample was examined in beam line 6ID-D at the Advanced Photon Source at Argonne National Laboratory. Each joint was exposed to the beam and the diffraction pattern was collected using a 16 bit image plate. An 8 bit weak-peak image of two such diffraction patterns are shown in Figs. 7 and 8 (further experimental details can be found in ref. [14]). Diffraction patterns show around 5-25 strong peaks, depending on the orientations of Sn present in the joint. To assess this method of characterization, one sample is being destructively serially sectioned to identify the 3-D microstructure using polarized light microscopy, and OIM maps are acquired on selected XPM sections. Two sections each of two balls are illustrated in Figs. 7 and 8. In many balls, there are 3 orientations present, which are made evident on the OIM maps.

Indexing of the diffraction pattern is challenging, as it is known that many balls have more than one orientation. The peaks are too strong and sharp to index with software developed for polycrystals, so an analysis technique under development to differentiate and index overlapping diffraction pattern for transmission electron microscopy was used [15]. The diffraction patterns for every Sn orientation in a 2° polar orientation space are predicted, and then a measured

diffraction pattern is compared with these solutions until a good match is found. To date, this program has been used with 8-bit images that favor weak peaks of the diffraction pattern. Of 14 balls, 11 show complete agreement with OIM results, with two examples illustrated in Figs. 7 and 8, where one of the three orientations in the OIM maps was not detected from indexing the diffraction pattern. Using weak peak images, it is not possible to estimate volume fractions of each orientation, and it is clear that some orientations are not detected (and some erroneous orientations may be identified). Future development of the program to use all 16 bits of intensity data will improve identification of orientations, as well as volume fraction information to be extracted from the measured data, and will be reported in a future paper.

Strain analysis and microstructure evolution

Recent work has illustrated how details of the peaks from the 16 bit diffraction pattern can be analyzed to identify internal strains as a function of thermal cycling temperature and cycle number. This work is briefly summarized here and the reader is referred to ref [14] for details and figures. In a center row of the same PBGA package shown in Fig. 1, where the silicon die is present, samples were measured as indicated above. The sample was then cycled for 450 cycles, and then re-examined in the synchrotron beam. The diffraction patterns obtained after 450 cycles showed nearly the same diffraction patterns, but in all joints, there were some peaks that were weakened or disappeared, and others that were strengthened or appeared. This indicates that after 450 cycles, there are changes in orientation and/or volume fraction of measured orientations.

In another experiment with a center slice of a single row from a 44x44 ball FCBGA package, diffraction patterns were obtained about every 5 minutes during a 70 minute thermal cycle with 15 minute hold times at 0 and 100°C. The strain evolution was determined for a single crystal joint during the first cycle and after the 50th cycle, and the strain history was *quantitatively* different but *qualitatively* similar during the 1st and 50th cycles. These measurements were even able to resolve small strain reductions during the hold times. After 50 cycles, the diffraction pattern did not show obvious orientation or intensity differences such as those observed after 450 cycles, suggesting that

changes in microstructure do not occur quickly, but that these changes are enabled later. It is known that the small Ag₃Sn intermetallic particles coarsen with time during thermal cycling, and the small size is more effective at pinning grain boundaries than large particles that develop as coarsening occurs by Ostwald ripening. It is known that the hardness decreases with thermal cycling, which can be attributed to particle coarsening [6,16]). The effective pinning of grain boundaries by small precipitates may account for the lack of measurable crystal orientation change during the first 50 cycles. Further analysis of microstructure evolution in these thermally cycled specimens is under way.

Conclusions

Crossed polarized light microscopy and orientation imaging microscopy provide similar statistical information, but the details differ due to the lack of accuracy in obtaining true grain orientation information that is inherent with optical microscopy. When the same joints were measured using synchrotron x-ray measurements and OIM measurements, good agreement is found, though there is still some uncertainty in the developing algorithms used to extract multiple orientations from overlapping diffraction patterns from a multi-crystal. Detailed analysis of peak intensities, shapes and shifts provide internal strain information from *in-situ* tests of thermally cycled specimens, and has shown that grain orientation changes develop after 450 cycles, but changes were not resolved after the first 50 cycles.

Acknowledgements

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