ESRF	**Experiment title:** Probing Sn whisker growth mechanisms	**Experiment number**: MA-1724
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Report:

This report describes the experiment and initial results for MA-1724 performed in July 2013 on BM32. This experiment investigated the use of 2D and 3D (by wire-scanning: differential aperture x-ray microscopy, DAXM) Laue diffraction to measure grain distributions and grain strains around “tin whiskers” growing from a tin layer over a copper substrate. The objective was to understand the driving forces for tin whisker growth to subsequently advance the concurrrent developments of a theoretical material model of such systems.

Measurements were performed on a sample of tin-plated copper that had been aged for about 5 weeks such that tin whiskers (single crystal tin “protrusions” from the sample surface) could be observed under an optical microscope. A region of the sample containgin a single evident tin whisker was identified for investigation using the optical microscope on the BM32 measurement stage. Using a 0.5 x 0.5 μm^2 beam, an area of 10x12 μm^2 around the whisker was scanned using 2D Laue diffraction to provide an initial mapping of the crystal structure. From the resultant data at each point, it was demonstrated that clear Laue diffraction patterns could be measured through the thickness of the tin layer down to, and including, the copper substrate. Furthermore, initial analyses showed that the Laue patterns associated with the tin grains could be identified and fitted for the local cell parameters. Figure 1 shows a map of the c-axes of the tin grains in this region, based on fitting of the Laue patterns (using the BM32 LaueTools software) to the strongest intensity family of diffraction spots; making the assumption that the strongest intensities correspond to the grain nearest the surface of the sample at each point. Figure 1 also shows the principal grain-strains at each of these points corresponding to the fitted cell parameters relative to an ideal unit cell. Clear regions of consistent c-axis orientations can be seen that probably reflect the tin grain structure at the surface. Furthermore, the strain maps show correlations of the strain field with the grain shapes in the c-axis plot and in some cases strain gradients can be seen within grains. These strain gradients could be related to the formation of a tin whisker (the approximate position of the main whisker is at x=6 μm and y=4 μm).

The results described above are preliminary, but already indicate that Laue Diffraction can be used to investigate the crystalography and strain through the thickness of the tin layer down to the copper substrate. Furthermore, indications of the intermetallic compound that forms between the copper and the tin could be seen in the diffraction data, which suggests that this interface might be mapped, in future experiments, and its topography could then be related to the strains in the tin grains (however full crystallographic and strain information may be difficult to extract for this phase).

In addition to the 2D mapping, two 3D-Laue wire scanning line scans were made (one in the y-direction, 10 μm length with 1 μm spacing, and one in the x-direction, 10.5 μm length at 0.7 μm spacing through the centre of the images in Figure 1). This wire scanning took approximately 1 hour/point. Analysis of the 3D crystallography and strains based on these data is under way; early results suggest that depth resolved information can be derived to correctly separate the signals from different depths (including seperating the diffractions from whiskers from those from the grains in the sample).

The described 3D-Laue wire scanning can only provide the deviatoric strains in the sample, but the phenomenon of interest is strongly linked to volumetric changes with the formation of the intermetallic compound. Therefore it is desirable to quantify the volumetric strain component of the deformation. An attempt was made to do this using a diamond momnochromator, this was however unsuccesful do to issues setting up the system.

Whilst promising results were achieved from this experiment, the experiment also revealed the complexity of the sample, a complexity that makes for a more difficult interpretation of the 3D resolved crystal strain and strain-gradient data in relation to whisker growth. This complexity is due to the fact that a significant number of whiskers appear to have formed and, whilst a zone with a relatively isolated whisker has been selected, a more in-depth analysis will be faciltiated by ensuring that there are minimal interactions between whiskers. Analysis of the optical microsopy and x-ray diffraction data suggests that this complexity might arise from the grain size in the copper substrate and also from the tin-surface preparation. For future experiments the sample preparation will be optimised, based on the current results and insight gained, to provide samples with spatially isolated whiskers. With such samples it should be possible to develop a a clear picture of the strain fields around individual whiskers (the existing data have proven the experiment concept and the possibiltiy of extracting good crystalographic and strain results from the Laue diffraction data, but greater insight to the processes requires a simplification of the mechanical system). Furthermore, measurements of the volumetric strain must be made, as this is an essential part of the phenomena of interest.

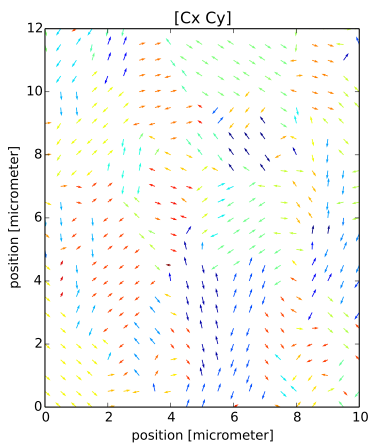

Figure 1: First results from analysis of 2D Laue scanning taking the strongest intensity family of diffraction spots (based on a preliminary assumption that these correspond to the grain nearest the surface) and fitting, using LaueTools, to determine the tin cell parameters at each spatial location. From these cell parameters the crystalographic orientations have been determined; left shows a plot of the c-axis orientations (colours indicate the out of plane angle). Furthermore, grain strains have been determined et each scan point and the principal components of the deviatoric strain tensor are presented below.