ESRF	**Experiment title:** 3D measurement of crystal orientations and strain around growing tin whiskers	**Experiment number:** MA-3359
Beamline: BM32	**Date of experiment:** from: 17 May 2017 to: 23 May 2017	**Date of report:** 29 August 2017
Shifts: 18	**Local contact(s):** Jean-Sébastien Micha & Odile Robach	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):
Johan Hektor*: Division of Solid Mechanics, Lund University, Sweden
Matti Ristinmaa*: Division of Solid Mechanics, Lund University, Sweden
Stephen Hall: Division of Solid Mechanics, Lund University, Sweden
Håkan Hallberg: Division of Solid Mechanics, Lund University, Sweden
Srinivasan Iyengar: Division of Materials Engineering, Lund University, Sweden

Report:
This report describes the experiment MA-3359 performed on BM32 during May 2017 and during in-house beamtime in October 2016. The aim of the experiment was to characterize the grain structure and the strain field around a tin whisker (a tin single crystal growing from the surface) in 3D. The general objective is to gain better understanding of the mechanisms involved in whisker growth and to aid in the development of theoretical and numerical models of the phenomena. The measurements were made using the Laue microdiffraction setup on BM32.
The studied sample consisted of a 6.5 µm thick layer of Sn deposited on a Cu substrate by means of electron beam evaporation. The sample was aged in ambient conditions for approximately four months prior to the experiment in October 2016 (11 months for the experiment in May 2017). The aging was long enough for whiskers to nucleate and grow to lengths of approximately 20 µm. A suitable region containing an isolated whisker was located using an electron microscope before the experiment. The same region was located using the optical microscope on the beamline. The same whisker was scanned in both experiments. During the in-house experiment we used Differential Apperture X-ray Microscopy (DAXM) to characterize the crystal orientation and deviatoric strain field around the selected whisker in 3D. Due to DAXM being a time consuming technique it was only possible to obtain 3D resolution along two lines, 16 µm and 8 µm long, intersecting in the whisker root. In fig. 1a and fig. 1b reconstructions of the grain orientation and the effective elastic deviatoric strain around the whisker is shown.

These results are presented in a paper currently being reviewed for publication in Scripta Materialia. Given the successful in-house experiment we decided to use our beamtime in May to measure the volumetric strain around the whisker (normally this can not be done using Laue diffraction because of the white beam) rather than repeating DAXM measurements. By measuring the energy of at least one Bragg peak the absolute values of the lattice parameter can be obtained. This allows the full (deviatoric+volumetric) strain tensor to be determined. On BM32 this is done using reverse monochromatic filtering from a diamond plate placed upstream of the sample. By scanning the filter over a range of angles ($-48° \pm 1.25°$) the intensity of the diffraction peaks as a function of the filter angle is measured. Figure 1c shows the intensity of 10 diffraction peaks belonging to a Sn grain close to the whisker as a function of the filter angle. The dip in peak intensity is used, together with a model describing which energies are removed from the beam at each filter angle, to determine the energies of the diffraction peaks from the sample. Once the energy of at least one peaks is known the full strain tensor can be determined. This data is currently being analyzed. Whiskers are believed to grow due to gradients in pressure (directly linked to the volumetric strain) driving diffusion of Sn atoms towards specific sites on the sample surface. Determining the volumetric strain is therefore a key ingredient in understanding the whisker growth phenomena.
A new, much faster, detector has recently been installed on BM32. This makes it possible to use DAXM to build a proper 3D map. It also makes simultaneous use of the DAXM and diamond filtering techniques more realistic.

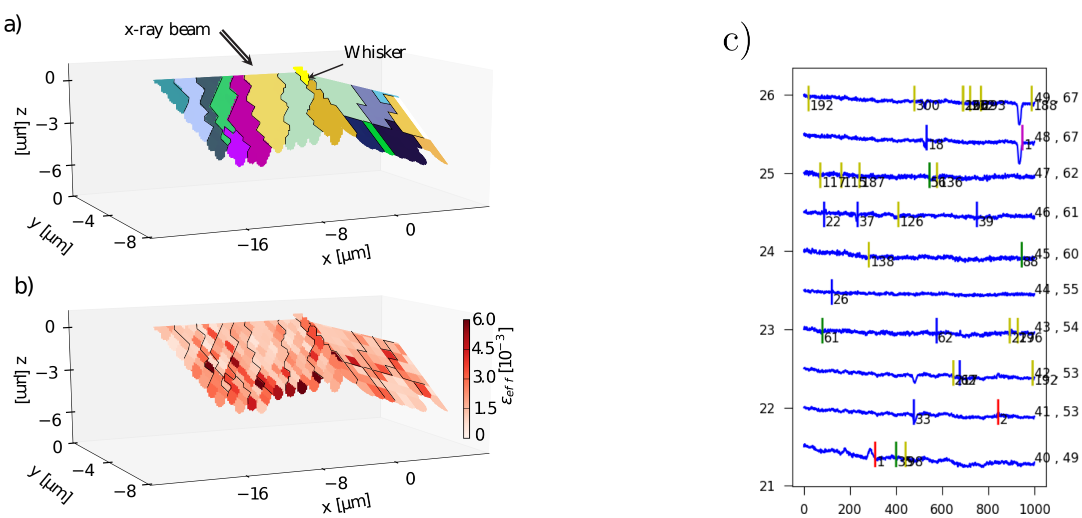

Figure 1: (a): 3D reconstruction of grain orientation around the whisker. (b): 3D reconstruction of the effective elastic deviatoric strain around the whisker. (c): Normalized peak intensity as a function of filter angle for some of the Bragg reflections from a Sn grain near the whisker. The plot also shows theoretical preditions of where the dips in intensity should be located (vertical lines).