Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF

	Experiment title: **In-situ X-ray Diffraction Contrast Tomography Analysis of Grain Boundary Sliding in a Tin-Bismuth Alloy**	**Experiment number**: MA2895
Beamline: ID11	**Date of experiment**: from: 06/07/2016 to: 12/07/2016	**Date of report**: 10/09/2016
Shifts: 18	**Local contact(s)**: Dr Wolfgang Ludwig	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Junnan Jiang[a]*, Samuel A. Mcdonald[b]*, Wolfgang Ludwig[c,d]*,
Philip J. Withers[b], Richard I. Todd[a], Angus J. Wilkinson[a]

[a] Department of Materials, The University of Oxford, Park Road, Oxford OX1 3PH, UK
[b] Henry Moseley X-ray Imaging Facility, School of Materials, The University of Manchester, Manchester, M13 9PL, UK
[c] ESRF – The European Synchrotron, 71 Rue des Martyrs – CS 40220, 38043 Grenoble, France
[d] MATEIS, INSA de Lyon, Université de Lyon, 7 Avenue Jean Capelle, 69621 Villeurbanne, France

Report:

Grain boundary sliding is one of the dominant deformation mechanisms of superplasticity and high temperature creep. Despite decades of experimental research, the mechanisms of grain boundary sliding still remain controversial. Conventional techniques such as SEM-EBSD in-situ experiments have shown grain neighbour switching geometry phenomena in 2D sections using surface marker grids. However, the surface behaviour does not necessarily reflect the bulk of the sample.

In the ID11 beamline, we have carried out a novel experiment in which diffraction contrast tomography (DCT) is exploited to investigate grain boundary sliding behaviour in 3D in the bulk material. DCT scans can be reconstructed to 3D maps illustrating the evolution of the grain structure and orientation. Constant displacement rate tensile test was carried out to induce deformation in the tin-bismuth sample. A low strain rate is applied so as to ensure that grain boundary sliding is the dominant deformation mechanism. Conventional phase contrast tomography (PCT) was also conducted to monitor and track the movements and shape changes of the specimen during tensile tests.

Specimens with various gauge dimensions have been tested with DCT scans. It was found that specimens with a narrow (200 μm width) gauge allowed stable mechanical testing and suitable number of grains for DCT scans. Figure 1 has shown an SEM image of the tensile specimen. The specimen underwent tensile deformation at a strain rate of 10^{-6} controlled by a piezo actuator. PCT scans were taken every 0.3% strain to monitor the change in the length with tensile deformation. For around every 5% strain, a diffraction contrast tomography (DCT) scan was taken in a small section of the gauge region. From the PCT scans, it was found that after 20% strain, the gauge started to have necking as shown in Figure 2. This leads to the necessity to carry out another test with improved experiment setup and surface finish of the specimen.

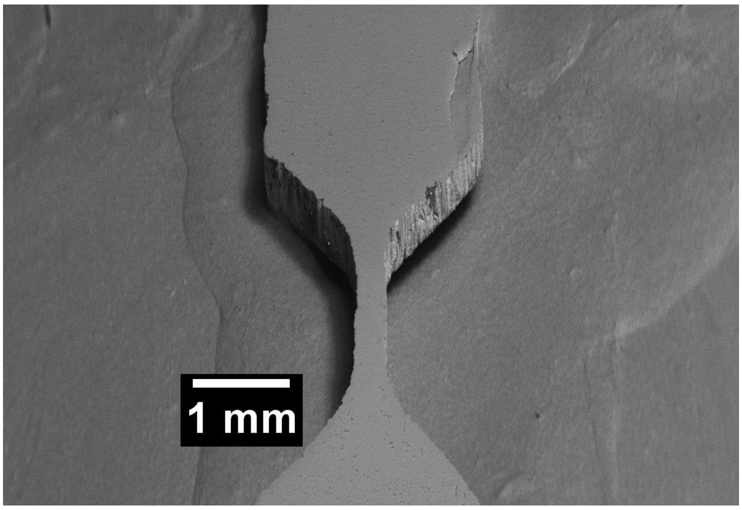

Figure 1. SEM image of the tensile specimen.

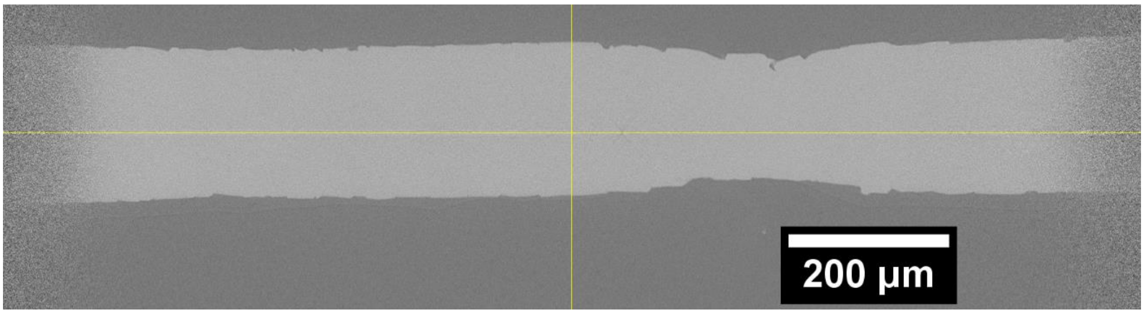

Figure 2. A phase contrast tomography (PCT) scan, showing the gauge region with necking locally occurring.

We have carried out preliminary analysis on the DCT data. The reconstructed 2D maps of the initial state (no deformation) and after 20% strain are shown in Figure 3 and Figure 4, respectively. Even though the two maps are from different slices in the gauge region, they illustrated the feasibility of the DCT reconstruction process. In Figure 4, most of the grains are able to be reconstructed even after a large strain of 20% on the sample. This is because grain boundary sliding contributed to most of the deformation, while dislocation motion plasticity took place in some individual grains. The 3D map reconstruction work is still in progress. When the 3D map is available, we would be able to track individual grains during the tensile test process to capture grain neighbour switching phenomenon.

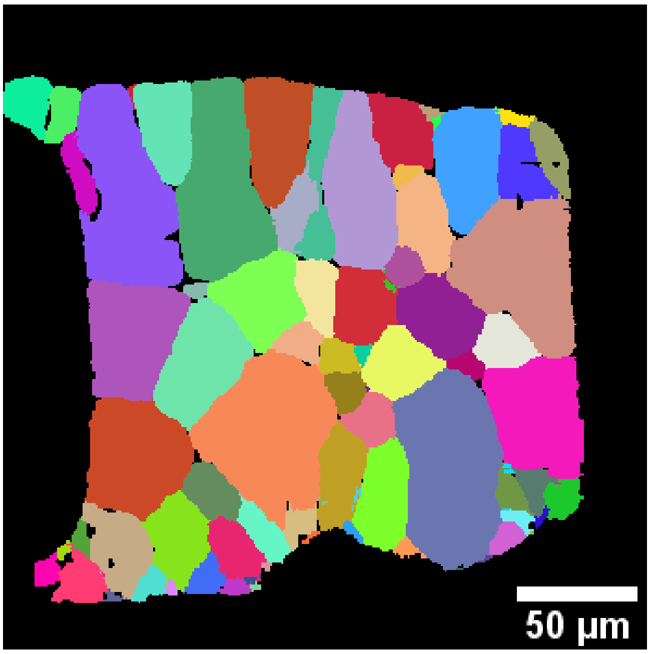

Figure 3. Reconstructed 2D map of a section of the gauge region at the initial state (no deformation).

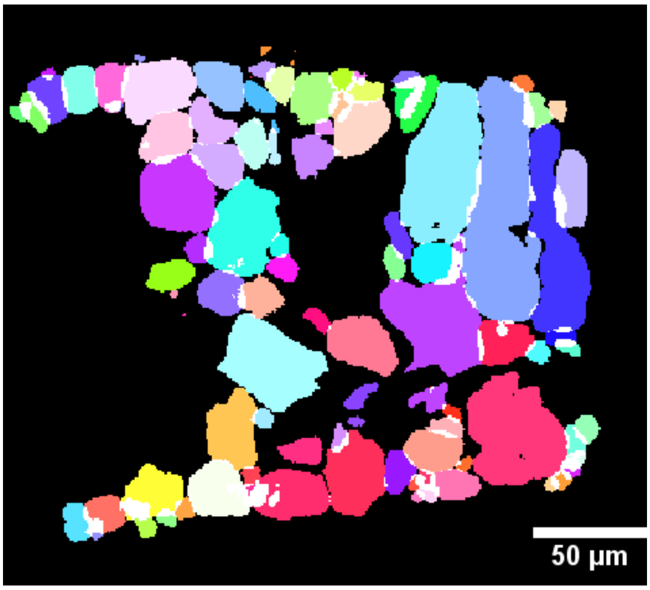

Figure 4. Reconstructed 2D map of a different section of the gauge region after 20% strain.

In order to capture grain neighbour switching phenomenon in 3D, the specimen has to undergo around 50% strain. This requires the following improvement to prevent the specimen from necking and ensure that grain boundary sliding is the dominant deformation mechanism:

1. Finer grain size: the previous experiment (MA2895) was on specimens with 30 microns grains, which can be further reduced. Finer grain sizes will promote grain boundary sliding, enabling better observation of grain neighbour switching phenomena. So the point is to adjust the heat treatment recipe to prevent grain growth and get smaller grains than the previous experiment.
2. The amount of contribution of grain boundary sliding to the total deformation is dependent on the strain rate. For the previous test on the sample with 30 μm grains, a strain rate of 10^{-6} could enabled grain boundary sliding to occur. If we could obtain smaller grains (10 μm) after heat treatment, we can speed up the tests to 2×10^{-6}, which takes less time to carry out experiments. An additional beamtime of four days would be enough to carry out another in-situ tensile test with 10 DCT scans (testing to 50% strain with DCT scans at 5% strain intervals).
3. Better surface finish: electro-polishing may be applied to clean up the gauge region of the specimen. This will prevent necking from initiating at the rough surfaces.