EUROPEAN SYNCHROTRON RADIATION FACILITY

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ESRF

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 using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now 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: Analysis of copper particle rotations during sintering applying SCT of focused-ion-beam marked particles**	**Experiment number**: MA - 1116
Beamline: 15A	**Date of experiment**: from: 8.12.2010 to: 13.12.2010	**Date of report**: 25.2.2011
Shifts: 15	**Local contact(s)**: Marco Di Michiel	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Introduction

Sintering of metals is usually explained by a two particle model which describes neck growth and centre approach by surface energy minimisation.In 3D samples in addition to the particle centre approach, cooperative material transport occurs - the movements of entire particles relative to surrounding particles, for example particle rotations [1]. These rotations are usually attributed to tensile stresses due to asymmetric interparticle contacts [2], inhomogeneous centre approaches [2] and the need to form low energy grain boundaries [3]. The existence of particle rotations was shown by Herrmann and Gleiter [4] using a sphere plate model, by Wieters using rows of spheres [1] and by Exner using 2D arrangements of monocrystalline spherical copper particles [5]. However, inside 3D specimens cooperative material transport was rarely investigated.
Computer tomography, especially high resolution synchrotron computer tomography (SCT), is a suitable method to investigate particle movements in 3D samples. First results were obtained by ex-situ [6, 7] and in-sit analyses [8]. These experiments proved the existence of particle rearrangements in the 3D case.
Furthermore, it became clear that the rotation of particles in 3D specimens can be analysed quantitatively using high resolution SCT in combination with photogrammetric image analysis. As shown in our previous experiment MA-290 and at BESSY II, it has become possible to measure rotation processes inside of 3D specimens.

Results

We successfully measured samples of the same material prepared in an identical way on PREVIOUS occasions (see introduction and report MA-290). The new experiment was supposed to allow for a more detailed analysis of the sintering processes. We prepared single crystal copper spheres with FIB marker holes to follow intrinsic rotations of the particles. THIS time the quality of the 3D images is not sufficient for an image analysis.
The only differences (beside the FIB marker holes) to previous experiments by us:
1. this time we used glue recommended by the local contact, (as we could use it (only) once before and had a very good experience of its quality),
2. extended sintering profile.
Both differences should improve the quality of the measurement. According to Dr. Marco Di Michiel the problem seems to be related to the presence of the in-situ furnace at high temperatures (provided by the ESRF) itself and the situation was improved by M. di Michiel, but during the subsequent measurement of samples it turned out to be not sufficiently resolved as was even present in samples without FIB marker holes, i.e. samples identical to the ones used in previous successful experiments. A more detailed analysis was not possible in the allocated beam time.

References

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