EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

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.

	Experiment title: Particle morphology, displacement, rotation and grain size evolution during powder compaction and sintering by diffraction contrast tomography	**Experiment number**: MA 1139
Beamline: ID11	**Date of experiment**: from: 18/10/2011 to: 23/10/2011	**Date of report**: 05/04/2012
Shifts: 15	**Local contact(s)**: Andrew King	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Samuel A. McDonald[1]*, Andrew King[2]*, Stefan Schmiederer[1,2]*, Steven Van Boxel[1]*, Philip J. Withers[1]

[1] Manchester X-ray Imaging Facility, School of Materials, University of Manchester, Grosvenor Street, Manchester, M1 7HS, UK
[2] European Synchrotron Radiation Facility, Grenoble, France

Report:

Sintering of loose or compacted granular bodies is an important final step in the production of components via the powder metallurgical route. Transport processes, motions such as translation and rotation of particles, have been shown to be important in contributing to the early stages of sintering. We have carried out a novel experiment in which individual particle displacements and rotations are inferred from mapping the shapes and crystallographic orientations of the individual crystallite grains contained within polycrystalline copper spheres, in three-dimensions and *in situ* during a sintering heat treatment. Conventional absorption contrast tomography was combined with diffraction contrast tomography (DCT); the former to monitor and track the lateral movements and shape changes of the particles, while exploiting DCT to map the grain shapes and orientations of the polycrystalline particles.

Figure 1 shows two 3-D isosurface representations of the copper particles, from absorption contrast tomography scans, in their loose as-filled state within the 0.5 mm diameter capillary and after a sintering heat treatment. The magnified images for the two steps illustrate the development of interparticle bonds or necks between neighbouring particles, indicated by the corresponding numbers in the two images for those on the cut surface of the cropped section. The arrows indicate necks formed between particles further into the interior of the cropped section and so showing their outer surfaces. It is the characteristics of the particle motions associated with and contributing to these shape changes occurring during sintering that we are interested in identifying. Figure 2 illustrates the information we are able to extract from the DCT data, with a spatial resolution of ~ 2 μm and grain orientation resolution of < 0.1 °. Changes in grain orientation reveal the particle rotations (Figure 1(a)), while the ability to characterise grain boundaries both between and within the particles is enabled (Figure 1(c)). In following the particle rotations over several steps during the early stages of a sintering heat treatment we are able to extract and plot statistical data such as the mean rotation speed of individual particles and the average of groups of particles, as shown in Figure 3. This infers the rotation speed to decrease, initially quite quickly and then getting more shallow, as sintering time progresses.

Further analysis of the data will focus on extracting specific characteristics for the driving forces for particle rearrangement, for example the mechanisms behind why some particles are more ‘active’ than others and the relationship to the number of contacting neighbours.

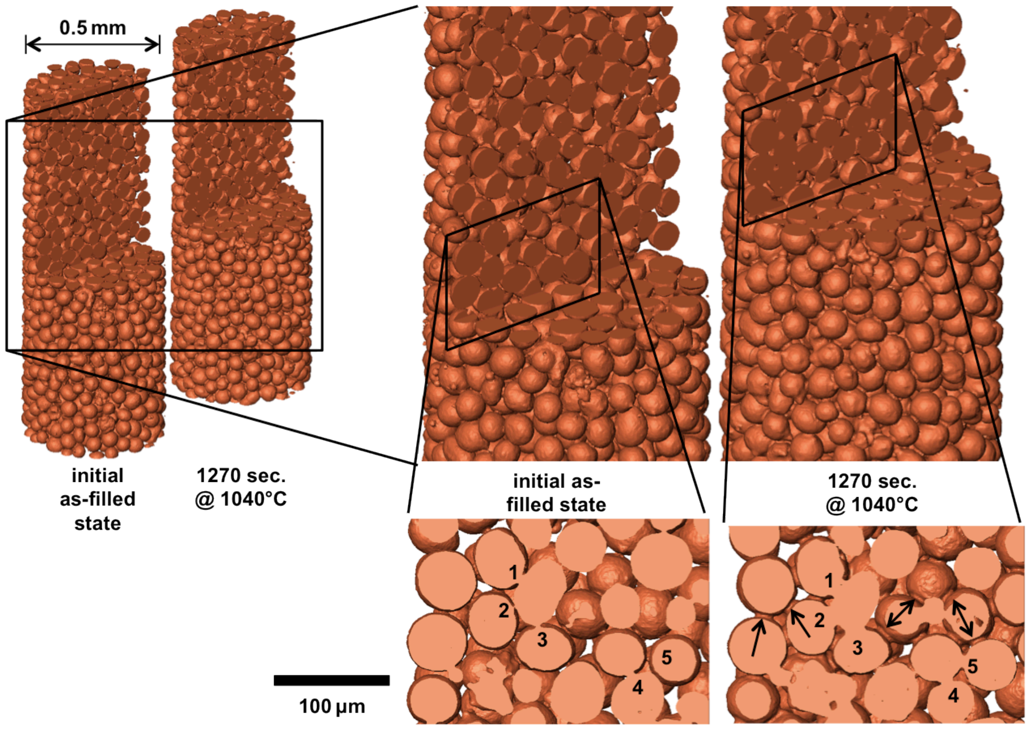

Figure 1. 3-D isosurface representations of the Cu particles in the initial as-filled state within the capillary, and after a sintering heat treatment of 1270 seconds at 1040°C. The arrows and corresponding numbers shown in the magnified images (bottom) indicate neck formation at interparticle contacts.

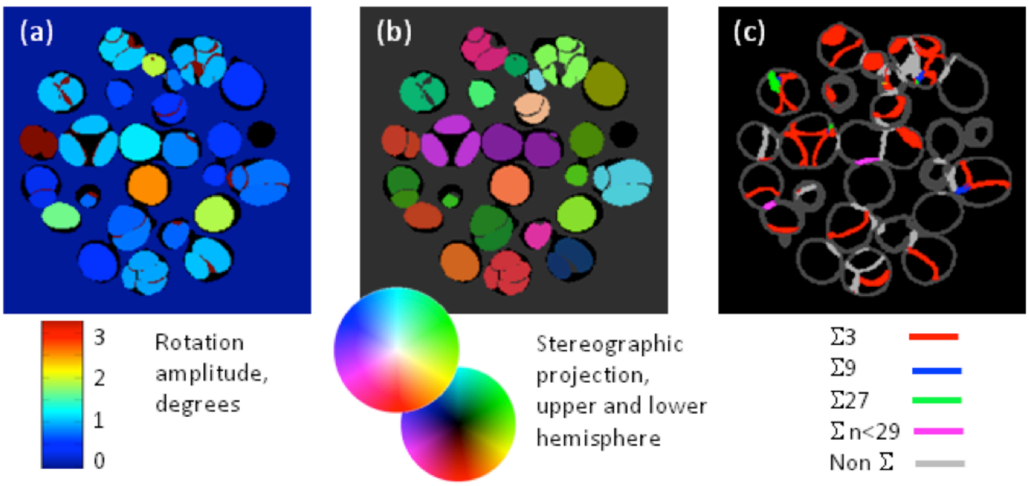

Figure 2. Slice cut through a DCT dataset and labelled with (a) sphere rotation amplitudes; (b) rotation axes; (c) grain boundary types.

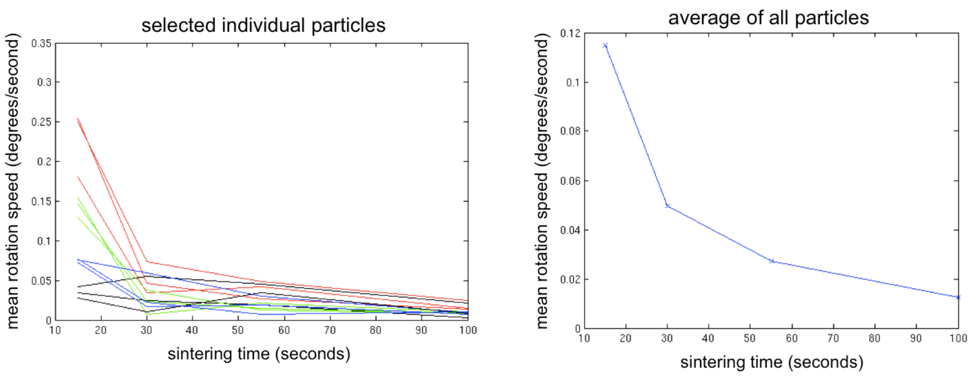

Figure 3. Plots showing the mean rotation speed of (left) selected individual particles and (right) the average of all particles.