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 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.

	Experiment title: Effect of particle size and shape on the internal microstructural evolution of Cu powder during compaction and sintering	**Experiment number**: MA-204
Beamline: ID15A	**Date of experiment**: from: 29/11/06 to: 02/12/06	**Date of report**: 01/09/07
Shifts: 9	**Local contact(s)**: Dr. Marco Di Michiel	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

* Dr. Samuel A. McDonald, University of Manchester, UK
Prof. Alan C. F. Cocks, University of Oxford, UK
Prof. Philip J. Withers, University of Manchester, UK

Report:

X-ray microtomography has been used on ID15, ESRF, to monitor the consolidation of loose powder particles during sintering in three dimensions. Two different copper powders, in terms of their manufacturing route and particle size, were investigated and subjected to a thermal cycle which included heating up to a temperature of 800 °C at a rate of 40 °C min^{-1} followed by a dwell time of 20 mins and then heating up to a maximum temperature of 1050 °C. Cooling of the sample occurred at a rate of 50 °C min^{-1} and the complete cycle was over a timescale of 4 hours. Figures 1 and 2 compare the microstructural evoluton of the two powders in the form of a sequence of 2D greyscale vertical slices extracted from the full diameter of the reconstructed volumes of the capillary holding the powder.

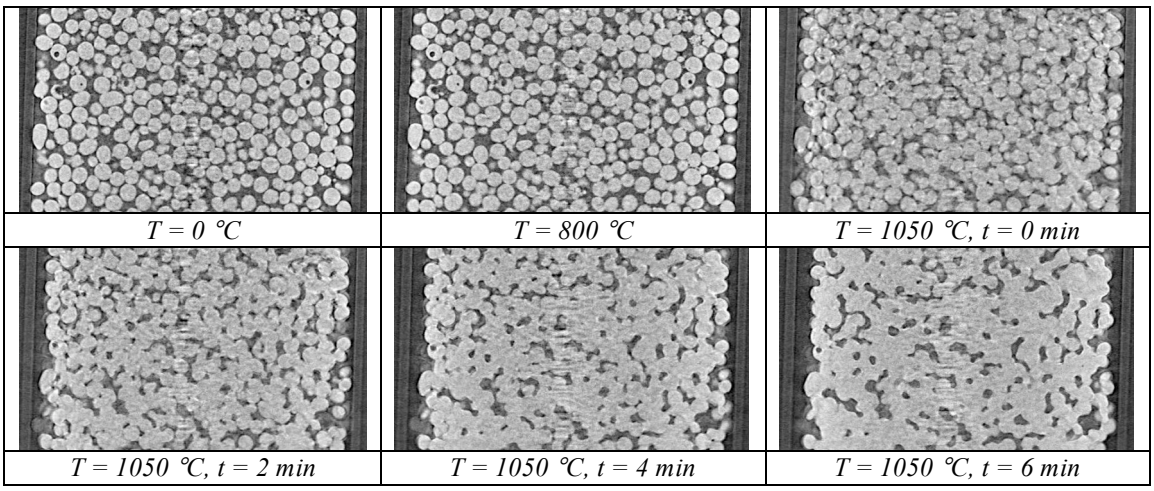

Figure 1. Showing the microstructural evolution during sintering of Cu powder 1 (60 μm particle size).

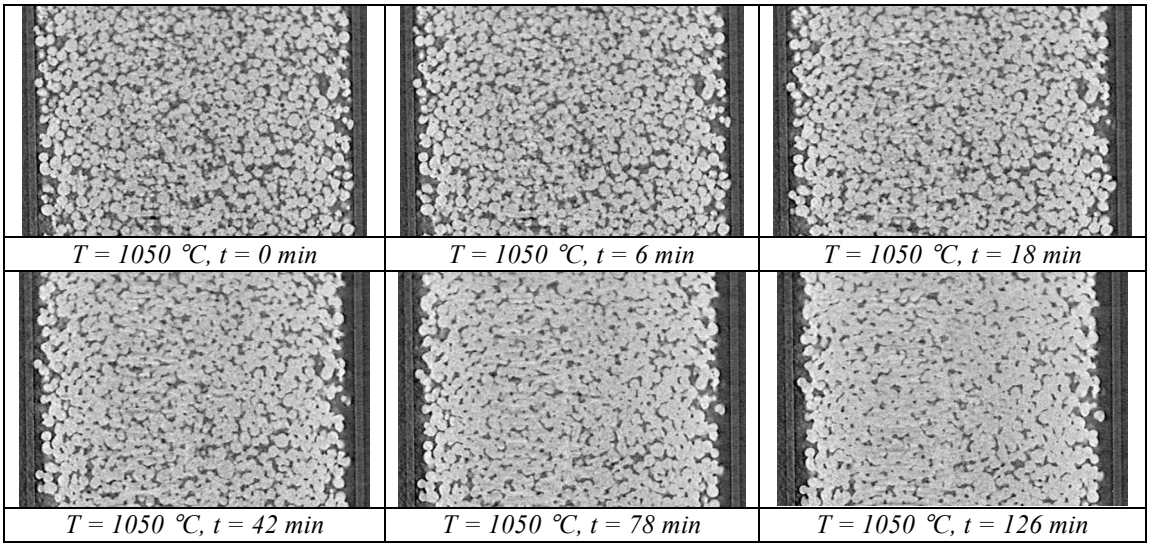

Figure 2. Showing the microstructural evolution during sintering of Cu powder 2 (35 μm particle size).

The densification process for the two bodies of powder material is illustrated, and how repacking of the particles has occurred to give a reduction in total porosity. It is clear that the process is much faster for powder 1 (Figure 1), observed simply from the fact that after just 6 mins at maximum temperature there is no individual particle structure, while necks are only just starting to form between particles of powder 2. The particles of powder 2 tended to form agglomerates after filling of the capillary, with large regions of pore space, making it more difficult to close them during sintering [1]. The particles of powder 1 were much more evenly distributed and uniformly packed.

Image correlation has been used to monitor the movement of the particles during the process (shown for powder 2 in Figure 3). Initially, contraction of the powder body occurs laterally (after 6 mins) towards the centre of the capillary, while later on (after 12 mins) there is some vertical consolidation. After 18 mins the vertical movement is much greater and it is clear that shrinkage of the particles is occurring towards a central point. Analysis of smaller sections of the tomographic volumes shows that particle rearrangement, due to the loose nature of the particles prior to sintering, clearly contributes to the densification process. Necks then form and grow between the contacting particles. Isolated pores are then left which, as shown by image correlation, have some rotation and rearrangement of their own as they shrink. Detailed image correlation maps are being used to compare to, and help develop, models of the sintering process [1, 2].

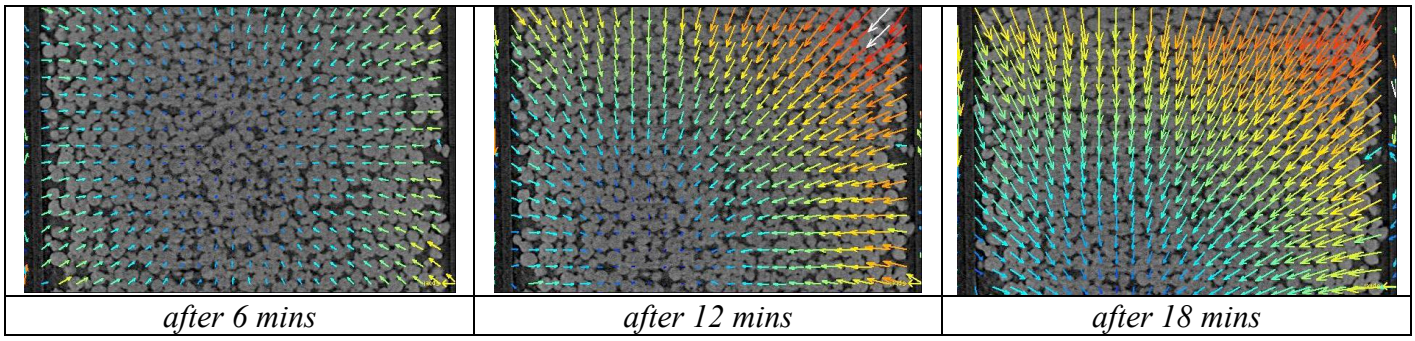

Figure 3. Vector displacement maps extracted from image correlation calculations performed on sequences of slices through the sintering cycle of Cu powder 2.

Further work will concentrate on performing such an experiment under load, as is done in industrial cases and particularly important for the study of irregular shaped particles, will enable the relationship between initial state, applied stress and porosity evolution to be determined. This will further aid the development of new 3D continuum micromechanical models for the sintering of powders.

[1] A.C.F. Cocks, 'Constitutive modelling of powder compaction and sintering', *Progress in Materials Science,* 2001, **46**, 201.
[2] J. Pan, 'Modelling sintering at different length scales', *Int. Mat. Rev.*, 2003, **48**, 69.