ESRF	**Experiment title:** In situ constrained sintering of aggregated and layered ceramic powders	**Experiment number:** MA-3254
Beamline: ID16B-NA	**Date of experiment:** from: 2016/11/18 to: 2016/11/21	**Date of report:** 2017/08/23
Shifts: 9	**Local contact(s):** Julie Villanova	*Received at ESRF:*

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Report:

Aims:. The general aim is the understanding of microstructural evolution of powders during constrained sintering. A particular interest was put on:

- liquid phase sintering for these experiments.
- glass beads sintering
- MCF layers sintering for SOFC applications

Experiments: We have looked at the in-situ sintering of three systems, glass beads and Cu-Sn powders (liquid phase sintering) and MCF layers.

Soda-lime glass particles were introduced in a quartz capillary having a 10 µm thick wall and a 100 µm inner diameter, which was glued on top of a mullite rod. An isothermal treatment at 670°C was imposed on the system. A total of 190 scans, lasting 33sec., were recorded during one hour and forty five minutes to follow the 3D microstructure evolution. The data reconstruction is performed in two steps for each scan: (i) single-distance phase-retrieval calculation on the projections and (ii) tomographic reconstruction using the retrieved phase maps. The phase retrieval calculation is based on a Paganin-like approach by assuming a homogenous ratio δ/β in the sample.

Cu-Sn powders were sealed in a capillary with reducing atmosphere and sintered at 700°C.

MCF layers were prepared and sintered at 700°C.

Results: Glass beads (were sintered at high temperature (670°C) and we could follow the evolution of necks between particles (Fig. 1).

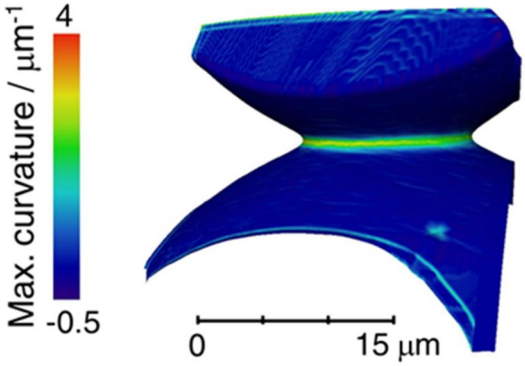

Fig. 1: Evolution of a neck curvature at increasing neck growth, illustrating the enlargement of the curvature distribution.

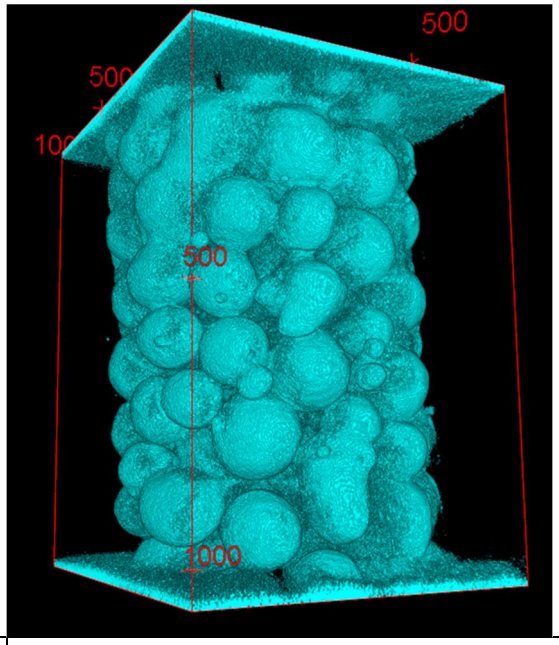

Fig. 2: Cu-Sn sintering

The experiments on Cu-Sn were not fully conclusive as we could not ensure that a liquid phase had formed. Some further work is needed to choose a more suitable powder system for liquid phase sintering. However we have successfully validated the encapsulation method, which allows powders to be sealed in the capillary filled with a reducing atmosphere.
Concerning MCF layers, cracks were observed during the thermal treatment and are still analyzed.