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

Deadlines for submission of Experimental Reports

Experimental reports must be submitted within the period of 3 months after the end of the experiment.

Experiment Report supporting a new proposal ("relevant report")

If you are submitting a proposal for a new project, or to continue a project for which you have previously been allocated beam time, you must submit a report on each of your previous measurement(s):

- even on those carried out close to the proposal submission deadline (it can be a *"preliminary report"),*
- even for experiments whose scientific area is different form the scientific area of the new proposal,
- carried out on CRG beamlines.

You must then register the report(s) as "relevant report(s)" in the new application form for beam time.

Deadlines for submitting a report supporting a new proposal

- 1st March Proposal Round - **5th March**
- 10th September Proposal Round - **13th September**

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.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report in English.
- include the experiment number 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: MI-SNAPCHAT – Role of microstructure on the Sn active phase transformations of Sn-Al phase Change Alloys: in-situ real-time tomography**	**Experiment number**: MA-3744
Beamline: ID19	**Date of experiment**: from: 21/2/2018 to: 23/2/2018	**Date of report**: 18/10/2021
Shifts: 4	**Local contact(s)**: Elodie Boller	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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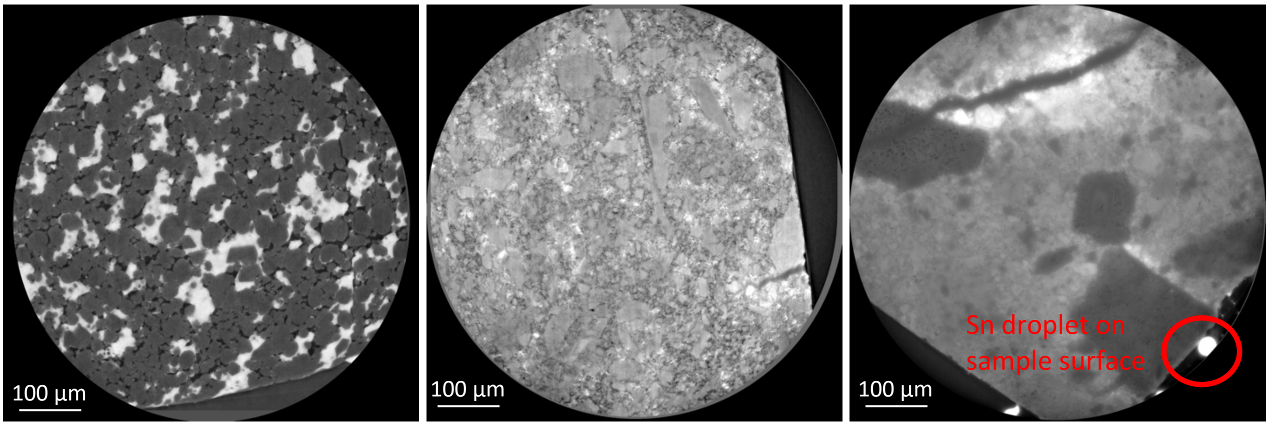
Report:

The investigation focused on the possibility to investigate the in-situ solid/liquid transformations in Al-based Al-Sn Phase Change Alloys (PCA) for Thermal Energy Storage (TES). In PCA, the heat storage/release during heating/cooling cycles occurs by means of active phase transformations and it is affected by microstructural features, properties, composition and morphology of phases as induced by the manufacturing route of the material. The investigated alloys were produced by simple mixing (SM) or ball-milling (BM) powders of the two elements or by means of combined SM and BM, followed by compression and sintering processes. Their microstructures, different from those of conventional casting processes, were promising for PCA as far as the overall material form-stability and molten phase leakage are concerned.

Thus, a series of heat cycles simulating service across the melting temperature of Sn phase (232°C) was performed on different alloys in a furnace developed at ESRF-ID19. Specifically, isochronal heating from RT to 300°C, followed by cooling at the same heating rate was performed. In some cases, the sintering heat treatment at temperature exceeding 500°C was simulated. During each test, high resolution (HR) micro-computed tomography (micro-CT) scans of the specimen were performed in heating and cooling at specific temperatures with 180° sample rotation. The micro-CT reconstructions were prepared by ESRF team, that supplied for each specimen/temperature a set of 2048 slices for further analyses.

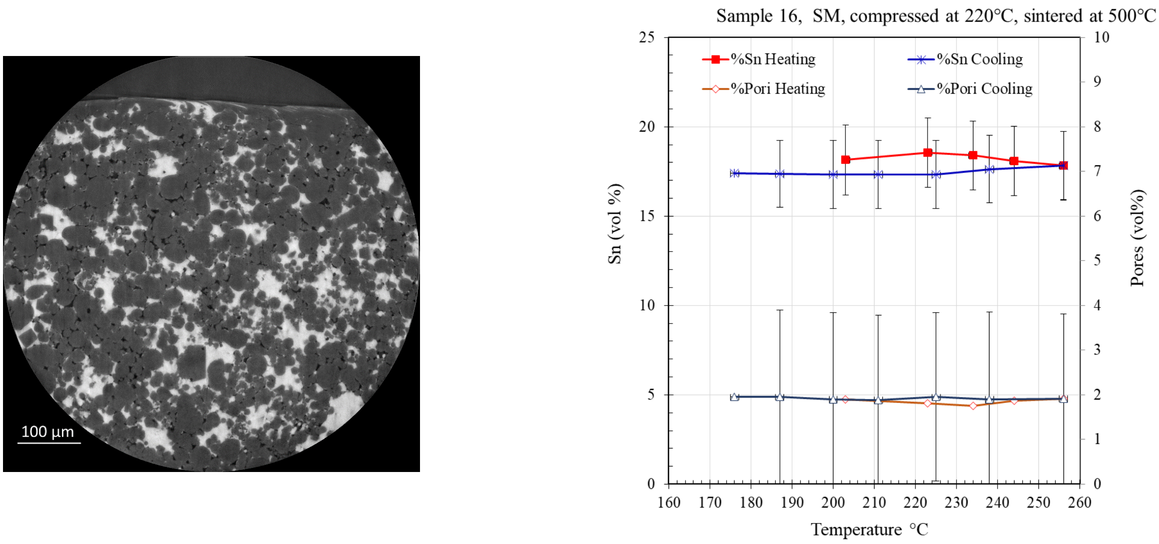
The analyses are still under completion, but some of the main results can be here mentioned.

1) A clear correspondence between the micro-CT scans and SEM analyses at the same magnification can be observed, specifically in samples where homogeneous Sn phase was greater than 10 microns (Figure 1a). This correspondence is useful for image analyses and comparison to previous SEM analyses. The same holds for finer microstructures, obtained from ball milling, where the Al and Sn content was only identifiable by phase brigthness (Figure 1b).
2) The tomographic analyses were suitable for the detection of Sn movement inside the samples, which also included different degrees of initial porosity. For example, Figure 2 shows the trend for Sn and pore percentage as function of temperature during the thermal cycle, for a sample produced though simple mixing of powders, hot compression at 220°C and sintering at 500°C.
3) In certain samples, the formation of small surface droplets on the external surface of the samples was also detected due to the accurate surface positioning within the volume of analyses (see Figure 1c).



(a) (b) (c)

Figure 1 HR Tomography slices of Al-Sn PCAs characterized by coarse microstructure obtained through simple mixing (a), by fine structure obtained through ball-milling (b), and formation of droplets on the surface of the samples heated above the melting temperature of the Sn phase (c).



(a) (b)

Figure 2 HR Tomography slice of a SM sample (a) and analysis of the Sn and pore average volume fraction in different regions a simple mixed sample during the heating and cooling cycle (b).