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.

ESRF	Experiment title: A detailed atomic investigation of the precipitations on a joint of a Friction-Stir-Welded Al-Cu-Li alloy (AA2198)	Experiment number : MA-4134
Beamline:	Date of experiment:	Date of report:
ID24	from: 07.06.2018 to: 12.06.2018	08.05.2019
Shifts:	Local contact(s):	Received at ESRF:
16	Frédéric De Geuser	
Names and affiliations of applicants (* indicates experimentalists):MSc. Danny Petschke University of Wuerzburg		
MSc. Frank Lotter University of Wuerzburg		
Ricardo Helm University of Wuerzburg		

Report:

The aim of the proposal was to investigate clustering and precipitation processes of Al-Cu based alloys in situ using small angle X-ray scattering.

Temperature ramps: To follow the precipitation path of Al alloy often differential scanning calorimetry is used as a standard method. With the in situ furnace at the station we tried to mimic the heating ramp conducted in our previous DSC measurements (20K/min, RT-673K) to get information on the precipitate size and volume fraction. In Fig. 1 one can observe the scattering images for an Al-Cu-Mg alloy during the heating ramp. The scattering signal in the first frame, which corresponds to 75°C, is completely isotropic. This suggests small non-oriented particles, e.g. pre-GPB-zones that are formed at this stage also represented by the exothermic peak in the DSC thermogram. At 115°C a faint and broad streak appears in the SAXS pattern, which is usually a sign of small and thin oriented particles such as the here suggested GPB zones. Going to 150°C only a slight change in the scattering signal is visible until the streak disappears almost completely approaching 200°C. Finally, at about 275°C the scattering image shows intense streaking originating from the rod-like S-phase, as described by the sharp peak in the DSC curve (Fig.2).



Figure 1 Precipitation sequence of an Al-Cu-Mg alloy.



Figure 2 DSC curves for the Al-Cu-Mg alloy. The sharp peak corresponds to the S-Phase formation. At the exact temperature the streaks originating from the rod-like precipitates appear.

Solute Clustering during room temperature: To investigate the effects of trace additions (100pm) of specific elements on the decomposition process of the Al-Cu-(Mg) alloys we followed the solute clustering in situ during room temperature ageing. Fig. 3 shows some of the obtained scattering curves with the respective model fits using the model of Ivanov et al. [1]. Fig. 4 displays the fitting parameters over time for the Al-Cu and Al-Cu-Mg alloys with and without additions of In or Sn. One can observe the significant difference between the Al-Cu and Al-Cu-In/Sn due to a vacancy binding effect of those elements. Suprisingly this effect does not show for the Al-Cu-Mg alloys. These data are already published and discussed in detail in [2].



Figure 3 Scattering curves for an Al-1.7 at.% Cu alloy with and without 100ppm Sn and the respective fits for the clustering model.



Figure 4 Fitting parameters for the investigated alloys during the very early stages of room temperature ageing.

References:

Ivanov, R., <u>Deschamps</u>, A. & De <u>Geuser</u>, F. (2017). J. <u>Appl. Cryst</u>. 50, 1725-1734.
Lotter, F., Petschke, D., De Geuser F., Elsayed M., Sextl G., Staab T. (2019) Scr. Mater. 168, 104-107.