EUROPEAN SYNCHROTRON RADIATION FACILITY

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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: **IN-SITU X-RAY DIFFRACTION DURING CASTING:** **DETERMINATION OF MECHANICAL COHERENCY**	**Experiment number**: MA-2122
Beamline: ID15B	**Date of experiment**: from: 30.04 to: 02.05.2014	**Date of report**:
Shifts: 6	**Local contact(s)**: Jessica Hudspeth	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Coalescence and rigidity temperature

Coalescence is an important transition in solidification of metallic alloys and corresponds to the formation of solid bridges between grains. It starts at the coherency point when the grains touch each other but are unable to sustain any mechanical load and ends up at the rigidity point when the structure is able to sustain macroscopic tensile strains and stresses. At the level of microstructure, rigidity temperature (T_{rig}) is reached when the solid phase is sufficiently percolated to transmit tensile and shear strains. This temperature is important as it determines the very instant when macroscopic stresses start to build up owing to thermally induced deformations. It is also an important parameter in the hot tearing (solidification cracking) resistance of some aluminium alloys. The aim of the experiments carried out at ID15 was to measure T_{rig} using *in situ* 2D diffraction during casting of Al-Cu alloys in a dog bone shaped mould. This allowed us to follow the changes of diffraction patterns resulting from lattice parameter variations.

In situ X-ray diffraction during castings

The device used to perform *in situ* casting was composed of a dog bone mould, a tundish and a crucible. Each part contained independent electric heating elements in order to control its temperature. The crucible was mounted on a tilting support commanded by a pneumatic pusher in order to remote control the movement. This allowed us to acquire diffraction patterns even before pouring the liquid. Conic holes were machined into the steel walls to let the beam go through. Alumina fiber pieces were used to plug these holes to avoid any liquid metal leakage. The temperature was recorded along the sample at a frequency of 3 Hz. One thermocouple was partially in the gauge volume. The X ray diffraction patterns were detected by the pixium 4700 solid state detector covering an area of 406 x 295 mm. The pixel size was 154 x 154 µm and the sample to detector distance was 1270 mm. We used a 87.2 keV beam energy and a 0.7 x 0.7 mm beam size to get the highest flux possible trough our 15 mm thick samples. A diffraction pattern acquisition (scan) was composed by a sum of five 0.4 s exposure images. We used a standard acquisition mode which took 4.62 s to save data, leading to an acquisition frequency of 1 scan every 6.62 s. During the 6 shifts, 16 castings of grain refined binary aluminium alloys containing 1, 2, 3, or 4,43 wt.% copper were performed. Figure 1 shows a micrograph of the grain refined Al-1 wt.%Cu alloy. The microstructure presents 100-150 µm equiaxed graind surrounded by eutectic resulting from fast cooling.
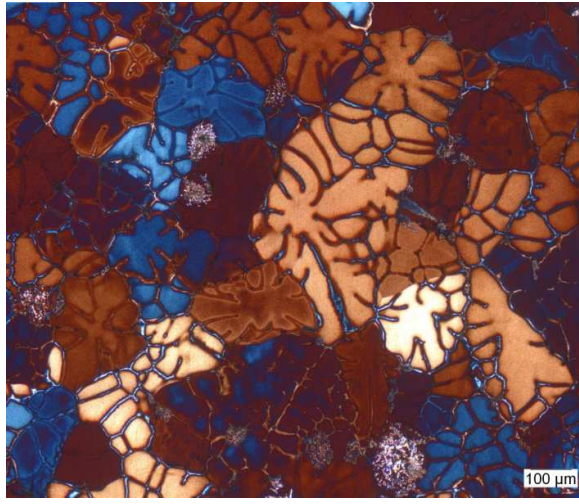
Figure 1: polarized light micrograph of Al-1 wt.%Cu alloy

Each alloy was cast in a "free to contract" (FC) and constrained, i.e. "dog bone" (DB) configurations. The DB configuration prevents the aluminium sample to shrink since it is constrained by the steel which has a coefficient of thermal expansion (CTE) around 16 $\mu m.m^{-1}\ K^{-1}$. Aluminium exhibits CTE in the order of 25 $\mu m.m^{-1}\ K^{-1}$ at low temperature. This difference causes a tensile strain at the hot spot located in the gauge volume. The FC configuration consisted in pouring liquid only in the central part to allow free contraction of solid.

Some of the alloys were not grain refined. Casting temperature varied between 713 °C and 750 °C and mould temperature was around 540 °C. For one casting, this temperature was 406 °C and a hot tear occurred. Figure 2 shows the casting device, the mould geometry and castings in DB and FC configurations.
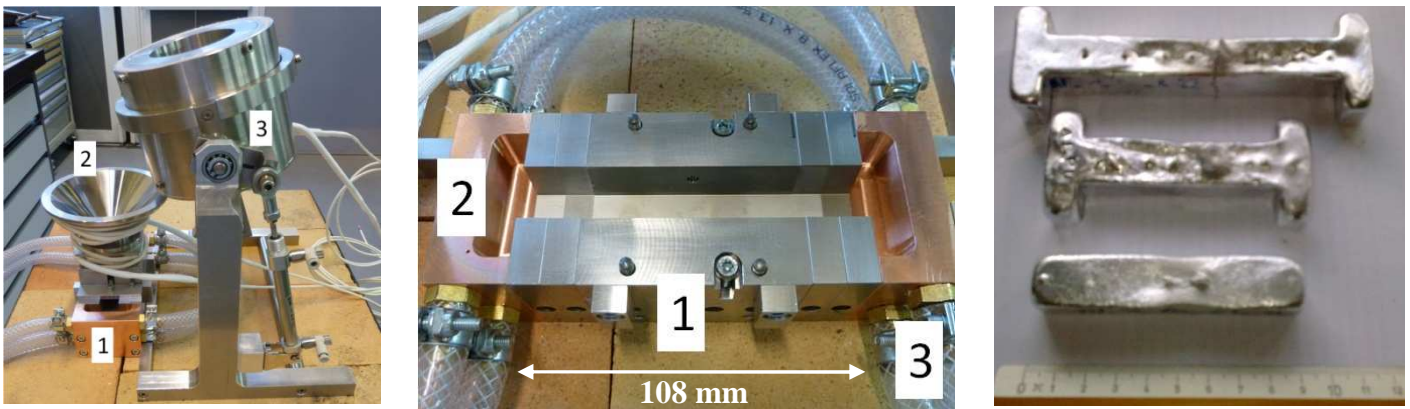

Figure 2: left, casting device (1 - mould, 2 - tundish, 3 - crucible); centre, mould (1 - steel body, 2 - copper chills, 3 - water pipes) and right, castings in DB and FC configurations.

Results

All diffraction patterns were analysed using Fit2D and TOPAS Academic[©]. The calibrant sample was CeO_2 powder. The lattice parameter of fcc aluminium copper solid solution was extracted as a function of the temperature at the hot spot, i.e. at the very centre of the casting. For alloy cast without grain refiner, the diffraction patterns appeared to be hardly exploitable owing to the low number of grains within the gauge volume and the loss of precision.

Several scans were acquired during the temperature plateau at the liquidus. The value of the lattice parameter at the liquidus is considered as the reference for all castings having the same alloy composition. The relative difference in lattice parameter between the FC and the DB configurations corresponds to the elastic strain.

Figure 3 shows the evolution of the temperature and lattice parameter relative to the Al-4,43wt.%Cu alloy for experiments in the FC and DB configurations. The cooling rate approaches zero at the liquidus (649 °C) and eutectic temperatures, and remains between -1 Ks^{-1} and -7 Ks^{-1} during solidification. As soon as the metal starts to solidify, some diffraction patterns forms owing to the presence of early crystallites.
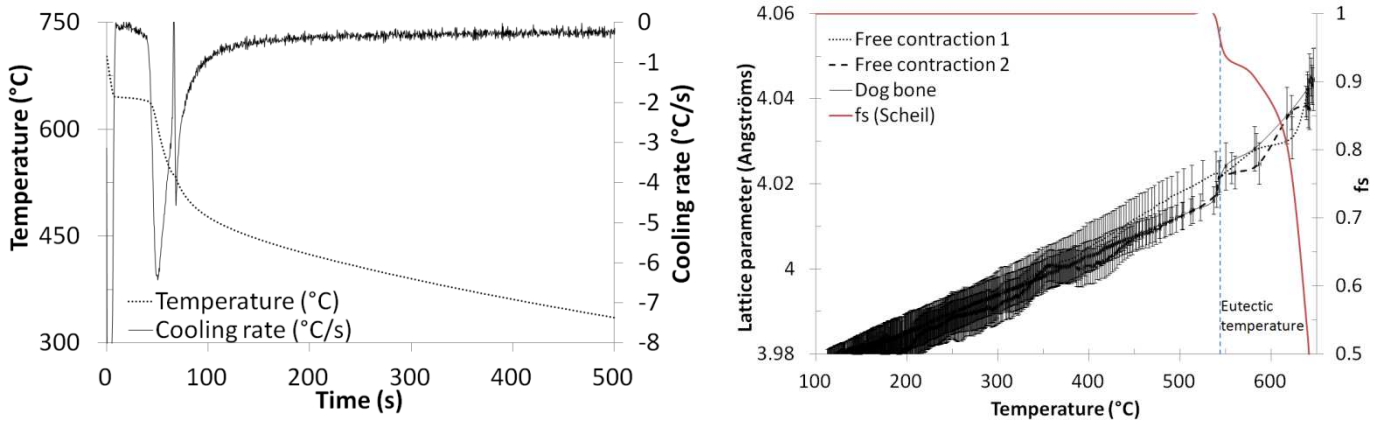

Figure 3: left, temperature and cooling rate at the hot spot for the Al-4,43wt.%Cu alloy and right, lattice parameter and solid fraction as functions of temperature for FC and DB configurations.

One can see that the relative difference between FC and DB configurations is inside the error bars. Consequently we are not able to retrieve stress value with these results. The lattice parameter decreases with a high slope (between 553 °C and 649 °C as a result of microsegregation) as the hot spot reaches the eutectic temperature (548°C) with a small undercooling, where it exhibits a drop around 0.3 %. It corresponds to a sudden 2 wt.% increase in average solute content in the solid. A CTE value between 26.4 and 29.2 μm.m^{-1}.K^{-1} is retrieved in the solid state which is typical for aluminium alloys. As all the curves are within the error bars and no clear tendency is seen in semi solid state, it is impossible to determine a rigidity point.

Conclusion

The standard saving mode (1 scan every 6.62 s) allowed us to acquire scans quicker than we performed previously with neutron diffraction (1 scan every 11 s), but we still have not enough points in the semi solid state to precisely determine a rigidity point. Increasing the acquisition rate and reducing cooling rate would allow seeing mechanical coherency above the eutectic point, *i.e.* the solid *α* solution ensures the stress transmission through the whole sample.

For some reason, several diffraction patterns were really noisy and unidentified materials were diffracting. Moreover the flux was so high that some diffracted beams were strong enough to diffract again in the plate protecting the detector. Our samples were 15 mm thick which contributed to peak broadening. This also brought complications during Rietveld refinement processing and Rwp were in the order of 35 % which explains the error bar size. All these cumulated imprecisions prevented us from determining the rigidity point of these alloys. We still have been able to extract CTE at high temperature (32.15 μm.m^{-1}.K^{-1}) which is slightly higher than the close to room temperature one (28.3 μm.m^{-1}.K^{-1}).

Further experiments will be performed using thinner samples, smaller beam size to increase measurement precisions, and lower scan duration to acquire more point in the semi solid state. Finding a way to avoid dead time between scan recordings would also help a lot.