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.

<b>ESRF</b>	<b>Experiment title:</b> 4D imaging of thermolelectric controlled solidification of Al-Si-Cu alloys	<b>Experiment</b> <b>number</b> : MA2989
Beamline:	Date of experiment:	Date of report:
	from: 29 Jun 2016 to: 03 Jul 2016	
Shifts:	Local contact(s):	Received at ESRF:
	Elodie Boller	
Names and affiliations of applicants (* indicates experimentalists):		
Dr. Biao Cai*, University of Manchester		
Prof. Peter d Lee*, University of Manchester		
Professor Koulis Pericleous, University of Greenwich		
Dr Andrew Kao*, University of Greenwich		

## **Report:**

### **Proposal summary**

We have designed and commissioned a new *in situ* advanced manufacturing rig to directionally solidify novel alloys whilst applying a permanent magnetic field (0.5T) and obtaining fast tomographic images. We now propose to use the ultra-fast X-ray tomography at ID19 to thoroughly study how magnetic fields affect solidification of Al-Si-Cu-(Fe) alloys, in particularly, how the dendrite morphology is disrupted. This experimental investigation, when coupled with our numerical models, can be used to demonstrate the potential of Thermoelectric Magneto-Hydro-Dynamics (TEMHD) convection during solidification, helping the design of a new class of alloys (lighter and stronger).

## 1. Integration of the rig with ID19.

An in-house build MagDS system, which contains two furnaces (up to 1000 °C) and an X-ray window in between (Figure 1), is capable of directionally solidifying novel alloys whilst applying a permanent magnetic field (0.5T). This MagDS system was integrated with ID19's high speed tomographic imaging system successfully (Figure 2) with the support of ID19's beamline scientists.







Fig. 2. The MagDS system in ID19 ESRF

## 2. Results

We have performed high speed tomographic imaging of solidification process of both Al-15wt%Cu and Al-5.5Si-3.4Cu (wt.%) alloys. The samples were first fully melted in the furnace then a thermal gradient was built up via varying the temperature of both furnaces. The directional solidification was triggered by cooling both furnaces down at a prescribed rate. Experimental conditions with varied cooling rates and thermal gradients were utilized. A magnet field of 0.5 T was applied to different samples with the same directional solidification conditions.

Analysis of the full time dependent 3D reconstruction is still underway, but the initial results already show the effect of magnet on solidification microstructures.

- (1) Experiments on 2mm diameter Al-15% wt.Cu samples have shown the formation of a helical microstructure (figure 3) when the magnet is on, and more importantly the growth morphology and segregation behavior are totally different with the magnetic field applied (figure 4). Our simulations reproduce the observed behavior as shown in figure 5.
- (2) There is also evidence that TEMHD can lead to significant grain refinement. Using the same experiment set-up (MagDS), experiments were conducted on a 319 Al alloy (Al-5.5Si-3.4Cu, wt.%), which is widely used in industry. Figure 2.7 shows the microstructure of 319 Al alloy with and without the application of the magnetic field. In the presence of a 0.5 T, the magnetic field the grains are around 20 times smaller.

Current results demonstrate that TEMHD has a significant effect on solidification microstructure. However, fully analysis of the experimental results is still ongoing. The 3D/4D quantification will provide us a better understanding of the underlying mechanisms. One example of 3D dendrite is shown in figure 7.



Fig. 3. Radiographies of directionally solidifying Al-15wt%.Cu dendrites at different rotation angles: 0, 90, 180°, showing the helical structure.



Fig. 4. 2D slice from 3D volume of directionally solidifying Al-15wt%.Cu dendrites. Top row: without magnetic fields; bottom row with 0.5 T magnetic field onto the sample.Significant difference in dendrite morphology and segregation can be observed.



Fig. 5 Numerical model of TESA showing the formation of a single turn of the helical structure. Time is relative, where 0s represents the start of the turn.



Fig. 6. 2D slice from 3D volume of a directionally solidifying 319 Al alloy (a) without magnetic fields, (b) with a 0.5 T magnetic field, showing 20 times significant grain refinement when magnet is on.



Fig. 7 A reconstructed equiaxed dendrite

## **3.** Publications

An oral presentation has been accepted at Defects and Properties of Cast etals in TMS2017, entitled '4D Synchrotron X-ray Imaging of Magnetically Controlled Al Alloy Solidification'. A few journal publications are expected, and one is in preparation.