ESRF	**Experiment title:** **Influence of crystal orientation relationships on the morphology of lamellar eutectic microstructures**	**Experiment number**: 32 02 812
Beamline: BM32	**Date of experiment**: from: June 28. 2018 to: July 2. 2018	**Date of report**: July 17, 2018
Shifts: 12	**Local contact(s)**: Jean-Sébastien Micha	*Received at ESRF:*

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Report:

- Objective & expected results: -

The aim of this project (Proposal number: 20171113 ; Experiment number: 3202812) is to establish a link between microstructural features of melt-grown metallic composites and the orientation of the co-growing crystals. This is key to improving our understanding of the formation of textures in eutectic materials. Fundamentally, the challenge is to develop theoretical tools for predicting the self-organizing growth dynamics of eutectics and the anisotropy of the interphase boundaries in the solid. In practice, micro-diffraction analyses at BM32 (J.-S. Micha) aim at determining the crystal orientation relationships (ORs) between neighbouring crystals of the two eutectic-solid phases [the cubic Al-rich terminal solution (Al), and the tetragonal intermetallic Al_2Cu] on a local scale in eutectic solidification microstructures of thin Al-Al_2Cu metallic films. Synchrotron radiation permits to make non-destructive measurements through a sapphire-plate substrate.

- Results and the conclusions of the study: -

Thin Al-Al_2Cu films (thickness ≈ 9—14 µm; lateral dimensions 8×50 mm^2) are enclosed between two single-crystal sapphire (Al_2O_3) plates. They were prepared by plasma sputtering and solidified at INSP. The thickness of one sapphire plate was reduced to 200 µm. Two solidification methods with real-time optical observation were employed: (i) directional solidification, and (ii) rotating directional solidification (RDS) that permits to vary continuously the orientation of the solid with respect to the growth axis.

We selected samples containing typically 2—4 lamellar-eutectic grains (each being supposedly uniform as regards the orientation of the crystals) of a few mm each. Non-destructive Laüe micro-diffraction allows one to map the orientation of the alternating (Al) and Al_2Cu crystals over selected regions, with good transmitted signal through the 200-µm thick Al_2O_3 substrate, and an actual resolution much better than the size of the smaller crystals (≈5 µm). This provides a means for distinguishing between different grains, and gaining information on the degree of mosaicity, if present, within a given grain.

Sample n°	Alloy	DS/RDS/NA	Sapphire cover	Scan names	Analysis date
Ech44	$Al-Al_2Cu$	DS	Yes	ROI01-ROI08 (tests) ROI01, ROI08-ROI21	06/28/2018 06/29/2018
Ech45	$Al-Al_2Cu$	DS	Yes	ROI01-ROI09 ; ROI20-ROI22	06/30/2018
Ech49	$Al-Al_2Cu$	DS	Yes	ROI01-ROI15	07/01/2018
Ech19	$Al-Al_2Cu$	DS	No	ROI01	06/30/2018
Ech28	$Al-Al_2Cu$	DS	No	ROI01-ROI08 ROI21-ROI28	06/28/2018
Ech62	$Al-Al_2Cu$	RDS	Yes	ROI01-ROI09	07/01/2018
Ech59	$Al-Al_2Cu$	NA	No	ROI depot	07/01/2018
Ech710	$In-In_2Bi$	DS	***	ROI01-ROI03	07/01/2018

*Table 1: List of samples and scans. DS: directional solidification. RDS: rotating directional solidification. NA: as-deposited sample. Note that Ech710 was a thin solidified $In-In_2Bi$ eutectic. *** ≈50µm glass.*

Each Region of Interest (ROI) was scanned, and an x-ray detector used to collect a characteristic fluorescence of copper for revealing the microstructure of the actually scanned area (Fig. 1). The raw output data are a set of Laüe patterns for each ROI. For each sample, we also measured a Laüe pattern on a reference Ge crystal fixed on the sample surface for fine angle-scale calibration, and another one on the sapphire plates.

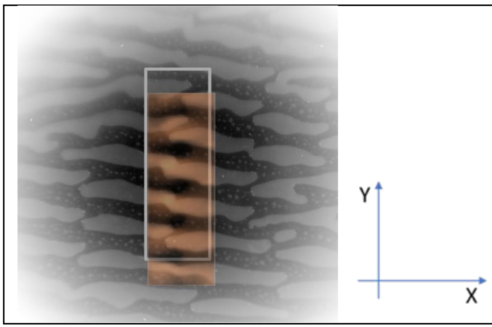

Figure 1: Optical micrograph (hor. dimension: 200 µm) corresponding to ROI05 of ech62 ; the Al_2Cu phase appears dark, the Al one is brighter. Black frame: nominal ROI. Superimposed color image: actual matching of the fluorescence pattern.

Let us now take Ech62 as an illustrative example (Fig. 2).

Fig. 3 shows a Laüe pattern measured at some point in an Al lamella. Note that, in all Laüe patterns, diffraction peaks coming from the Al_2O_3 single-crystal substrates could be easily identified. Using the LaueTools software, we could locate Al peaks (see mosaic image, Fig. 3b), and successfully run a best-fit procedure (Fig. 3c). It is thus shown, in particular, that a (111) plane is tilted by a few degrees off the substrate. A similar analysis for a neighbouring Al_2Cu lamella is shown in Fig. 4. We will extract the orientation of the crystals (rotation matrix and/or Euler angles), and look for special ORs.

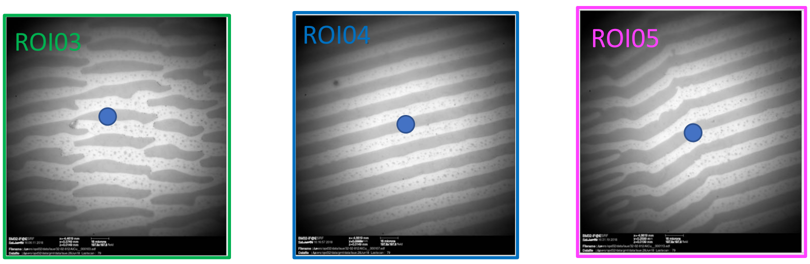

Figure 2: Sample Ech62: examples of ROIs (horizontal dimension: 200 µm). The blue dot signals a given Al_2Cu lamella that we followed over a long distance in the microstructure.

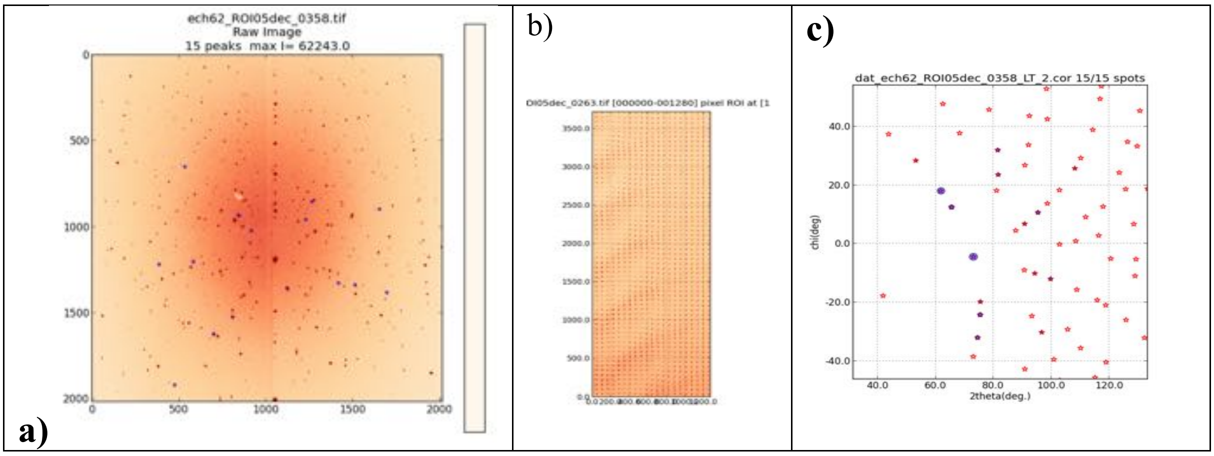

Figure 3: a) Laüe pattern in an Al crystal. b) Mosaic image that shows that the variation of the intensity of a particular peak also follows the alternation of the lamellae. c) Best fit of the selected peaks (blue). The high-intensity (222) peak is close to the normal to the sample plane (2theta=80deg; chi=0 deg).

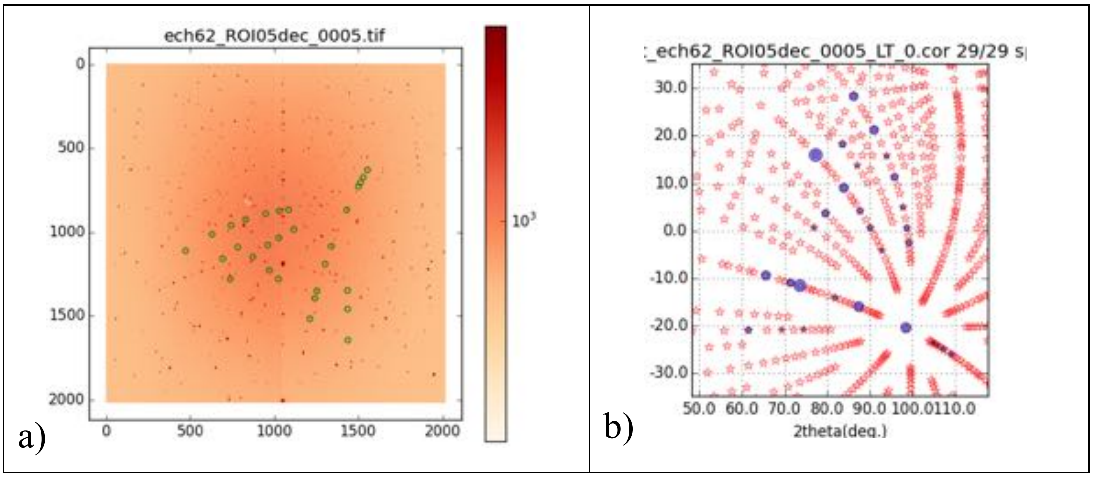

Figure 4: a) Laüe pattern in an Al_2Cu crystal. b) Best fit of selected peaks (blue). The (100) peak is clearly visible.

In addition to the main problem (link between crystal orientation and micrsotructural features), various questions will be tentatively addressed:
1-Is there a variation of the orientation of the lamellae during growth ? **2**-In a given eutectic grain, all the lamellae of one phase are known to originate from a single crystal seed. We have to check, however, if there is, as is often suspected, a progressive rotation, up to a few degrees, of the crystal lattice of the crystals across a eutectic grain. **3**-Ddiffraction peaks are often not circular (Fig. 5). This feature can evolve from one interphase boundary to the other, inside a given lamella. A detailed interpretation of that is not straighforward.

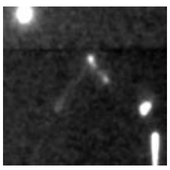

Figure 5: Detail of a Laüe pattern (ech44, ROI21, image 535) showing a circular Al_2O_3 spot (top, left), a distorted Al spot (center), and an elongated Al_2Cu spot (bottom right).

- Justification and comments about the use of beam time: -
The use of the synchrotron radiation was need for nondestructive analysis through a sapphire plate. Micro-diffraction at ESRF in addition provides spatially resolved series of measurements, including crystal distortion, on a local scale with an unprecendented accuracy.

- Conclusion: -
In conclusion, our micro-diffraction measurements on Al-Al_2Cu solidification microstructures through a sapphire plate have been successful. The use of a reference sample without sapphire was helpful for spatial calibration. Both Al and Al_2Cu crystals can be nicely indexed. The analyses will be performed more systematically. This requires not only the use of the LaueTools software, but also some image-analysis

development. The distortion of diffraction peaks seems to indicate small-scale strain: do mechanical stresses develop during growth at a small distance from the solid-liquid interface, or at a much lower temperature, far from the solidification front ? This question cannot be addressed ex situ. It would encourage, and motivate one to develop in situ micro-diffraction during solidification.