ESRF	**Experiment title:** In situ study of directional solidification of metallic materials by combined synchrotron X-ray radiography/topography	**Experiment number**: MA-413
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Scientific objectives

The characterization of the solidification microstructure requires a precise knowledge of the solute segregation profile and the influence of solidification parameters such as the growth rate and the temperature gradient. Most of the phenomena involved during crystal growth are dynamical and cannot be described using classical post-mortem characterization techniques. As a consequence, the use of X-ray techniques which provide in situ and real time observation of the solid-liquid interface is necessary to understand the solidification dynamics and the solute profile formation during the initial transient. Therefore, we decided to apply synchrotron X-ray radiography and topography to investigate directional solidification of Al-4wt% Cu and AlPdMn. However, X-ray topography could not be performed during this session for technical reasons, then only X-ray radiography was used during the whole experimental session. Consequently, the main objectives of this session were:

- To develop a method to measure solute distribution during the initial transient of directional solidification.
- To validate an experimental procedure to perform equiaxed growth in nearly isothermal conditions.
- To study peritectic transformation during AlPdMn directional solidification

Experiments

Two Al-4wt% Cu samples and one AlPdMn sample were prepared at IM2NP (Marseille). Sixteen experiments on Al-4wt% Cu and four experiments on AlPdMn were carried out in a vertical Bridgman set-up with both sample and furnace vertically fixed. *Temperature Gradient Decrease* (*TGD*) and *Isotherms Displacement* (*ID*) techniques were applied to induce respectively columnar and equiaxed growths. In the *TGD* technique, the hot zone temperature is decreased at a given cooling rate R, while the cold zone temperature is maintained constant. In the *ID* technique, the temperatures of both hot and cold heaters are simultaneously decreased at a given cooling rate, which yields the sample solidification.

The main surface of the sample was perpendicular to the incident monochromatic X-ray beam. The energy was adjusted to 17.5 keV (resp. 20 keV) for Al-4wt% Cu (resp. AlPdMn) alloys. The FReLoN camera was used as detector and the optics were chosen to obtain a good compromise between a large field of view ($15x6mm^2$) and a good resolution (pixel of 7.46 µm).

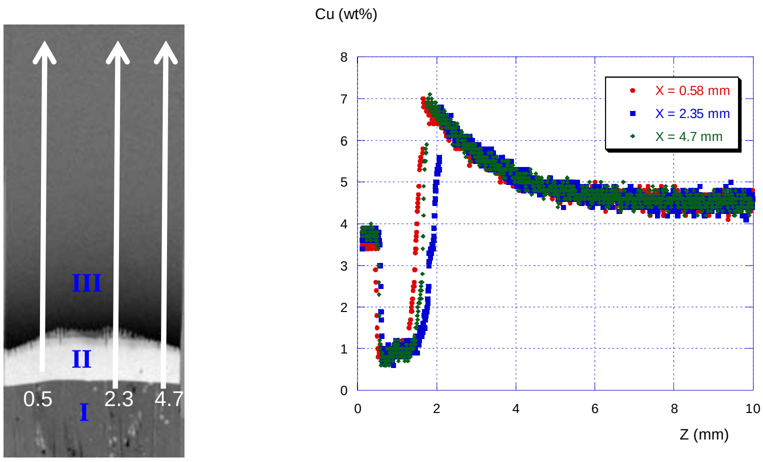
Results

1. Solute profile during the initial transient of directional solidification of Al-4wt% Cu

The as-recorded Al-Cu images show a poor contrast and few defects, originating either from the crucible itself or from the X-ray beam, therefore an image processing technique was required and has been developed to improve their quality. The result is an enhanced contrast and a defects free image as can be seen in fig.1.a & fig.2. We have then developped an image analysis procedure, based on the measurement of contrast distribution on the processed images, to determine the variation of solute content in both solid and liquid phases during columnar growth of Al-4wt% Cu (Fig.1.a&b). This outstanding result is no doubt a breakthrough in the use of synchrotron X-ray radiography for the analysis of solidification process. The application of this analysis to a planar solidification front has given very promising results, which must be now verified more systematically. The subsequent step should be to extend this analysis to more complex cases, such as the columnar and the equiaxed dendritic growth.

Fig.1*: (a) Microstructure after 24 min of solidification,*
(I) unmelted part of the sample,
(II) solidified part,
(III) liquid phase,

(b) Three longitudinal solute profiles, calculated for different transverse positions x.



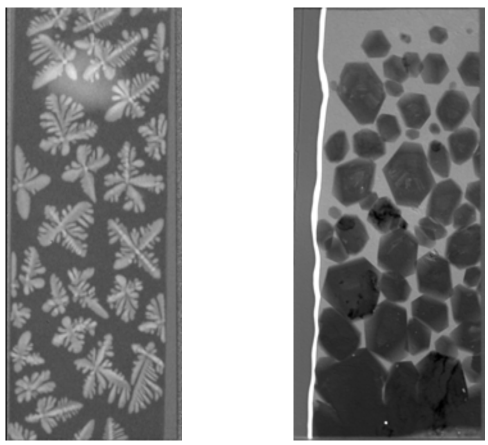
2. Equiaxed growth of Al-4wt% Cu in isothermal conditions

In this experimental session, we have also proved that it is possible to perform a fully equiaxed growth in nearly isothermal conditions (fig.2a) with our experimental set-up. This is achieved by the application of the *Isotherms Displacement* (ID) technique. Prior to the solidification, the temperatures of the furnace heaters are adjusted to reach nearly isothermal conditions (i.e. no temperature gradient). Then, those temperatures are simultaneously decreased at a given cooling rate, ranging from 0.2 K/min to 1.5 K/min, until the nucleation and growth of equiaxed grains. These preliminary experiments open a new research area which is of great interest for industrial application (casting).

Fig.2 : *X-ray images during directional solidification of Al-4wt% Cu and AlPdMn alloys :*

(a) A fully equiaxed growth in nearly isothermal conditions at a cooling rate of 0.2K/min (Isotherms Displacement)

(b) A columnar & equiaxed growth of a quasicrystal (AlPdMn) at a cooling rate of 0.2 & 0.4K/min (Temperature Gradient Decrease)



3. Quasicrystals (AlPdMn) growth

The quasicrystals solidification microstructure (fig.2b) was also observed by synchrotron X-ray radiography. Solidifications were induced by *TGD* technique. The complete analysis of these experiments and additional post-mortem measurements are still in progress.