ESRF	**Experiment title:** Application of combined radiography and topography to the characterization of Al-based solidification process	**Experiment number**: MA-514
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Scientific Objectives

The MA-514 experiments were dedicated to the study of directional solidification of Al-Cu alloys in order to deepen the results obtained during the MA-413 experiments. During alloy solidification, when increasing the growth rate, the smooth solid-liquid interface becomes unstable, consequent to the Mullins-Sekerka morphological instability theory *[Mullins and Sekerka, J.Appl.Phys.35 (1964) 444]*, and transforms to an array of cells or dendrites. The physical origin of this instability is the "constitutional supercooling" consequent to a continuous solute rejection that occurs during phase transition. The main objective of MA-514 experimental session was to complete and improve the preliminary analysis of the solute profiles through X-ray radiography image analysis procedure started during our previous session.

Another objective was to characterize the equiaxed solidification in isothermal conditions. Preliminary tests carried out in MA-413 have shown that it is possible with our experimental set-up to perform solidification in isothermal conditions. Therefore, we intended to check the reproducibility of our technique in order to study the interaction between neighbouring dendrites and, if possible, the solute distribution around grains which requires images with higher resolution. For this reason a "3µm" optics was tested but due to the poor efficiency of the scintillator employed, the test was not successful. We plan to perform a new test using up to date devices (scientillator, FreLon camera) in a future experimental session.

Initially, X-Ray topography was planned in order to study the crystallographic orientation of the grains, the strains and other crystalographic defects which are important paramers in materials properties. However, for technical reasons the set-up could not be installed during this experimental session.

Experimental Procedures

Al-4wt% Cu and Al-10wt% Cu samples were prepared at IM2NP (Marseille). Solidifications were induced by applying the isotherms displacement technique previously described in Ma-413 report. Two different types of experiments were performed:

- Columnar solidification with a planar interface: In these experiments, the temperatures of the two thermal zones (hot and cold) of the furnace were adjusted to achieve a desired temperature gradient.

Then, cooling rates as low as 0.3, 0.2 and 0.1 K/min were applied to both hot and cold zones of the furnace in order to maintain a smooth planar solidification front for a long duration.

- Fully equiaxed growth: After adjustment of the hot and cold zone temperatures to achieve a nearly zero temperature gradient, the same cooling was simultaneously applied to both zones of the furnace at the rates of 0.3 K/min and 0.5 K/min until the achievement of nucleation and growth of equiaxed grains.

Results

1- Improvement of the procedure for the measurement of solute profiles

- To better reveal the microstructure and the solute segregation, an image treatment fully described in *[Buffet, A., Ph.D thesis, 2008]* was required. During the image analysis process, we found that it was very crucial to take into account the variation (up to 14% in one of the experiments) of the incident monochramatic beam intensity with time. For this reason, the value of the transmitted beam was modulated with a correction factor in order to measure the precise local variation of concentration.

- Solute distribution was measured as well as the time evolution of the solute concentration in the liquid ahead of the solidification front for several experiments. The results were in very good agreement with the predicting models as can be seen from fig.1 *[Nguyen Thi, H., et al., Fifth International Conference, Solidification and Gravity 2008, Miskolc, Hungary, Sept.1-4, 2008]*.

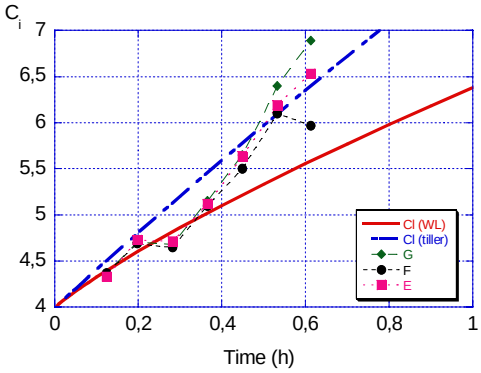

Fig.1. *Time evolution of the interface solute concentration in the liquid during the initial transient of planar growth. The experimental values are compared with Warren-Langer [Warren, J.A. and J.S. Langer, Physical Review E, **47**(4) (1993) 2702] and Tiller [Tilleret al., Acta Metallurgica, **1** (1953) 428]*

2- Solutal interaction between neighbouring dendritic equiaxed grains

From the recorded X-ray radiographs, the length of the dendrites arms could be measured directly at every time throughout the solidification. For instance the growth rates of two interacting dendrites (fig.2a,2b) are plotted in fig.2c. As can be seen, the decrease of both growth rates started at t ≈ 50 s, it is assumed to be a consequence of the interaction between their solute layers. Moreover, at t ≈ 200 s one of the grains stopped while the other one was still growing. Finally the two grains remained separated by a small liquid channel. These results suggest that the solutal interaction is dominant between equiaxed grains.

Fig.2. *Interaction between two dendritic equiaxed grains during growth of Al-10wt% Cu in nearly isothermal conditions.*

(a) t_1=28 seconds
(b) t_2=196 seconds
(c) growth rates of the two interacting dendrites