| ESRF | Experiment title: **Analysis of fragmentation and columnar-to-equiaxed transition in Al-Cu alloy by the combination of synchrotron X-ray radiography and topography** | **Experiment number**: MA-746 |
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## Scientific Objectives

In metal casting, dendritic equiaxed grains are the most common solidification morphology. After the nucleation stage, a growth phase leads to the formation of the fully equiaxed grain microstructure. In the very early stages of solidification and for small growth rate, each grain can be considered as isolated or growing freely. As solidification proceeds, grain interaction becomes the key mechanism that controls the microstructure formation. Several attempts have been made to model grain interaction by an average approach, which have resulted in many interesting predictions for casting. However, many open questions remain, especially regarding the nature (thermal or solutal) of the interaction between grains and the variation of their dendrite tip velocities during the entire growth phase. Therefore, a deeper understanding of the interaction occurring during the equiaxed grain formation is crucial for the prediction of the resulting microstructure and for modelling the evolution of the grain structure.
The main objective of MA-746 session was to analyze the equiaxed growth in nearly isothermal conditions. Preliminary tests carried out in MA-413 and MA-514 have shown that it is possible with our experimental set-up to perform such type of solidification experiments. Therefore, we intended to check the reproducibility of our technique in order to study the interaction between neighbouring dendrites. For this reason a “3µm” optics was tested during this experimental session. Initially, X-Ray topography was planned in order to study the crystallographic orientation of the grains, the strains, etc…, which are important paramers in materials properties. However, for technical reasons the set-up could not be installed during this run.

## Experimental Procedures

Al-4wt% Cu and Al-10wt% Cu samples were prepared at IM2NP (Marseille). Solidifications were induced by applying the *Powerdown technique*. In this technique, adjustment of the hot and cold zone temperatures were first performed to achieve a nearly zero temperature gradient. Then, the same cooling *R* was simultaneously applied to both zones of the furnace until the achievement of nucleation and growth of equiaxed grains. Different cooling rates within the range [0.1 – 24] K/min were applied for each sample.

# Results

Using radiography, outstanding results were obtained on the dynamics of equiaxed grain formation during this experimental session. A very short abstract is given in the report. These results were presented in:

- International conferences:
  - E-MRS (8-12/Jun/2009, Strasbourg, France)
  - ICSSP4 (20-23/Nov/2009, Chennai, India)
  - CSSC (3-5/Feb/2010, Hokkaido, Japan)
  - Int. Workshop on X-ray imaging of solidification of metallic materials (8/Feb/2010, Japan), Invited talk
- Publications:
  - *Nuclear Instruments and Methods in Physics Research B* 268 (2010) 394–398
  - *Transactions of The Indian Institute of Metals*, Vol. 62, Issues 4-5 (2009). 427-431

## 1- Self-poisoning effect during equiaxed grain formation

From the recorded X-ray radiographs, the length of the dendrites arms *L(t)* could be measured directly at every time throughout the solidification, and then the growth rates *V(t)*. Measurements were performed for a selection of grains like in Fig.1, with four arms growing at right angles to each other along <100> direction. The unexpected difference of the arm growth velocities at the beginning may be attributed to a "self-poisoning of parts of the grain. Indeed, as solidification proceeds, the rejected Cu is carried downwards by gravity driven fluid flow, leading to the velocity decrease of the dendrite arms situated aside and below.

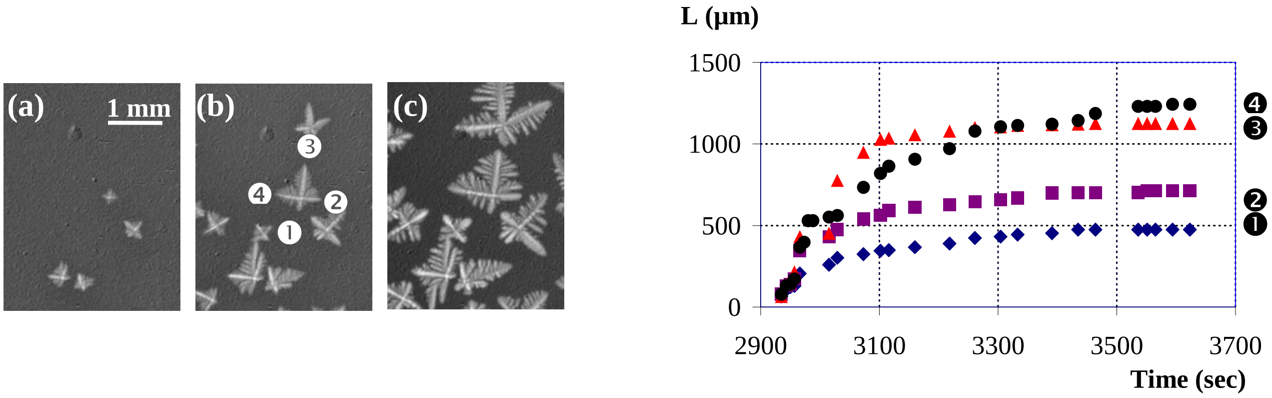


*Fig. 1: Sequence of three radiographs showing the growth of an equiaxed grain of Al-10 wt% Cu, for a cooling rate of 0.5 K/min (a) t=2957sec, (b) t=3037sec and (c) t = 3362 sec. (d) Curves of the time variation of dendrite arms lengths. The curves are referenced by the numbers given on the right hand side of the graph.*

## 2- Solutal interaction between neighbouring dendritic equiaxed grains

Several couples of grains with dendrite arm growing towards each other were analyzed. The growth rates of two interacting dendrites (Fig.2a and 2b) are plotted in fig.2c. The accelerating part of the curve is the period when the dendrite arms grow freely, far from any neighbouring grain. The decelerating part of the curve approaching zero is the period when the two dendrite solute boundary layers overlap, which gradually evens out the Cu concentration between the two dendrite tips. Consequently, the concentration gradient in between dendrite arms decays toward zero, leading to the growth being stopped. Hence, the solutal interaction between the two grains is confirmed by the fact that $V_1/V_2$ reach their maximum values roughly at the same time. The maximum value of $V_1$ is lower than that of $V_2$, due to the solute enriched liquid rejected on $L_1$ (i.e. solutal poisoning) by a third grain just above (Fig.2b).

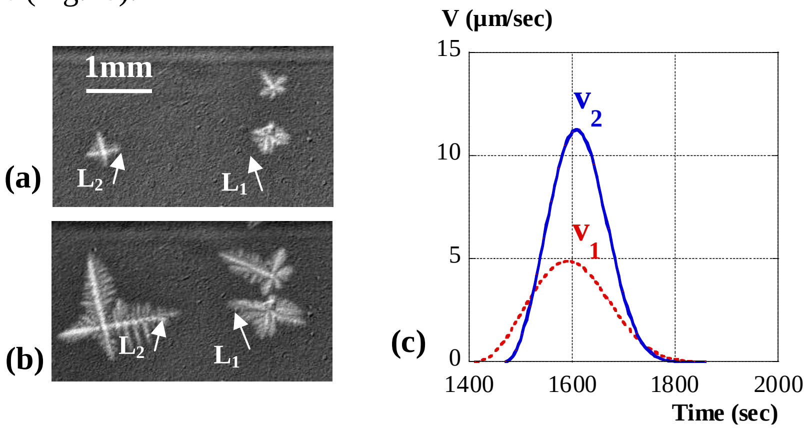


*Fig.2: (a) Image showing two Al – 10 wt% Cu dendrite arms $L_1$ and $L_2$ growing towards each other at the early stage of solidification when there is no interaction between the grains (R = 0.5 °C/min; t = 1559 sec). (b) Image showing arms $L_1$ and $L_2$ effectively interacting (t = 1642 sec).*

*(c) Time evolution of the growth rates $V_1$ and $V_2$.*