ESRF	**Experiment title:** Analysis by X-ray imaging techniques of dynamical phenomena during directional solidification of metallic alloys	**Experiment number**: MA-213
Beamline: ID19	**Date of experiment**: from: 10/04/2006 to: 10/07/2006	**Date of report**: 05/15/2007
Shifts: 15	**Local contact(s)**: Dr. Juergen HAERTWIG	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

*NGUYEN-THI Henri**, L2MP, Campus Saint-Jérôme, Case 142, 13397 Marseille Cedex 20, France
*JUNG Hyejin**, L2MP
*MANGELINCK-NOEL Nathalie**, L2MP

*SCHENK Thomas**, LPM, Ecole des Mines de Nancy, Parc de Saurupt, 54042 Nancy cedex, France

BUFFET Adeline, PhD (ESRF), BARUCHEL José (ESRF), *HÄRTWIG Jürgen** (ESRF)

Scientific background

The solidification microstructure, with the accompanying solute segregation profile, largely and often definitively controls the properties and quality of grown materials. Thus, a precise understanding of growth processing, from the microscopic to the macroscopic scale, is essential to tailor reproducibly products of specified quality.

Experimentally, most studies on metallic alloys have characterized the solid-liquid interface by post-mortem analysis (morphological observations and compositional measurements on samples after completed or interrupted solidification). As most of phenomena involved during solidification are dynamical, our objective is to perform in situ and real-time investigations of the solidification of Al-based alloys by using synchrotron imaging techniques. By this way, we intend to provide benchmark experiments both for the metallurgical and the material sciences communities.

Aims of the experiments

The MA-213 experiments was divided in two parts. During this first set of experiments, noted MA-213a, we adressed mainly on the columnar growth and the Columnar to Equiaxed Transition (CET) during directional solidification of Al - 3.5 wt% Ni and Al-7.0wt%Si alloys. Three samples were prepared: an Al-3.5wt% Ni, a refined Al-7.0wt% Si, a non-refined Al-7.0wt% Si. These samples were typically 40 mm in length, 6 mm in width and 200 µm in thickness.

Observations were performed only by X-Ray radiography, in a "16 bunch" mode, the Synchrotron intensity was varying between 60 - 95 mA. The low number of photons explains the poor quality of recorded pictures, compared to experiments in uniform mode (see previous reports). The X-Ray energy was initially fixed at 13.5 keV like in previous experiments.

Results

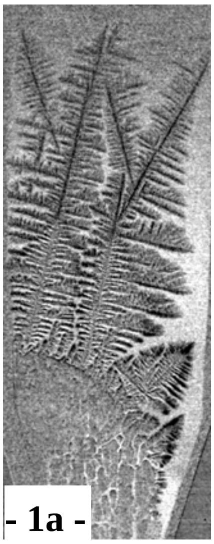
[1] Study of columnar growth and CET during Al-3.5 wt% Ni alloy solidification

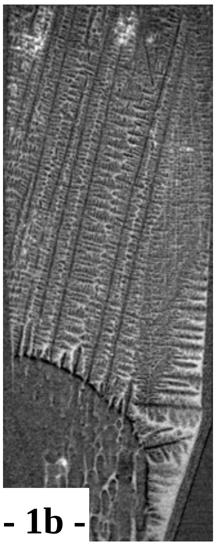
Some preliminary columnar growth were first carried out with control parameters identical to previous experiments, in order to verify the thermal profile in the furnace and then in the sample In a second step, the temperature gradient G in the liquid phase ahead of the solid-liquid interface was gradually decreased down to 6 K/cm, to obtain conditions close to the casting process. This value was the minimal value we were able to apply in the sample. For each value of G, the solidification of the sample was performed by decreasing the temperature of both heaters elements with a rate of R = 1 K/minute, which induced an upward motion of isotherms and consequently the sample solidification. The growth rates were measured after experiments from recorded pictures. In all these experiments, the CET was never observed, may be due to the still high value of the temperature gradient (Fig.1a and 1b).

[2] Study of columnar growth and CET during refined Al-7.0 wt% Si alloy solidification

From previous experiments, we knew that it was possible to observe the solidification microstructure in the case of Al-7.0wt% Si samples, even if the two elements are neighbouring in the Periodic Table. Indeed, even if nothing is visible on the direct recorded picture of the sample, it is possible to reveal the changes in the solidification microstructure by either dividing or subtracting two successive pictures (Figure 2). It is worth to notice that this image processing implies that the sample is fixed in the field of the Frelon camera. However, with the "16 bunch" mode, this image processing only gave us poor quality pictures of the microstructure. We tried to improve the picture quality by varying the X-ray energy and the best result was obtained for 17.5 keV.

In spite this limitation, a great number of solidification experiments were performed. Solidifications were induced by decreasing temperatures of both heaters elements at various rates (R = 1 to 6 K/min). At low growth rate, a columnar microstructure is observed as expected. For higher growth rate, only few equiaxed grains nucleated on the crucible wall, even with refining particles in the alloy. Thus, the main conclusion is that the efficiency of refining particles in the case of Al-7.0wt%Si alloy is much lower compared to experiments with refined Al-Ni samples. This is in agreement with both literature results and experiments carried out with this alloy in our laboratory, that show a poisoning effect of refiners by silicon in Al-Si alloy.





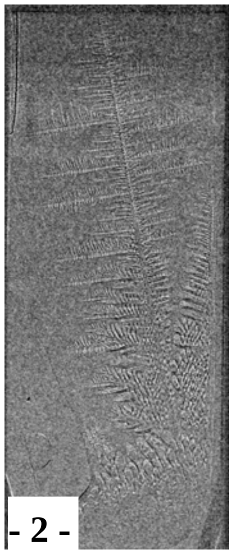


Figure 1a: Columnar growth of Al-3.5wt%Ni, G = 6.7K/cm, V = 24.7 µm/s, **1b**: Columnar growth of Al-3.5wt%Ni, G = 7.8K/cm, V = 85 µm/s, **Figure 2**: Columnar growth of Al-7.0wt% Ni. This picture is obtained after image processing.

[3] Study of columnar growth and CET during non-refined Al-7.0 wt% Si alloy solidification

During Al-Si casting, Columnar to Equiaxed Transition could be observed even in non-refined alloys. Detachment of dendrite fragments, created in the mushy zone of the columnar front and then carried into the liquid phase, is the most plausible origin for equaixed grains. In order to study this point, dedicated experiments with a non -refined alloy were performed.

A great number of experiments were carried out, with the same experimental procedure as described above. In these experiments the poor quality of the recorded pictures did not allow us to conclude if fragmentation is a usual feature in Al-7.0% Si alloy. Thus, we planned to study the fragmentation phenomena with better imaging conditions, in the second part of MA-213 series of experiments.