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: 02/08/2007 to: 02/11/2007	**Date of report**: 05/22/2007
Shifts: 15	**Local contact(s)**: Adeline BUFFET (PhD student)	*Received at ESRF:*

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Scientific background

See ***Ma-213a report***

Aims of the experiments

The MA-213 experiments were divided in two parts. For this second set of experiments, noted MA-213b, three samples were prepared: a refined Al-3.5wt% Ni, a non-refined Al-7.0wt% Si and a quasicrystal AlPdMn. These samples were typically 40 mm in length, 6 mm in width and 200 µm in thickness. The first sample was devoted to thermal calibration (owing to some modifications of the furnace), tests of experimental set-up for X-Ray topography (wheel, post-specimen shutter, beam-stopper) and X-Ray radiography (adjustement of the post-specimen monochromator). The second sample was dedicated to the study of the Columnar to Equiaxed Transition (CET) during directional solidification of non - refined Al - 7.0wt%Si alloys. The main idea was to analyze the mechanical behavior of the dendritic columnar microstructure before and during the fragmentation phenomena, which is a key point during CET. Finally, we intended to deepen our study of the QC quality in their early stages of growth but with a small layer of boron nitride between the sample and the graphite crucible. This layer could change the wetting behavior between the QC sample and the crucible and thus could improve the crystallographic quality of the growing QC if the previously observed defects were related to the interaction with the crucible walls.

Observations were performed by both X-Ray radiography and X-ray topography, in a "16 bunch" mode, the Synchrotron intensity was varying from 95 to 60 mA. The spatial resolution of X-ray topographs is not sensible to the low number of photons but we had to increase the exposure duration by roughly a factor two compared to experiments performed in uniform mode. For the X-ray radiography it seems that, due to the larger bandwidth of the post-specimen monochromator compared to the optical hutch monochromator, the quality of recorded pictures is a little bit better than in the first series of experiments MA-213a, performed in "16 bunch mode" with a monochromatic beam. The X-Ray energy was initially fixed at 13.5 keV like in previous experiments.

Results

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

As usual some preliminary columnar growths with an Al-Ni sample 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) and to adjust the whole experimental set-up for X-Ray radiography and Topography.

In a second step, several solidifications were performed. During these experiments, the temperature gradient G in the liquid phase ahead of the solid-liquid interface was gradually decreased, by decreasing the temperature of the "hot" heater element at a rate of 0.5, 1.0 and 0.1 and 0.2 K/minute whereas the power of the "cold" heater element is fixed. The temperature gradient and growth rates were measured after experiments from recorded pictures. In these experiments CET and fully equiaxed growth (Fig.1) were obtained depending on the value of the cooling down rate. The results are in agreement with previous ones.

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

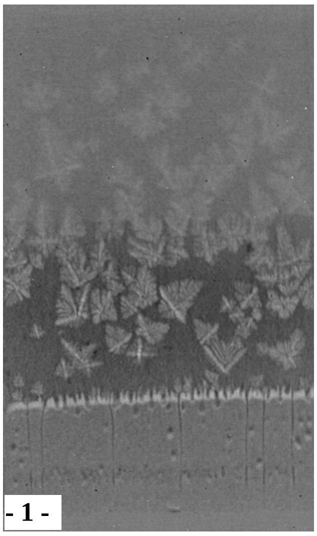
During the first part of MA-213 experimental session, the poor quality of the recorded pictures did not allow us to conclude whether fragmentation is a usual feature or not in non-refined Al-7.0% Si alloy. Thus, we planned to study the fragmentation phenomena with better imaging conditions in this second part of MA-213 experiments. It is worth to recall that it is only possible to reveal the Al-Si solidification microstructure by either dividing or subtracting two successive pictures owing to the small difference of Al and Si absorption coeffcients. The X-Ray energy was adjusted to 17.5 keV.

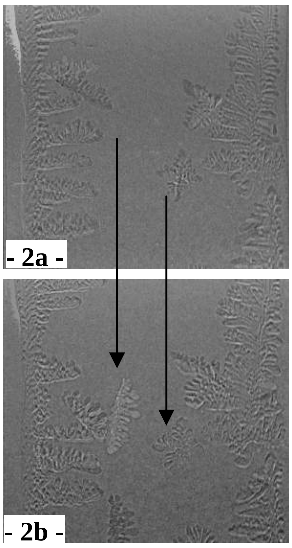
Several solidification experiments were performed, with the same experimental procedure as described above. In experiment at R = 0.5 K/min, a great number of fragmentation were successfully observed and the settling of the dendrite fragment was followed in real-time (Fig.2). These fragments felt down because they are denser than the surrounding liquid and can drag other secondary branches during their motion. Finally, a central equiaxed core was created like in casting process.

[3] Study of AlPdMn alloy solidification

The characterization of the crystallographic quality of small QC grains, during their early stage of development, was performed by X-Ray topography, combined with X-Ray radiography. The post-specimen monochromator was adjusted for an X-Ray energy of 24.0 keV. Two solidifications were performed.

First, it seemed from topographs that the quality of the small QC grains was better in these experiments owing to the boron nitride layer. However, these small QC grains were still not free of strains. Secondly, a striated phase was seen at the end of the growth like in previous QC samples, but for the first time its in situ formation was observed by X-Ray radiography (Fig.3). Complementary post-mortem analysis are in progress to determine the composition of this phase and thus its location in the phase diagram.





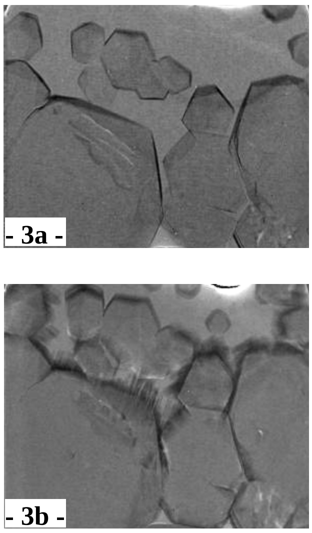


Figure 1: Equiaxed growth of Al-3.5wt%Ni, G = 28.6 K/cm, V = 10 µm/s

Figure 2: Fragmentation and subsequent motion of secondary arms during columnar growth of non-refined Al-7.0wt% Si

Figure 3: Growth of QC AlPdMn: Early stage of equiaxed grain growth and formation of a new striated phase.