ESRF	**Experiment title:** Microtomography of the columnar to equiaxed transition in Al-Ni and Al-Si alloys	**Experiment number**: MA-293
Beamline: BM05	**Date of experiment**: from: 04/18/2007 to: 04/21/2007	**Date of report**: 02/29/2008
Shifts: 9	**Local contact(s)**: Dr. Paola Coan	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

MANGELINCK-NOËL* Nathalie, IM2NP, UMR 6242 CNRS - Universités Paul Cezanne, Campus de Saint Jérôme, Service 142, F -13397 Marseille Cedex 20
JUNG* Hyejin, IM2NP
BOLLER*, Elodie, ESRF
NGUYEN-THI Henri, IM2NP
BILLIA Bernard, IM2NP
BARUCHEL José*, ESRF
HÄRTWIG Jürgen*, ESRF

Scientific background

The objective of research and development in materials engineering science is the optimisation of the materials performance and properties. To reach this aim, the control of the final microstructure must be improved. As a consequence, it is mandatory to deepen the quantitative knowledge of the basic physical phenomena which, from the microscopic scale to the macroscopic scale are governing the microstructure formation in elaboration processes by solidification. The growth forms observed in aluminium alloy casting by industrial processing cover a range of different type of dendritic microstructures from columnar (growth oriented in one direction) to equiaxed grains (grains growing in all space directions). Depending on the application, one type of grain is preferred and thus favoured. In car engines, a fine dendritic equiaxed microstructure is required leading to a material with more isotropic macroscopic mechanical properties and a more homogeneous composition field than columnar microstructure. Conversely, columnar growth is desired in turbine blade casting. As a consequence, it is critical to understand the physical mechanisms which control the transition from columnar to equiaxed growth (CET).

Comparison of experiments with models is necessary to deepen our understanding of the phenomena involved. To be successful, this comparaison must be sustained by the most precise experiments characterisation. Moreover, modelling research work in this field is now moving to 3D simulation of the grain structure. In that frame, 3D characterisation is essential.

Experiments

Directional solidification experiments were performed at IM2NP on both Al-3.5 wt% Ni and Al-7wt%Si alloys to investigate the CET and the equiaxed growth. Al-3.5wt%Ni is chosen as a model alloy. On the other hand, Al-7wt%Si is chosen because it is an alloy of interest for industry. These alloys can be either refined or not as classically done in industrial plants. They are refined when particles acting as germs for equiaxed grains are added. In the frame of CETSOL (Columnar to Equiaxed Transition in SOLidification processing) ESA (European Space Agency) project, microgravity experiments were achieved. The microgravity results are compared to ground experiments to confirm and better understand the effect of natural convection and sedimentation phenomena on the CET.

During the ESRF experiment, we used the experimental device on beamline BM05 to perform X-ray microtomography.

We first performed some microtomography on a refined Al-3.5wt%Ni alloy sample. In the case of this alloy, the contrast is obtained only by absorption because of the high density difference between Nickel and Aluminium. For this alloy, an energy of 40keV was used which gave a satisfactory contrast.

We then moved to several refined and non refined Al-7wt%Si alloys solidified in the same laboratory directional solidification furnace (one refined and four non refined samples) or during a microgravity experiment in a sounding rocket (three non refined samples). In the case of Al-Si7wt%, phase contrast had to be used because the two constituting element of the alloy have similar absorption coefficients. The best compromise to get good quality images was obtained for an energy of 30KeV.

For all experiments, the Frelon camera with the 5µm optics was used. This optics was used to be able to resolve the dendrites secondary brances (arouns 10µm).

Results

For this experiment, we purposedly chose samples which were subjected to solidification conditions leading to a columnar to equiaxed transition. This renders their characterisation even more challenging because there is a transition between grains elongated in one preferred direction and grains growing isotropically in all space direction. Morever, the size of both microstructures is different. The CET was not studied in previous works that focused either on columnar microstructure or on a single equiaxed dendrite [1] and which were limited to Al-3.5wt%Ni alloy.

Nevertheless, this preliminary work showed that it is possible to characterise the microstructures obtained in both alloys (Al-3.5wt%Ni and Al-7wt%Si) at the CET as presented in the following example.

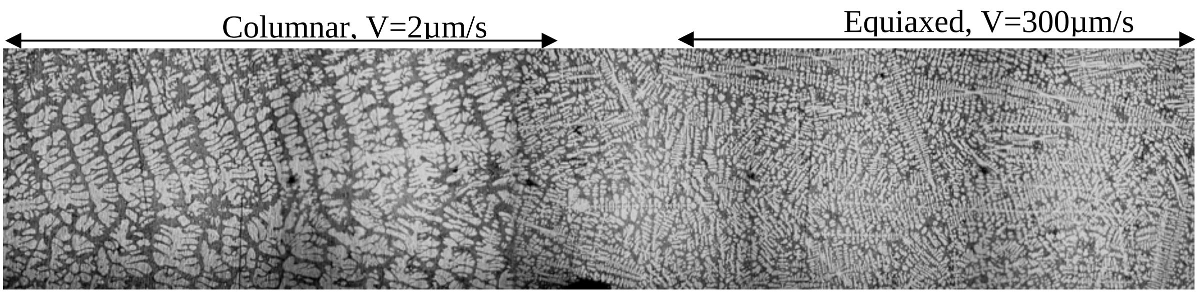
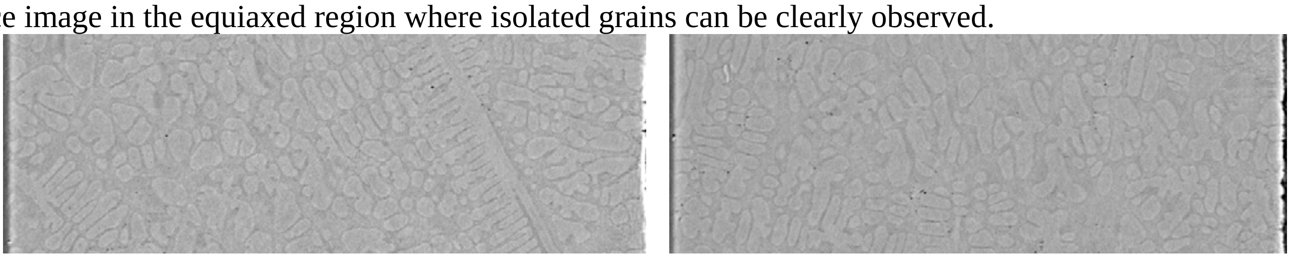


Figure 1: Longitudinal section in the middle of a cylindrical (8mm diameter) sample of refined Al-7wt%Si (G=15K/cm, V=2µm/s to V=300µm/s). Solidification from left to right.

Figure 1 is an image resulting from the mechanical polishing of a refined Al-7wt%Si alloy directionally solidified at a constant temperature gradient of 15K/cm and submitted to a pulling rate jump (V=2µm/s to 300µm/s) to provoke CET. Using this current characterisation method, we are limited to a cut inside the cylindrical sample ususally at its center. CET is observed but with this kind of characterisation, we are limited to a 2D observation of the microstructures arrangement whereas phenomena are truly 3D.

Figure 2 shows two examples of the images that were obtained using microtomography on the same sample. Figure 2.a is a longitudinal slice image in the transition region where isolated grains are competing with some columnar grains (see one columnar grain on the right of figure 2.a). Figure 2.b is a longitudinal slice image in the equiaxed region where isolated grains can be clearly observed.



a) b)

Figure 2: X-ray microtomography in a cylindrical (8mm diameter) sample of refined Al-7wt%Si (G=15K/cm) a) Transition zone: columnar and equiaxed grains V=2µm/s to V=300µm/s, b) Equiaxed grains V=300µm/s. Solidification from bottom to top.

As can be seen above, using the different slices, the 3D arrangment of the equiaxed and columnar microstructures in the whole sample can be characterised precisely with microtomography. Further quantitative measurements are foressen as e.g. eutectic fraction, microstructures size, branching factor and elongation but require important computer performances.

1. M.D. Dupouy, D. Camel, J.E. Mazille, Colloque matériaux, Poitiers, France (2002).