ESRF	Experiment title: **Planar and cellular growth in the Al-Zn alloy system and fragmentation and columnar-to-equiaxed transition in Al-Sn alloy by synchrotron X-ray radiography.**	**Experiment number**: MA-1004
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I. Planar and cellular growth in the Al-Zn alloy system

A dilute alloy of Al-Zn was examined using synchrotron radiography. Owing to the low concentration of solute, solidification experiments were performed with reduced solutal convection effects. Whilst many studies on the nature of the planar solid liquid interface have been carried out using transparent materials that freeze like metals, these experiments provide some of the first data on the planar growth of real metals. The experiments enable the *in situ* and real time study of the breakdown of a planar interface to form cellular and dendritic structures [*W.W. Mullins & R.F. Sekerka, J. Appl. Phys.,* ***35*** *(1964)*] in metals, for the first time in nearly diffusive conditions.
Solidifications were induced by applying the *Powerdown technique (see MA-746 report for instance).* Different cooling rates within the range [0.5 – 15] K/min were applied and Fig.1a shows a typical cellular microstructure obtained during a solidification experiment with a cooling rate of 0.7K/min. Fig.1b shows a succession of images obtained from the same solidification run. The images have been processed heavily to show the interface: image division processing [A. Buffet *et al*, Materials Science Forum, **649** (2010) 331-336] accounts for the 'double' interface in each time step. An edge detection algorithm has been applied to the images so that the interface is fully revealed. The results show an initially planar interface (t = 0), after which depressions emerge at grain boundaries (t = 15s, t = 45s), before protrusions s either side of the grain boundary (t= 81s). A depression then emerges either side of the protrusions (t = 123s). The interface then breaks down to form cells (t = 165s, t = 186s).
According to these observations, the effect of grain boundaries on pattern formation was studied. Grain boundaries have a strong effect on the patterns formed in two ways: the Mullins-Sekerka instability emanates from grooves at grain boundaries, and the instability must match up with grain boundaries, just as with a particle-in-a-box wave solution. This leads to a set of discrete frequencies of the Mullins-Sekerka instability that are optimally stable. Theoretical work is ongoing to rationalize this hypothesis.

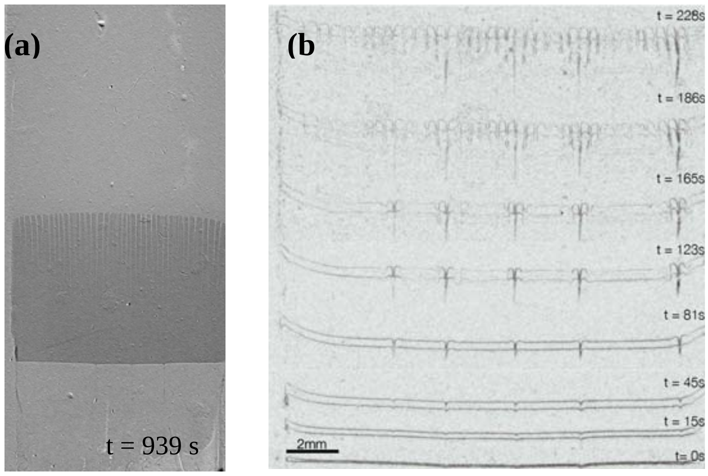

Fig. 1:
(a) Typical cellular microstructure obtained during a solidification experiment with a cooling rate of 0.7K/min.

(b) Succession of images showing the breakdown of the planar interface (t = 0), and the formation of the cellular microstructure.

II. Fragmentation and columnar to equiaxed transition in Al-Sn alloy system

The solidification of an Al -14 wt% Sn sample was characterised by using X-ray synchrotron radiography. Solidification was induced by the *Powerdown technique*. Cooling rate from 0.2K/min to 16K/min was applied to the upper heating zone starting from a temperature gradient of about 30 to 40K/cm.
Numerous fragmentation events were observed during these experiments for all cooling rates but with a higher frequency at higher cooling rates (Fig.2(a)). This is in favor of a mechanism of local solute and heat accumulation that can lead to a local remelting of the secondary branches attaching neck [*K. A. Jackson et al., Trans. Am. Inst. Min, Engrs,* ***236*** *(1966) 153*]. Indeed, at higher cooling rate the rejection of solute and of latent heat are expected to be higher. However, fragmentation was not limited to secondary arms but primary trunk detachment could also be observed (Fig.2(b)). The observation of the detachment of primary trunk in the case of this alloy and composition for which the solid is lighter than the surrounding liquid suggests that there is also a buoyancy mechanism.
In a second step, due to buoyancy, the fragments or primary trunk pieces are flowing upwards, where the liquid is hotter so that some fragments are remelted. Other fragments or detached primary trunk pieces are stopped in their ascension and are growing leading in some cases to the columnar to equiaxed transition (CET) (Fig.2(c)).

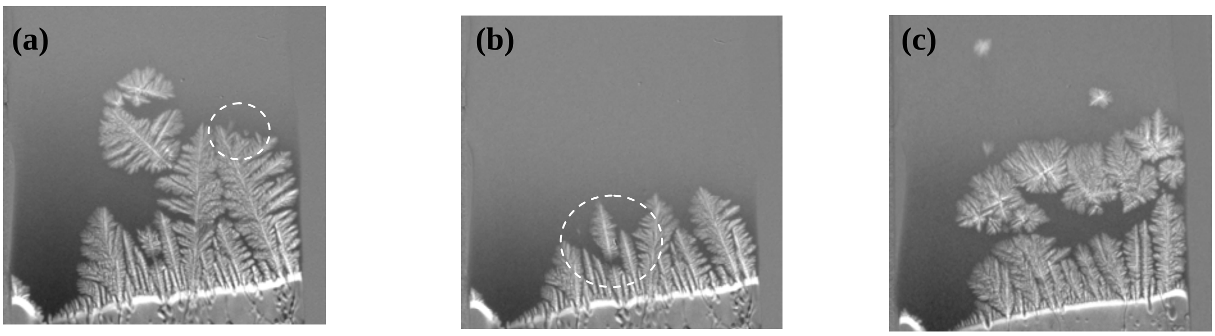

Figure 2: a) Secondary arm fragmentation. b) Primary trunk detachment. a) and b) Al-14wt%Sn solidified with a cooling rate of 8K/min. c) CET in Al-14wt%Sn solidified with a cooling rate of 16K/min.(width of the images= 6mm)

These experiments give incepts of the fragmentation phenomena in metallic alloys and prove that CET can be achieved by fragmentation of the columnar front and subsequent growth of the fragments. The mechanism of fragmentation is still a subject of discussion and a key point to control the formation of the equiaxed structure. These experiments show that several mechanisms are implied (local solutal or thermal remelting, buoyancy). Deeper analysis and further experiments are needed in particular to try to understand the localisation of the fragmentation. Indeed, one pending issue is to know if the localisation of the fragmentation or detachment is linked to mechanical fragile points. This point will be studied in future experiments by combining X-ray synchrotron radiography and topography.