	Experiment title: In situ observation of the nucleation kinetics and the mechanism of grain refinement in Al-Si alloys	**Experiment number:** **MA 709**
Beamline: ID11	**Date of experiment:** From: 30 January 2009 to: 2 February 2009	**Date of Report:** 21 October 2009 Received at ESRF: 22 October 2009
Shifts: 10	**Local contact(s):** Jonathan P. Wright (email: wright@esrf.fr)	

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Report:

Mechanical properties of as-cast alloy are very dependent on the solidification process. It is well-known that grain refinement enhances properties of castings. It is also known that Al-Ti-B master alloys are the very effective grain refiners. For Al-Si foundry alloys, those TiB_2 containing grain refiners without excess titanium such as Al-3Ti-3B are found to be more effective than those with excess titanium (Al-5Ti-B). This is attributed to the effect of alloying elements of Si and Mg. It is notable that there are interesting discussions on the refinement mechanism on this type of alloy. In-situ studies can help advancing the nucleation theories substantially.

Grain nucleation and grain growth for few different casting conditions of a commercial aluminium alloy (A356: Al-7Si-0.4Mg-0.1Fe-0.1Ti wt%) were investigated using three dimensional X-ray diffraction microscope (3D/XRD) located at ID11. A monochromatic hard X-ray beam (energy of 70 keV) with a beam size of 200×200 μm^2 was used. Samples were placed into a cylindrical container of glassy carbon (height: 25 mm, inner diameter: 5 mm, wall thickness: 1 mm). This container was placed into a Quartz tube, which was vertically aligned inside a furnace. X-ray beam was transmitted through an orifice placed on the furnace. The furnace was fixed to a rotation table enabling sample rotation alongside the vertical axis. However, this rotation was less than 10 degree. Heating and cooling of the samples were operated under vacuum of 10^{-5}. The x-ray diffractions from liquid state give two rings on the 2D detector, as the solidification begins spots, representing the grains appear and grow. Collecting and measuring their numbers and intensities gave important data about the grain size and solid phase fraction.

To study solidification, samples were cooled from 650°C to 400°C at different cooling rate of 2.5 K/min to 25 K/min following a 15 min holding time at 650°C. Samples were: not grain refined and grain refined with different amount and type of grain refiners: (0.05 and 0.1 wt% Ti as Al3TiB; 0.1 and 0.4 wt% Ti as Al5Ti). Samples grain refined with Al5Ti did not show significant amount of grain refinement compared to the rest and alloy with 0.1 wt% (TiB_2) gave the best refining effect.

Figure 1 shows the evolution of nucleation and the solid fraction for two different alloying conditions of non-grain refined and grain-refined with 0.1 wt% Ti (as Al-3Ti-B) under two different cooling rates of 2.5 K/min and 7.5 K/min.

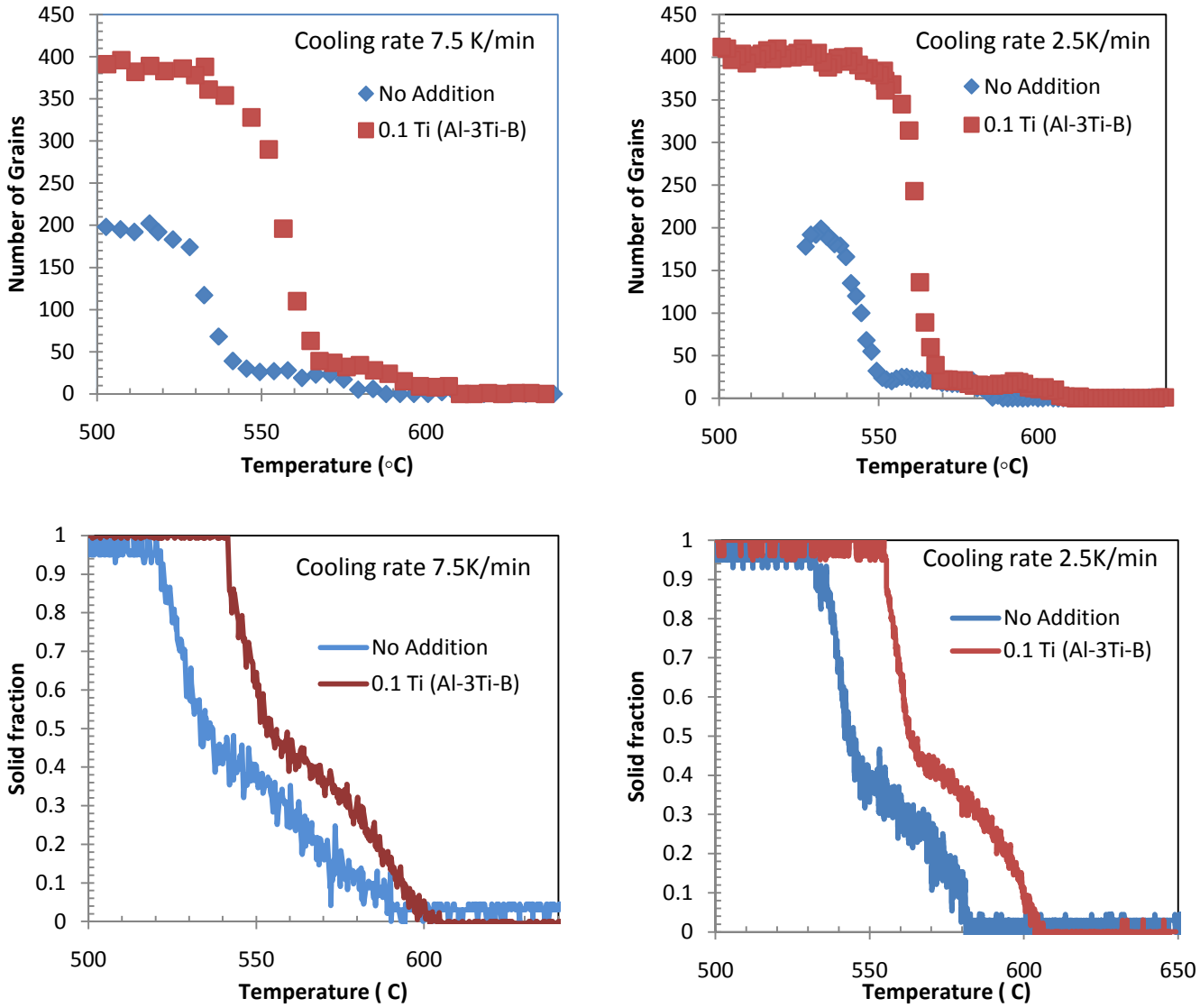

Figure 1. Effect of grain refinement on the nucleation of primary phase in a commercial alloy of A356 at two different cooling rates of 2.5 and 7.5K/min.

Comparing the evolution curves, shows that how adding 0.1 wt% Ti (TiB_2) lifts the nucleation temperature of the primary phase by ~20 K. Interestingly it also advances the eutectic reaction temperature by ~20 K. These unique observations of microstructure evolution during solidification are the first experiments of such for a commercial Al-Si foundry alloy. To have better understanding of the alloy's solidification, further investigation with different type/amount of additives (eutectic modifier as well as grain refiners) is suggested.

Publications resulting from the experiments:

[1]. M. Faraji, J.P. Wright and L. Katgerman, Materials Letters 64 (2010), pp. 1016-1018
[2]. M. Faraji, J.P. Wright and L. Katgerman, to be submitted to Journal of Materials Science