ESRF	**Experiment title:** Nucleation kinetics in liquid aluminium alloys with TiC grain refiners during solidification	**Experiment number**: HD-283
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Report:

Grain refinement plays an important role in the liquid to solid phase transformation of aluminum alloys. Generally TiB_2 and TiC nucleating particles are added to the aluminum alloys to refine the grain size of the solidified product. These alloys contain microscopic TiB_2 and $TiAl_3$ nucleating particles, which act as a substrate for heterogeneous nucleation of solid aluminium grains during solidification. This results in a reduced grain size of the solid aluminum, at room temperature. The mechanical properties are significantly improved by the decrease in the average grain size. The grain refining particles are effective, only in the presence of a small amount of solute titanium in the melt. Our previous in-situ experiments at ID11, for the study of grain refinement process in aluminum alloys containing TiB_2 particles along with solute titanium, have investigated the mechanism of grain refinement quite in detail. The current experiment aimed (1) in the comparative study of of grain refinement process in aluminum alloys with TiC and TiB_2 particles, so as to determine the grain refinement efficiency of these two different type of particles (2) how does the grain refinement depend on the size of the added particles? Therefore three different aluminum alloy samples were prepared, such that the first sample contained 0.1wt. % of TiC particles (with average particle size 40 nano meter), 2^{nd} alloy contained 0.1wt. % of TiC particles (with average particle size 2 micrometer) and 3^{rd} alloy contained 0.1wt. % of TiB_2 particles (with average particle size 2 micrometer). Also each sample contained 0.1 wt. % solute titanium.

The X-ray diffraction measurements were performed using the three dimensional X-ray diffraction microscope (3DXRD) at beam line ID11 in transmission geometry. A monochromatic X-ray beam with an energy of 70 keV (wavelength of 0.177 $Å^{-1}$) and a beam size of 200×200 μm^2 illuminated the 5 mm diameter of the sample (with a height of 10 mm) that was mounted in a glassy carbon container within the vacuum furnace. A continuous sample rotation of 1^o around the vertical axis (perpendicular to the beam) gives rise to a diffraction pattern on the two-dimensional detector that is placed behind the sample. This pattern gives direct information on both the liquid and solid phases during the solidification process.

Each sample was heated to 973 K, and then cooled with different cooling rates. The X-ray diffractions from the liquid phase resulted in two broad rings on the 2D detector, indicating the maxima in the liquid structure factor resulting from short-range order of the aluminum atoms. In the mixed phase, during solidification, the intensity of the broad rings reduces, and a limited number of diffraction spots from the solid grains is observed at the diffraction angles corresponding to reflections of the face-centered cubic lattice structure of aluminum. In the solid phase the broad rings of the liquid phase completely disappear and the diffraction spots show an increase in number and intensity. According to standard diffraction theory the number of spots detected is proportional to the number of illuminated grains and the intensity of each spot is proportional to the volume of the grain from which it originates. Figure 1, illustrates the evolution of grain nucleation and the solid phase fraction for three alloy samples, during solidification, at two different cooling rates of 40 K/min and 10 K/min.

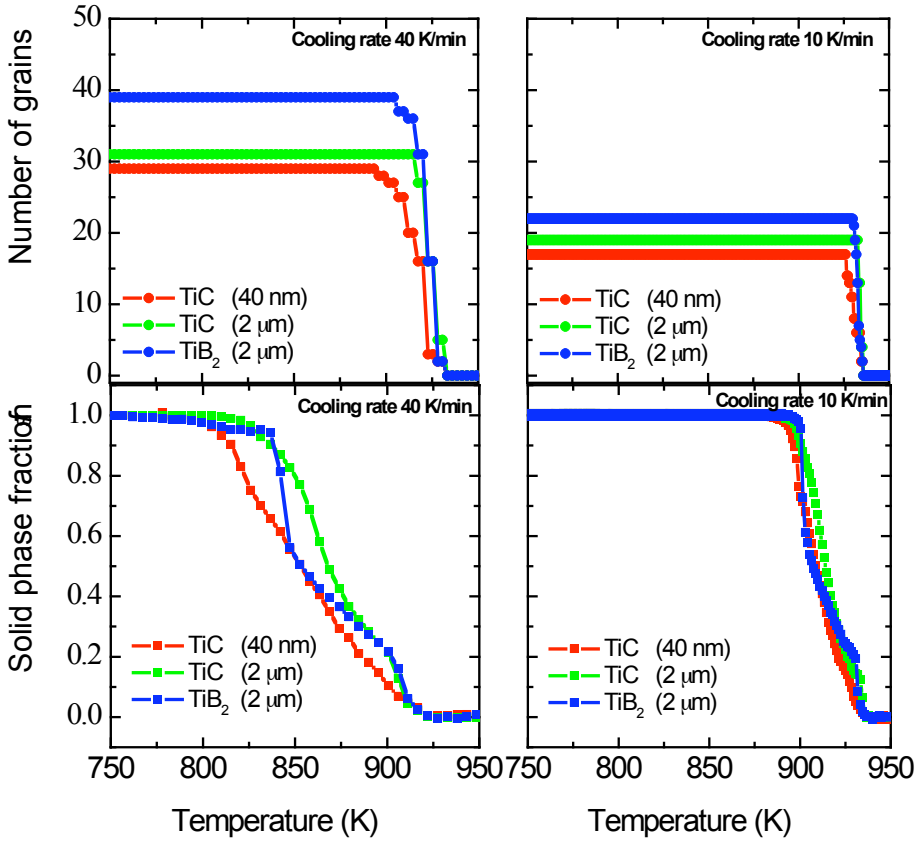

Fig. 1. *Grain nucleation and solid phase fraction of three aluminum alloys during solidification, with TiC (40 nanometer and 2 micrometer particle size) and* TiB_2 (2 micrometer particle size) *grain refining particles, as a function of temperature for different cooling rates. Each alloy contains 0.1 wt. % of solute titanium.*

The comparison of grain nucleation curves for both the cooling rates, clearly demonstrate that the micrometer size TiB_2 particles exhibit enhanced grain nucleation during solidification, compared to the TiC particles of the same size. Also the TiC particles with 2 μm size, appear to be efficient grain refiner compared to TiC particles of average size about 40 nanometer. Further, our measurements confirm that the nucleation process is limited to the initial stage of the solidification and is complete at a solid phase fraction of about 20% for all samples.

Publications resulting from the experiment:

[1] N. Iqbal et al., submitted to Scripta Mat.
[2] N. Iqbal et al., to be submitted to Acta Materialia