ESRF	**Experiment title:** Ultrafast in situ microtomography during solidification of Al alloys submitted to ultrasonic melt treatment	**Experiment number**: MA2984
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Report

Aims:

The processing of Al-based Metal Matrix Nano-Composites (MMNCs) requires the application of Ultrasonic Melt Treatment (UST) in order to prevent the natural agglomeration of the ceramic nano-particles and improve their dispersion inside the Al alloy. This experiment aimed to capitalize on the efforts made to develop an *in situ* setup designed to apply UST prior to solidification and investigate the particles behaviour towards the solidification front for different experimental conditions.

Experiments:

Based on previous results obtained at the ID15 high energy beamline, the already existent set-up, especially the crucible container was adapted to the high flux 19 keV configuration of the ID19 beamline. However, despite sustained efforts and partial achievements of the experiment, no undoubtful results could be obtained with this configuration. It is believed that the necessary reduction of the crucible section (due to low incident beam energy) prevented the full propagation of the ultrasounds inside the melted area to be probed.

Fortunately, we could adapt another set-up dedicated to the investigation of Mg-based MMNCs processing but without the possibility to perform UST as the samples are placed inside a sealed quartz tube filled with Ar gas to prevent self-ignition of the material during heating. Two *in situ* experiments were however performed using a resistive furnace:

- One in which the the sample was partially remelted at slow heating rate (5K min-1 then 0.75 K min-1 once semi-solid), isothermally held at about 575°C for 30 min and then solidified at a cooling rate of 17K min-1
- The other in which the sample was fully remelted and then solidified at a cooling rate of 5 K min^{-1}

The material was a Mg-based alloy Elektron 21 (El21) containing 1 wt% of yttria particles (size ~500 nm). The beam energy was set to 19.6 keV, the detector was a PCO Dimax camera, the pixel size was set to 1.1 µm and the field of view was 1008 x 720 pixels.

Results:

Figure 1 shows a solidification sequence acquired during the case of the second experiment (from the fully liquid state). The recorded 3D images allowed us to study and analyse the complexity of the particle – solidification front interactions taking during dendritic solidification of the material. We were able to classify the different mechanisms undergone by the particles (pushing, engulfment, entrapment etc…) as a function of the different experimental conditions leading to different Solid-Liquid interface (SLI) shapes and velocities.

Such *in situ* experimental investigation surprisingly revealed several discrepencies with the expected particles behaviour predicted by the different models encountered in the literature. One of the main unconsistency lies in the fact that almost all particles were observed to be pushed by a high velocity SLI whereas models foresee engulfment by the solid phase in this case. Such observation can be explained when taking into account several parameters that are often omitted in models (SLI shape, gravity, liquid flow etc…)

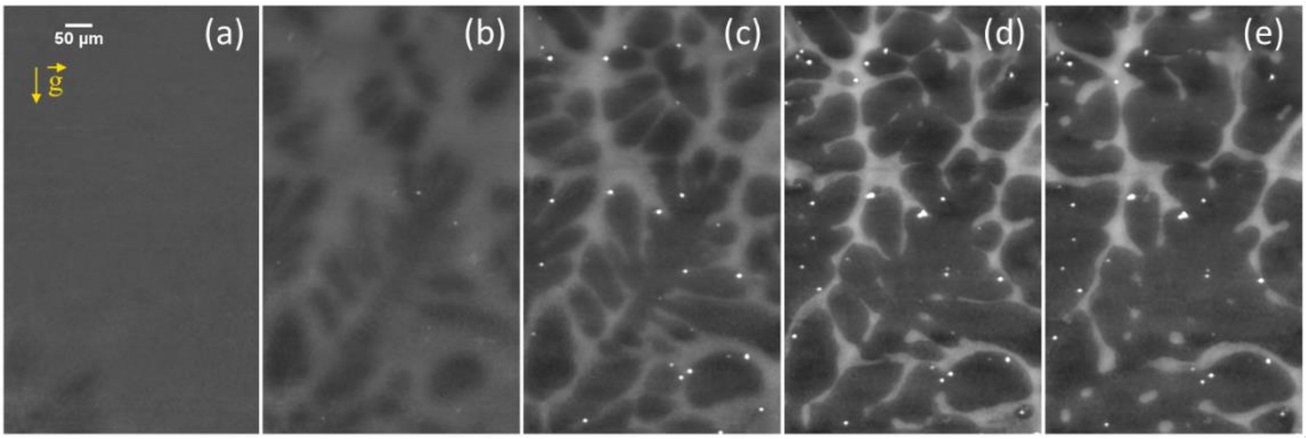

Figure 1: Sequence showing the solidification after reheating the matrix to the fully liquid state. The particles are engulfed and entrapped during the later stage of solidification

Valorisation:

This experiment was involved in the now closed european project ExoMet that aimed to study the fundamental phenomena occuring during MMNC processing. Despite the non-achievement of the expected experiments on Al-based MMNCs processing submitted to UST, the experimental results obtained on Mg-based MMNCs are highly valuable for the project, especially to the theorerical community for further improvement of the different models. These results will be submitted to the Acta Materialia journal in a very near futur.