



	<b>Experiment title:</b> Dendrite fragmentation for microstructure control using pulsed electromagnetic fields during solidification	<b>Experiment number:</b> MA-2035
<b>Beamline:</b> ID19	<b>Date of experiment:</b> from: 30 -10-13                      to: 3-11-13	<b>Date of report:</b> 28-08-14
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**Report:**

In-situ synchrotron X-ray radiography studies of the solidification of Al-Cu alloys under the influence of a pulsed electro-magnetic field (PEMF) were carried out at ID19 during beamtime MA-2035.

Videos of the formation of both columnar and equiaxed dendrites under various solidification conditions were recorded with and without the presence of the PEMF. A total of 264 sequences were collected corresponding to 3.5 TB of data. The beamtime objective can be summarized as follow:

- 1- To investigate solute segregation effects in the melt on fragmentation efficiency under a PEMF;
- 2- To study the solute field destabilization effect induced by different type of pulse of the PEMF;
- 3- To undertake PEMF experiments on grain refined alloys with equiaxed microstructure.

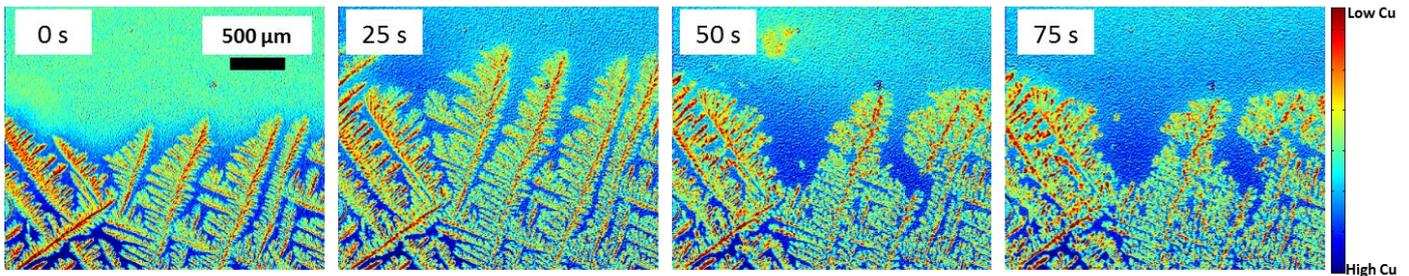
The experiment were performed using our bespoke solidification rig that was designed to study the dynamic of solidification phenomena using X-ray synchrotron radiography. Its portable and flexible design includes a Bridgman furnace combined with an external Pulse Electro-Magnetic Field (PEMF) device capable of generating a variety of solidification and fluid flow conditions.

Al-Cu alloy foil samples with 10, 15, 20 and 25 wt%Cu content were prepared by melting and homogenizing high-purity Al (99.999%) and Cu (99.99%) and adding 0.1 Al-5Ti-1B to the specimens dedicated to the study of equiaxed growth. The samples were then sliced and polished both side to obtain 10 x 25 x 0.2 mm<sup>3</sup> foils which were covered with a layer of BN powder and encapsulated into a quartz cell.

Two main experimental set up were used: one for the study of fragmentation of a columnar array of dendrites and the second designed to study equiaxed growth. In the first the solidification was directed parallel and anti-parallel to gravity and a thermal gradient was imposed to induce the dendrite to grow from the cold towards the hot side. In the second set up the hetares were aligned horizontally and the samples cooled isothermally at a constant cooling rate varying between 0.3 and 1.5 K/s. All the experiments were repeated with and without PEMF and at least three times in each condition.

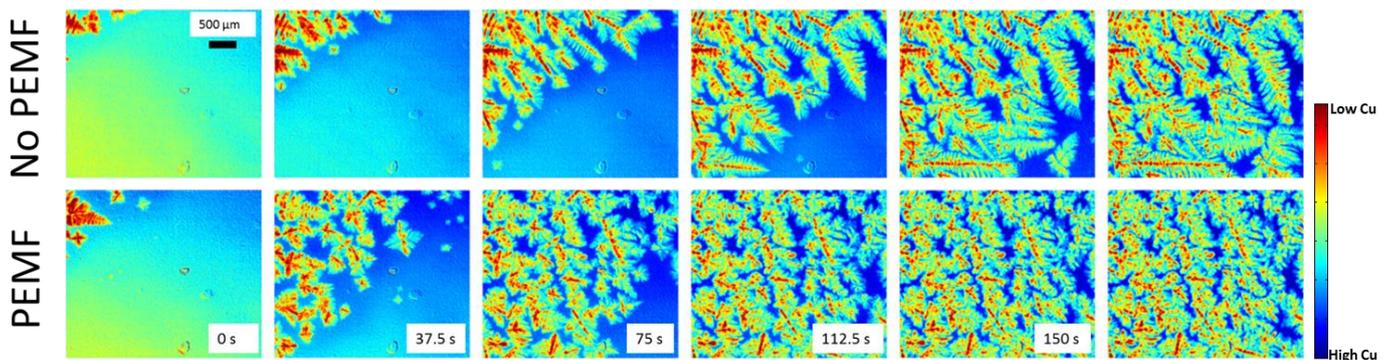
## Results

**Columnar growth:** The fragmentation of a columnar array of dendrites was studied in a quasi-static arrangement where the dendrites were induced to grow until they reached approximately the middle of the field of view and then the PEMF was switched on. Fig. 1 is an example of one of the solidification sequences with PEMF showing the fragmentation of the mushy zone. Also, there was enough absorption contrast to investigate the effect of the PEMF on the solute field within the liquid in the interdendritic regions. The data shows that the PEMF heavily influenced the solute field well deep into the mushy zone destabilizing the equilibrium condition at the solid liquid interface enhancing the detachment of dendritic arms.



**Fig. 1:** image sequence of the fragmentation of a dendritic array of Al-25Cu induced by PEMF.

**Equiaxed solidification:** A wide range of stirring patterns and solidification conditions were tested in order to reproduce realistic casting scenarios. Some preliminary results suggest forced convection can induce clustering of the small floating nuclei and creates ‘*nucleation free zones*’ which then lead to inter-granular segregation and elongated grains formation. However, fluid flow can be manipulated in such a way to enhance nucleation and limit grain growth to obtain a more uniform grain size distribution as shown in Fig. 2. The applied Lorentz force acted along the vertical direction pushing the liquid up and down with a frequency of 0.1 Hz. The stirring action induced a fluid flow which destabilized the solute distribution in the liquid in front of the solid liquid interphase and enhanced the nucleation of dendrites and their impingement on the solid front which as a consequence obstructed the growth of the previously formed grains. The final microstructure obtained with PEMF retained a smaller and more equiaxed grains compared with the one solidified without PEMF.



**Fig. 2:** image sequences of the solidification of Al-25Cu + 0.1 TiB<sub>2</sub> with and without PEMF. Isothermal cooling, cooling rate = 0.1 K/s, oscillatory stirring generated with a sine wave pulse at 0.1 Hz.

## Conclusion and future work

Data analysis is underway and requires the quantification of the fragmentation in the columnar experiments and the measurement of the nucleation rate and growth in the equiaxed. An automatic algorithm has been developed within our group for the extraction of meaningful quantitative information from the videos. Preliminary results from MA-2035 were already presented at 2 international conferences (EMRS 2014, Lille, France – ICASP4, London, UK), a first publication regarding the equiaxed growth is currently in preparation for submission to a high impact journal and will be followed by a series of other publications on the topic of dendrite fragmentation, electromagnetic stirring of Al-Cu alloys and equiaxed solidification.