	Experiment title: **Time-resolved X-ray imaging of equiaxed dendritic growth in Al-Cu alloys**	**Experiment number**: ME595
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Report:

Dendritic growth in 9 different alloys of the AlCu system has been studied *in-situ* by time-resolved X-ray radiography. Samples were prepared with three constitutions at 10, 15 and 20wt%Cu, with particular precautions taken in the casting process to minimize gas porosity. It has been found in our earlier work that gas pore formation leads to severe degradation of sample contrast and lifetime. For each alloy constitution separate batches were made with additions of TiB_2 grain refiners in concentrations 0.05 and 0.2 wt%, respectively. Grain refiner additions increase nucleation rates, thus promoting equiaxed over columnar dendritic growth. Furthermore, when nucleation site density is increased, growing grains will start to interact with adjacent neighbors at an earlier stage, which gives a prominent effect on the terminal grain size distribution. Accordingly the three batches prepared for each constitution provided the basis for three different growth modes to be studied: columnar dendritic (batch without grain refiners), coarse equiaxed dendritic (0.05 wt%TiB_2) and finer equiaxed dendritic (0.2 wt%TiB_2).

Monochromatic energies at 13 and 15 KeV were used together with the FReLoN 2000 detector setup in the 4-channel read out with a 2×2 hardware binning of the CCD, employing a medium resolution 12 μm thick, transparent scintillator. All together this configuration provided nominal resolution of about 2.5 μm and a read-out dead time of about 150 ms, with a direct computer memory storage capacity of 300 frames per sequence. The samples were mounted in a custom made furnace system rig, designed to carry out directional solidification. The solidification processes were controlled by adjustments of furnace temperatures, furnace inter spacing and sample pulling velocities, yielding imposed thermal gradients, G from 12 to 120 K/mm, and global cooling rates, dT/dt, from 0.3 to 9 K/s.

In total 97 video sequences of solidification were acquired, constituting about 60 GB of raw data.

The primary aim of the experiment was to study interactions between adjacent equiaxed grains, and in particular how such interactions regulate the growth process. The grain refined alloys at 10,15 and 20 %wtCu provided data on growth, interaction and mushy zone grain coagulation over a broad range of grain size and density distributions. By varying the magnitude and direction of G (parallel and anti-parallel with gravity) the differences in mass densities between crystallized α-dendrites (< 3wt%Cu) and their parent melts provided data sequences over a large variation in crystal mobility by floatation and thermo-solutal convection, both of major importance with respect to grain coagulation and the mushy zone permeability. Apart from the high level grain refined 10wt%Cu samples, where gas porosity caused inadequate sample lifetimes to obtain data over a satisfactory variation of solidification parameters, lifetimes and contrast preservation throughout consecutive cycles of solidification and remelting were generally satisfactory. In contrast to our previous experience, slight contrast degradation was observed due to the Born-Nitride sample spray coating. New measures will be taken to avoid this problem in the future. Analysis of the vast material on equiaxed growth and grain interactions is on its way.

Samples without grain refiners were used to carry out a more dedicated study on columnar dendritic growth with emphasis put on fragmentation, which we have observed phenomenologically in previous X-ray experiments. Fragmentation refers to the detachment of solid fragments from the mushy zone, which due to either a lower density than the melt, or to liquid flows tend to float out of the mush and interrupt the columnar growth front. The full nature of fragmentation is still under debate, the most common explanation being that fragmentation occurs by the remelting of ternary dendrite arms at the root due to recalescence deeper in the mush. Another possible model is a thermo-mechanical combining local stresses caused by convective flows with the effect explained above. Evidence to support this model has been found in ME595, and also yet another possible nucleation mechanism for fragmentation was observed. Gas porosity which settles on the surface of primary crystals fronting the mush, can cause a local reduction of heat capacity, and also in our confined 3d geometry, introduce a narrowing of liquid channels in the mush. Thermal pulses or local liquid flow brought by convection can thus induce a local detachment of fragments either by remelting or by mechanic forces. It has also been observed that fragmentation, once nucleated, escalates. Thus, the on-set of fragmentation will eventually result in so-called columnar to equiaxed transitions, a transition which in many casting processes is sought for in order to achieve better component performance characteristics.

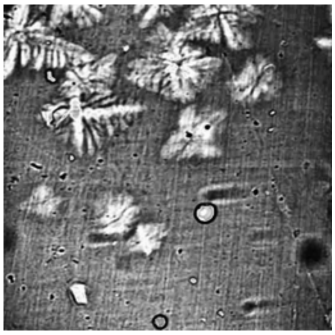

Figure 1. Grain refined equiaxed growth in Al-15wt%cu. Grains at lower left grows while floating up and then coagulatewith other grains.

The results from ME595 will be presented at intl. conferences on solidification, and will combined with our previous results be presented in two papers to be prepared for journal submission. The data on equiaxed crystal growth and coagulation in convective flows will be of importance in verification and guiding of process modeling. Fragmentation is a hot topic in solidification science. The results already obtained, combined with possible future experiments will offer an unprecedented opportunity to study the only known potent grain refinement mechanisms for many alloy systems.

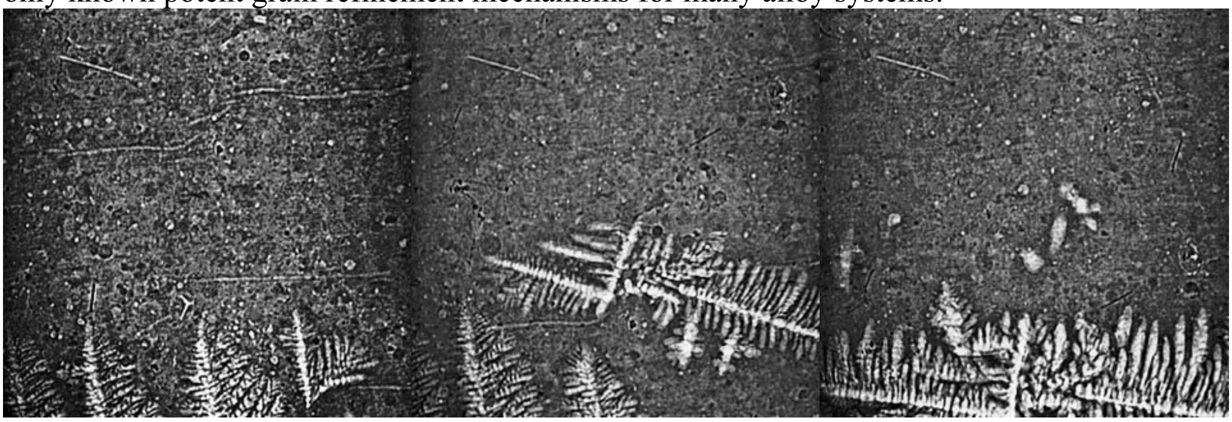

Figure 2. From left to right.1) Detatched ternary branch growing rapidly in the melt. 30 frames (10 s.) later where the fragment has grown to a large crystal preventing further growth of the columnar front. Two other fragments have loosend and coagulated at the colder side of the primary fragment 3) 45 frames (15 s) later: the columnar front has been replaced by the equiaxed crystal, five new fragments have just detatched from the new front.