ESRF	**Experiment title:** **The interaction of ceramic particles with a dendritic solidification front**	**Experiment number**: MA 88
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Report:

An interesting problem in metal physics is the interaction of a solid / liquid interface with inert particles. In case of a dendritic interface the scenarios of an interaction with the particles are: (i) engulfment of particles by primary dendrite tips, (ii) engulfment of particles by secondary dendrite tips, (iii) entrapment of particles between dendrites, (iv) pushing of particles by a single dendrite, (v) cooperative pushing of large particles by two or more dendrites.

We conducted an experimental investigation of the growth of dendrites under directional solidification conditions using a Bridgman-type gradient furnace for processing the alloys at ID 19. Absorption contrast radiography was used to observe dendrite growth *in-situ* in thin foils of two alloy systems: (i) $Al_{90}Cu_{10}$ containing Al_2O_3 particles (2 samples) and (ii) $Al_{90}Cu_{10}$ containing ZrO_2 (4 samples) at high spatial and temporal resolution. The aim of the experiments was to identify the above mentioned interaction scenarios. In a previous campaign, we had used samples, which were coated with a layer of boronnitride and were processed inside of a container made of thin carbon foils (thickness 130 and 250 µm). The carbon container had been identified as the reason for a speckled background in the images. To avoid this non homogeneous background we tested BN containers in this campaign.

Results: (i) $Al_{90}Cu_{10}/Al_2O_3$: Only a few hours were spent for the visualization of Al_2O_3 in $Al_{90}Cu_{10}$ alloy. The experiments were not successful and further experiments were postponed in favor of the much more promising and successful experiments with $Al_{90}Cu_{10}$ plus ZrO_2 particles.

(ii) $Al_{90}Cu_{10}/ZrO_2$: Solidification experiments with additional particles in the melt are quite ambitious from the viewpoint of in-situ visualization with x-rays. Contrast has to be established between dendrite, melt and particles. This aim was achieved with the system $Al_{90}Cu_{10}$ with ZrO_2 particles (Figure 1). The dendrites (bright) can be distinguished clearly from the melt (grey) and the particles (dark). The resolution for particles is around 20 µm. It is remarkable that the background noise arising from the BN containers is much weaker than with the carbon container in previous experiments.

The directional solidification of the alloy underlies, of course, the parameters temperature gradient and pulling velocity. By variation of these parameters the dynamics of the solidification has been varied from equiaxed growth to growth of a single dendritic front. As a special operation mode of the furnace, we used to lower the temperature of the lower heating element. This led to stable solidification conditions with the advantage that no sample movement is necessary. In this case image analysis can be performed directly during the experiment. An example is given in Figure 2. The image has been produced by substracting two images, which are separated by 440 s. The parts of the dendrites, which have been grown in the meantime appear bright. Particles, which have not been moved are given with faint contrast. Those, which have been moved, appear as a pair of black-white particles.

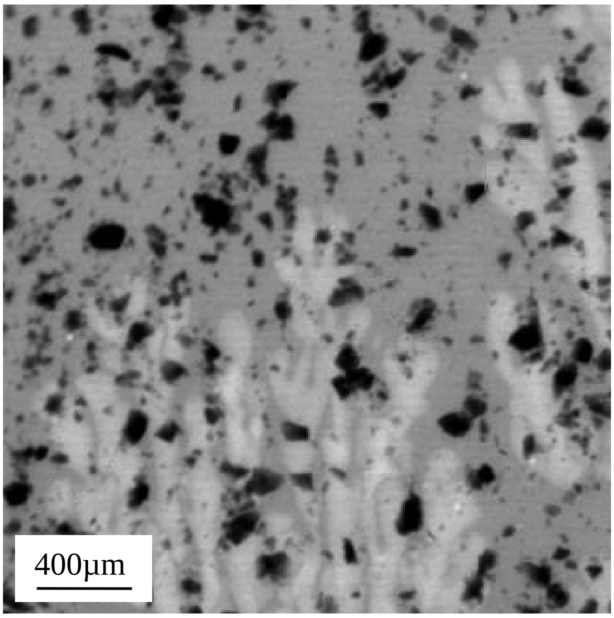

Figure 1: In-situ radiography of $Al_{90}Cu_{10}$ + ZrO_2 particles (P5) taken with 18.3 keV at ID 19. Al-rich dendrites (bright), Al-Cu melt (grey) and ZrO_2 particles (black) are resolved.

Several typical scenarios for particle-dendrite interaction could be analyzed by using this technique. The particle marked by an arrow in Figure 2 might be associated to dendrite splitting. In other cases the splitting of the dendrites happens without involving a particle. The reason is the low anisotropy of the surface energy of the Al-dendrite, which leads to spontaneous splitting.

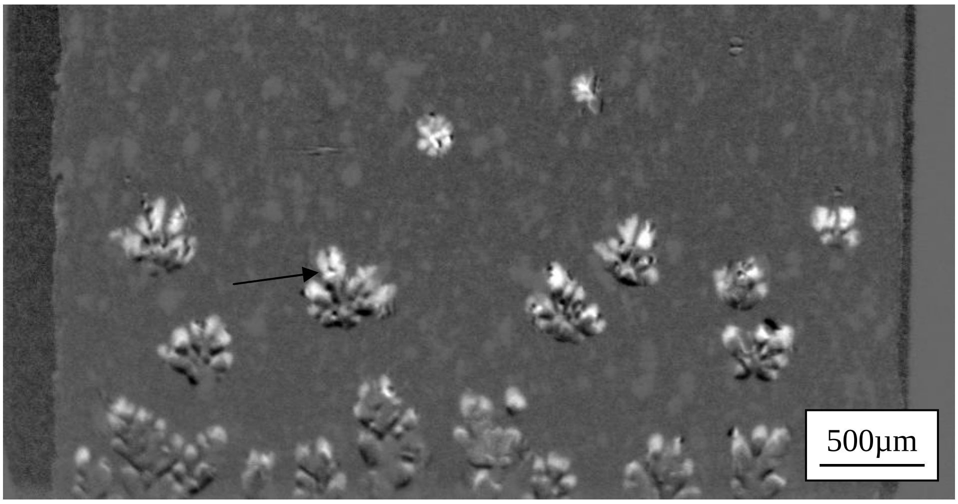

Figure 2: Equiaxed growth of $Al_{90}Cu_{10}$ with ZrO_2 particles. The image is the difference between two images taken 440 s after each other. Bright features have appeared *after* the first image has been taken.

Conclusions: The Bridgman furnace at ID 19 and the recommended sample holder design were suitable to directionally solidify the proposed metal/particle systems. The replacement of carbon by BN as container material reduces background noise. Growth of dendrites was observed in $Al_{90}Cu_{10}$/ZrO_2. Particles, dendrites and melt show sufficient contrast. Scenarios of particle-dendrite interaction as tip splitting could be observed. A publication is planned after further analysis of the received data.