	Experiment title: **The interaction of ceramic particles with a dendritic solidification front**	**Experiment number**: ME 921
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Report:
Many casting processes require a detailed knowledge on dendritic growth during directional solidification. MMC material (metal matrix composite) is usually produced by expensive powder metallurgy. An alternative MMC production process, which solidifies the material directly from a particle containing melt, requires an understanding of the interaction of the ceramic particles with the advancing solidification front *and* the growth conditions. Interaction scenarios between particles and advancing dendrite are the following:

- Engulfment of particles by primary dendrite tip
- Engulfment of particles by secondary dendrite tip
- Entrapment of particles between dendrites
- Pushing of particles by a single dendrite
- Pushing of large particles by two or more dendrite tips

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) $Cu_{90}Sn_{10}$ containing Ta_2O_5 (1 sample) at high spatial and temporal resolution of 2 µm and 2 Hz, respectively. The aim of the experiments was to identify the above mentioned interaction scenarios. The samples were coated with a layer of boronnitride and were processed inside of a container made of thin carbon foils (thickness 130 and 250 µm). In order to evaluate the influence of the container on image quality, we compared images at high resolution taken from the solidified sample *with* and *without* carbon foil, which is due to the structure of the foil. To avoid this non homogeneous background we plan to test quartz or BN containers in a further campaign.

Results: (i) $Al_{90}Cu_{10}/Al_2O_3$: The experiments were successful in the sense that the entire process of dendritic solidification has been recorded from initial dendrite nucleation followed by dendrite growth to complete grains in the Al-based system. An example is given as a sequence in Figures 1a-c. The advancing Al-rich

dendrite is clearly to observe, the contrast between Al-rich dendrite and residual Al-Cu liquid is sufficient. Interesting parameters as dendrite velocity, dendrite tip radius, dendrite trunk diameter and secondary dendrite velocity can be directly determined. The Al_2O_3 particles (Ø 10-30 µm) are not visible under this recording conditions. A systematic determination of the interesting parameters as mentioned above for individual dendrites is on the way.

The feeble contrast between alloy and particles in the system $Al_{90}Cu_{10}/Al_2O_3$ does not allow to visualize the particles. This is not surprising, because of the similar X-ray absorption of Al-rich dendrites and Al_2O_3 particles. The reason for choosing this system was the low bouyancy of the Al_2O_3 particles in the melt. Hence, the solidified samples have to be investigated ex situ by complementary metallographic techniques in order to determine the locations of Al_2O_3 particles. These investigations are currently carried out. We have observed dendrite tip splitting in-situ, a reason could be the interaction with a particle. In such a case, the particle will be embedded at the bottom of the splitted tip.

(ii) $Cu_{90}Sn_{10}/Ta_2O_5$: The second system under investigation was $Cu_{90}Sn_{10}$ with Ta_2O_5 particles (Ø 0,5-20 µm). The particles were clearly visible, but the contrast between the Cu-rich dendrites and the residual melt is too weak, so that dendrite growth cannot be observed in general. However, in few sequences a weak contrast from dendrites could be observed, but it was not stable. It is thought that the incident beam and crystal planes of the dendrite were in the vicinity of a Bragg angle, which gave rise to a weak diffraction contrast when the dendrite moved slightly during solidification of the sample.

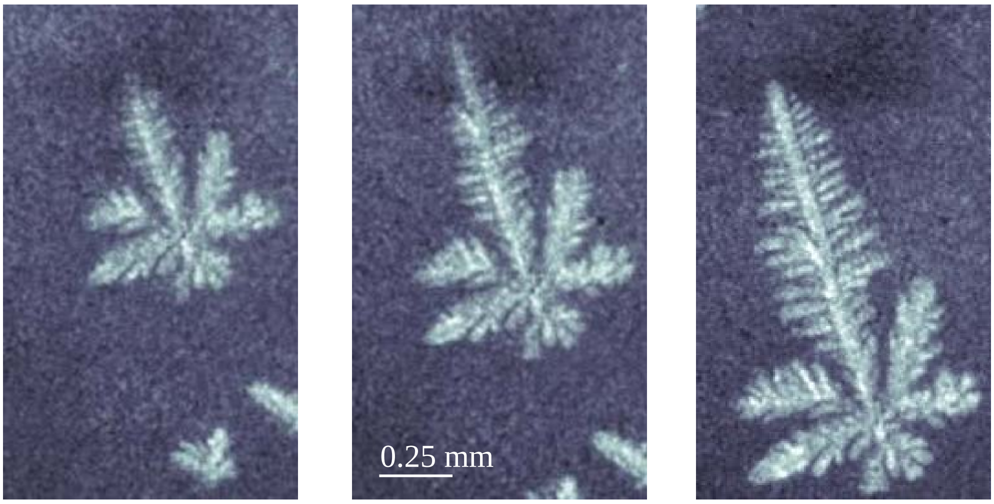

Fig. 1a-c: Sequence of growing Al-rich dendrite in the $Al_{90}Cu_{10}/Al_2O_3$ system. The hot zone is on top, the sample is pulled out downwards. The temperature gradient is 10 K/cm, the pulling speed 6 µm/s.
From left to right the times are 0 s, 44 s, 159 s.

Conclusions: The Bridgman furnace at ID 19 and the recommended sample holder design was suitable to directionally solidify the proposed metal/particle systems. The growth of dendrites was observed in $Al_{90}Cu_{10}/Al_2O_3$, but the particles do not show sufficient contrast. We conclude to replace them by particles with higher atomic mass (ZrO_2, CeO_2,...), which have to be stable in the Al melt. In parallel, we propose to improve the sample holder by replacing the carbon foils of the crucible by BN or SiO_2 with BN protection layer against the Al melt. This could reduce the speckled background, which originates from the carbon foils. The $Cu_{90}Sn_{10}/Ta_2O_5$ system has been discarded due to the low contrast between dendrite and residual melt.

Future publications: We plan to present results from our ESRF investigations on 2 conferences in 2005.
(i) *12th International Conference on Rapidly Quenched & Metastable Materials*, August 21-26, Jeju (South Korea), 2 abstracts submitted
(ii) *International Conference on Solidification Processes*, June 7-10, Stockholm (Sweden), 1 abstract submitted