ESRF	**Experiment title: In situ and real time analysis of the mushy zone evolution in a fixed temperature gradient by X-ray radiography**	**Experiment number**: MA-1277
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Scientific Objectives:

This work is the very first step in the analysis of accidents in nuclear power plants, where a mushy zone could be formed between the corium and a cold concrete walls [*Seiler J.M., Froment K., Multiphase science and Technology, 12 (2) (2000) 117*]. Despite the great importance of this issue, a large number of open questions are still unsolved owing to the complexity of the problem. This series of experiments tackles the "more simple" analysis of a mushy zone evolution in a fixed temperature gradient, when solidification is stopped during the mushy zone formation. The mushy zone is defined as the partially solid/liquid zone, formed during the directional solidification of an alloy and which consists of a dendritic solid network and the remaining melt.

The main objective of MA-1277 was to perform *in situ* and real-time analysis of the evolution of a mushy zone in a fixed temperature field in a model case of binary Al-based alloys. The purpose was to characterize the morphological changes of a mushy zone as a function of time and determine the key physical mechanisms (with their characteristic times) that control its evolution. It has been recently shown [*H. Nguyen Thi et al, J. of Crystal Growth, **310** (2008) 2906*] that synchrotron X-ray radiography is a powerful technique, perfectly adapted for such a study.

Experimental method:

Experiments were performed at BM05 in a Bridgmann furnace described in detail elsewhere [*H. Nguyen Thi et al, J. Crystal Growth, **310** (2008) 2906*]. Thin Al - 3.5 wt% Ni, Al – 4 wt% Cu and Al – 20 wt% Cu samples (40 mm × 6 mm × 0.2 mm) were first prepared at IM2NP and inserted inside the furnace. These Al-based alloys were chosen in order to achieve a good contrast in radiographs between the dendritic solid network (weak absorption) and the solute-enriched liquid phase (high absorption).

The mushy zone is formed by directionally solidified the sample with a *power-down method*, which consists of decreasing either both furnace heaters (with roughly constant temperature gradient) or only the hot furnace heater (with a slight temperature gradient decrease). Solidifications were carried out vertically upward with a temperature gradient about 30 K/cm. As soon as the mushy zone was formed in the field of view, solidification was stopped and the microstructural changes of the mushy zone observed as a function of time. For each sample, 2-3 experiments were carried out, with various cooling rate ranging from 0.5 to 3 K/min to have slightly different mushy zone morphologies.

It is worth noting that the duration of each experiment is typically about several hours, because all phenomena are mainly control by solutal diffusion. This explains the large number of shifts required for this work and asked for in the proposal.

Preliminary results:

Fig.1 shows the morphological changes of the mushy zone as a function of time for an Al – 4 wt% Cu alloy during one typical experiment of this campaign. It is shown that several phenomena act simultaneously with different magnetudes depending on the location in the MZ. The position of the highest point of the mushy zone is noted Z_{MZ}. Three successive regimes are observed:

1- In a first phase, solidification leads to the MZ formation with a sharp increase of Z_{MZ}. Fig.1a shows the MZ when the solidification phase is stopped at $t = 837\ s$.

2- Immediately following the end of solidification end, a decrease of Z_{MZ} is observed (regress of the mush-liquid interface), due to the dendrite tip remelting. This phenomenon is induced by the solute diffusion in the liquid phase from the inside part of the MZ towards the top of the MZ, which form a solute layer ahead of the MZ and thus markedly change the equilibrium temperature at the MZ top. Concomitantly, solute migration in the MZ induces the formation of an Al-enriched solid layer at the bottom of the MZ (white layer with increasing thickness in Fig.1). Fig.1b and 1c show the effect of these two phenomena on the MZ morphology.

3- After the solid-mush and mush-liquid interfaces merge the MZ disappears and the solid-liquid interface moves upward again due to the diffusion of the solute in the melt, which causes a resolidification at Z_{MZ}.

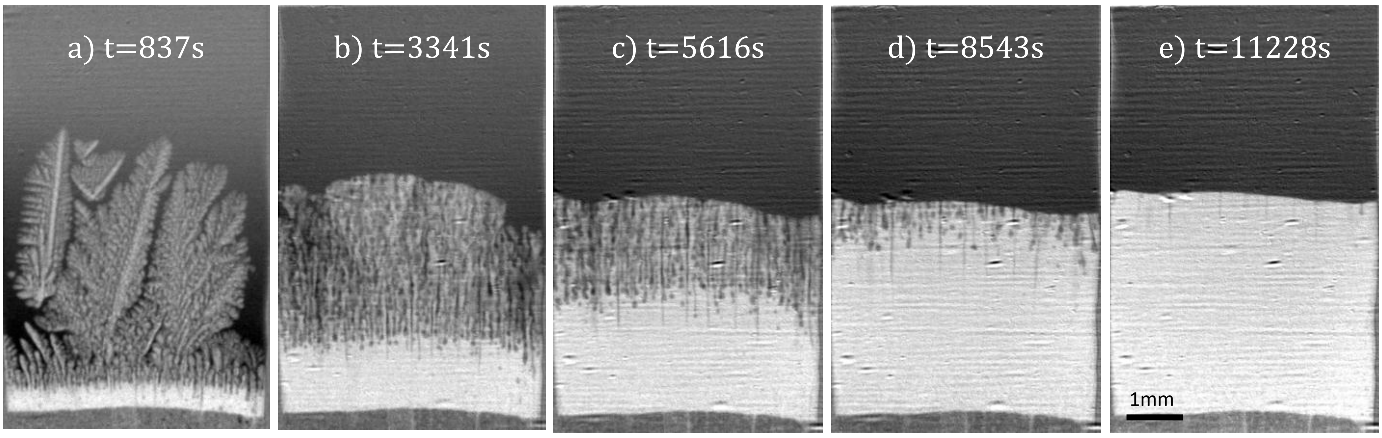

Fig.1. Radiographs of the evolution of the mushy zone in an Al-4wt%Cu experiment

Future work

In a next step, further measurements will be performed to quantitatively characterize all the morphological changes of the mushy zone. We plan to measure:

- Characteristic times and velocity of the almost pure aluminium solid crust.
- Temperature gradients along all of the sample and locally in regions of interest.
- Solute concentration using image treatment and Electron Microprobe analysis.

Observations will be analysed with the help of existing models in order to characterize the main phenomena responsible for the MZ evolution.