ESRF	**Experiment title: In situ and real time analysis of the directional solidification under static (DC) magnetic field by X-ray radiography**	**Experiment number**: MA-1378
Beamline: BM05	**Date of experiment**: from: 02/11/2011 to: 06/11/2011	**Date of report**: 31/08/2012
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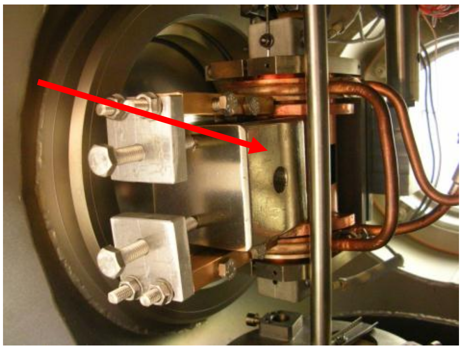
Scientific Objectives:

It has been wellknown that magnetic fields have potential to control fluid flow and then solidification microstructure in metallic alloys. Using static magnetic field to damp convection during solidification was the first application of magnetic field to solidification process [*Chedzey H. A. et al, 1966. Nature, 210 (1966) 933*]. However, some unexpected results suggested that apart from damping convections, certain flows may be induced as well. Tewari et al. [*Tewari S.N. et al, Metallurgical and Materials Transactions A, 25 (1994) 1535*] found that a 0.45T magnetic field had no effect on the macrosegregation of PbSn alloy, but at very low pulling rates the cellular was severely distorted. This interesting phenomenon was firstly studied by Alboussiere et al. [*Moreau R. et al, Materials Science and Engineering A, 173 (1993) 93*] under the primary theory exploration by Shercliff [*Shercliff J.A. Journal of Fluid Mechanics 91 (1979) 231*]. In our previous work [*Xi L. et al, Metallurgical and Materials Transactions A, 42 (2011) 3459*], an experimental and numerical simulation investigations have been done on the topic by post-mortem investigations. However, a large number of open questions are still unsolved owing to the complexity of the problem.
The main objective of MA-1378 was to perform *in situ* and real-time analysis of directional solidification with and without a static magnetic fields by synchrotron X-ray radiography. The purpose was to characterize the morphological changes of the solidification microstructure induced by the static magnetic field. 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 Sn -3 wt% Pb and Al – 4 wt% Cu samples (40 mm × 6 mm × 0.2 mm) were first prepared at IM2NP and inserted inside the furnace. These 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 static magnetic field was created by a magnet put inside the ultra-vacuum chamber (indicated by a red arrow on the figure), with a maximum intensity about 0.08 T. The direction of the magnetic field is parallel to the X-ray beam and in the same direction, perpendicular to the main surface of the sample.



The samples were directionally solidified by using the *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, and the microstructural changes of the solid – liquid interface were observed as a function of time. For each sample, 2-3 experiments were carried out, with various cooling rate ranging from 0.3 to 1 K/min to have slightly different mushy zone morphologies. It is worth noting that the duration of each experiment is typically about 1-2 hours. This explains the large number of shifts required for this work and asked for in the proposal.

Preliminary results:

Figure 1 shows a sequence of three radiographs during the directional solidification of Al – 4 wt% Cu sample. This figure displays the liquid/solid interface shape that was modified by Thermo-Electric Magnetic Effect (TEMC). Indeed, in a presence of a temperature gradient on two mediums with different absolute thermoelectric power (in our case the liquid and solid phases), a thermoelectric current could be genereted in both mediums as shown in figure 1a. The global TEMC redistributed the solute ahead of the interface (from the left side towards the right side of the sample) and then modify the interface shape. In this experiment, the solid-liquid interface became flater and flater as solidification proceeded.

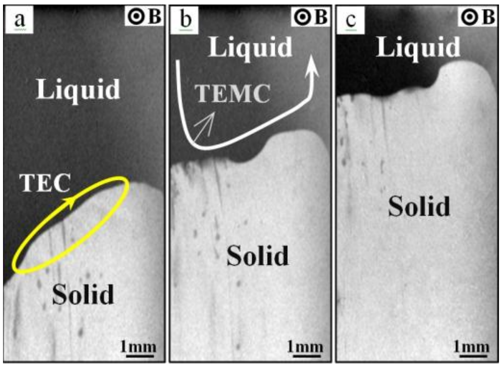


Figure 1. A sequence of three images at successive times during directionally solidifying Al-4wt%Cu alloy under a 0.08T static magnetic field (R=0.46µm/s; G=30K/cm): (a) t ≈ 270min; (b) t ≈ 330min; (c) t ≈ 390min.

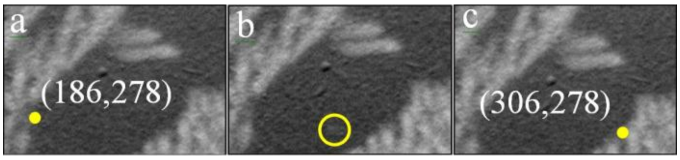


Figure 2. The tracing of a fragment.
(a) t = t_0; (b) t = t_0+4.2s; (c) t = t_0+7s.

Figure 2 shows a sequence of three radiographs during another experiment on the same alloy (Al – 4 wt% Cu). In this sequence, dendrite fragmentation occurred. However, on contrary to the case without magnetic field where the dendrite fragments sedimented, the detached fragment in this case moved horizontally. The initial and ending point were pointed out in figure 2a and 2c. Comparing the real velocity measured through in-situ observation and analytical calculation confirms that the TEMF acted on the dendritic fragments directly.

Future work

In a next step, further measurements will be performed to quantitatively characterize all the morphological changes of the solidification microstructure induced by the static magnetic field. A direct comparison will be performed with numerical simulations conducted at SIMAP.