Investigation of gravity effects on solidification of binary alloys with in situ X-ray radiography on earth and in microgravity environment

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Abstract. As most of the phenomena involved during solidification are dynamic, in situ and real-time X-ray imaging should be retained as the method of choice for investigating the solidification front evolution of metallic alloys grown from the melt. On Earth, natural convection in the melt is well known to be the major source of various disturbing effects which can significantly modify or mask important physical mechanisms. Microgravity environment is an efficient way to eliminate buoyancy and convection to provide benchmark data for the validation of models and numerical simulations. In addition, a comparative study of solidification experiments carried out on Earth and in space can also enlighten the effects of gravity. In the frame of the ESA - MAP programme entitled XRMON, an experimental set-up has been developed to perform directional solidification in microgravity conditions with in situ X-ray radiography observation. In the first part of this paper, we will present a brief review of some effects induced by gravity on the solidification process and investigated by mean of synchrotron X-ray radiography at ESRF (European Synchrotron Radiation Facility). In the second part of this paper, we will describe some results obtained with a prototype of the XRMON-Gradient Furnace set-up. These preliminary results show the large capabilities of the experimental set-up in terms of thermal behaviour, as well as X-ray observation.

1. Introduction

Structural material properties are directly related to their solidification microstructures, so that a precise control of growth process is crucial in engineering. As most of the phenomena involved during solidification are dynamic, in situ and real-time X-ray imaging should be retained as the method of choice for investigating the solidification front evolution of metallic alloys grown from the melt [1-3]. Elsewhere, an important issue to clarify in the solidification process is the role of convection on macrosegregation and on microstructure formation. The coupling between fluid flow and solidification
has been the subject of a great deal of experimental, theoretical and numerical works [4,5]. The main conclusion of these studies is that natural convection in the melt is the major source of various disturbing effects which can significantly modify or mask important physical mechanisms on Earth (1g). It has been shown that microgravity (µg) environment is an efficient way to eliminate buoyancy and convection to provide benchmark data for the validation of models and numerical simulations [6-10]. In addition, a comparative study of solidification experiments at 1g and µg can also enlighten the effects of gravity. Therefore, in the frame of the ESA - MAP programme entitled XRMON (In situ X-Ray MONitoring of advanced metallurgical processes under microgravity and terrestrial conditions), an experimental set-up was recently developed by SSC (Swedish Space Corporation) to perform directional solidification in microgravity conditions with in situ X-ray radiography observation. The first solidification experiment in microgravity using this facility is scheduled during MASER 12 sounding rocket mission, in autumn 2011.

This paper is divided is two parts. In the first part, we will present a brief review of some effects induced by gravity on the solidification process and investigated by mean of synchrotron X-ray radiography. Experiments were carried out at ESRF (European Synchrotron Radiation Facility) in thin Aluminium-based alloys. In the second part of this paper, we will describe the breadboard tests carried out in Stockholm (Sweden) with a prototype of the XRMON-Gradient Furnace set-up. Preliminary results show the large capabilities of this experimental set-up in terms of thermal behaviour, as well as X-ray observation.

2. Analysis of the influence of convection on solidification process by synchrotron X-ray radiography

The synchrotron experiments are carried out at the European Synchrotron Radiation Facility (ESRF). Solidifications are performed inside a Bridgman furnace, which was already described in details [11]. It has been shown [12,13] that this furnace can be used either for directional solidification (i.e. with a vertical temperature gradient) or for equiaxed growth (i.e. in nearly isothermal conditions). In situ and real time observation of the solidification process is possible by the use of synchrotron X-ray radiography. The principle of this technique is quite simple: The main surface of the sheet-like sample (40 x 6 mm$^2$) is set perpendicular to the incident monochromatic X-ray beam, with an energy in the range 14-18 keV. The beam energy is chosen as a function of the alloy composition (Al – 4 wt% Cu or Al – 10 wt% Cu alloys) and the sample thickness (200 µm). The transmitted beam is recorded by a CCD–based (FReLoN) camera placed behind the sample. Absorption is the main source of the image contrast and depends on the atomic number of the elements and the solute content. In all radiographic pictures, the solid aluminium microstructures appear in white and the copper-rich liquid in dark grey. The optics is chosen to obtain a good compromise between a large field of view (15 x 6 mm$^2$) and a sufficient resolution (pixel size: 7.46 µm). For all experiments, the image quality was improved by applying an image processing described in [14].

2.1. Convection effects on solid–liquid interface during the initial transient of solidification

It is well known that even when solidification is performed under both thermal and solutal stabilizing configuration, convection in the melt can nevertheless be strong due to residual transverse gradient, in particular at low growth rates [9,15]. Indeed, in real experimental configurations there are generally small departures of the initial interface from planarity, even after a long thermal stabilisation phase. As solidification proceeds, the solute is rejected at the solid–liquid interface. Due to gravity, the denser Cu-enriched liquid flows towards depressed regions of the front, which results in solute accumulation in those regions. According to the phase diagram, solute accumulation in depressed regions retards solidification and further deepens the solid–liquid interface modulations. This leads to a large
macroscopic deformation of the solidification front. In addition, the transverse solute gradient gives rise to a transverse microstructure gradient, as already shown by post-mortem analysis [9].

Figure 1 shows a sequence of four images recorded at successive times during a solidification experiment at a cooling rate of 0.3 K/min and an average temperature gradient \( G \) of 35.5 K/cm. In this experiment an initially slightly tilted smooth interface (figure 1a) is observed. A crude estimation of the transverse temperature gradient \( G_y \) can be obtained from the expression \( G_y = (\Delta z_0/d)G \), where \( \Delta z_0 \) is the distance between the highest and the lowest levels of the curved front at \( t = 0 \) (before solidification starts) and the corresponding transverse distance \( d = 5 \) mm. In the presented experiment, \( \Delta z_0 = 0.45 \) mm, which gives \( G_y = 3.2 \) K/cm. When the cooling of the sample begins the growth rate gradually increases as solidification proceeds, leading to the destabilisation of the interface first on the left-hand side of the sample (figure 1b). The propagation of the morphological instability towards the right-hand side of the sample is visible in figure 1c and figure 1d, as well as the transverse gradient of microstructure. Concomitantly, macroscopic distortion of the solid–liquid interface becomes larger and larger with time, due to fluid flow in the melt. The amplitude of the front distortion increases from \( \Delta z_0 = 0.45 \) mm at the beginning to nearly 4 mm at the end of the experiment (figure 1d).

![Figure 1. Sequence of four images showing the increasing macroscopic deformation of the solid-liquid interface during the initial transient of directional solidification of Al – 4 wt% Cu (cooling rate = 0.3 K/min; \( G = 35.5 \) K/cm. (a) \( t \approx 17 \) min; (b) \( t \approx 34 \) min; (c) \( t \approx 70 \) min; (d) \( t \approx 88 \) min.](image)

In figure 1c and figure 1d, it can be seen that the liquid phase on the right-hand side of the sample just ahead of the solid–liquid interface becomes darker as solidification proceeds, which confirms an increase in the liquid solute concentration there. At the same time, the solute amount in the liquid phase on the left-hand side of the sample also increases but at a slower rate. Recently we developed a novel image analysis technique which enables measurement of the long-range solute concentration profiles in the liquid phase [14]. By using this procedure, solute concentration were measured at three different \( y \)-abscissae during the initial transient. Figure 2 shows the time evolution of the concentration at the solid–liquid interface for the three different abscissae \( y_1 = 0.7 \) mm, \( y_2 = 2.6 \) mm and \( y_3 = 4.0 \) mm (\( y \)-axis reference was chosen as the left-hand side of the sample). It can be seen that in the early stages of solidification (\( t < 8 \) min) the solute concentration variation at the solid–liquid
interface along the y-axis $\Delta C_{\text{int}}(t) = C_{\text{right}} - C_{\text{left}}$ is close to 0, indicating that the liquid phase is almost homogeneous at the beginning of the solidification. $\Delta C_{\text{int}}(t)$ then gradually increases during the initial transient and finally reaches a value of 2.0 wt.% at the end of the experiment ($t = 70$ min).

![Figure 2](image.png)

**Figure 2.** Evolution of the concentration at the solid–liquid interface for the three different abscissae $y_1 = 0.7$ mm, $y_2 = 2.6$ mm and $y_3 = 4.0$ mm during the initial solidification transient of Al – 4 wt% Cu, $G = 35.5$ K/cm, cooling rate = 0.3 K/min.

### 2.2. Self-poisoning effect during equiaxed growth

In a typical equiaxed growth experiment, the temperatures of both heater elements are adjusted in order to achieve nearly isothermal conditions all over the sample. After a stabilization period of two hours, solidification is triggered by applying simultaneously the same cooling rate on both heaters [12,13]. For the experiment presented in this paper the cooling rate applied was $R = 0.5$K/min. In situ and real time observation of the solidification process was made possible by the use of synchrotron X-ray radiography, from $t_0 = 0$ corresponding to the application of the cooling rate to the end of experiment when lengthening of the dendrite arms was no more detectable with naked eye.

Length measurements are performed for the four arms of a single grain (figure 3), growing roughly at right angles to each other along the $<100>$ direction. As can be seen in figure 3d, the behaviors of the four dendrite arms are not identical: the initial slopes (or growth velocities) of the four curves as well as the values of their respective plateaus are different. For instance, at the beginning dendrite arm 3 has the highest growth rate, while the longest length is achieved by dendrite arm 4. The difference of the arm growth velocities at the beginning is attributed to a “self – poisoning” of the grain, already revealed in the case of the faceted growth of an equiaxed grain [16]. In fact, the grain is initially isolated from the surrounding grains (figure 3a) and we can assume that there is no influence from other grains. As solidification proceeds, the rejected solute (Cu) is carried downwards by gravity driven fluid flow, leading to the velocity decrease of the dendrite arms situated aside and below. Then, the disparity in the asymptotic values of the arm lengths can be attributed to the interaction with neighboring grains in the late stage of solidification (figure 3b-3c). Owing to the grain interaction, the growth is hindered and is finally stopped, although there is no mechanical impingement of the dendrites as it will be shown later. In addition, the grain interaction is highlighted on the curve representing the growth of the dendrite arm 4, where a plateau is distinctly visible following the initial slope of the curve (figure 3d). This plateau indicates that the arm has stopped growing for this short period of time, due to the influence of another grain falling in its vicinity.
Figure 3.
(a) Equiaxed grain of Al – 10 wt% Cu initially isolated from the surrounding grains at the early stages of solidification (t = 2957sec, R = 0.5 K/min).
(b) The grain having its four dendrite arms growing at right angle to each other (t = 3037sec).
(c) The same grain surrounded by other grains at the final stage of solidification (t = 3362 sec).
(d) Time variation of dendrite arms lengths. The curves are referenced by the numbers given on the right hand side of the graph.

2.3. Sedimentation and Columnar-to-Equiaxed transition

Sedimentation and rotation of the equiaxed grains are two key concepts to understand the final grain arrangement and can only be analyzed by in situ observation [17-20]. For instance, the gravity-settling behaviour of few equiaxed grains is shown in figure 4, with a sequence of radiographs recorded during AlCu growth. In the beginning of the experiment, nucleation of small grains occurs in different area of the field-of-view, as well as in the region just above the field-of-view. Most of them are stuck on crucible wall, but one grain falls down due to gravity (figure 4a to 4d). In addition, live monitoring also shows that the equiaxed grain rotate because of uneven lengthening of their dendrite arms. Finally, the grains stop to move as soon as their size is comparable to the crucible thickness. It should be stressed that the falling down of equiaxed grains is enhancing grain packing at the bottom of the sample. At the end of the experiment presented, another grain moves upward, in a opposite direction of the first grain, as shown in figure 4e to figure 4h. The reason of this movement could be: (i) either there is a convective roll in this region of the sample or (ii) the grain becomes buoyant with respect to the fluid owing to local segregation in this region of the sample. Indeed, since the liquid become denser owing to the solute rejection, the relative difference of density between the grain and the surrounding liquid gradually changes. For Al-Cu alloys, the solid formed is denser than the bulk liquid below a nominal composition of about 10 wt% Cu, whereas it is lighter for higher concentrations. This explains the change in the direction of the movement between the two grains.

The grains settlement could be critical in the Columnar-Equiaxed Transition. In experiments dedicated to CET carried out on refined Al–3.5 wt% Ni alloy, we first applied a low pulling rate to establish dendritic columnar microstructure. After that, the pulling rate is at reference time t₀ jumped to a higher value to trigger CET. Rapidly, equiaxed grains become visible in the solute-rich boundary layer adjacent to the columnar structure (figure 5a). Under gravity the falling down of equiaxed grains is enhancing grain packing above the columnar dendrites and eutectic front. According to this, CET is
expected to occur earlier on earth than in microgravity. Columnar growth is thus stopped and equiaxed growth regime installs and propagates (figure 5c), according to scenario proposed by Martorano [21].

Figure 4. Sequence of radiographs recorded during Al-10wt% Cu equiaxed solidification showing the sedimentation and floatation of equiaxed grains.

Eventually, equiaxed grains get trapped in the eutectic phase, as it happens for the first fallen grains in figure 5b and 5c, giving the false impression of a mixed columnar–equiaxed structure. This sedimentation effect can obviously be misleading in post mortem analysis of the grain structure, as well as in the feedback to modeling of the solidification sequence. After the CET, equiaxed growth is self-sustaining, and a fully equiaxed mushy zone formed by closely packed equiaxed grains (figure 5d). Equiaxed growth proceeds by the propagation of the forefront of this mushy zone with the velocity of leading dendrite tips nearing the pulling rate. In the melt just above, new grains repeatedly nucleate, grow and fall down, screening the current leading grains. This operating cycle is maintained till the end of the sample (figure 5d).

Figure 5. Sequence of radiographs of refined Al-3.5wt% Ni solidification, recorded during the CET induced by a sharp increase of the pulling rate from 1.5 to 15 µm/s at \( t = t_0 \), \( G = 30 \text{ K/cm} \).

(a) \( t_0 + 63 \text{ sec} \), (b) \( t = t_0 + 87 \text{ sec} \), (c) \( t = t_0 + 111 \text{ sec} \) and (d) propagation of the equiaxed microstructures.
3. XRMON – GF project

3.1. XRMON-GF device

The conceptual design of the breadboard setup is given in figure 6. It consists of a gradient furnace system for solidification of AlCu-based alloys and an attached high-resolution X-ray diagnostic system. The most challenging tasks for the development came from the distance between the source and the sample, which must be about 5 mm. This short distance required special thermal insulation to avoid heat leakage from the sample to the cooled target. In addition such a thermal insulation had to be transparent to X-rays as much as possible to guarantee maximum intensity.

The metallic samples had a sheet-like geometry with a length of 50mm, a width of 5mm and a constant thickness of less than 0.2mm along the sample. Each sample was first mechanically polished down to the desired thickness (with a surface rugosity of 1µm), and then spray coated with boron nitride (BN). Then it was sandwiched between two rectangular glass plates, welded together. The BN coating was required because of its combination of properties: i) it prevents chemical reaction between the Al-Cu sample and the glass plates and ii) it is transparent to X-rays in the range of energy of our interest.

To investigate the dynamics of solidification processes, the field of view for the X-ray diagnostics was about 5 x 5 mm² with a spatial resolution of 3-5 µm and a temporal resolution of 2-3 Hz. The X-ray source was based on a microfocus transmission target in molybdenum using polychromatic radiation with a peak at 17.5 keV.

![Conceptual design of the breadboard set-up](image1)

**Figure 6.** (a) Conceptual design of the breadboard set-up, (b) picture of the breadboard set-up showing the X-ray source (left), the furnace (very close to the microfocus source) and the detector (right).

3.2. Initial transient during directional solidification of Al - 10 wt% Cu alloy

Figure 7 shows a sequence of six images taken during the initial solidification transient of an Al - 10 wt% Cu alloy for a temperature gradient of 43 K/cm and a cooling rate of the hot heater element at 6 K/min. This figure shows the time evolution of the interface pattern, from the breakdown of the planar interface to the subsequent developments of the microstructure. For this experiment, the solidification microstructure was photographed every 2 seconds with an exposure time of 2 seconds. In figure 7a (t = t₀ + 150s, with t₀ the reference time, just before the beginning of the solidification) the first disturbances are visible along the whole interface, following the planar front breakdown induced by the Mullins–Sekerka instability [22]. One can already see in figure 7a that side branches begin to grow on most of the perturbations, which indicates the inception of the formation of dendrites. At this stage, it is possible to determine an average wavelength \( \lambda_i \) for the microstructure, \( \lambda_i \approx 180 \mu m \). Further
development of the pattern occurs with a progressive increase of the amplitude of the disturbances (figure 7b, \( t = t_0 + 170s \)), while the liquid ahead of the columnar front becomes darker and darker, due to the solute rejection during the liquid-solid transformation. In the subsequent stages, both the amplitude and lateral size of dendrite increase concomitantly, with solute screening causing a strong decrease of the growth of the neighbouring dendrites. In our experiments, a steady state was never achieved for several reasons: (i) the power-down technique used to trigger the solidification, (ii) the low value of the partition coefficient \((k \approx 0.14)\) which implies a long transient regime, and (iii) the short sample length (about 50 mm). Nevertheless, the general shape of the microstructure does not change drastically in the latest stages of solidification and is mainly composed of dendrites protruding markedly into the liquid phase with an average final primary spacing \( \lambda_f \approx 1.260\mu m \) (figure 7f).

3.3. Fragmentation phenomenon during solidification of Al – 20 wt% Cu alloy

One mechanism for the formation of equiaxed grains during the Columnar-to-Equiaxed Transition [23] is the dendrite fragmentation, which is believed to be at the origin of the central equiaxed core region in casting processes. Fragmentation occurs when dendrite branches are detached from the main primary trunks. If these fragments are transported ahead of the columnar front by buoyancy forces or convection, they can continue to grow and then form equiaxed grains that can stop the advancing columnar front. Fragmentation is favoured by the dendrite ripening process but also by the initiation of local remelting due to the local pile-up of solute within the partially solid-liquid sample region usually called the “mushy zone” [24-26]. Fragmentation is an essential feature of the CET in non-refined alloys; however, the details of the fragmentation phenomenon are insufficiently understood and controlled. Thus, it is compulsory to confirm the predominance of the fragmentation phenomenon in CET and also to improve the characterisation and understanding of its mechanism, in particular the role of gravity at each step of CET.

To achieve this goal, an efficient approach is to perform a comparison between solidification experiments carried out at 1g and in µg, in both cases with \( \textit{in situ} \) characterization by means of X-ray radiography. Several experiments were performed on a single non-refined Al – 20 wt% Cu sample, for various high temperature gradients (200 - 400 K/cm) and cooling rates in the range 1 – 20 K/min. For
the experiment presented in figure 8, the high nominal solute concentration lead to a higher contrast than in previous experiment with an Al – 10 wt% Cu. Therefore, the exposure time was reduced to one second, and the evolution of the solidification microstructure was recorded every second. In figure 8a one can see the gradual establishment of columnar growth, as described in the previous section. In this experiment, an equiaxed grain nucleated ahead of the columnar front, on a small heterogeneity. This grain remained fixed and simply continued to grow in interaction with the columnar front. In figure 8b and figure 8d, the black circles pinpoint the presence of several dendrite fragments. For Al – 20 wt% Cu, the solid (mainly composed of aluminium) being much lighter than the surrounding liquid (Cu - enriched), dendrite fragmentation was promoted by buoyancy forces acting on secondary dendrite arms. After the fragmentation, the dendrite pieces were immediately carried away to the upper part of the sample. In this experiment, fragmentation occurred continuously during the growth process. Radiographs made it possible to have a rough estimation of the fragments velocity, about 60µm/s in this experiment. It is worth noticing that these dendrite fragments could not promote CET, because they were carried up far into the liquid where they were re-melted. Furthermore, the fact that all Al-enriched dendrite fragments were transported by buoyancy forces into the upper part of the sample was at the origin of a strong segregation along the sample as the solidification front advanced.

![Figure 8](image)

**Figure 8.** Sequence of radiographs during columnar growth of Al – 20 wt% Cu showing several dendrite fragmentations (cooling rate = 20K/min; G = 400 K/cm) (a) t=t₀+50s; (b) t=t₀+100s; (c) t=t₀+150s; (d) t=t₀+168s. t₀ is the reference time, just before the beginning of the solidification. Image width=5 mm.

In figure 8c, the fragmentation of a dendrite primary trunk can be noticed, indicated by a black ellipse. Its size being large compared to the sample thickness, the primary trunk fragment could not move far into the liquid phase. This phenomenon was rarely observed compared to the secondary dendrite arm fragmentation, certainly because of the difference in diameter between primary trunk and secondary arms. It has recently been observed by using combined radiography and topography that the rotation of some tenths of degrees around the growth axis of the entire primary trunk during the growth process can occur [27]. This rotation has been interpreted as a consequence of a torque induced by shear stress that builds up with the growth [28]. This suggests that stress accumulates there to finally provoke the trunk to crack. In our experiments, it was also observed that this phenomenon occurred mainly for a specific crystallographic dendrite direction <110>, which suggests an effect of dendrite morphology.

4. Conclusions

This paper reports on a selection of results obtained during metallic alloy solidification experiments characterized in situ and in real time by X-ray radiography. Experiments were carried out with both a Synchrotron source (ESRF) and a microfocus tube, with a dedicated novel experimental set-up developed by SSC in the frame of the ESA-MAP XROMON. These results emphasize the great impact of gravity and convection on the dynamics of the solidification front by showing the influence of convection on the solid-liquid interface, the solutal self-poisoning during equiaxed growth, the
sedimentation/buoyancy of grains, the bending/fragmentation of dendrite secondary arms. The next step of the project is to perform an experiment in microgravity environment to obtain purely diffusive transport conditions and then to provide benchmark data for the validation of models and numerical simulations. In addition, a comparative study of solidification experiments at 1g and µg will also enlighten the effects of gravity. This is the goal of the solidification experiment scheduled during MASER 12 sounding rocket mission, in autumn 2011. For the future, several research projects could also benefit greatly from this novel experimental setup, which enables X-ray characterization in microgravity experiments.

Acknowledgments
This research work is supported by the XRMON project (AO-2004-046) of the MAP program of the European Space Agency (ESA) and by the French National Space Agency (CNES).

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