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# IN SITU X-RAY STUDIES OF DIRECTIONAL SOLIDIFICATION OF METAL ALLOYS IN MICROGRAVITY CONDITIONS

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## ABSTRACT

The performance of structural metallic materials is associated with the solidification microstructures, which are strongly dependent on gravity effects. Experimentation in a microgravity environment is a unique way to suppress these effects and to provide benchmark data for testing current theories of grain and microstructure formation. This contribution presents a summary of results obtained for directional solidification of Al-Cu alloys within the framework of the XRMON project. It is focussing on *(i)* the first ever microgravity experiment on solidification with in situ monitoring of metal alloys performed on board a sounding rocket and *(ii)* solidification experiments performed on board parabolic flights, where the effects of varying gravity level have been studied.

## 1. INTRODUCTION

The most common solidification microstructure is the dendritic grain, which can be either columnar or equiaxed [1]. The grain structure is called columnar if the growth is preferentially oriented in a direction close to the heat flux, whereas equiaxed grains are growing in all space directions, leading to a material with more isotropic macroscopic mechanical properties and a more homogeneous composition field. Depending on the application, one type of grain structure is usually preferred, e.g. equiaxed grains in automotive components and columnar grains in turbine blades. Elsewhere, an important issue is to clarify the role of convection on macrosegregation and on microstructure formation during the solidification process.

The coupling between gravity effects and solidification has been the subject of a great deal of experimental, theoretical and numerical work. The main conclusion of all these studies is that gravity is the major source of various disturbing effects, which can significantly modify or mask important physical mechanisms on Earth (1g) [2]. Numerous experiments in microgravity conditions have shown that the microgravity (µg) environment is a unique and efficient way to eliminate buoyancy and convection to provide benchmark data for the validation of purely diffusive models and numerical simulations [3, 4]. In addition, a comparative study of solidification experiments at 1g and µg can also enlighten the effects of gravity [5, 6].

### **1.1.** In situ characterization during solidification of metallic alloys

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 [7], in particular on the effects induced by gravity [2]. In this non-destructive technique, the contrast in the recorded image is due to local changes in the amplitude of the X-ray beam transmitted through the sample. A (monochromatic) X-ray beam illuminates the sample and a 2D-detector (photographic film or CCD camera) is placed close to the sample to record the transmitted beam. In alloy systems, contrast mainly results from segregation of the chemical species and is generally weak and therefore difficult to reveal with conventional X-ray sources.

Recent developments of more powerful laboratory X-ray sources, as well as modern X-ray detectors, have opened up new perspectives for the application of X-ray radiography to microgravity experiments. Indeed, it has become conceivable to design facilities dedicated to the study of alloy growth processes with in situ characterization on board microgravity platforms.

### **1.2.** Microgravity relevance

On Earth, gravity has important additional effects on the solidification process. Firstly, gravity induces natural convection in the melt, due to density variations following the local temperature and concentration [4].

The main impacts of convective flows on the solidification microstructure are the macroscopic deformation of the solid/liquid interface, the micro- and macro-segregation in the sample, and the modification of the primary arm spacing (Fig.1a). Secondly, due to solute rejection during the liquid to solid transition, growing grains and surrounding liquid have generally different densities. Consequently, buoyancy force can act on the solid grains, which lead to their sedimentation or flotation in the liquid phase (Fig.1b). Therefore, equiaxed microstructures on Earth and in microgravity are dramatically different. Thirdly, gravity is at the origin of mechanical effects, in particular on the secondary arms. It has been shown that these mechanical effects can induce the bending of secondary arms when they are long enough (Fig.1c). In some cases, the bending phenomenon can precede the dendrite fragmentation [8]. Finally, a less addressed issue related to gravity is the hydrostatic pressure in the melt, despite its strong impact on the human body during space travel. It is well known that hydrostatic pressure applied during solidification significantly reduces the formation of porosities. On the contrary, under microgravity conditions, the liquid shape is only determined by the surface tension and the wetting behaviour of the melt on solid surfaces. Hence, the loss of hydrostatic pressure in the melt can cause shrinkages or the formation of voids along the sample during microgravity experiments [9]. Therefore, a deeper understanding of gravity effects on solidification microstructure formation is of great importance for scientists and industrialists alike.

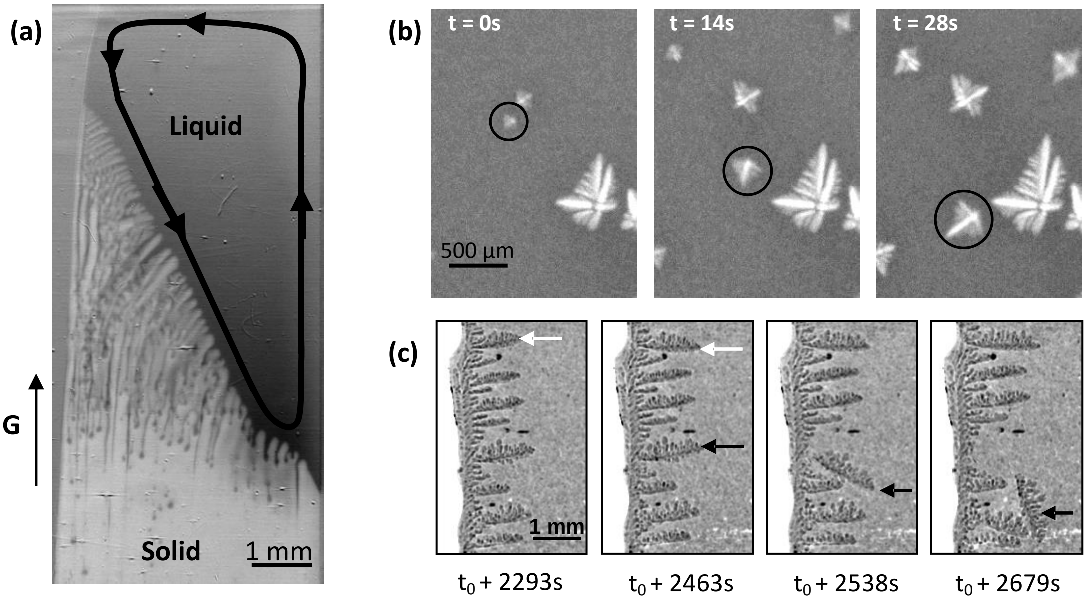

Figure 1. *In situ observation of gravity effects during direction solidification: (a) Deformation of the solid–liquid interface due to convection during directional solidification of Al–4wt.% Cu (cooling rate is 0.3 K/min, temperature gradient is 35.5 K/cm); (b) Sequence of radiographs recorded during Al-10wt.% Cu equiaxed solidification showing the sedimentation of equiaxed grains; (c) Radiographs showing bending and fragmentation of secondary arms during the development of a columnar dendrite of Al-7wt.%Si (cooling rate is 0.5 K/min, temperature gradient is 15 K/cm)*

### **1.3.** XRMON project

Since 2004, the European Space Agency (ESA) has been supporting investigation of gravity effects on solidification by promoting in situ X-ray monitoring of the solidification of aluminium alloys on microgravity platforms and on earth. The ESA-MAP (Microgravity Application Promotion) entitled XRMON (In-situ X-ray monitoring of advanced metallurgical processes under micro gravity and terrestrial conditions) aims to develop and perform in situ X-ray radiography experiments on metallurgical processes related to solidification phenomena, as well as solute diffusion in liquid metals, in microgravity and terrestrial environments [10]. The specific research subjects are closely linked to subjects of interest in several other active MAPs, but nevertheless unique as they deal with in situ X-ray studies which are not included in the other running projects.

Within the framework of the XRMON project, we have designed and developed new facilities dedicated to the study of Al-based alloy solidification on board microgravity platforms, with in situ X-ray radiography. The aim of this paper is to give an overview of the in situ X-ray studies of metal alloy solidification, carried out in sounding rocket and during ESA Parabolic Flight campaigns. The presented experiments mainly focussed on directional solidifications, and equiaxed growth experiments [11, 12] are out of the scope of this paper.

## **2.** XRMON-GF (Gradient Furnace) and XRMON-PFF (Parabolic Flight Facility) apparatus

The XRMON-GF set-up was developed within the framework of the XRMON project by SSC (Swedish Space Corporation) to perform directional solidification with in situ X-ray radiography observation in microgravity conditions. The gradient furnace is of Bridgman type, with two identical heaters for the “hot” and “cold” zones (Fig.2a) that are independently regulated by a PID-regulator. This furnace enables directional solidification with thermal gradients within the range of 5-15 K/mm. The heater gap has a “hole” of 5mm x 5mm for the X-ray radiation transmission. Solidification of the sample is made by cooling the two heaters at the same cooling rate $R$ to keep a temperature gradient $G$ constant.

The experiment samples were made of Al-10wt.% Cu and Al-20.wt% Cu. The high amount of solute was chosen to ensure a high contrast between the grown solid and the surrounding liquid phase but also to have a short solidification interval which could enable us to visualize the whole mushy zone in the Field-of-View (FoV). The sample dimensions were 50 mm (length) x 5 mm (width) and 150 µm in thickness (drawn in white in Fig.2b). The sample was enclosed in a crucible, made of two 150 µm thick glassy carbon (Sigradur K) sheets, which were sewn together with a 200 µm silica thread along the long edges and left open in the short ends.

The X-ray diagnostic device comprises two main parts: The X-ray source and the camera (Fig.2c). A microfocus transmission-type X-ray tube with 3 µm focal spot was used in order to meet the spatial resolution requirement (roughly 5 µm) and a sufficient photon flux to ensure a good image contrast. The two peaks in energy are $K_\alpha$ = 17.4 keV and $K_\beta$ = 19.6 keV, which are adapted to Al-20wt.% Cu alloys. It is worth noting that, contrary to synchrotron sources, which provide a parallel beam, microfocus sources deliver a cone-beam. This leads to a geometrical magnification of the image, depending on the respective source-sample and source-camera distances. In our geometry, the magnification is about 5 and, a pixel size of the camera of 20 µm corresponds to a “real” pixel (i.e. at the level of the sample) of 4 µm.

The camera system comprises a digital camera (Vosskühler 11000) with a 24x36 mm CCD-sensor adapted for X-ray usage by the integration of a 50 mm thick fiber optical plate that protects the sensor from radiation. A scintillator plate placed in front of the optical fiber converts X-ray radiation to visible spectrum light [13].

To perform directional solidification experiments during ESA parabolic flight campaign, a duplicate of the XRMON-GF was built, named XRMON-PFF (Parabolic Flight Facility). This facility is quite similar to the XRMON-GF, with only minor changes to be fitted in the Airbus A300 Zero-G operated by Novespace (www.novespace.fr) [14].

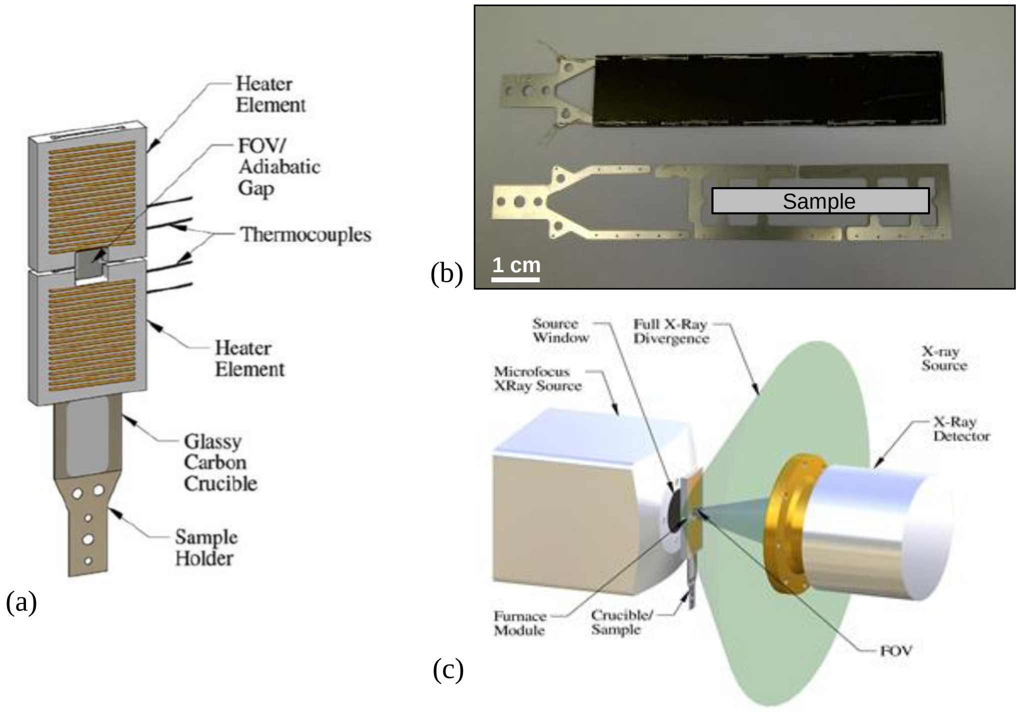

Figure 2. (a) *Sketch of the Bridgman furnace, (b) Pictures of the crucible-sample system and (c) X-ray device facility.*

## 3. INFLUENCE OF GRAVITY ON DENDRITE FRAGMENTATION

The first solidification experiment in microgravity using the XRMON-GF facility was successfully carried out during the MASER-12 sounding rocket mission on February 13th, 2012 at Esrange (Sweden). The initial objective of this microgravity experiment was to demonstrate the new opportunities that X-ray radiography offers for future microgravity experiments. In addition, some interesting results were obtained and described in details elsewhere [13, 15].

Since the microgravity duration during a MASER sounding rocket is limited to 6 min, a suitably adapted experimental timeline was defined [13]. After a short stabilization period (about 20 s), the sample directional solidification was triggered by applying successively three increasing cooling rates to both heaters (0.15 K/s → 0.7 K/s → 3 K/s). Two ground-reference tests were carried out with the same experimental profile and on a fresh sample, for two different sample orientations: in the first reference test, the growth direction was perpendicular to the gravity vector, while it was parallel and in the opposite direction to the gravity vector in the second ground-reference test.

Fig.3 displays two sequences of radiographs recorded during the solidification experiment in microgravity conditions and for the reference experiment at normal gravity. These sequences of radiographs unveil the time evolution of the interface pattern during the slowest cooling rate (R = 0.15 K/s), with a temperature gradient of about 15 K/mm between the heaters.

The sample in the field of view (FoV) was fully liquid at the end of the melting phase and then nucleation of the first solids occurred below the field of view. After a while, dendrite tips appeared at the bottom of the FoV (left column in Fig.3) and formed a very disordered dendritic pattern (second column in Fig.3). Gradually the grain competition gave a more regular array of dendrites (third column in Fig.3).

During the columnar solidification in microgravity conditions, nucleation of one equiaxed grain ahead of the columnar front is visible (Fig.3a2, bottom right), most likely on a small heterogeneity of the sample oxide layer. This grain slightly rotated during the solidification, which clearly showed that it was not stuck on the sample walls. However, due to the microgravity environment, this equiaxed grain remained at the same altitude and were progressively engulfed by the columnar front and then completely incorporated into the columnar microstructure.

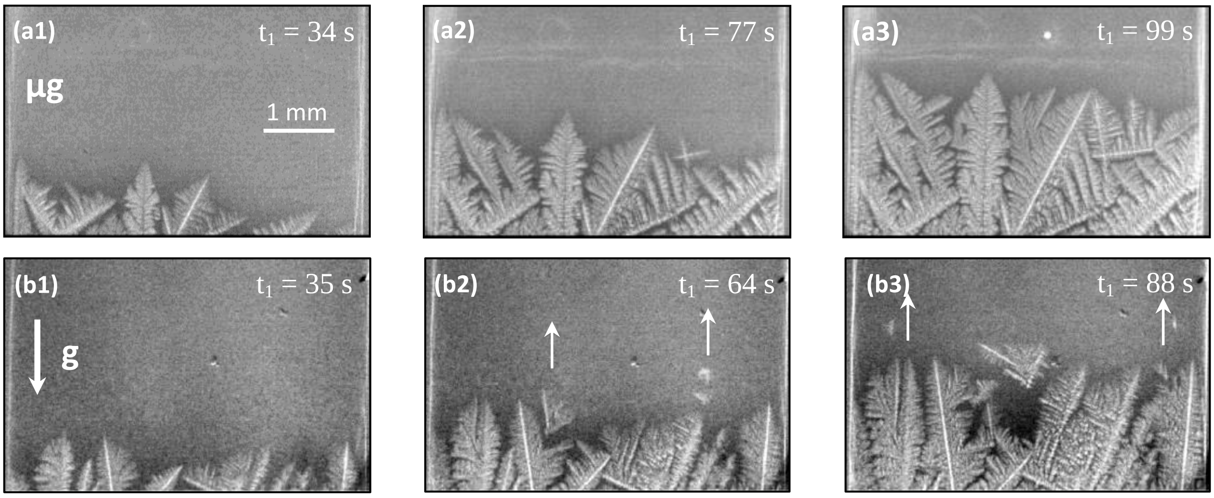

Figure 3. *Columnar solidification of Al-20wt.% Cu with a temperature gradient of about 150 K/cm between the two heaters and a cooling rate of 0.15 K/s on both heaters: (a1-a3) in microgravity conditions, (b1-b3) sample in vertical position (same reference time for the three experiments).*

For the upward solidification experiment on Earth, the most important feature is the multiple fragmentations in the mushy zone region (Fig.3b2 and Fig.3b3, indicated by arrows), in particular at the top of the columnar front. The fragmentation density in this region is about twenty times larger than in the deep mushy zone as displayed in Fig.4 For the microgravity experiment, dendrite fragmentations also occurred in the whole mushy zone but the difference between the top of the mushy zone and the deep mushy zone is less marked than for 1g-experiment (only 5-6 times larger). These measurements are qualitatively in agreement with recent results of others groups [16, 17]. This dramatic difference of behaviour between µg and 1g experiments may be attributed to two effects: *(i)* the presence of gravity-driven convection in the inter-dendritic liquid regions in the top of the mushy zone, where the liquid fraction is relatively high [17, 18] or/and *(ii)* the buoyancy force acting on secondary arms of dendrites [19, 20].

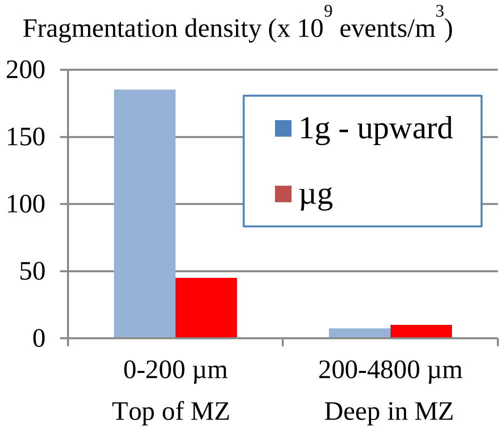

Figure 4. *Fragmentation density measured for 1g (blue bars) and µg (red bars) experiment, respectively for the top and deep regions of the mushy zone.*

For 1g conditions, most fragments moved upward after their detachment, due to the buoyancy force because the solid density is lower than the density of the surrounding liquid. Some of those fragments were free to float to the hot region of the sample (white arrows in Fig.3). During their upward motion, the size of the dendrite fragments decreased because they gradually melted, forming a final white cloud, which corresponded to the melting of the aluminium-rich dendritic fragment. It is worth noting that these dendrite fragments could not promote columnar-to-equiaxed transition, because they were carried up too far into the liquid where they were re-melted. In addition, a strong segregation along the sample occurred during solidification because all Al-enriched dendrite fragments were transported by buoyancy forces into the upper part of the sample and mixed in the liquid phase after melting. In absence of gravity, dendrite fragments moved towards the cold part of the mushy zone after their detachment, carried by the liquid movement induced by the sample shrinkage.

## 4. COLUMNAR-EQUIAXED TRANSITION TRIGGERED BY GRAVITY LEVEL VARIATION

Directional solidification experiments on refined Al–20 wt.%Cu and refined Al–10wt.%Cu samples were also carried out during several ESA Parabolic Flights (PF) campaigns. The succession of periods with different gravity levels offered by parabolic flight allowed the investigation of the effects of gravity level variations on the columnar-to-equiaxed transition. Experiments were carried out in the XRMON-PFF apparatus for a wide range of cooling rates and a constant temperature gradient. As mentioned in Section 2, this facility is simply a duplicate of the XRMON-GF used during the MASER-12 experiment, which has been adapted to the Airbus A-300 and then A-310, operated by Novespace (Bordeaux-France). Solidification is induced by the power-down method, which consists of applying the same cooling rate on both heater elements to keep a constant temperature gradient during the process. X-ray radiography was successfully used to observe the microstructure evolution following the variations of gravity level. The solidifications of two samples were carried out, an Al–20 wt.%Cu sample and Al–10 wt.%Cu sample, both inoculated with AlTiB grain refiners.

According to the parabolic trajectory of the plane, the gravity level changes during each parabola approximately from 1g → 1.8g → 0g → 1.8g →1g (Fig. 5), with approximately 24s and 22s at 1.8g and 0g, respectively [21]. During the course of the flight, the parabola is repeated a total of 31 times. In this section, we present the solidification experiments for the refined Al–20wt.%Cu sample alloy at slow cooling rate $R$ = 0.05 K/s that extended over five parabolas.

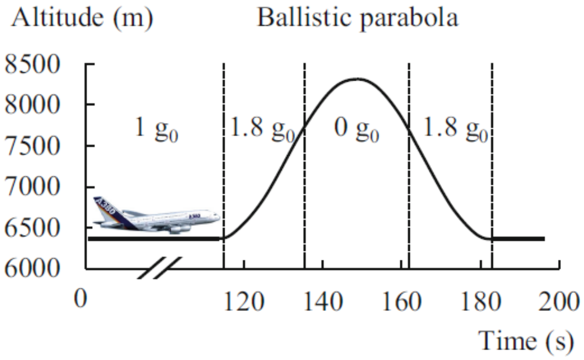

Figure 5. *Parabola profile, showing the gravity level variations during the course of the plane.*

Figure 6 displays a sequence of radiographs recorded during a part of the solidification experiment of refined Al-20.wt% Cu sample under varying gravity level. During the 1g period, a development of a columnar microstructure was observed at the bottom of the field of view (Fig.6a). Fragmentation phenomena continuously occurred along the solid/liquid interface, and the dendrite fragments floated from the bottom to the top due to buoyancy force (Fig.6b). During their upward motion, the fragments gradually melted and eventually disappeared, like in experiments performed in terrestrial laboratory conditions. In this parabolic flight experiment, a few grains additionally nucleated on the crucible wall.

The most striking effect was observed at the step-change of gravity level. Indeed, when the gravity level suddenly increased to 1.8g at the beginning of the parabola, a sudden nucleation of a large number of equiaxed grains ahead of the columnar front was observed (Fig.6c and Fig.6d). This explosive nucleation phase was obviously provoked by the sharp increase of the gravity level. The activation of refining particles ahead of the columnar front was likely triggered by an increase of the magnitude of liquid undercooling ahead of the columnar front, which is itself due to a decrease of the liquid composition ahead of the columnar structure. Indeed, the increase of the hydrostatic pressure of the melt when the gravity level changed from 1g to 1.8g generates a downward flow of Cu-poorer liquid toward the columnar front, which modifies the liquid composition ahead of the columnar front [14]. After their nucleation, the equiaxed grains started to float and then melted when they reached the hot region of the liquid (Fig.6d and Fig.6e). Some grains remained stuck between the sample walls, because their size was too large compared to the sample thickness (Fig.6f).

As soon as the gravity level reached 0g, the equiaxed grains stopped moving upward because the buoyancy force vanished (Fig.6e and Fig.6f). During the microgravity period, the grains were dissolved due to an upward copper-enriched fluid flow, which is visible in Fig. 6f as a dark region in the liquid ahead of the microstructures. This is particularly visible for the big dendrite fragment stuck on the right side of the sample. This upward fluid flow is due to vanishing of the hydrostatic pressure in absence of gravity.

After the reduced gravity period, the gravity level increased again to 1.8g and the explosive nucleation phenomenon occurred again (Fig.6g and Fig.6h), but in a less intensive manner. All these phenomena were repeated in the following parabolas. It is worth noting that these observations were also made in the experiment with the same sample, the same temperature gradient but at a higher cooling rate (R = 0.15 K/s) [14].

Moreover, the same phenomena were observed during the solidification experiment on refined Al-10.wt%Cu sample, which confirms the reproducibility of the observations. However, contrary to the Al-20wt.%Cu sample, the nucleated equiaxed grains moved slightly downwards and formed a closely-packed layer of equiaxed grains ahead the columnar front. This change in the behaviour of equiaxed grains is expected for an alloy of composition Al-10wt.%Cu, since the density of the solid is higher than the density of the liquid [22] Therefore, a columnar-to-equiaxed transition is achieved at this alloy composition. During the subsequent increase of gravity level from 0g to 1.8g period, the explosive nucleation phenomenon occurred again [14].

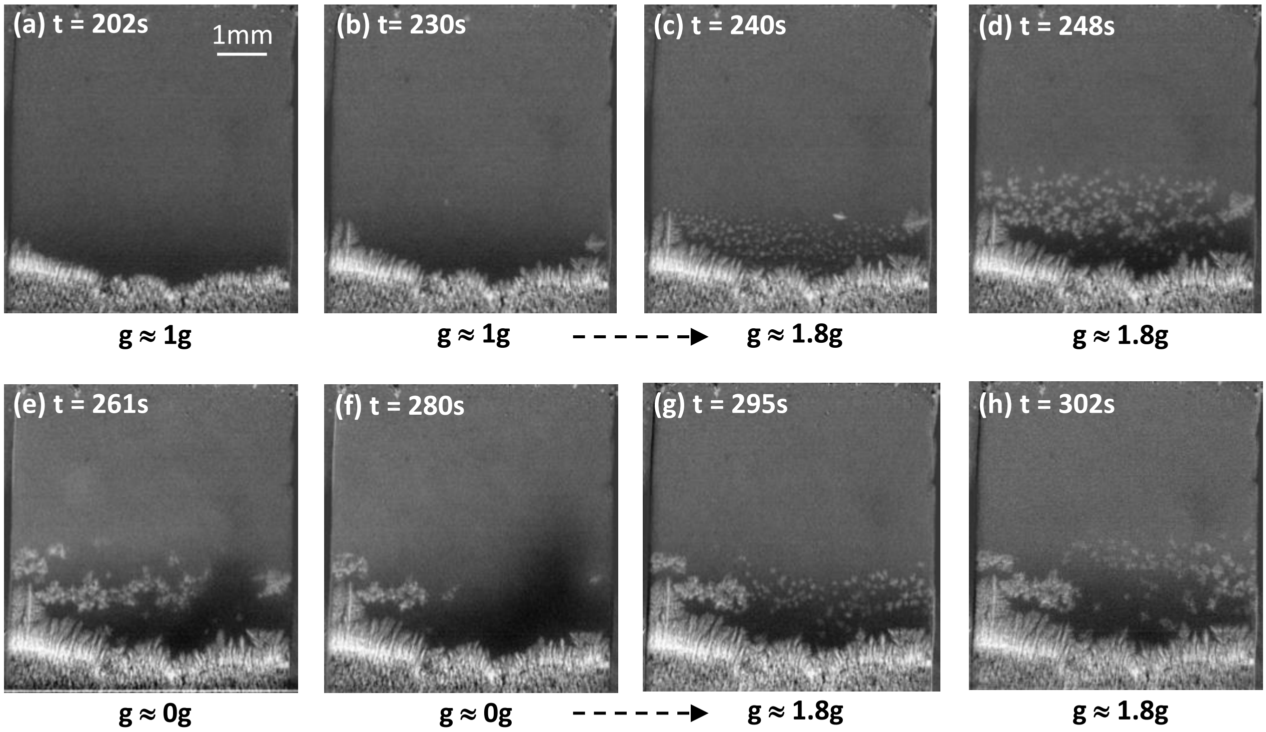

Figure 6. *Sequence of radiographs recorded during the solidification of a refined Al–20.wt.% Cu for a low cooling rate* ($R_1$ *= 0.05 K/s) and temperature gradient (G = 15 K/mm) under varying gravity level (parabolic flight). The gravity vector (in* $1g_0$ *and* $1.8g_0$ *periods) points vertically downwards relative to the field of view.*

## 5. CONCLUSIONS

X-ray radiography (or X-radiography) was used during Al–Cu solidification experiments carried out in terrestrial conditions and on board of microgravity platforms (sounding rocket and parabolic flight campaigns).

The MASER 12 solidification experiment was the first solidification experiment with in situ and real-time characterization by X-ray radiography on metallic alloys in microgravity conditions. The results obtained during the MASER 12 mission, as well as the two ground-reference tests, were very promising and validated the experimental set-up in terms of thermal behaviour and X-ray imaging, which were very challenging issues at the beginning of the project. From a scientific point of view, these results demonstrate the capability of the X-ray device developed within the framework of the XRMON project to provide a real-time diagnostic technique during solidification or melting of Al-based alloys.

It was observed that gravity level variations can have a significant impact on the microstructure formation. The variation of g-level induces a variation of the liquid composition ahead of the solid/liquid interface which affects the constitutional undercooling. For a refined alloy, this undercooling increase can provoke an explosive nucleation of equiaxed grains ahead the columnar front, yielding a composition-dependent columnar-to-equiaxed transition.

## 6. ACKNOWLEDMENTS

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