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Overview of *In Situ* X-ray Studies of Light Alloy Solidification in Microgravity

D.J. Browne[1,*], F. García-Moreno[2,3], H. Nguyen-Thi[4], G. Zimmermann[5], F. Kargl[6] , R.H. Mathiesen[7], A. Griesche[8], O. Minster[9]

Abstract

Gravity has significant effects on alloy solidification, primarily due to thermosolutal convection and solid phase buoyancy. Since 2004, the European Space Agency has been supporting investigation of these effects by promoting *in situ* X-ray monitoring of the solidification of aluminium alloys on microgravity platforms, on earth, and in periodically varying g conditions. The first microgravity experiment – investigating foaming of liquid metals – was performed on board a sounding rocket, in 2008. In 2012 the first ever X-ray-monitored solidification of a fully dense metallic alloy in space was achieved: the focus was columnar solidification of an Al-Cu alloy. This was followed in 2015 by a similar experiment, investigating equiaxed solidification. Ground reference experiments were completed in all cases. In addition, experiments have been performed on board parabolic flights – where the effects of varying gravity have been studied. We review here the technical and scientific progress to date, and outline future perspectives.

[1] School of Mechanical and Materials Engineering, University College Dublin, Belfield, Dublin 4, Ireland.

[2] Institute of Applied Materials, Helmholtz-Zentrum Berlin für Materialien und Energie GmbH, Hahn-Meitner-Platz 1, D-14109 Berlin, Germany.

[3] Technische Universität Berlin, Structure and Properties of Materials Department, Hardenbergstr. 36, D-10623 Berlin, Germany.

[4] IM2NP & Université d'Aix-Marseille, Campus St-Jérome, Case 142, 13397 Marseille Cedex 20, France

[5] Access e.V., Intzestrasse 5, D-52072 Aachen, Germany.

[6] Deutsches Zentrum fur Luft- und Raumfahrt (DLR), Institut fur Materialphysik im Weltraum, Linder Höne, 51147 Köln, Germany.

[7] Institut for Fysikk, Norsk Teknisk-Naturvitenskapelige Universitet (NTNU), 7491 Trondheim, Norway.

[8] Bundesanstalt für Materialforschung und –prüfung (BAM), FB 9.4 Weld Mechanics, Unter den Eichen 87, 12205 Berlin, Germany.

[9] Physical Sciences Office HRE-UP, Science Department, Human Spaceflight and Robotic Exploration Directorate, ESTEC, European Space Agency, BP 299, 2200AG Noordwijk, The Netherlands.

*corresponding author: david.browne@ucd.ie

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Introduction

The microstructure and resultant properties of light alloys, as with many other metallic alloys, may be determined during a solidification process along the manufacturing route. Traditionally, solidification science, as applied to metals, has included theory and computational modelling, but experimental observation of the progress of solidification (nucleation, growth and impingement of crystalline grains) was not possible due to the opacity of metals. As a result, validation of theory and computational models was mostly limited to post-solidification metallography. However, nearly 20 years ago pioneering researchers began to use high brilliance X-rays at large-scale synchrotron facilities – such as the European Synchrotron Radiation Facility (ESRF) in Grenoble, France – to observe the progress of solidification in alloys which provided sufficient transmission contrast between solid and liquid phases [1,2]. Al-Cu, Al-Ni or Al-Ge have proven to be popular alloy systems for such research. Important gravitational effects such as buoyancy and motion of equiaxed grains [3,4] and fragmentation of columnar dendrites [5] were observed *in situ*, providing a challenge to computational modellers to simulate such behaviour accurately.

In order to study gravitational effects, such as buoyancy and thermosolutal convection, it is best to compare experiments with and without the effects of gravity. The latter, however, can only be achieved in microgravity conditions – such as those in free-falling environments or in space. So the European Space Agency (ESA), near the start of the millennium, considered supporting a scientific community to use X-rays to study alloy solidification in microgravity conditions. This would require the development of specialised and compact equipment, consisting of X-ray source, solidification furnace, camera and detectors, which could be deployed on board microgravity vehicles. Along with feasibility studies on hardware, ESA started an academic Topical Team network of interested scientists and engineers, in 2004, to start planning experiments. Candidate microgravity facilities (with microgravity period) included parabolic flights (seconds), sounding rockets (minutes) and the International Space Station (days). Although the ISS has been used for alloy solidification experiments, e.g. [6-8], it has not yet hosted *in situ* X-ray experiments. The research consortium to date, which includes the authors, has concentrated on unmanned sounding rocket and manned parabolic flights. The sounding rockets, with many minutes of microgravity time, are particularly suited to solidification studies at ~0 g. Parabolic flight campaigns, on the other hand, provide about 20s of microgravity per altitude parabola, but each such period is directly preceded by hypergravity periods of about the same duration but in which ~2g conditions are reached [9]; such flights are therefore suited to research on the effects of g variation on phenomena of interest. The current contribution selects some highlights from the ESA sounding rocket and parabolic flight campaigns. The programme of research is known as XRMON: *in situ* X-ray monitoring of advanced metallurgical processes under microgravity and terrestrial conditions. Hardware developed during the research is given the XRMON label.

Sounding Rocket Experiments

ESA has sponsored a series of MASER (Material Science Experimental Rocket) sounding rocket flights to support the alloy solidification research. MASER is a sub-orbital microgravity research rocket that is launched by the Swedish Space Corporation from Esrange in northern Sweden. The rocket can provide between 6 and 7 minutes of high quality microgravity, depending on weather conditions.

MASER 11: Foaming of Metals

Metallic foams are very promising materials for certain industrial applications due to their extraordinary properties like low density, high energy absorption, high specific strength and stiffness, damping, etc. [10]. However, large-scale production has been hindered by an incomplete understanding of the processes by which metal foam melts stabilize and solidify, and how these processes influence the final structure of the foam. This is still a major challenge, as examining the molten system *in situ* and verifying proposed mechanisms requires sophisticated experimental set-ups.

The flow of liquid metal, due to gravity, induces changes in foam density distribution. This affects foam evolution by influencing, for instance, the thinning of cell walls which in turn induces cell wall ruptures as is known in the case of aqueous foams [11]. Under microgravity we can separate some of the key effects which govern foam evolution, namely drainage, flow, coarsening and coalescence in order to improve models of the foaming metals.

An X-ray transparent foaming furnace combined with an *in situ* X-ray imaging system was designed and constructed by the Swedish Space Corporation, and launched successfully in the XRMON module of MASER 11 in May 2008. The microgravity foaming setup comprised a 80 kV microfocus source, the foaming furnace and a flat panel detector. The furnace was nearly X-ray transparent. Inside the heating zone was situated the crucible, with inner dimensions of 20 x 20 x 10 mm^3. It was machined from one piece of boron nitride together with a closing lid. A Pt wire wound around the BN crucible allowed heating of the sample up to 700 °C. Thermocouples were placed on the crucible walls to record the temperature profile. The sample foaming procedure and the heating profile, from the starting point of heating until the switching off of the heater, were completely automatic and followed the profile pre-set before the experiment.

The sample precursor was a foamable thixocast Al- 6.0wt.%Si - 4.0wt.%Cu + 0.5 wt.% TiH_2 alloy, of dimensions 20 x 10 x 4 mm^3. It expanded in the course of heating to 600°C at a rate of 200 K/minute to around 5 times its volume, completely filling the crucible with foam. This is caused by the melting of the alloy and the parallel action of the gas source provided by the blowing agent TiH_2.

During MASER 11 a homogeneous wet metal foam with round pores and without collapse could be produced; Fig 1. There was no gravity-induced drainage but an unexpectedly strong coalescence rate. This sounding rocket mission allowed us to study for the first time cell coarsening under microgravity. We discovered for metallic foams, besides the expected effect of gravity-induced drainage and cell wall thinning, the pronounced effect of the blowing agent on coalescence, influencing foam stability considerably in terms of cell wall ruptures [12,13]. As a consequence we concentrated in the past years on the development of foams free of external blowing agents following the powder metallurgical route, such as AlMgCu or AlMgZn alloys. The gas source is intrinsic in these foams, i.e. included in the alloy powders, leading to improved gas nucleation. Two patents followed this work and related research carried out on ESA parabolic flights.

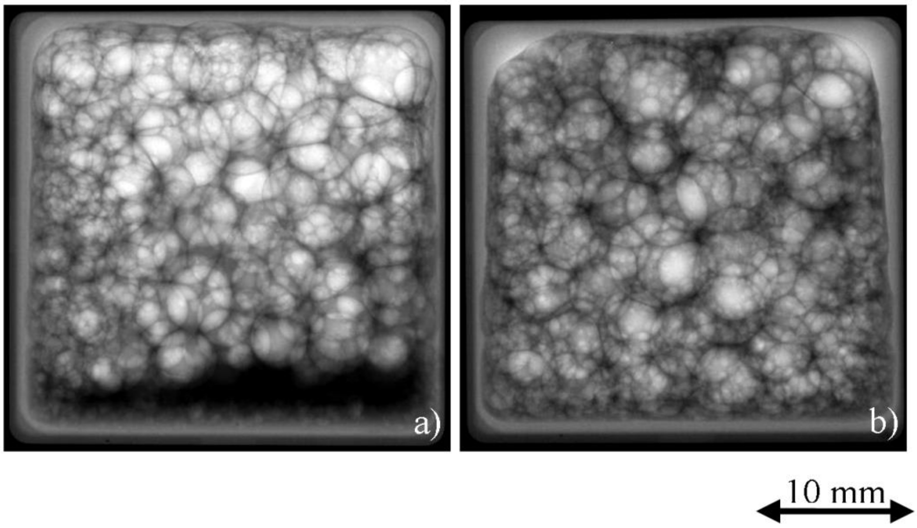

Figure 1. Thixo AlSi6Cu4 sample after 200 s in the liquid state foamed (a) on Earth and (b) during the MASER 11 flight. On Earth liquid flow due to gravity-induced drainage leads to higher foam density at the bottom of the sample (darker region) and thinner cell walls in the top part, which induce increased bubble rupture. Under microgravity an homogeneous wet metal foam with round pores can be produced.

MASER 12: Columnar Solidification

A novel facility was developed for the study of directional solidification of aluminium – based alloys, with *in situ* X-ray radiography on board microgravity platforms. This new facility, named XRMON-GF (GF for Gradient Furnace), was successfully deployed during the MASER 12 sounding rocket campaign, in spring 2012 [14,15].

The experiment

The gradient furnace is of Bridgman type, with two heaters: one for the “hot” zone and one for the “cold” zone, which can be adjusted independently. This enables directional solidification with thermal gradients within the range of 1-10 K/mm and solidification of the sample is made by cooling the heaters. The Al - 20wt% Cu sample was 5 mm in width, 50 mm in length and 150 µm in thickness. More details of XRMON-GF are available elsewhere [14,15].

For the X-ray radiography system, a microfocus X-ray tube with 3 μm focal spot was used, with two peaks in energy at K_α = 17.4 keV and K_β = 19.6 keV, which are adapted to Al – 20 wt% Cu alloys. The effective spatial resolution is about 4-5 μm, which is satisfactory for studying evolving solidification microstructures.

The MASER 12 sounding rocket was launched on 13 February 2012 at Esrange, Sweden. Since the microgravity duration during a MASER sounding rocket is limited to six minutes, a suitably adapted experimental timeline was defined [14]. After a short stabilization period (about 20s), the sample 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.

Results

Figure 2 displays three sequences, of three images each, taken during the solidification of an Al - 20 wt% Cu alloy in microgravity conditions (first row), and for the two reference experiments at normal gravity (second and third rows). These sequences of radiographs show 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. 2) and formed a very disordered dendritic pattern (second column in Fig. 2). Gradually, the grain competition gave a more regular array of dendrites (third column in Fig. 2).

During the columnar solidification in microgravity conditions and for a horizontal sample, nucleation of equiaxed grains ahead of the columnar front is visible, most likely on a small heterogeneity of the sample oxide layer. This grain slightly rotated during the solidification, which clearly showed that they were not stuck on the sample walls. However, due to either the microgravity environment or the horizontal position of the sample, those grains remained at the same altitude and were progressively engulfed by the columnar front and then completely merged into the columnar microstructure.

For the upward solidification experiment, the most important feature is the multiple fragmentations in the dendritic tip region, at the top of the columnar front (bottom row, right column in Fig. 2). After their detachment, most fragments moved upward due to the buoyancy force since, in the case of Al - 20wt% Cu alloys, the solid density is lower than the density of the surrounding liquid [16]. Some of those fragments were free to float to the hot region of the sample (indicated by white arrows in Fig.2). 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 aluminum-rich dendritic fragment. It is worth noting that these dendrite fragments could not promote columnar-to-equiaxed transition [17-19] or CET, because they were carried up 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. For the sake of completeness, it worth mentioning that the dendrite fragmentations were also observed during the 1g-horizontal and µg experiments, but in these cases deep in the mushy zone. After their detachment, the dendrite fragments moved towards the cold part of the mushy zone, probably carried by the liquid movement due to the sample shrinkage [18].

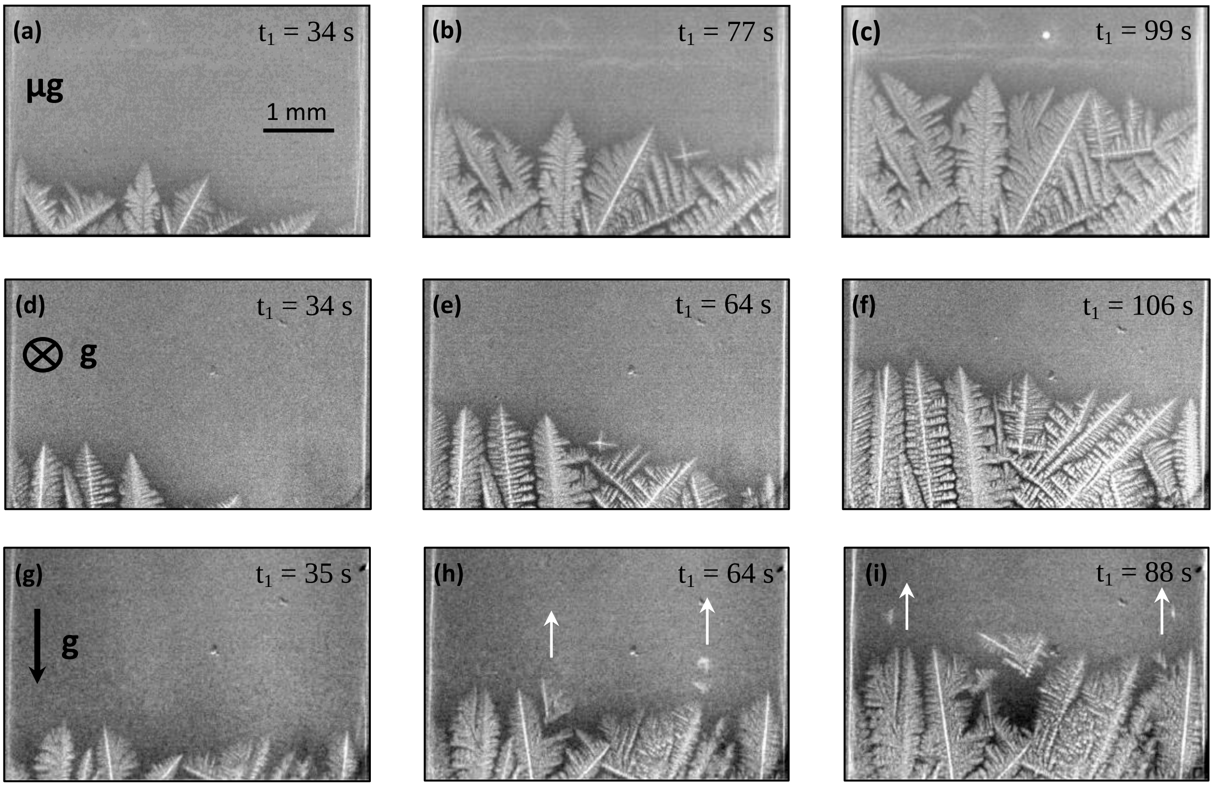

Figure 2: Columnar solidification of Al-20wt.% Cu with a temperature gradient of about 150 K/cm between the two heaters and a cooling rate of 0.15 K/s on both heaters: (a-c) in microgravity conditions, (d-f) sample in horizontal position, (g-i) sample in vertical position (reference time is arbitrary). During upward solidification, after their detachment from the primary trunks, some dendrite fragments were free to float to the hot region of the sample (white arrows in Fig.2h and 2i).

Based on the radiographs, it was possible to measure the growth rate as a function of time for each experiment (Fig. 3). For each experiment, a representative dendrite well-oriented with respect to the temperature gradient, close to the centre of the sample and that crossed the whole height of the FoV, was chosen and its tip position was measured as a function of time. In each case, the three successive growth rates are clearly visible and an average value can be deduced. The values of the growth rate for the microgravity experiment (Fig. 3a) are closer to those of the 1g-upward solidification (Fig. 3c) than to those of the 1g-horizontal solidification (Fig. 3b), in spite of the multiple fragmentations which occurred during 1g-upward solidification.

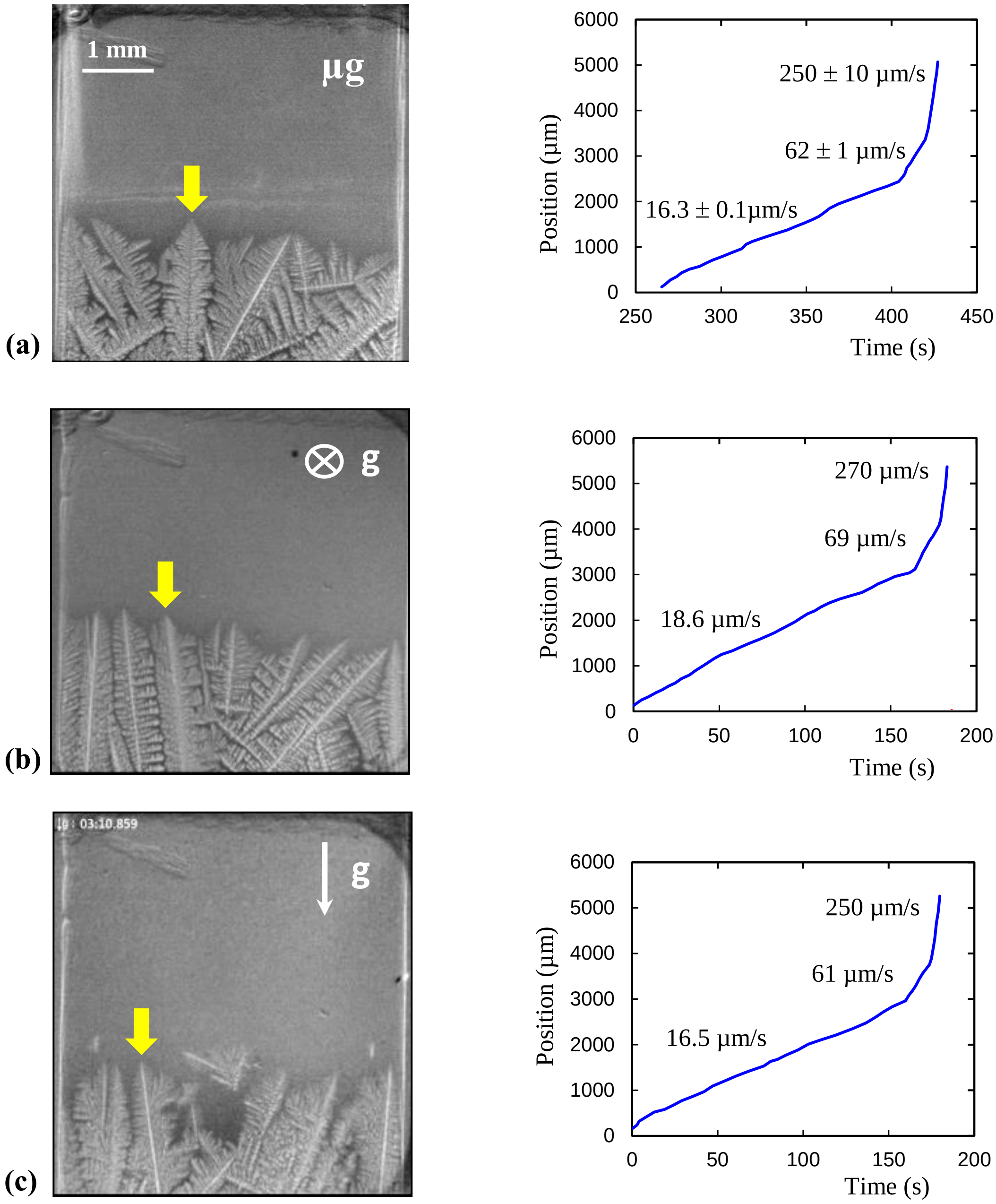

Figure 3: (right column) Front position as a function of time showing the three successive growth rates for (a) the microgravity, (b) 1g-horizontal solidification and (c) 1g-upward solidification. On the radiograph at the left of every plot, a yellow arrow indicates the dendrite chosen for measuring the position of the solidification front as a function of time.

This observation can be explained by considering the fact that solidification in the 1g-upward case is performed in both stable thermal and solutal conditions (hot zone placed above the cold zone and solute denser than the solvent), which minimise the occurrence of natural convection, in particular for high axial temperature gradient and high solidification velocities like in these experiments. Actually, it has been shown that high values of these two control parameters

prevent convective flow induced by residual transverse temperature gradient in this configuration [20].
On the other hand, for the sample solidification in 1g-horizontal position, the convective roll induced by thermal convection and/or the rejection of heavy solute within the thickness of the sample may be strong enough to modify the thermal or the solutal fields, thus increasing the growth rates. Indeed, in the case of horizontal Bridgman growth, thermal convection occurs as soon as there is a temperature gradient along the sample (i.e. no threshold). This qualitative explanation was confirmed by an order of magnitude analysis performed by Nguyen-Thi et al [21].

Conclusions
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 behavior 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 in the frame of the XRMON project to provide a real-time diagnostic technique during solidification or melting of Al- based alloys.

MASER 13: Equiaxed Solidification

Due to the unconstrained nature of equiaxed solidification, buoyancy effects can be significant. To achieve equiaxed solidification which is evenly distributed across the FoV, a locally spatially isothermal sample is needed. These conditions were nearly achieved by setting the hot and cold sections of the XRMON-GF [14] to the same temperature. In terrestrial experiments, using grain-refined Al-20wt.% Cu, we demonstrated the effects of gravity by altering the orientation of the sample with respect to gravity. For the horizontal sample, gravity effects were minimal, but grain buoyancy effects were dramatic with the sample plane vertical [22]. Due to the nature of XRMON-GF, however, nucleation events were not randomly distributed throughout the FoV but a front of equiaxed grains moved across from one side of the sample (see Fig. 2 of [22]). As a result it was decided to design and build a new furnace which could create more isothermal conditions in the sample.

The new furnace, XRMON-SOL, was designed to have rotational symmetry, and the samples were thin discs. The furnace and equipment, designed to fit within the MASER experimental module, are described in detail in [23], and shown in Fig. 4. The isothermality of the new system was excellent, as attested to by the even equiaxed solidification achieved, and the furnace was tested within the MASER sounding rocket module to prove the hardware for the space flight [23]. The test schedule was synchronised with the rocket time-line so that the sample was completely melted, and re-solidified, within the notional microgravity window. Although the effects of gravity were minimised using a horizontal sample orientation, the results suggested that such effects could only be eliminated in a microgravity environment.

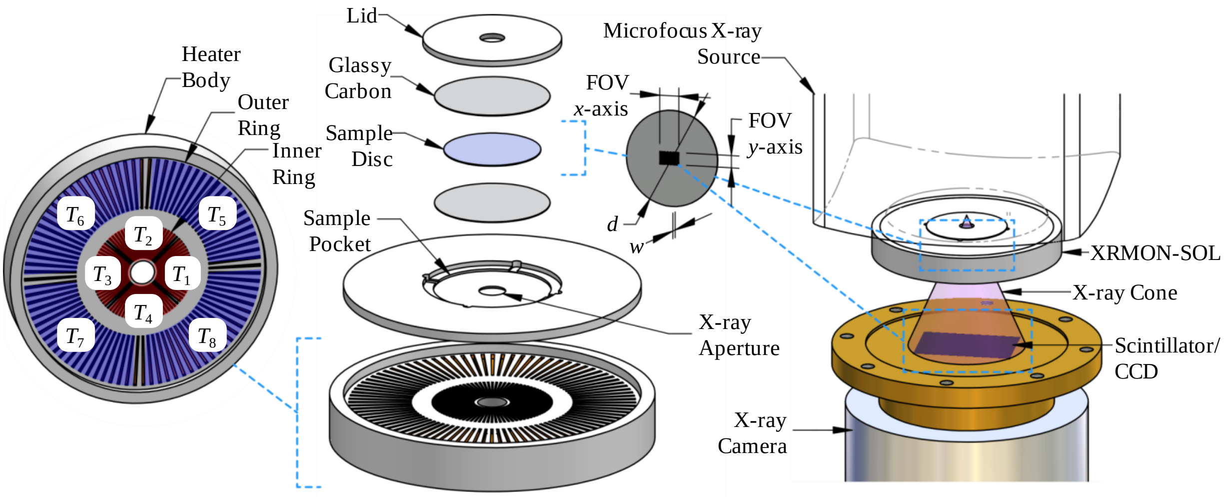

Figure 4: Schematic illustration of XRMON-SOL construction and integration with in situ X-ray diagnostics. Labels $T_{1\rightarrow 8}$ indicate the relative location and arrangement of eight independently regulated heater coils. Dimensions d and w denote the sample diameter (21 mm) and thickness (0.2 mm), respectively. FoV x-axis and y-axis represent the physical extent of the X-ray field of view relative to the sample diameter, horizontally (~ 4.1 mm) and vertically (~ 2.7 mm), respectively.

Following proving of the XRMON-SOL capability, it was incorporated into the MASER 13 rocket, which launched on 1 December 2015. Prior to launch, and using the flight sample in the flight furnace, a ground reference test was performed, with the sample in horizontal orientation. The microgravity flight, and the solidification experiment, were successfully completed as planned. The X-ray video sequence has been published openly, along with a report of the experiments [24]. Figure 5 shows an image from the sequence. The microgravity results were compared to the ground reference ones. Because the sample was horizontal in the terrestrial reference case, the differences between the two experiments were small; similar nucleation distribution, dendrite growth rates and final eutectic solidification were observed in both. However in microgravity the equiaxed grains were completely immobile during the majority of their growth [24] – in comparison to gravity-driven motion and rotations of some grains in the ground experiment which are currently being quantified, along with the evolution of the solutal fields, using an approach developed by Becker et al. [25]. Towards the end of solidification, in both experiments, grains moved due to solidification-induced shrinkage and interdendritic flow of the liquid phase.

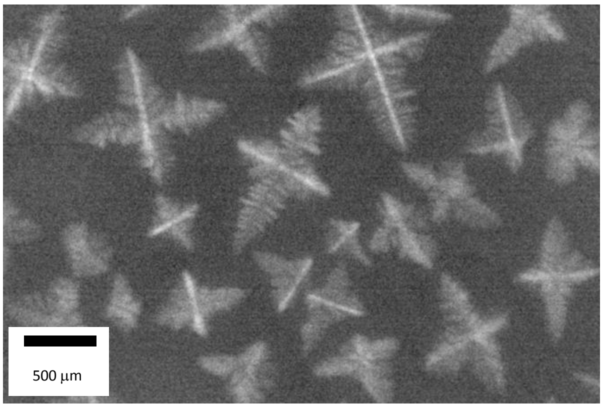

Fig. 5: still image form the MASER-13 microgravity experiment, furnace temperature = 582 °C

The results provide, for the first time, benchmark data for computational modelling of equiaxed metallic alloy solidification in a gravity-free environment, and isolate shrinkage effects so that they are the only cause of grain motion towards the end of solidification.

MAXUS 9: Diffusion in Liquid Metals

A special furnace, XRMON-DIFF, is currently being tested for flight on the MAXUS sounding rocket, which is larger than the MASER one, with a higher apogee enabling about 12 minutes of microgravity time. The experiment will be on diffusion in liquid metals in the absence of natural convection, and launch is planned for 2017.

Chemical diffusion coefficients of liquid alloys are important input parameters for modelling microstructure evolution during solidification. Traditionally, measurements have been carried out by post-mortem analysis of melted, annealed and solidified diffusion couples leading to large uncertainties in the data. A major improvement is the use of the rather sophisticated shear-cell technique [26] which enables melting of the different alloys separately and separating of the liquid column into individual slices prior to cooling. Experiments were even carried out under microgravity conditions to avoid buoyancy convective-flow [27]. However, uncertainties in the data can still often exceed 30%. In recent years by *in situ* monitoring the diffusion process using X-ray radiography the accuracy of the data has been greatly improved [28]. Using a linear shear-cell technique even materials that sediment on melting or that show largely different melting and liquidus temperatures can be handled [29,30]. The shear-cell was successfully operated aboard the DLR sounding rocket MAPHEUS-4 in the MIDAS-M setup investigating the influence on cross correlations on interdiffusion in Al-rich Al-Ni alloys [31]. By implementing a newly developed ultra-high temperature shear-cell chemical diffusion in binary Al-Ti and Si-Ge alloys will be investigated aboard MAXUS-9 in spring 2017 using the XRMON-DIFF MAXUS-9 setup. Results will be reported in a later publication.

Parabolic Flight Experiments

ESA parabolic flight campaigns were carried out on board a specially converted Airbus A300 commercial turbofan aircraft, operated by Novespace in Bordeaux, France [9]. The experiments are manned by the scientific investigators during the flights, each of which consists of about 30 parabolas. The findings of some of the XRMON campaigns are presented here.

Parabolic Flight experiments: columnar solidification

Directional solidification experiments on refined Al – 20 wt% Cu and refined Al – 10 wt% Cu samples were 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 impact of the effects of gravity level variations on the columnar-to-equiaxed transition. The influence of these gravity level variations on the CET was investigated using *in situ* and real-time X-ray radiography.

Experiment

Experiments were carried out in a dedicated apparatus entitled XRMON-PFF (Parabolic Flight Facility) [32] for a wide range of cooling rates and a constant temperature gradient. 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 (Fig. 6a). 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 solidification of two samples were carried out, an Al – 20 wt.% Cu sample and Al – 10 wt.% Cu sample, both inoculated with AlTiB grain refiners. The sample dimensions were 50 mm in length, 5 mm in width and about 200 µm in thickness, and is in the vertical orientation in the furnace.

According to the parabolic flight trajectory, the gravity level changes during each parabola from 1 g → ~1.8 g → ~0 g → ~1.8 g → 1 g (Fig.1b), with approximately 24s and 22s at ~1.8 g and ~0 g respectively [9]. During the course of the flight, the parabola is repeated a total of 31 times. In this work, we present the solidification experiments for both alloys at slow cooling rate R = 0.05 K/s that extended over five parabolas.

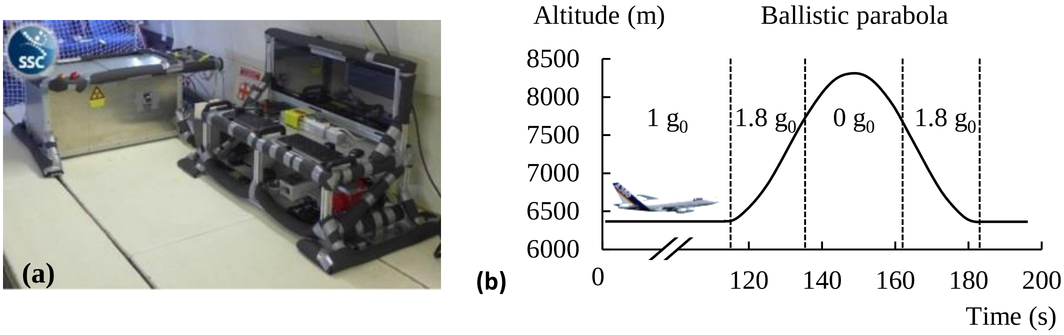

Figure 6: (a) XRMON-PFF facility in Novepace Airbus A300 Zero-G, (b) Parabola profile showing gravity level variation with time

CET triggering by variation of gravity level

Figure 7 displays a sequence of radiographs recorded during a part of the solidification experiment of refined Al – 20wt% Cu sample under varying gravity level. During the 1 g period, a development of a columnar microstructure was observed at the bottom of the field of view. Fragmentation phenomena continuously occurred along the solid/liquid interface at the top of the mushy zone, and the dendrite fragments floated from the bottom to the top due to buoyancy force (Fig. 7a). During their upward motion, the fragment gradually melted and eventually disappeared, like in experiments performed in terrestrial laboratory conditions.

However, when the gravity level suddenly increased to ~1.8 g at the beginning of the parabola, a sudden nucleation of a large number of equiaxed grains ahead of the columnar front was observed (Fig. 7b). The large number of grains nucleated ahead of the columnar front was likely triggered by an increase of the liquid undercooling ahead of the columnar front, which is itself due to a decrease of the liquid composition ahead of the columnar structure. This composition decrease is attributed to the increase of the hydrostatic pressure of the melt when the gravity level changed from 1 g to 1.8 g. As the two thin glassy carbon sheets that compose the crucible are flexible, the hydrostatic pressure rise caused a larger bulging of the sample at the solid-liquid interface. As a consequence, this bulging increase induced a downward flow of less concentrated liquid toward the columnar front [32]. The new grains started to float and then melted when they reached the hot region of the liquid (Fig. 7c).

As soon as the gravity level reached ~0 g, the equiaxed grains stopped moving upward because the buoyancy force vanished (Fig. 7d). After the reduced gravity period, the gravity level increased again to 1.8 g and the explosive nucleation phenomenon occurred again (Fig. 7e). All these phenomena were repeated in the following parabolas. It is worth noting that these observations were also observed in another experiment, with the same temperature gradient but a higher cooling rate (R = 0.15 K/s) [32].

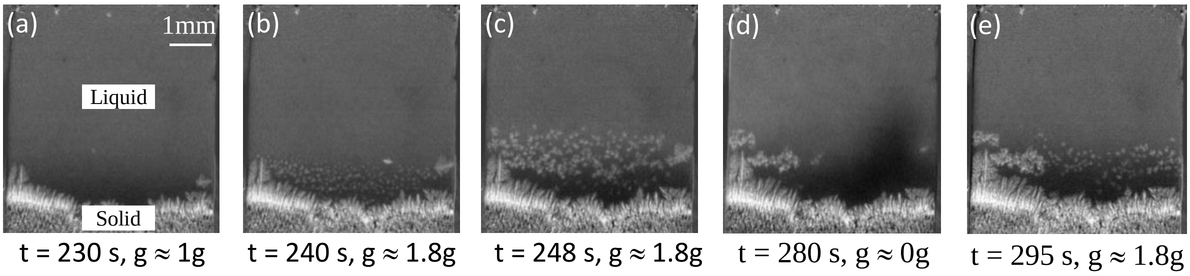

Figure 7: Sequence of radiographs recorded during a part of the solidification experiment for the refined Al – 20 wt% Cu, with a low cooling rate (R = -0.05 K/s) and a high temperature gradient (G = 15 K/mm) and under varying gravity level. The gravity level points vertically downwards relative to the FoV.

A sequence of radiographs recorded during a part of the solidification experiment on refined Al – 10wt% Cu sample is shown in Fig. 8. Figure 8a shows the initial columnar growth, with a few nucleations of equiaxed grains ahead of the columnar front during the 1 g period. When the gravity level suddenly increased to 1.8 g a large number of equiaxed grains nucleated ahead the solidification front (Fig. 8a), like for the Al – 20wt% Cu sample. The large number of grains nucleating ahead of the columnar front was triggered by an increase of the liquid undercooling ahead of the columnar front due to a downward flow of higher purity liquid toward the columnar front. However, contrary to the Al-20wt% Cu sample, these grains moved slightly downwards and formed a closely-packed layer of equiaxed grains ahead the columnar front, making it easier to form a CET. This change in the behavior of equiaxed grains is expected for an alloy of composition Al – 10 wt% Cu, since the density of the solid is higher than the density of the liquid [16]. During the subsequent increase of gravity level from 0 g to 1.8 g period, the explosive nucleation phenomenon occurred again (Fig. 8d).

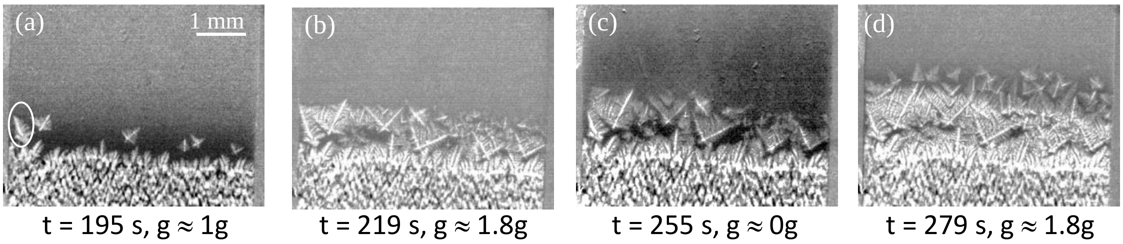

Figure 8: Sequence of radiographs recorded during the solidification of a refined Al – 10 wt.% Cu for a low cooling rate (R = 0.05 K/s) and temperature gradient (G = 5.5 K/mm) under varying gravity level (parabolic flight). The gravity vector (in 1 g and 1.8 g periods) points vertically downwards relative to the field of view.

Conclusion

X-ray radiography was used during Al-Cu solidification experiments carried out in terrestrial conditions and during parabolic flight experiments. 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.

Parabolic Flight Experiments: other

On separate parabolic flights, the effects of varying g conditions on equiaxed solidification were investigated [33]. In this work, a grain-refined Al-20wt%Cu alloy was solidified, near-isothermally, also using XRMON-GF, with the hot and cold zones set to the same temperature. Solidification was controlled such that nucleation occurred coincident with the onset of microgravity. This allowed for the effects of microgravity on equiaxed nucleation and initial growth, followed by continuing solidification in hypergravity, to be observed, as well as the effect on the semi-coherent grain structure when transitioning between the two. Under nominally 0 g conditions, equiaxed grains were observed to move together within the FoV, in an opposite direction to the g-level fluctuations recorded during solidification: at levels slightly above 0 g the unconstrained dendrites moved downwards, and vice-versa. This was unexpected, as the dendrites are lighter than the average liquid for this alloy [16]. It seems as though their motion was due to advection in the denser Al-Cu liquid. Once the 0 g → 1.8 g transition occurred, this liquid motion caused packing of the equiaxed grains towards the bottom of the sample, at which time they formed a rigid coherent structure [33]. Due to the flexibility of the two thin glassy carbon sheets that form the crucible walls, bulging of the bottom of the sample at high g occurs due to the downward flow of both liquid and advected solid. This is the cause of the apparently anomalous solid motion. Normal behaviour (solid buoyancy) would be expected if a rigid crucible were used.

On parabolic flights in which the XRMON foam unit was used, we found out that under microgravity conditions, imbibition of liquid metal in the foam structure due to capillarity forces dominated. The previously induced drainage due to gravity disappeared. This effect could be observed especially after gravity transition from 1.8 to 0 g. The flow back of liquid during the transition from 0 g to 1.8 g enabled calculation of the effective viscosity and surface tension of the liquid foams for the first time [34].

Conclusions and Outlook

Microgravity experiments have been used to deepen our knowledge on processing of liquid aluminium alloys – both on foaming and solidification phenomena. However, due to the limited opportunities afforded to date, only a small number of conditions have been investigated. It is expected that more microgravity flights will be run in the future, enabling expansion in the number of cases studied, and the investigation of new phenomena. Development of an experimental facility on the ISS, or some other space station, would enable multiple experiments to be performed using the same equipment because the microgravity time on board is not limited by nature.

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