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X-Ray Radioscopy of Liquid Metal Foams under Microgravity

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Abstract

Results obtained from ground-based reference experiments, a parabolic flight campaign (PFC) and the sounding rocket MASER 11 campaign are presented. X-ray analysis that allows us to follow the entire foaming process *in-situ*, was employed for qualitative and quantitative analysis of local changes in density, pore size distribution and the frequency and location of cell wall rupture. In the PFC, we found that under microgravity imbibition of liquid metal into the foam due to capillarity forces dominates, and even a pre-existing gravity-induced drainage disappears. This effect could be observed specially after gravity transitions from 1.8 to 0 g. During MASER 11, a homogeneous wet collapse-free metal foam with round pores could be produced. Obviously, there was no gravity-induced drainage, but an unexpected strong coalescence rate was observed. We conclude that gravity-induced drainage is not the only factor that leads to cell wall rupture as previously assumed.

1. Introduction

Metallic foams, especially Al-foams, are becoming very interesting for industrial applications due to their extraordinary properties like light-weight, high specific strength and stiffness, energy absorption, damping, etc. But many fundamental questions are still open and have to be clarified to understand the phenomena governing foam growth and cell wall stabilization, and to be able to improve the quality of the foam structure.

Gravity driven drainage makes an investigation of foam coarsening and of the influence of surface active elements and viscosity-enhancing additives very difficult. 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 it is known in the case of aqueous foams [1]. Under microgravity we can separate some of the key effects which govern foam evolution, namely drainage, flow, coarsening and coalescence in order to improve the models of the foaming metals. In metal foams produced by the powder metallurgical route flow of bubbles and coarsening are not significant as can be observed from the radioscopy images. Using microgravity we can influence drainage and therefore study coalescence. Preliminary results were already presented at the Eufoam 2008 conference organized by ESA at Estec, Holland and published [2].

The PFC was also used as test of the hardware for the sounding rocket experiment, being the first time that an imaging X-ray diagnostic system was flown under microgravity conditions. The hardware is also considered by ESA as a multipurpose system that will fly in future with different material science experiments like diffusion in melts or metal solidification experiments [3,4].

In this work we present further results obtained from the 46th ESA PFC, showing metal liquid imbibition and changes

in the gravity induced drainage. The focus is concentrated on the results of MASER 11, where the coalescence was studied in more detail.

2. Materials and methods

For the preparation of the MASER 11 campaign and testing of the hardware system the need of a parabolic flight measurement was identified. The microgravity tests should also provide useful information concerning the selection of the most appropriate sample for the sounding rocket campaign, and how the metal foaming kinetics is influenced during microgravity. Moreover, the parabolic flight should give scientific results observing the drainage and imbibition capability of the liquid metal foam during microgravity and during the transition from 0 g to the highest gravity, i.e., 1.8 g.

Series of X-ray images during foaming of samples were recorded. Therefore qualitative analysis of foam evolution was possible. But also quantitative analysis like, e.g. foam density and coalescence rate was performed with the help of the self developed software Axim [5]. In particular, the cell wall ruptures were calculated comparing consecutive images of the sequence with a corresponding threshold. Changes in absorption intensity were recognised as single cell wall ruptures taking the bubble size dimension into account. Only ruptures that occurred after the foam was fully expanded and before cooling started were considered.

2.1. The 46th ESA Parabolic Flight Campaign

The μ G-furnace for foaming of metals built by the Swedish Space Corporation (SSC) was tested in combination with the X-ray system available at TU Berlin. The furnace was validated in Berlin. Further ground tests were performed with

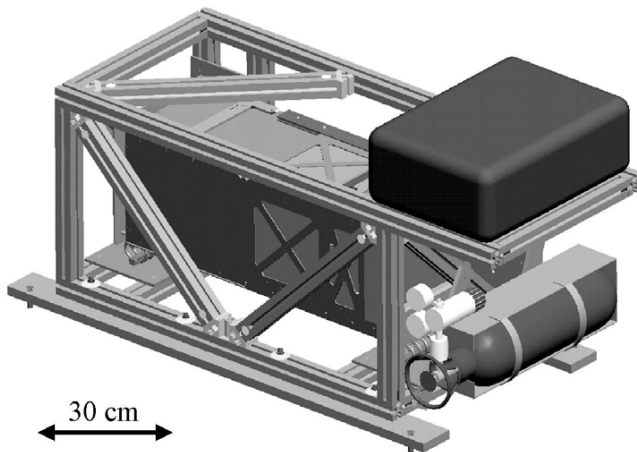


Fig. 1 : CAD of XRMON module on the 46th ESA PFC.

the flight setup to optimize the heating sequence and to obtain reference results on ground, after it was completely ready for the PFC.

A sketch of the X-ray module is given in Fig. 1. It was composed mainly of a radiation protected chamber covered with lead. In opposite ends a microfocus X-ray source and a flat panel detector from Hamamatsu were placed. In between an exchangeable furnace loaded with a precursor was placed. In order to have a maximum number of experiments, six furnaces, each loaded with one sample, were stored in a secured box attached to the setup. They were exchanged during the interval between two sets (each consisting five) of parabolas. X-ray images of the experiments, temperature, gravity and further control parameters were recorded during the experiment. Different temperature ramps were programmed in advance in order to reach the foaming at the right time to exploit as much microgravity time as possible. Small adjustments of ± 5 s were possible after each experiment by selecting pre-programmed ramp. A gas cylinder with pressurized Ar was used for the cooling of the samples. The scientists could observe and interact with the experiment from a separate computer pool control. The setup was also described in [2].

2.2. XRMON module on MASER 11

The software and hardware was tested during the parabolic flight under microgravity conditions. Some discrepancy in the heating profiles was found compared to the ground experiments, induced by the different gravity conditions and corresponding modified heat contact conditions between sample and crucible. The difference to reach the end temperature was found to be at maximum ± 5 s, what was not a problem for MASER 11.

One modification (improving the insulation of the heating wire) of the furnace was found to be necessary and it was implemented by SSC. To avoid overpressure in the crucible, that will influence foaming, it was necessary to make an opening in it. Due to this opening liquid melt droplets could also come out and damage the furnace. Under microgravity this melt could once damage the heater. After improvement this problem was solved for MASER 11.

The setup was adapted for MASER 11 removing the PFC supporting frame and introducing it in a cylindrical metal casing, that is part of the MASER 11 payload (Fig. 2). The external computers and power supply were replaced by an integrated flight computer and battery packs, respectively. The recorded images were stored during flight in a solid state

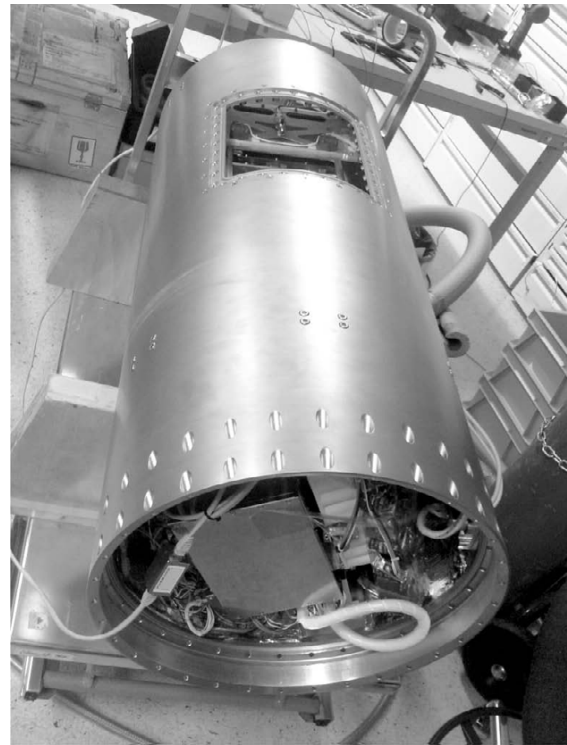


Fig. 2 : MASER 11 XRMON metal foam module.

hard disk and recovered after the flight.

After the module acceptance review in February 2008, the ready module (Fig. 2) was already mounted and prepared for flight. Reference tests were done using the same configuration, samples, temperature profiles, batteries, etc. The results of the tests on Earth were later compared with the sample made under microgravity. The flight was performed on 15th Mai 2008.

3. Results

Although the parabolic flight was focused on testing the setup for MASER 11, some relevant results concerning the absence of drainage and the rapid changes in gravity level could be obtained.

3.1 46th ESA PFC

Thixo AlSi6Cu4 + 0.6wt% TiH₂ samples foamed under 1.8 g short before the microgravity period experienced an imbibition of the previously drained melt into the foam structure, cell walls and Plateau borders after reaching 0 g. This led to a well distributed foam density of $\sim 40\%$ in the whole height of the foam. In contrast, during 1.8 g the density was in the range of 20–100%.

We can see in Fig. 3, at the end of the microgravity period, how in just one second – during the transition from 0 g to 1.8 g – a large amount of liquid metal flows through the foam to the bottom of the sample. It is most prominent at the top part marked with a broken ellipse, where the curvatures of single bubbles can be observed at $t = 71$ s in opposite to $t = 70$ s, where the outer skin has a single curvature. At the bottom of the foam, the drained liquid is also visible as dark shadow in the X-ray image.

Another interesting effect is a cell wall rupture observed in Fig. 4 after 92 s leading to the coalescence of 2 bubbles during the 1.8 g phase. The result is a coarser bubble, but with almost no significant change of the arrangement of the neighbouring bubbles.

3.2. MASER 11

The selected sample was a Thixo AlSi6Cu4 + 0.6wt% TiH₂ precursor. These precursors showed the largest drainage influence during the parabolic flight and therefore they allow to study better drainage induced and related effects. Fig. 5 shows radiosopic images of the reference sample foam on Earth (left) and of the sample foamed under microgravity (right).

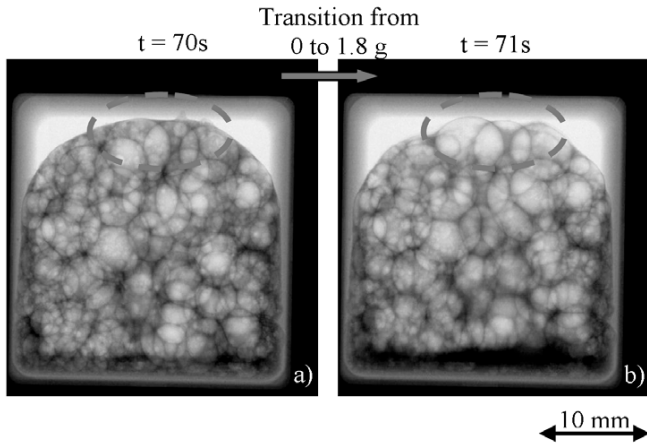


Fig. 3 : Liquid flow from top to bottom during transition from (a) 0 to (b) 1.8 g in a Thixo AlSi6Cu4 foam. Red ellipses denote the region of interest.

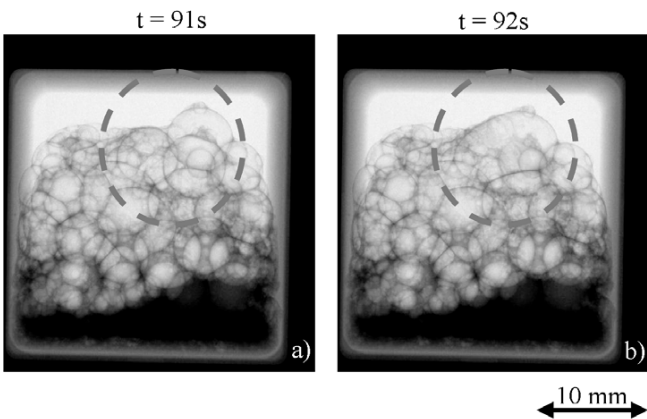


Fig. 4 : Cell wall rupture and consequent coalescence of 2 bubbles on the top of a liquid Thixo AlSi6Cu4 foam under 1.8 g at (a) $t = 91$ s and (b) $t = 92$ s. Broken circles show the region of interest.

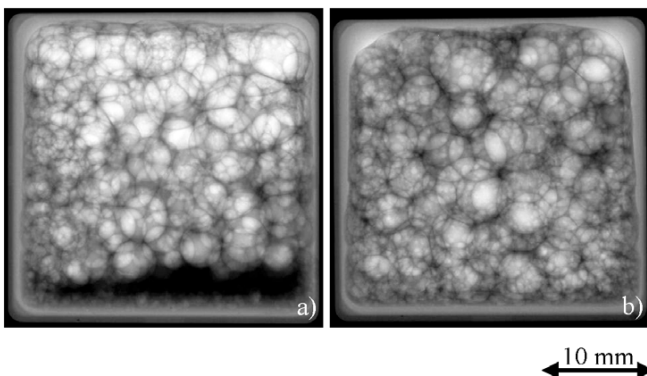


Fig. 5 : Thixo AlSi6Cu4 sample after 200 s in the liquid state foamed (a) on Earth and (b) during the MASER 11 flight.

Under microgravity a more homogeneous structure with no drainage at the bottom, round pores and no corrugated pores at the top can be achieved. Under gravity, a top part with lower density and a bottom part with almost 100% of density can be observed. Also an irregular pore structure can be seen.

But the most exciting result is shown in Fig. 6, where the spatial distribution of cell wall ruptures in the expanded foam during around 4 minutes (231 s) of microgravity and of gravity for the reference sample in the liquid state are plotted. The total number of ruptures and the coalescence rate under gravity (477 rup, 2.05 rup/s) compared to the absence of gravity (500 rup, 2.16 rup/s) could be measured. For them no significant difference could be found, although an obvious spatial redistribution due to the absence of bulk melt at the bottom is obvious. That means less cell wall ruptures can be registered in the bottom and more on the top of the sample foamed under gravity conditions.

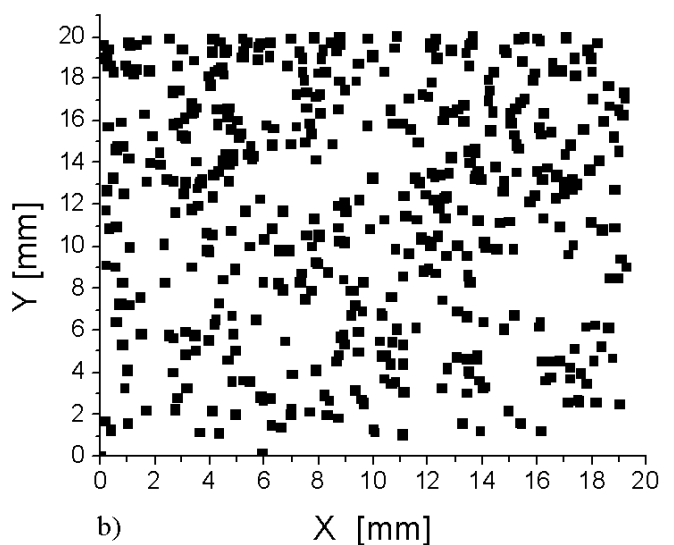
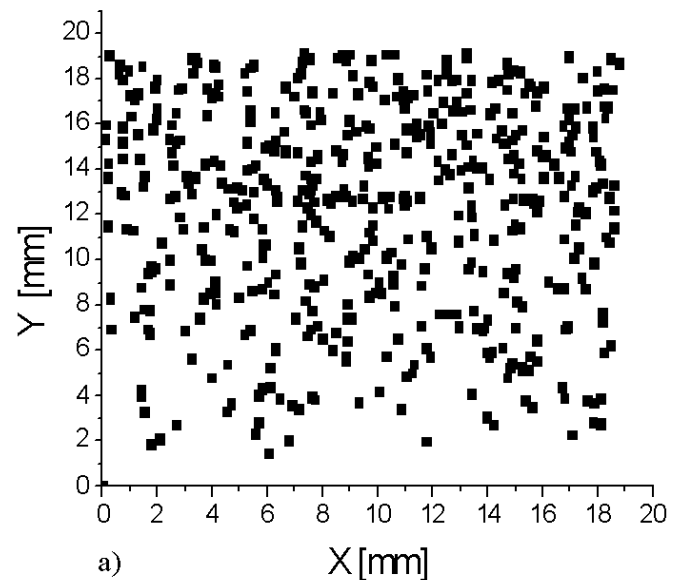


Fig. 6 : Cell wall rupture map of the Thixo AlSi₆Cu₄ foam in the liquid state during almost 4 minutes (a) under gravity and (b) during MASER 11 extracted from the sequence of X-ray images.

4. Discussion

It was a great challenge to fly an imaging X-ray equipment for the first time under microgravity conditions. Although the parabolic flight was a test for MASER 11, we could already achieve relevant scientific results. But nevertheless, for the bubble coalescence analysis, a larger number of ruptures are required to provide enough statistics. That is not the case during the around 20 s of a parabolic flight. Therefore, that was the major goal of MASER 11, with almost 6 minutes of microgravity in total.

4.1. 46th ESA PFC

The imbibition of liquid metal in the foam structure demonstrates that the capillarity forces of the cell walls and Plateau borders is higher than the non gravity driven drainage forces, namely the ones produced by the curvature of the cell walls.

The quick transitions between 0 and 1.8 g showed that the internal viscosity of the liquid metal foams should be lower than expected. This is contradictory with the idea, that cell walls are stabilised by particles or network of oxides connected to each other. In that case very high viscosities should exist. A rheology effect that plays a great role determining an effective dynamic viscosity will be the consequence.

We were able to observe cell wall ruptures by means of X-ray imaging, especially on the top of the foams. The observation could corroborate the detected events measured and counted by the software, but the short time frame of a parabolic flights experiment does not provide enough statistics for a reliable statement in comparison with ground experiments.

4.2. MASER 11

A more homogeneous foam evolution under microgravity achieving rounder bubbles was expected and found. That is a clear example of the influence of gravity on the process. A coalescence rate gradient from top to bottom could be found only for the sample made under gravity. Following, a natural redistribution of the cell wall ruptures under gravity due to the density differences is obvious. But unexpected was the fact that almost no significant reduction of the total coalescence rate was found under microgravity. That means,

there are other factors that influence coalescence essentially more than cell wall thinning due to the influence of gravity. This opens the door for other theories that should be proved and studied in future.

5. Conclusions

For the first time, we could fly a metal foam experiment with X-ray imaging diagnostics under microgravity. The parabolic flight was a successful test for MASER 11 that delivered scientific output such as the imbibition of a melt into metal foam during microgravity, rapid liquid flow during gravity transition and rupture of cell walls under microgravity. The sounding rocket experiment showed that the foam made under microgravity has rounder pores and is more homogeneous. It also showed an important, unexpected result, namely that there was not much difference between the coalescence rate under gravity and microgravity, proving that the total coalescence rate in liquid metal foams is not influenced by gravitational drainage significantly as it was supposed previously. Only bubble coarsening is influenced locally, namely by an increasing bubble rupture rate from bottom to top.

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