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2003 J. Phys.: Condens. Matter 15 S427

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Stability of metallic foams studied under microgravity

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Received 28 October 2002

Published 16 December 2002

Online at stacks.iop.org/JPhysCM/15/S427

Abstract

Metal foams are prepared by mixing a metal powder and a gas-releasing blowing agent, by densifying the mix to a dense precursor and finally foaming by melting the powder compact. The foaming process of aluminium foams is monitored *in situ* by x-ray radiography. One observes that foam evolution is accompanied by film rupture processes which lead to foam coalescence. In order to elucidate the importance of oxides for foam stability, lead foams were manufactured from lead powders having two different oxide contents. The two foam types were generated on Earth and under weightlessness during parabolic flights. The measurements show that the main function of oxide particles is to prevent coalescence, while their influence on bulk viscosity of the melt is of secondary importance.

1. Introduction

Metal foams have come to be a popular field of research owing to the technological relevance of such lightweight materials which exhibit an unprecedented spectrum of properties and also due to the interesting physics which governs the foaming process of metals [1, 2]. The processes during metal foam formation are still not very well understood. Detailed insight into the process is difficult to achieve due to the non-transparency, reactivity and high temperature of liquid metals. Series of snapshots of the ongoing foaming process can be obtained by freezing emerging foams at certain stages and by analysing the structure of the solid foam afterwards, e.g., by means of metallography, x-ray tomography or ultrasmall-angle neutron scattering [3, 4]. *In situ* observations during foam formation have been carried out using x-ray radiography and yield a wealth of new information [5], but are difficult to carry out.

Frequently, the physics of metal foaming is discussed in close analogy to what is known from aqueous foams [6]. Indeed, the phenomena during foaming are similar: *drainage* leads to a steady removal of liquid from the foam due to gravitationally induced

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flow. *Coalescence* caused by spontaneous rupture of films separating bubbles changes foam morphology discontinuously. The two phenomena occur simultaneously, which makes their investigation difficult and is the main motivation for using microgravity in this context. A closer analysis of the foaming process shows that there are differences between the foaming of aqueous liquids and that of metallic liquids, which is not surprising taking into account the differences in chemistry. In liquid metals there are no surfactants acting via electrostatic forces as there are in tenside-stabilized aqueous systems, indicating that the stabilization mechanism must be quite different. Furthermore, foaming of metals relies on the presence of finely dispersed non-metallic particles in the melt, which must play a crucial role in foam stabilization. Their action has been explained in various possible ways:

- (a) they could reduce surface tension of a melt, thus contributing to foam stability;
- (b) they could increase bulk viscosity of melts and therefore hinder flow of metal out of the foam films;
- (c) they could delay or prevent rupture of films separating bubbles.

It is the purpose of this paper to discuss the effect of these stabilizing particles by analysing microgravity experiments.

2. Preparation of metal foams

Metal foams can be produced by liberating gas in a specially prepared metallic melt. One very practical possibility is to first compact a mix of metal powders and a gas-releasing blowing agent to a dense precursor material. This precursor is then foamed in a second step by melting it, thus releasing the propelling gas within the matrix and forming a foam [7]. Two metals were considered in this paper. Aluminium alloys are technologically relevant and were included here because they can be easily penetrated by x-rays. Lead foams do not have a practical application to date but are a convenient model system because of the low melting point of lead which allows it to be foamed in the short time available in microgravity experiments. Moreover, drainage is quite pronounced in lead due to its high density.

Aluminium alloy consisting of 93 wt% Al and 7 wt% Si was foamed using 0.5 wt% TiH_2 powder as blowing agent, which releases hydrogen above about 400 °C. Lead was foamed using 2 wt% lead (II) carbonate, which is already liberating CO_2 and H_2O at about 300 °C. In both cases the powder blends were densified by extrusion at temperatures below the melting point of the metal, namely at about 450 and 280 °C, respectively. No non-metallic additives had to be added, since the metal powders were processed under normal atmosphere and therefore contained oxidized surfaces which automatically led to the presence of finely dispersed oxides in the compacted precursor. This is an advantage of using powders as starting material over other processing approaches where melts are foamed directly and which rely on the addition of quite high volume fractions of stabilizing particles [8]. Two lead powders with different properties were used to investigate the importance of the oxide content: one powder with an average particle diameter of 82 μm had a measured oxygen content of 0.16 wt% (called *type N* in the following); the other had rather coarse particles between 400 and 600 μm and an oxygen content of 0.06 wt% (*type L*). The corresponding oxide contents, assuming that only PbO is present, are 1.7 and 0.85 wt%, respectively. The aluminium powder used contained 0.7 wt% oxygen.

3. Visualization of coalescence in metallic foams

In order to gain insight into the physical processes during foaming, the formation process of AlSi_7 foam was observed by *in situ* x-ray radioscopy. For this, foams were created in a

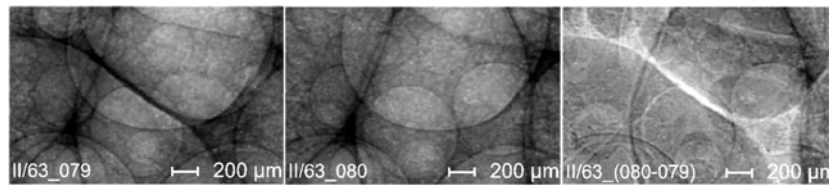


Figure 1. Rupture of a cell wall in an Al foam with 7 wt% Si. The images on the left and in the centre show two stages 500 ms apart; the image on the right shows the difference between the two images.

specially designed furnace which allowed a synchrotron-generated x-ray beam to pass through the evolving sample. Up to 18 absorption radiographs with $10\ \mu\text{m}$ spatial resolution were taken per second (for a description of the experiment, see [5]; for some movies, see [9]). The furnace temperature was set to $750\ ^\circ\text{C}$. The resulting series of images were analysed in various ways. First, in order to quantify drainage effects, the vertical density gradient as a function of time was obtained by integrating line by line over the horizontal extension of each image. Then, coalescence processes were identified by calculating difference pictures for all pairs of adjacent images and identifying visually the features which had changed. It was found that drainage was not very pronounced but that coalescence was very important especially in early stages of foaming. Figure 1 shows an example of a bursting cell wall.

Further analysis revealed that there is a critical rupture thickness of cell walls: whenever a film is stretched beyond a limit corresponding to a thickness of about $50\ \mu\text{m}$, rupture occurs and two or more bubbles join to form a bigger one [10].

4. Foam formation under varied gravitational conditions

4.1. Experimental set-up

Microgravity experiments were carried out during parabolic flights using the A300 zero-g aeroplane operated by Novespace in France. The time of relative weightlessness is 22 s in these flights, which is sufficient for creating a fully developed lead foam. The experimental set-up that is installed in a plane for parabolic flights contains two main modules: the furnace and the sample storage. The furnace chamber is equipped with a heating jacket to allow for pre-heating the atmosphere in the chamber to $250\ ^\circ\text{C}$. The samples are transferred from the storage—which can hold up to 18 samples—into the furnace chamber by means of a transfer bar, where they are deposited on the sample substrate and allowed to acquire the ambient temperature. After this, the samples are heated to the preselected foaming temperature (usually $380\ ^\circ\text{C}$) by an infrared lamp. The samples have a size of $20 \times 20 \times 1.8\ \text{mm}$ prior to foaming and expand mainly perpendicularly to the substrate. The temperatures of the sample and surrounding air are continuously monitored. In addition, the foaming process is recorded on a video tape. Foaming is initiated shortly before microgravity sets in and can be completed in about 5 s. The foam then further evolves in weightlessness but remains attached to the substrate due to some undercuts in it. Shortly before microgravity ends the samples are cooled by a stream of cold air, which is directed towards the foam.

4.2. Terrestrial reference experiments

In a first step the two lead precursor materials were foamed under $1\ g$ using the equipment described in the previous section. This was firstly done to tune the experimental settings—

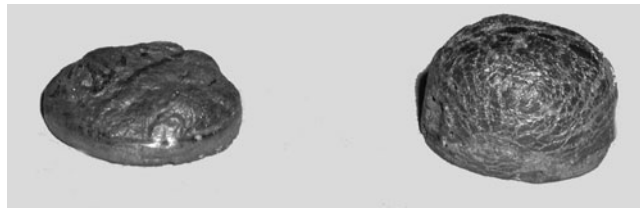


Figure 2. Comparison of foams of type L (left) and type N (right).

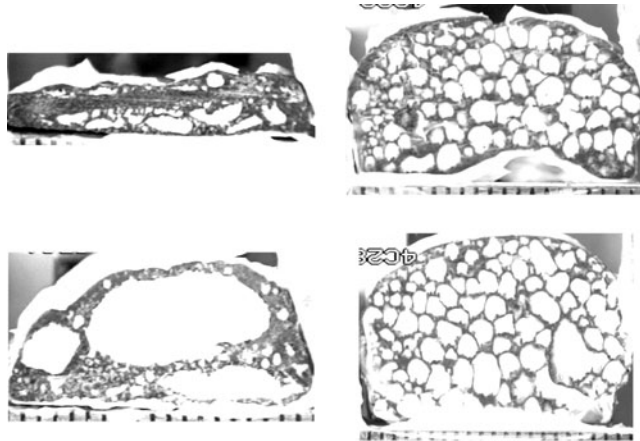


Figure 3. Sections of foams processed under normal gravity conditions (first row) and microgravity (second row). Left: type L material; right: type N material.

temperatures, times etc—but also to obtain reference samples processed under conditions equivalent to the microgravity experiments to be described later. The resulting foams as they look after solidification are shown in figure 2.

The foaming time—defined as the time from the first visible build-up of a bubble to the onset of cooling—was 30 s, the preheating at $T_0 = 250\text{ °C}$ was for 5 min, the heating rate was $\dot{T} = 2\text{ K s}^{-1}$ with a maximum temperature of 380 °C measured at the bottom of the sample with a thermocouple. Quite clearly, type L material, with less oxide content, does not evolve into a well expanded foam as material N does. Recalling the foaming process from the video recording, one observes that the evolution of material L is a sequence of expansion and collapse phases. It can be assumed that this behaviour is due to the evolution and destruction of large bubbles inside the melt. Because of drainage, the films on top of such bubbles are thinned quickly, thus leading to rupture and coalescence. In contrast, sample N expands continuously.

Additional insight is gained from sections through the solidified foams as seen in figure 3, top row. While foams of type N show a very uniform pore structure and little effect of drainage, type L foams have completely collapsed and mainly consist of solidified liquid with only a few pores conserved.

The experiments clearly show that non-metallic particles within the metallic melt are a prerequisite for a stable foam. Explanations given for this finding often address the influence of such particles on bulk viscosity of melts [5, 11, 12]. As viscosity is increased by dispersed particles, this would slow down the extraction of melt from the films by gravitational and capillary forces. In thin films the effect of particles could be even more important due to

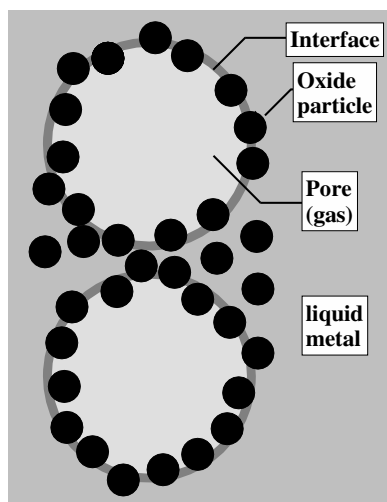


Figure 4. Non-metallic particles forming layers at liquid–gas interfaces in metallic foams (after [15]).

surface effects and a hypothetical build-up of particles near the thinnest section causing a jamming of liquid.

4.3. Microgravity experiments

If the viscosity-increasing effect of solid particles were the dominant mechanism, it should be possible to create a foam from the low-oxide material under weightlessness, because of the elimination of the driving force for drainage. We therefore foamed the same kinds of material—types N and L—once more under microgravity and observed that type N material still rose to a foam with just the final height a bit larger than under 1 *g* (figure 3, second row). Therefore the level of gravity has little effect on the stable foam. In contrast to this, sample L showed a different behaviour compared to the 1 *g* experiment: a marked expansion was observed which took place smoothly without intermediate collapse phases. A technical observation was that the sample is more sensitive to the cooling system as sample N. The section shows that type L foam is made up of a very small number of large bubbles. Such large bubbles can only survive under microgravity where there is no drainage. Therefore, in spite of the absence of gravity, no stable foam structure has developed, which shows that the effect of stabilizing particles must go beyond the viscosity effect proposed.

We suggest that, possibly in addition to the viscosity effect postulated previously, the main function of stabilizing particles is to prevent coalescence. Presumably the oxide filaments float to the surface of the liquid metal films and create layers at the liquid–gas interface. Due to the wetting conditions for oxide particles and liquid metal—probably partial wetting—the particles require a force to be totally pulled into the liquid as well as being removed from the interface. This could lead to the formation of a stable layer of particles at each gas/liquid interface (see figure 4) or a double layer of particles on each side of a foam film.

Surface films create the so-called disjoining pressure [13, 14] in thin aqueous foam films via the interaction of polar molecules. This pressure counteracts the attractive forces trying to pull the interfaces together, which alone would lead to the disappearance of films. In addition to stabilization originating from surface films of polar molecules, finely distributed

particles can also contribute to the stability of both foams and emulsions. In the so-called Pickering emulsions, solid particles present in the carrier liquid adsorb to the surfaces of the dispersed drops. A mechanically stable shell is formed in that way, which prevents droplets from merging if they get into contact. It can be observed that such emulsions are stable for months [16] against coalescence, although drainage can still be present. The same applies to foams (see e.g. [11, 17]) where particles act as surfactants. The mechanism of film stabilization is still under investigation.

We might suspect that metal foams could be stabilized in the same way. However, it is not quite clear how a disjoining pressure could be created by layers of silicon carbide particles on both sides of a metal film. Electrostatic forces which act in aqueous films have to be ruled out. Effects of the particles on film curvature have been held responsible [11], but a proof is still lacking. Finally, even the existence of such films has not yet been proved in all cases. Although particle-decorated surfaces have been observed in some solid metal foams [8, 12], foam films not covered by such particles at the surface can also be found [18]. In some cases the particles are quite uniformly distributed in the bulk of the Plateau borders without much preference for the surface. A possible explanation for this is that during solidification of the foam, the particles are dragged inwards, so the solid foam does not reflect the configuration found in the liquid. One should therefore attempt to look at liquid metal films *in situ* which, however, is quite a challenge.

5. Summary

The paper discusses the role of solid particles for the stability of liquid metallic foams. It was found that a certain oxide content is required for metal foam to be stable. In a microgravity environment, however, a notable foam expansion is observed also for the material known to be unstable in the presence of gravity, but coalescence creates very coarse bubbles. From the different structures of the solidified samples, we conclude that oxide particles not only hinder drainage by increasing the viscosity of the melt, but also suppress the coalescence of pores, possibly by generating a disjoining force analogous to that in aqueous foams.

Acknowledgments

We thank the German space agency DLR (grant 50 WM 0126) and the German DFG (grant Ba 1170/3-2) for funding this project and the European space agency ESA for providing microgravity flight opportunities.

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