

Metal foam evolution studied by synchrotron radioscapy

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(Received 20 October 2000; accepted for publication 18 December 2000)

High-intensity synchrotron x-ray radioscapy was used to obtain real-time images of foaming metals, thus allowing the formation, growth, and decay of such systems to be studied. Bubble generation, foam coalescence and drainage of an aluminum-based alloy foam were investigated. Although the foaming process appears to be very similar to the formation of aqueous foams, the observed rupture behavior of thin metal films suggests that the processes responsible for metal foam stabilization and destabilization must be quite different. © 2001 American Institute of Physics.

[DOI: 10.1063/1.1350422]

Foams are liquid–gas mixtures that evolve in a complex way governed by an interplay of viscosity, surface tension, and gravity. The physics of foams is still a disputed area and, e.g., drainage^{1,2} or foam flow phenomena^{3,4} are just being explored. Foams can be made from a variety of substances such as water, polymers, glass, or even metals. Solid metallic foams have attracted much attention because of their potential applications, e.g., in automotive and aerospace industries, where their specific structure makes them useful for lightweight construction or crash energy absorption.^{5–7} Although metal foams are rapidly becoming industrially important, surprisingly little is known about the physical processes governing their formation. Further improvement of such foams, however, requires more information about their liquid counterpart, which is difficult to obtain owing to the intransparency, reactivity, high temperature and low electrical resistivity of metal melts. Observation methods which are useful for studying aqueous foams such as, e.g., resistance, fluorescence, or light scattering measurements are therefore not applicable. In contrast, using synchrotron radioscapy an unprecedented insight into evolving metal foams could be gained.

Aluminum–silicon (AlSi7) alloys were foamed by releasing hydrogen gas in metal powder compacts.⁸ For this a metal powder blend Al+7 wt% Si (purity 99.5%, –100 mesh) was mixed with 0.6 wt% of powdered titanium hydride (TiH₂) after which the mix was hot pressed at 450 °C and 120 MPa. The resulting tablets were virtually dense (<0.75% porosity). Foaming of the alloy was triggered by heating tablets to the melting range of AlSi7 (577–620 °C) in normal atmosphere. This caused partial melting of the alloy, hydrogen release by the blowing agent, and formation and inflation of bubbles and a corresponding volume expansion to 5–10 times of the original volume. Metal foams are in many respects similar to aqueous foams: they are arrangements of films intersecting in edges—the Plateau borders—

which themselves meet in nodes. However, some differences are also apparent: there is a lower limit for the liquid fraction of about 10% which makes metal foams rather “wet” compared to “dry” aqueous foams that can have less than 1% liquid fraction.⁹

In order to observe the evolution of the internal structure of metal foams in real time, foams were generated in a furnace which was equipped with two water-cooled Al windows through which a synchrotron x-ray beam could pass (see Fig. 1). The beam, monochromatized to 33.17 keV, generated an absorption radiograph which was captured with an electronic detector system based on a 1024×1024 pixel CCD camera with 40 μm pixel size.¹⁰ The CCD camera was read out at frequencies between 2 and 3 Hz. The entire foaming experiment took a few minutes, corresponding to 500–900 radiographs for each of the 60 individual experiments carried out. Synchrotron beams had to be used because of their high intensity and very low divergence. Conventional microfocus x-ray tubes have been used for real time observations of liquid metals,^{11,12} but they do not allow for obtaining sufficiently sharp images of the thin structures or for exposure times well below 1 s.

The radioscopic images showed a great wealth of details of the foaming samples in various stages of evolution. In particular, if the samples were thin in the direction of the ray, the internal structure would be resolved very well. Phenom-

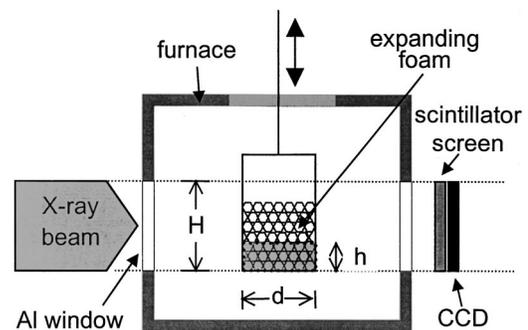


FIG. 1. Experimental setup for real-time radioscapy. The beam is $H = 15$ mm high and 40 mm wide, the samples were between $h = 4$ and 7 mm high before foaming, their thickness ranged from $d = 6$ to 20 mm.

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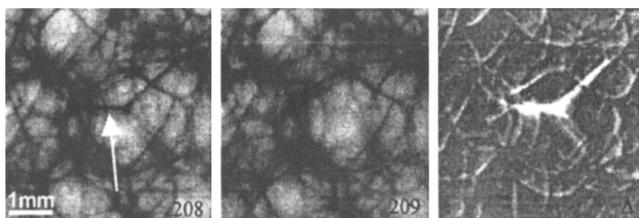


FIG. 2. Cell rupture observed in an AlSi7 foam. The two successive radiographs were taken from a series of 800 and show a closeup of the original total image area of $30 \times 15 \text{ mm}^2$ (left and middle). Time separation between the images is 500 ms. The structure that ruptures is marked by an arrow and is made visible in the difference picture (right); foaming temperature was 700°C . The sample was $d=6.5 \text{ mm}$ thick which leads to an initial x-ray transmission of 21.3%.

ena such as bubble creation and growth, rupture of films, local topological rearrangements, etc. can be studied. Figure 2 gives an impression of the possibilities, showing two close-up images of a fully expanded AlSi7 foam (15% liquid fraction) where $d=6.5 \text{ mm}$, corresponding to two to three bubble layers in the direction of the beam. Figure 3 shows six different full-field radiographs of another expanding AlSi7 foam in different stages of evolution ($d=10 \text{ mm}$). The various evolution steps can be divided into two phases: (I) the first four frames show a sample rising from its initial thickness to maximum height, (II) the last three frames a foam with an almost constant volume in which internal processes lead to changes in the bubble structure.

Looking at phase (II) one sees that the foam structure gets *coarser* with time and there is a slight tendency for metal flowing downwards under the influence of gravity. However, this *drainage* effect is rather weak: the vertical density profile remains fairly constant with time and most of the observed material rearrangement is associated with rupture phenomena which lead to short pulses of metal flow. Only an increase of temperature to at least 750°C in further experiments led to considerable and more continuous drainage. Analysis of the entire movie reveals that spontaneous cell rupture is the sole mechanism for coarsening and that there is no sign for gradual gas diffusion between the cells. The cell walls rupture in less than 500 ms. One rupture process is seen in Fig. 2: the left frame shows a plateau border and corresponding films which are about 2 pixels ($80 \mu\text{m}$) thick. They have disappeared in the second frame 500 ms later, thus demonstrating that aluminum films cannot be stretched beyond this limit but become unstable. This is very different from the behavior of most aqueous films.

The irrelevance of diffusion becomes understandable if one roughly estimates the time for diffusion of hydrogen through a film of $80 \mu\text{m}$ thickness: using a diffusion constant of $D=3.2 \times 10^{-3} \text{ cm}^2/\text{s}$ for H_2 in Al,¹³ and a surface tension of $\gamma=0.91 \text{ N/m}^{14}$ —both at 660°C —one finds that bubbles with, e.g., a radius of $R=3 \text{ mm}$ have excessive pressures of $2\gamma/R \approx 600 \text{ Pa}$. A typical pressure difference between two adjacent bubbles of, e.g., $\Delta p=100 \text{ Pa}$ would give rise to a gas flow of $j=5 \text{ nmol cm}^{-2} \text{ s}^{-1}$ which would empty the bubble only after more than 1 h.

The apparent difference between metal foams and aqueous foams requires some explanation. In aqueous foams the films are stabilized by surfactant molecules which cover their surface and form a monolayer of oriented dipoles. Films can

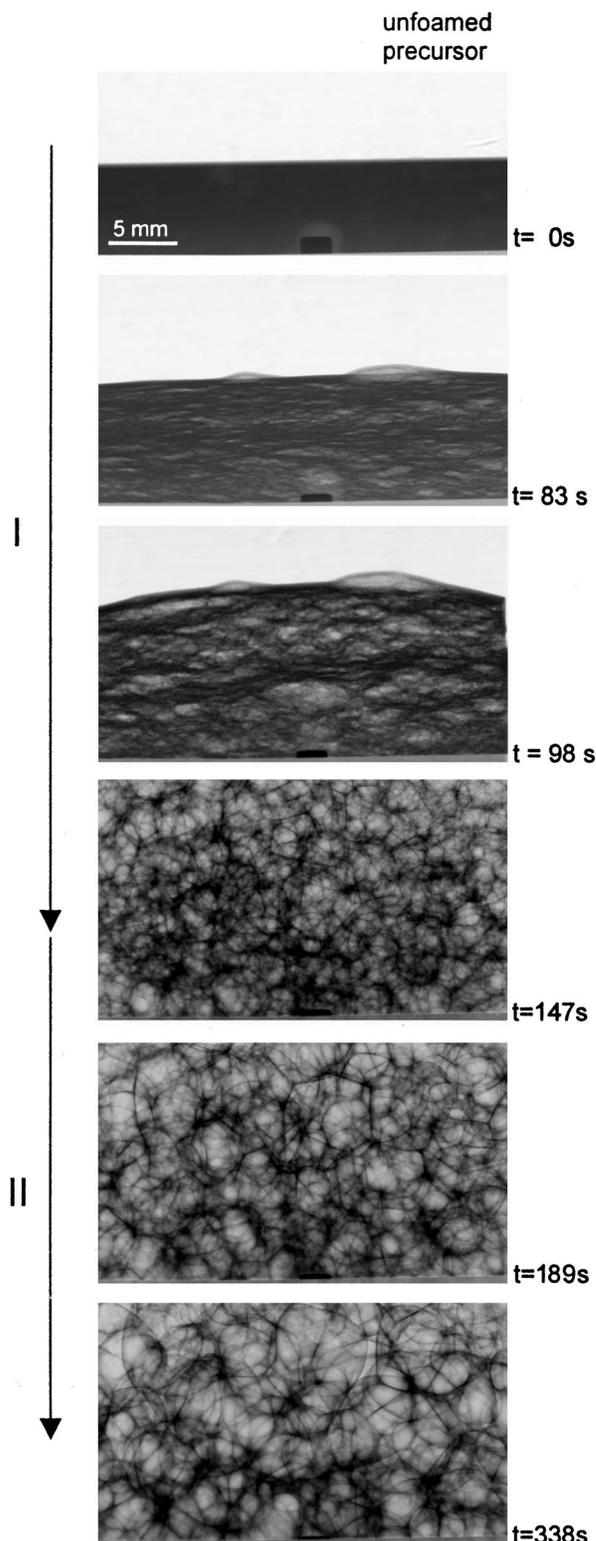


FIG. 3. Series of expansion stages of an AlSi7 alloy monitored by x-ray radioscopy. The original sample (frame 1) was $d=10 \text{ mm}$ thick (x-ray transmission of 9.1%). In frames 4–6 the foam has risen to more than 15 mm height and is therefore truncated. I marks phases of rising foam, II stages of decaying foam at nearly constant volume. The times given refer to a state at which expansion started which was about 120 s after inserting the sample into the furnace preheated to 700°C .

get as thin as 10–30 nm as liquid drains out under the influence of gravity. Thin films are stabilized by electrostatic forces between the two dipole layers and the Gibbs–Marangoni effect.^{9,15} As thin films allow gas to diffuse across them, small bubbles with a higher internal pressure

are prone to disappear. Liquid metal films, in contrast, are stabilized differently because the electrostatic forces are screened. It is widely accepted that a metal can only be foamed if fine solid particles are suspended in the melt. Such particles can be either added to the melt before foaming,^{16,17} be formed in the melt by *in situ* oxidation¹⁸ or, as in the foams described in this letter, be either a solid component of the particular alloy while melting (Al-rich phase in the Al–Si eutectic), or metallic oxides which permanently reside in the foaming material and that are remnants of the former thin (≈ 30 nm) oxide layers on the aluminum powders. An oxide content of 0.7 wt % was measured.⁸ The way solid particles act is still in dispute. In direct analogy to aqueous foams it was suggested that solid particles are only partially wetted and cover the surface thus increasing surface viscosity.^{16,17} Moreover, as suspended solid particles increase the apparent viscosity of a melt, a bulk effect is usually presumed.^{16,17}

The real-time observations of metal foams of this work allow to assess these pictures in various ways. First, the visibly weak drainage at moderate temperatures shows that gravity-induced flow is indeed very limited as long as temperatures do not get into a range where the solid phase is dissolved and fluidity increases too much. The solid particles can be thought to build up mechanical barriers in the thin films and hinder flow. Thus, the postulated bulk viscosity effect is certainly of paramount importance. Second, however, the sudden rupture of films at a critical thickness of about 50–80 μm suggests that particles can also be destabilizing rather than maintaining the film, 60 μm is the approximate size of the largest Al-rich precipitates in the melt as seen from micrographs of solidified foams.⁸ Prolonged stretching and corresponding thinning of metal films below 50–80 μm can therefore lead to a situation where the particles extend over the entire film diameter. Instead of wetting and bridging the particles the liquid can recede from them if the wetting conditions between particles and melt are unfavorable (“pinchoff effect”). Collapse of the entire film follows. Such effects are known from emulsions and aqueous foams^{15,19} and can be used to deliberately destabilize foams, e.g., in oil recovery.²⁰ Last, observing cell walls near the sample surface shortly after rupture revealed that repeated remnants of cell walls remained visible for some seconds as small arcs which were then slowly absorbed by the remaining liquid. This shows that one further effect is important: as the moving melt is prone to oxidation, thin but very rigid alumina skins are formed on the surface of the very reactive metal, thus leading to a high apparent viscosity which helps

to stabilize the films. Therefore, there might be a surface effect which, however, has no analogy in aqueous foams.

Turning to the first four images in Fig. 3 one sees that foam creation and further expansion is also rather different from what one would expect from an analogy with aqueous foams: the evolving hydrogen gas causes the formation of highly oblate cracklike voids rather than nucleating into small round bubbles. These voids round off as the metal expands and finally become fairly equiaxed after about 120 s. The direction of foam rise is always parallel to the short axis of the voids which in turn is parallel to the pressing direction of the powder mix. Therefore, the texture created during pressing of the powders must be responsible for this anisotropy. This implies that the very first pore formation already takes place in the solid state. Development of a blowing agent with a higher decomposition temperature could avoid this anisotropy.

This work was partially funded by Deutsche Forschungsgemeinschaft (DFG) within Priority Program 1075 and supported by European Synchrotron Radiation Facility (ESRF, beamline ID19).

¹D. Weaire, N. Pittet, and S. Hutzler, *Phys. Rev. Lett.* **71**, 2670 (1993).

²S. A. Koehler, S. Hilgenfeld, and H. Stone, *Phys. Rev. Lett.* **82**, 4232 (1999).

³D. J. Durian, *Phys. Rev. Lett.* **75**, 4780 (1995).

⁴A. Kraynik, *Annu. Rev. Fluid Mech.* **20**, 325 (1988).

⁵J. Banhart, *Prog. Mater. Sci.* **47**, 2001 (2000).

⁶L. J. Gibson and M. F. Ashby, *Cellular Solids* (Cambridge University Press, Cambridge, 1997).

⁷M. F. Ashby, A. G. Evans, N. A. Fleck, L. J. Gibson, J. W. Hutchinson, and H. N. G. Wadley, *Metal Foams-A Design Guide* (Butterworth-Heinemann, Boston, 2000).

⁸I. Duarte and J. Banhart, *Acta Mater.* **48**, 2349 (2000).

⁹D. Weaire and S. Hutzler, *The Physics of Foams* (Clarendon, Oxford, 1999).

¹⁰As the samples were smaller than the detector area, the actual images covered only about 800×300 pixels.

¹¹J. N. Koster, T. Seidel, and R. Derebail, *J. Fluid Mech.* **343**, 29 (1997).

¹²P. D. Lee and J. D. Han, *Acta Mater.* **45**, 4155 (1997).

¹³G. Altenpohl, G. Ibe, and H. Wittig, in *Gases and Carbon in Metals*, edited by E. Fromm and E. Gebhardt (Springer, Berlin, 1986).

¹⁴T. Iida and R. I. L. Guthrie, *The Physical Properties of Liquid Metals* (Clarendon, Oxford, 1988).

¹⁵D. Exerowa and P. M. Kruglyakov, *Foam and Foam Films* (Elsevier, Amsterdam, 1998).

¹⁶S. W. Ip, Y. Wang, and J. M. Toguri, *Can. Metall. Q.* **38**, 81 (1999).

¹⁷V. Gergely and T. W. Clyne, *Adv. Eng. Mater.* **2**, 175 (2000).

¹⁸L. Q. Ma and Z. Song, *Scr. Mater.* **39**, 1523 (1998).

¹⁹P. B. Binks and S. O. Lumsdon, *Langmuir* **16**, 2539 (2000).

²⁰V. Bergeron, in *Proceedings of the International Workshop on Foams and Films, Leuven Belgium*, edited by D. Weaire and J. Banhart (MIT, Bremen, 1999).