

Aluminium Based Metallic Films

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ABSTRACT

The present paper deals with a novel experimental technique to study liquid metal foam stability. This method is based on making single films from liquid Al and Al alloys with and without stabilizing particles and to show how foams stability could be studied on a simplified system such as a single film instead of having to deal with a multitude of different structural elements present in a foam. It is found that merely weakly stable films can be made from commercially pure Al and no stable films from Al-Si alloys which do not contain any particles. In contrast, thin stable films without rupture could be made from aluminium alloy containing particles such as SiC and TiB₂. Significant thinning of film is observed when the particle concentration decreases in the melt.

INTRODUCTION

Metal foams are processed using a variety of methods. The liquid processing routes for making closed cell metal foams are popular and widely used because they are economically attractive and a wide range of metals and alloys can be foamed. Phenomena such as liquid drainage and melt distribution associated with these processes are major issues during bulk foam production [1]. It is essential to have a clear scientific understanding of the mechanism of aluminium foam stabilisation in the liquid state in order to make the production technology more reliable and reproducible. The term 'foam stability' is related to the avoidance of cell wall rupture resulting mainly from foam perturbation and drainage. To produce stable liquid-metal foam by gas injection into the melt a minimum amount of solid particles is necessary [2]. During foaming process variables such as particle size, particle fraction, and foaming temperature determine cell size and cell wall thickness [3]. The particle content in the liquid metal increases both melt viscosity, slowing down

the vertical motion of liquid, and the surface properties of film/gas interfaces as particles segregate there [4]. Ceramic particles such as SiC, Al₂O₃, MgO, and TiB₂ are mainly used to increase the stability of liquid aluminium foam. However, no systematic study has been carried out to understand the physics of foam stability in the field of metal foams. Numerous experimental studies on the phenomenon of foam stability in aqueous foams are available in the literature, see. e.g. Refs. 5,6. Due to the complexity of foam systems most attempts to study the fundamental mechanisms of aqueous foaming have been carried out using simple model experiments [7]. One of these simple experiments is vertically pulling a single soap film from a liquid bath using a wire frame which is used to mimic the basic cell wall in a foam and hence serves to study drainage and stability of liquid foams. From the literature available about such model studies of aqueous films it is clear that the same factors which play a role in foam stability such as film thickness, elasticity, etc., also appear to have a decisive influence on the stability of isolated soap films [6]. It is known that films of liquid metal cannot be stretched to the extent of aqueous films. Rather, metallic films and liquid-metal cell walls usually rupture below the thickness of 30-150 μm [4]. It was found that cell wall thicknesses in solidified aluminium foams depend on particle size [3] and the composition of the alloy [4,8,9]. Systematic studies are still required to clarify the effect of particles on the cell wall thickness of single films. The present study is undertaken in an effort to understand foam stability and related phenomena using single films pulled from liquid metal or alloy. This attempt mainly focuses on metallographic studies of metal film cross sections, in the presence and absence of the stabilizing particles.

EXPERIMENTAL

Metal films are made by pulling circular and square wire frames from a liquid metal maintained at constant temperature. For this purpose, commercially-pure aluminium, AlSi9.5, AlSi9.5/SiC composite (containing 20 vol.% SiC of mean size 10 μm - supplied as 'F3S20S' by Alcan, Canada), and Al-6 vol.% TiB₂ composites are chosen for study. Here 'AlSi9.5/SiC/20p' means: 20 vol.% SiC particles ('p') in the alloy Al-9.5wt.% Si. For each experiment, 150 g of the precursor was melted in an alumina crucible using an electrical resistance furnace in an open atmosphere. After the melt had reached the desired temperature, a stainless steel wire frame, wire thickness 0.2 or 0.4 mm, was inserted into the skimmed liquid metal. After holding the wire frame inside the liquid for some time to achieve equilibrium in temperature, the frame was gently pulled out from the liquid and allowed to solidify. The film pulling was done manually, and the pulling velocity was kept very low, to achieve thin and stable films. Films were also made from composites having lower volume fractions of SiC and TiB₂ particles, made by dilution of the standard composites. For consistency, an average of 10 films was pulled for each experimental condition. The films produced were sliced vertically through the cross section for metallographic analysis. The surfaces of the films were also examined using scanning electron microscopy.

RESULTS and DISCUSSION

Films of Al and AlSi9.5 without particles

Fig. 1 shows a photograph of typical Al films made from commercially-pure Al using both circular and square wire frames. The stainless steel wire frame size is of 10 or 20 mm diameter in the circular case, and 10 or 20 mm² in the square case. The pure Al liquid bath was kept at 700°C and several thin films were pulled at this temperature. The pure Al films obtained after solidification appeared to be stable and possess uniform thickness throughout the frame area. However, hairline ruptures and transparent regions due to stretching of the film were observed at the film surfaces which probably developed during solidification. The surface of one such pure Al film reveals transparent regions appearing like stretch marks formed during thinning. These transparent regions take the appearance of distinct fibres or columns, arranged parallel to the pulling direction. In some places, these fibres are found to be broken due to excessive stretching. Although the melt does not contain particles the surface oxide of the Al melt, which forms as a skin over the surface of the film during withdrawal of the frame, must stabilize the pure Al films.

Fig. 2 shows the top part of the film cross section, including the node, Plateau border and the film. Subsequent thinning of film is observed from the top

towards the centre. The direction in which the film was pulled is indicated in the micrographs. The film thickness measured in this case is 12 μm at the top and 4 μm at the centre of the film. The bottom of the film has a thicker Plateau border, in comparison to the top. This can be attributed to drainage of liquid metal during film pulling. Thinning of films could take place due to drainage of liquid Al between the oxide skins on the film surfaces. Due to the absence of particles, liquid drains in excess, causing maximum thinning of film and leading to rupture or cracking before or during solidification. Various temperatures between 650 °C and 700 °C were tried for film pulling from the alloy AlSi9.5 without particles, but in no case a stable film could be made. This could be attributed to poor melt surface oxide, which make Al-Si film pulling impossible in the absence of particles. Moreover, the fluidity of the near eutectic AlSi9.5 melt may be too high and facilitate drainage during pulling.

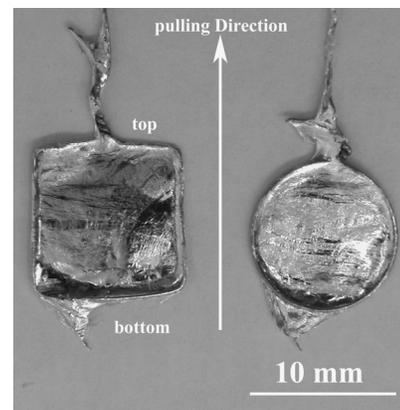


Figure 1. Image of metallic films pulled from pure Al with square and circular wire frame.

AlSi9.5 alloy films with SiC additions

AlSi9.5/SiC/20p composite films have been made using circular wire frames of 20 mm diameter. The melt in the furnace was maintained at 650°C during film pulling. Thick stable and uniform films could be made without rupture. The surfaces of AlSi9.5/SiC composite films appeared rough, unlike the surface of pure Al films. Wrinkles and small elevations are also observed on the surface of the film. Wrinkles observed in the film surfaces reflect an oxide layer, and the surface elevations or dimples in the film are due to SiC particle agglomeration before solidification. The bottom regions of the films appear to be thicker near the frames, in comparison to the top parts. This can again be attributed to drainage of liquid which accumulates at the bottom of the frame and solidifies there. The Plateau border contains a large number of SiC particles, uniformly distributed in the Al-Si matrix. It is sometimes observed that the SiC particles are arranged in the film such that two or three SiC particles form a column which connect the opposite film surfaces. However, it

seems that the number of particles present in the alloy and their size determines the thickness of the film. The concentration of SiC particles (20 vol.%) in our composites was decreased to 15, 10 and 5 vol.% by dilution with AlSi9.5. The size of the stainless steel wire frames was 20 mm, and the diameter of the wire was 0.2 mm. Stable and thin films, without rupture, were produced from the composite melts containing 20, 15 and 10% of SiC particles. However, film making was difficult with only 5 vol. % of SiC particles. Figs. 3a–c shows optical micrographs of the cross sections of composite films of different volume fraction (20, 15, and 10 vol.%) of SiC particles. The cross-sectional thickness measured in the case of AlSi9.5/SiC composite film (containing 20 vol.% SiC) ranged from 21 μm to 45 μm except for the top and bottom Plateau borders. This could be attributed to a large number of SiC particles present in the film. Decreasing the SiC particle concentration to 15% caused a significant decrease in film thickness, to 13–24 μm . It is interesting to note that the film produced from the 10% SiC composite is the thinnest among all SiC particle concentration studied, with minimum and maximum film thicknesses of 10 μm and 23 μm , respectively. Unlike in the 20 and 15% SiC composites, only single SiC particles are found between the film surfaces, through the entire cross section of the 10% composite film (Fig.3c).

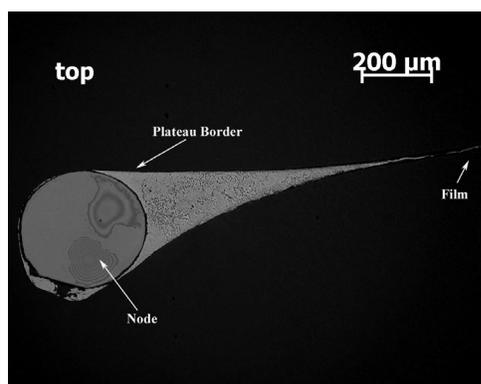


Figure 2. Optical micrograph of a pure Al film cross section depicting the top part of the film, showing node, Plateau border curvature and a film.

Single films could be made successfully from AlSi9.5/SiC with different particle volume fractions. Particle accumulation in a foam cell wall plays a key role in the stabilization process by increasing either bulk or surface viscosity of the liquid metal and thus retarding film drainage. Secondly, the particles are partially wetted by the melt and therefore preferentially go to the metal/gas interfaces and give rise to surface forces parallel to the surface [10]. The oxide films on top of the surfaces which may also increase stability cannot be overruled. It is clear from the results obtained that the SiC volume fraction plays a major role on the thickness of the film and its stability. Thin, stable and smooth films

without much surface relief are obtained by decreasing the volume fraction of SiC particles from 20 vol.% to 15, 10 and 5 vol.%. This could be attributed to a decrease in melt viscosity due to the decrease in particle concentration which facilitates the liquid flow between the film surfaces, but also to different surface properties of the films which are less densely populated with particles at the metal/gas interface. In addition to the absolute value of thickness, a better uniformity in film thickness is observed when decreasing the volume fraction of the SiC particles. Thinning of the film below the size of the SiC particles (10 μm) tends to rupture, suggesting the influence of particle size as well. Single particles would bridge the entire film in such cases thus leading to puncture and rupture.

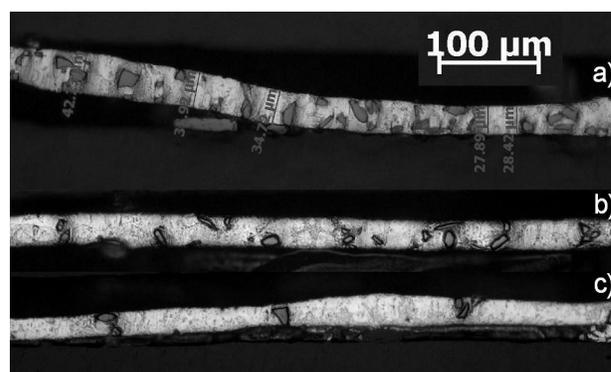


Figure 3. Optical micrograph of the cross-section of AlSi9/SiC composite films containing (a) 20 (b) 15, (c) 10 vol.% of SiC particles. Ring diameter 20 mm, stainless steel wire, 0.2 mm thick.

Al films with TiB₂ additions

Al/TiB₂/6p in-situ metal matrix composite prepared in the authors' laboratory was used for making single films. The composite contains submicrometre-size TiB₂ particles, ranging from 0.5 to 2 μm , in a pure Al matrix. For making films, the molten Al/TiB₂/6p composite was kept at 675°C. This temperature was chosen because no stable film could be made at higher temperatures. Stable, smooth and thin films could be pulled from this melt without rupture. The TiB₂ particles form networks or clusters due to their small size and high concentration in the Al matrix. These clusters seem to stabilize the film rather than individual particles. The clusters are found to segregate to the grain boundaries. In order to investigate the influence of TiB₂ particle concentration on film thickness, single films were made from composites made by diluting with particle-free alloy, leading to Al/TiB₂/3p, Al/TiB₂/1.5p and Al/TiB₂/0.6p composites. Stable films without rupture could be made even from the lowest concentration (0.6 vol. %) of TiB₂ in Al. The surfaces of the Al/TiB₂/0.6p and Al/TiB₂/1.5p films were observed to be smooth, without elevations or surface roughness, in comparison to the surfaces of Al/TiB₂/3p films. Al/TiB₂/0.6p is the thinnest film

measured among the three volume fractions studied, as shown by the polished sections in Fig. 5. The minimum film thicknesses measured were 30 μm for 6%, 37 μm for 3 %, 24 μm for 1.5 %, and 16 μm for the 0.6 % composite. The thickness variation was largest for 6% TiB_2 and therefore the corresponding value for the minimum thickness is less significant. A panoramic view of the metallographic cross sections (Fig.4) confirms that the thickness of the Al/ TiB_2 /0.6p film is uniform throughout the cross section, and its lower thickness is highly significant in comparison to the Al/ TiB_2 /3p film. From this observation it is clear that the viscosity of the melt plays a major role on thinning of the film. At higher concentrations (3 vol.% TiB_2), the viscosity of the melt is high and thus the frame catches more liquid. Therefore, there is no sufficient time for the liquid to flow out before solidification. This leads to the formation of films with non-uniform thickness. In contrast, lower concentrations (0.6 vol.%) of TiB_2 particles in the melt decrease the melt viscosity and increase liquid flow between the two film surfaces, causing film thinning before solidification. It should be noted that film thinning is restricted to the particle or cluster size, beyond which the film ruptures.

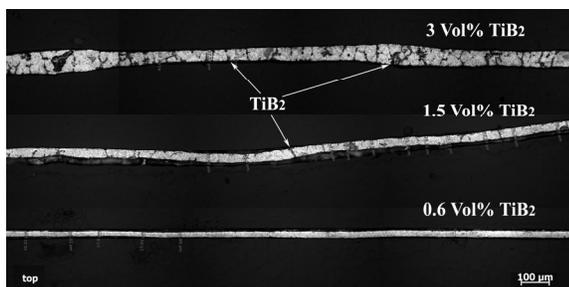


Figure 4. Optical micrograph of the cross-section of Al/ TiB_2 composite film at different volume contents of SiC particles. Ring diameter 20 mm, stainless steel wire, 0.2 mm thick.

SUMMARY and CONCLUSIONS

- Pure aluminium single films could be made, but they were very thin and fragile. The surface oxides cover the film and stabilize it partially, even in the absence of particles. No film could be made from Al-Si alloy without stabilizing particles.
- AlSi9.5/ SiC composite containing 20 vol.% SiC of 10 μm size gives a thick stable film. Reduction in SiC particle concentration leads to thinner films. This can be attributed to a decrease in melt viscosity which increases liquid drainage out of the film prior to solidification.
- AlSi9.5/ SiC composite containing 5 vol.% SiC does not lead to stable films due to a lack of particles.
- SiC particles between the film surfaces are uniformly distributed. This plays a key role in stabilizing the film.

- Although TiB_2 particles are known to have poor wetting with Al, stable, smooth and thin films could be made from Al-6 vol.% TiB_2 composites made by in-situ reaction synthesis. Reduction in the particle concentration to 3, 1.5 and 0.6 vol.% resulted in stable and uniform thin films. Along with the melt viscosity, a decrease in TiB_2 cluster size is believed to be responsible for this change.
- Al-0.6 vol.% TiB_2 particles can give stable, thin, and smooth films. The reduction in thickness obtained in the case of Al-0.6 vol.% TiB_2 , in comparison to Al-3 vol.% TiB_2 films, is highly significant.

REFERENCES

1. V. Gergely, T.W. Clyne, *Drainage in standing liquid metal foams: modelling and experimental observation*, Acta Mater. (2004) 52:3047–3058
2. S.W. Ip, Y. Wang and J.M. Toguri, *Aluminium foam stabilisation by solid particles*, Can. Met. Quart. (1999) 38:81–92
3. D. Wang, Z. Shi, *Effect of ceramic particles on cell size and wall thickness of aluminium foam*, Mat. Sci. Eng. (2003) A361:45–49
4. J. Banhart, *Metal Foams: Production and Stability*, Adv. Eng. Mater., 8:781–794
5. G. Verbist, D. Weaire, A.M. Kraynik, *The foam drainage equation*. J. Phys: Condens.Matter (1996) 8:3715–3731.
6. V. Carrier, S. Hutzler, D. Weaire, *Drainage of foam with regularly spaced parallel soap films*, Colloids and Surfaces A: Physiochem. Eng. Aspects (in press)
7. R.J. Pugh, *Foaming, Foam films, antifoaming and defoaming*, Adv. Coll. Interface Sci. (1996) 64:67–142
8. B. Kriszt, P. Cekan, K. Faure, *Foamability of the Al-Si system*, In: Cellular Metals and Metal Foaming Technology (Ed.: J. Banhart, M.F. Ashby, N.A. Fleck), Verlag MIT, Bremen (2001) 77–82
9. A. E. Markaki, T.W. Clyne, *Characterisation of impact response of metallic foam / ceramic laminates*, Mat. Sci. Tech. (2000) 16:785–791.
10. A. Haibel, A. Rack, J. Banhart, *Why are metal foams stable?* Appl. Phys. Lett. (2006) 89: 154102/1-3