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The effect of preform processing on replicated aluminium foam structure and mechanical properties

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Abstract

In replication processing of open-pore aluminium foams, the relative density can be varied by densifying the NaCl preform before infiltration. This can be done either by cold pressing or by sintering; we compare the two processes, show that the former yields superior foam modulus and flow stress, and explain why.

Keywords: Aluminium; Foams; Mechanical properties; Cold isostatic pressing (CIP); Infiltration

1. Introduction

The properties of foamed and highly porous metals are of interest both for practical applications and for the study of the fundamental behaviour of the class of materials as a whole. Many methods exist for the production of metallic foams [1,2]. Amongst these the replication process offers a simple and versatile way to produce open-cell foams, also called metal sponges [3]. The process, the basis of which was developed in the 1960s [4], uses a leachable preform (NaCl particles work well for aluminium), into which a molten material is infiltrated under argon gas pressure and solidified, before leaching of the preform to leave an open-celled structure.

This technique has been shown to permit the manufacture of pure aluminium and alloy foams [5], and also foams from a variety of other materials (see e.g. Refs. [6,7]), and to offer good control over the cell size [8–10] and cell shape [10,11]. The foam relative density can also be varied [9,12]; one approach to this end is to vary the preform density and so, in the inverse sense, the density of the foam. This, in turn, can be achieved either by cold isostatic pressing (CIP) of salt particles in flexible silicone rubber moulds [9], or by sintering the salt particles in air prior to infiltration [12,13].

We present here a comparison of the two processing routes. Replicated open-pore pure aluminium foams with pores 400 or 75 μm in average diameter produced from preforms that were predensified using either of these two processes are characterized for their structure and their uniaxial mechanical properties. One process yields less regular, yet stiffer and stronger, foams than the other; physical reasoning gives a simple reason for this somewhat counter-intuitive result.

2. Experimental

NaCl preforms were made using either sintering or cold isostatic pressing. Two NaCl particle types were used: (i) particles of mean diameter 400 μm (>99.5% pure, supplied by Fluka Chemie GmbH, Buchs, Switzerland) and (ii)

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ground particles (CP1 grade, >98% pure, supplied by Salines de Bex, Bex, Switzerland) sieved between 63 μm and 90 μm grade sieves (ISO 565) to obtain a mean size of 75 μm. Preforms of unpressed close-packed particles were densified either by sintering or by cold isostatic pressing.

Sintering was conducted in graphite-coated alumina crucibles under a flowing argon atmosphere at 755 °C. The sintering time was varied between 2 and 76 h to vary the preform relative density.

For cold isostatic pressing, the particles were poured into a flexible silicone rubber mould and vibrated to ensure good filling. The mould was cold isostatically pressed at various pressures from 5 to 60 MPa, increasing the pressure at 0.5 MPa s^{-1}, holding at the maximum pressure for 60 s and lowering the pressure at the same rate, to give preforms with a range of densities.

After densification, both preform types were used for the manufacture of aluminium (99.99%) foams, following the replication method described in Ref. [3]. In all samples of this work infiltration was carried out at 710 °C under an argon pressure of 8 MPa (80 bar).

Cylindrical samples of diameter and height 20 mm were machined and tested in compression using a screw-driven testing machine with a constant cross-head speed of 0.005 mm s^{-1}. As these foams do not display a curve with initial purely elastic deformation, a series of load–unload cycles were conducted near a nominal strain of 0.2%, the Young's modulus being found from the gradient of the straight lines of this part of the curve.

Further samples were machined into flat dogbone tensile test specimens of rectangular cross-section 9 mm × 4 mm and gauge length 43 mm. These were tested on a screw-driven testing machine with a constant cross-head speed of 0.005 mm s^{-1}, and once again load–unload cycles were used to determine the elastic response at a nominal strain of 0.2%. The strain to failure was also recorded, being taken to be the strain at which the maximum load was reached.

3. Results and discussion

3.1. Preform and foam structure

Fig. 1 shows optical micrographs of cross-sections through preforms of NaCl made by sintering particles of 400 μm and 75 μm diameter at 755 °C for different periods of time. As seen, while there are significant differences in the structure of the 75 μm preforms as the sintering time is extended, the 400 μm preforms do not show a large difference, even after extended sintering times.

Previous work has shown that the sintering of NaCl particles of a diameter greater than about 150 μm is dominated throughout the vast majority of the normal sintering process by the evaporation of material from the particle surface and the condensation of this material at the neck [13]. This mechanism changes the local neck architecture but does not alter the overall density. By contrast, the mechanism that dominates the sintering process for particles smaller than about 150 μm is the diffusion of material from the grain boundary formed between the two particles to the particle neck [13]; this both alters the neck shape and causes densification.

It is thus understandable that in the 400 μm particle size preforms sintering has brought about only a limited increase in density at even long sintering times (the highest density preform obtained was 0.76 of the theoretical

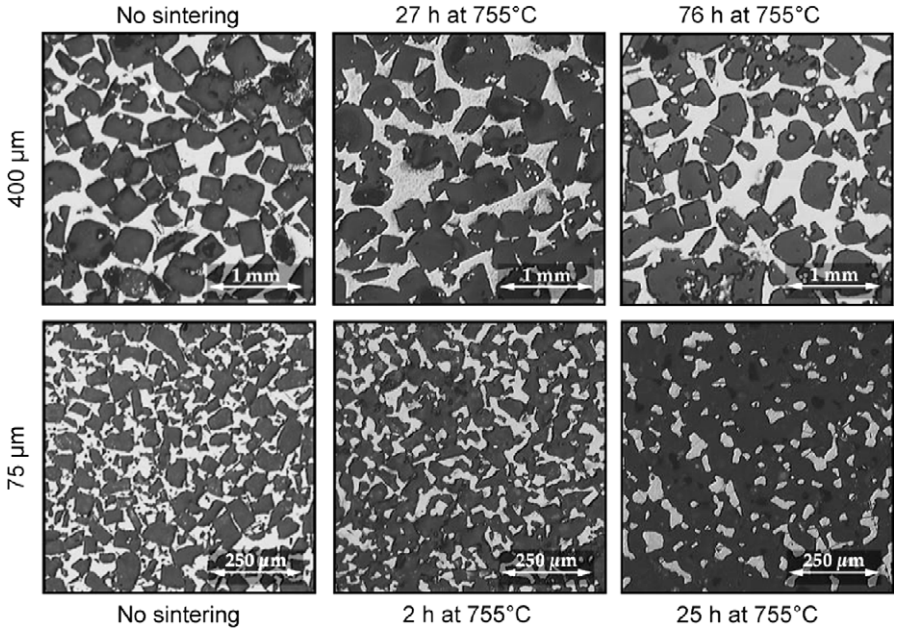

Fig. 1. Optical micrographs of polished sections through resin-infiltrated preforms of NaCl of 400 μm and 75 μm diameter, made by sintering at 755 °C for different periods of time. The dark phase is NaCl and the light phase is mounting resin.

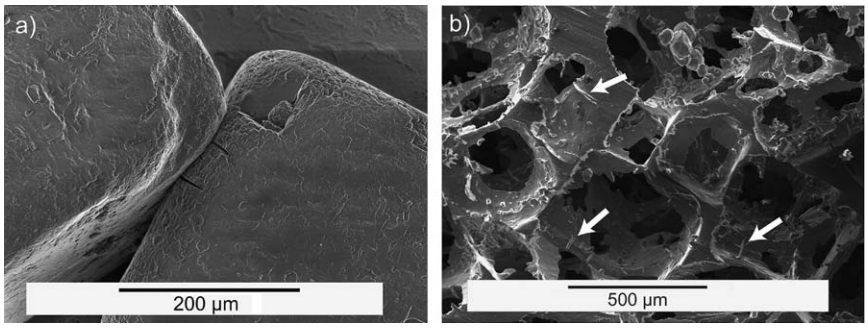

Fig. 2. Secondary electron SEM images of (a) the contact point between particles of 400 μm NaCl after CIP at 28 MPa, and (b) the resulting foam after infiltration under 4 bar pressure ($V_f = 0.2$), with replicated cracks indicated by the white arrows.

maximum density ρ_0, relative to a starting density of 0.64 ρ_0). That any densification was possible is due to the fact that the grain boundary diffusion mechanism does operate even in larger particles during the very early stages of sintering. By contrast, a large range of densities, up to $0.87\rho_0$, is possible with the 75 μm salt particles. Such limits on the achievable preform densities evidently place limits on the densities of foams achievable with sintering. Using cold isostatic pressing, on the other hand, densities varying over the range 0.64–$0.9\rho_0$ were easily produced using both sizes of NaCl particles.

As well as changing the density of the NaCl particle preform, the nature of the particle–particle contact point (the neck) will be influenced by sintering or CIP treatment. Fig. 2a shows a scanning electron microscopy (SEM) micrograph of the contact point between two particles after cold pressing. Signs of a small amount of local plastic deformation in the neck region can be discerned, along with some cracking. These cracks apparently survive heat-up prior to infiltration to 710 °C (relative to a NaCl melting temperature of 800.7 °C [14]), and are sometimes observed to have been replicated in the final foam structure (see Fig. 2b).

The general structure of foams made with cold-pressed preforms may be seen in the SEM image of a foam made with an unsintered preform in Fig. 3a. The structure retains its angular character as the density is varied.

After preform densification by sintering there is a change in the shape of the necks between particles, particularly at longer sintering times. This is clearly seen with the 75 μm particle preforms, the structure of which becomes gradually smoother and more regular, Fig. 1. This of course is expected from a process that minimizes surface energy. This difference is reproduced in the foams made from these materials, as is shown in the SEM images in Fig. 3. As the preform is sintered to higher densities the

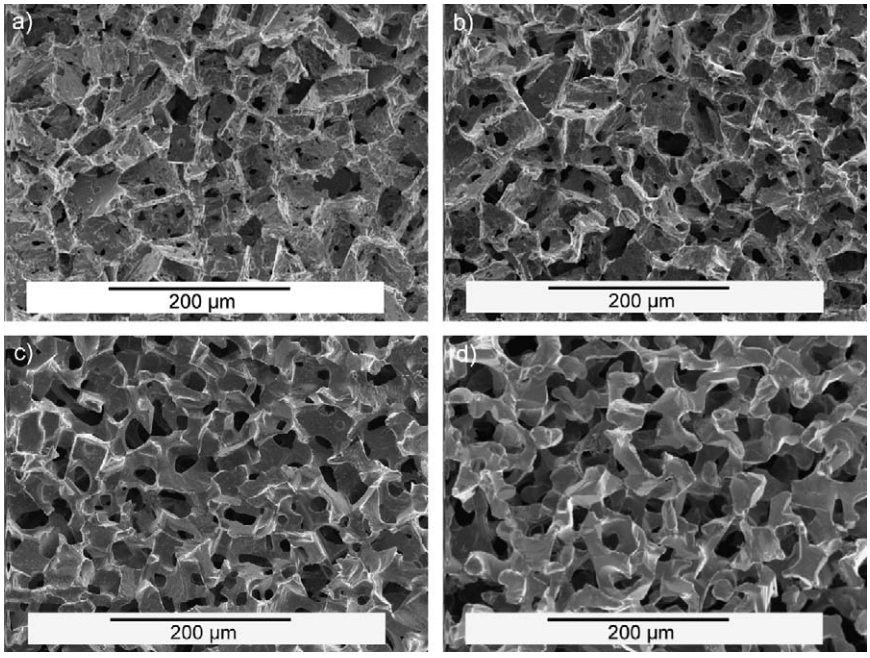

Fig. 3. Secondary electron SEM images of pure aluminium foams made by the replication method using preforms of 75 μm NaCl particles: (a) unsintered ($V_f = 0.4$), and sintered at 755 °C for (b) 2 h ($V_f = 0.37$), (c) 9 h ($V_f = 0.32$) and (d) 25 h ($V_f = 0.25$).

contact points between particles grow by diffusion and the particle–particle neck size is increased, resulting in a more smoothly rounded structure than in foams produced using cold isostatic pressing.

3.2. Foam mechanical properties

The Young's modulus and the 0.2% flow stress in both tension and compression for pure aluminium foams made using preforms densified by sintering or CIP of NaCl particles 400 μm and 75 μm in diameter are shown in Fig. 4. There is some scatter in the data; however, there is a noticeable trend for the results of foams produced by sintering to show slightly lower properties. This may seem surprising when the greater regularity of the sintered foam structure is considered (e.g. by comparing Fig. 3a with the other images in Fig. 3), but makes sense on further consideration. Indeed, sintering, which rounds the cross-section of struts delineated by the surface of neighbouring NaCl particles, decreases the moment of inertia of beams in the foam at given strut cross-sectional area. In other words, despite the more irregular structure of the foams produced with cold-pressed NaCl preforms, material is more efficiently placed in less regular foams where the preform has been densified by CIP. Fig. 5 illustrates the point with a sketch of three touching spherical particles drawn in cross-section through the plane containing their centres. Sintering, which is driven by capillary forces, will gradually smoothen and round the cross-section of struts running between the particles, resulting in foam struts having a near-circular strut cross-section. Conversely, CIP will tend to leave or even accentuate the sharp angles between the grains, generating struts of cusped and irregular cross-sections (in the idealized case of Fig. 5, a cusped triangle results). The latter struts have a higher second moment of inertia at constant cross-sectional area, and therefore a greater bending stiffness in all directions around the strut. As the elastic and plastic deformation of these foams are dominated by bending of the struts [12,15], these more efficient, albeit less regular, cross-sectional shapes should

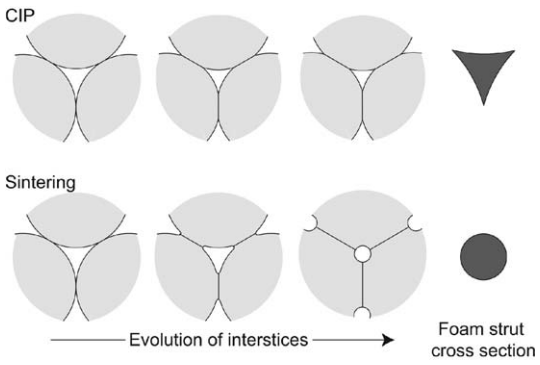

Fig. 5. A schematic diagram of three particles, showing a cross-section through the plane containing their centres, during sintering and CIP, illustrating the difference in the foam strut shapes produced.

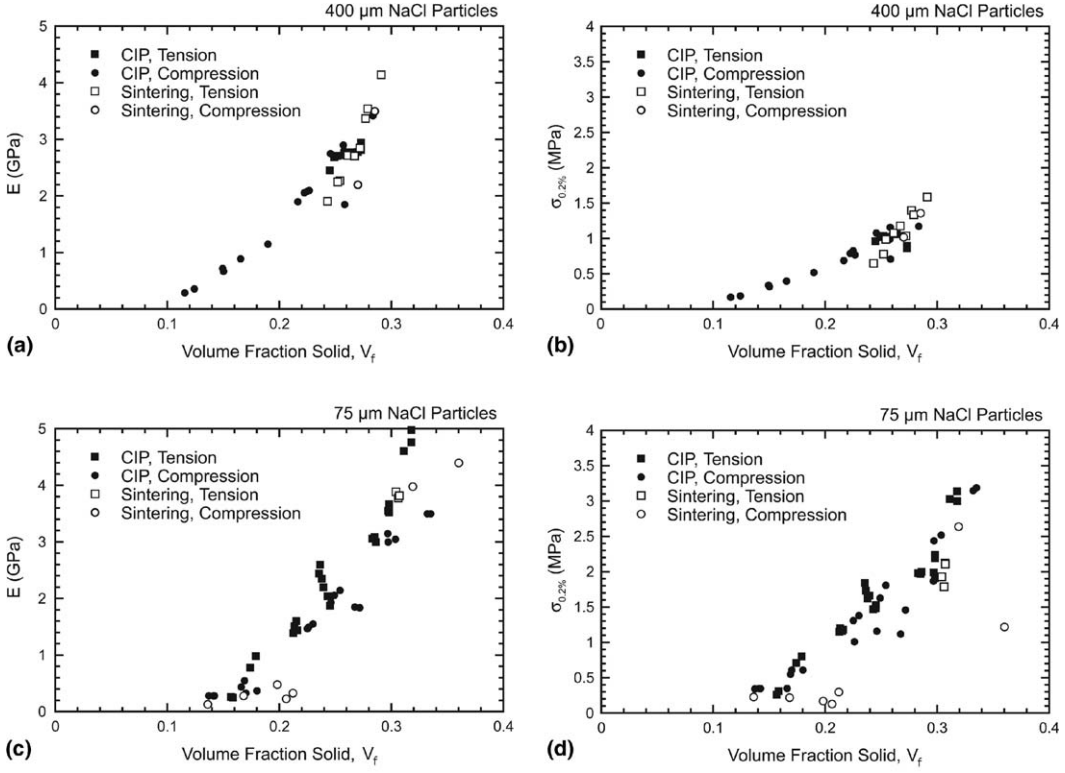

Fig. 4. The Young's modulus (graphs (a) and (c)) and 0.2% yield stress (graphs (b) and (d)) of pure aluminium foams made using preforms densified using sintering or CIP from 400 μm (graphs (a) and (b)) and 75 μm (graphs (c) and (d)) diameter NaCl particles, tested in tension and compression. Estimated error in testing and data analysis procedures is around 2% of the measured value; hence it roughly corresponds to the size of the points on the curves.

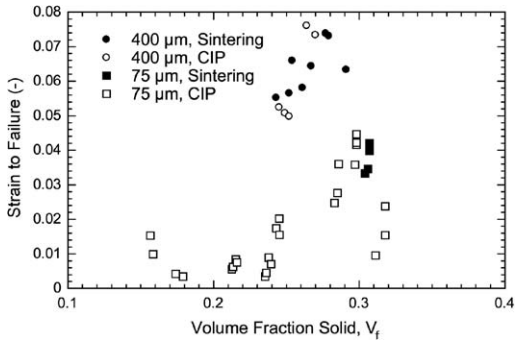

Fig. 6. Tensile elongation of foams made using preforms densified by sintering and CIP.

indeed increase the foam stiffness and flow stress, as observed. Between the two idealized shapes of Fig. 5, the difference in moment of inertia, and hence in foam modulus, is a factor of 1.68 (the cusped triangle has the higher value) [16]; this is of the same order as the difference between properties of the two foam series. It is also interesting to note that this effect, namely that smoother foam mesostructures are inferior from the standpoint of foam modulus and flow stress, is consistent with what was found in another study where the foam structure was varied by changing the infiltration pressure [17] (the lower the infiltration pressure is for a given preform, the less narrowly curved and hence smoother is the foam surface).

Plotting the maximum tensile elongation for the foams, Fig. 6, indicates on the other hand that sintered samples are more consistently ductile. Elongation values are above 3% for all samples from sintered 75 μm salt preforms and above 5% for all samples from sintered 400 μm salt preforms. Tensile elongations at failure for foams from cold-pressed preforms are erratic, varying from well below 1% to values that approach those for foams from sintered preforms. Although more samples would be needed for the trend to statistically solid, this tendency would be expected since the tensile ductility of these materials is dominated by the accumulation of internal damage [5], which should be minimized in the smoother and more regular struts produced from sintered preforms, Fig. 3. In summary, the sintered preforms produce foams that are weaker but more ductile.

4. Concluding remarks

Both sintering at elevated temperatures (>750 °C, $0.95T_m$), and cold isostatic pressing can change the density of NaCl preforms used for replication processing of metal foams. Foams produced from sintered preforms have a more rounded and regular structure than foams produced by cold-pressing. The latter, however, which are more irregular and jagged in structure, display generally superior stiffness and strength values. This somewhat unexpected result highlights the importance of distributing matter optimally in the struts making the open-pore foams; this fact that was pointed out in an earlier analysis [16].

Acknowledgements

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