Investigation of the influence of blowing agent and alloy composition on the foaming behaviour of thixocast AlSi6Cu4 precursor material

Francisco Garcia-Moreno*, John Banhart*, Marco Haesche**, Kanathala Vignodhar***, Jörg Weise***

*Hahn-Meitner Institut, Berlin and Technische Universität, Glienicker Str. 100 14109 Berlin, Germany

**Universität Bremen, Bibliothekstrasse 1, 28359 Bremen, Germany

***Fraunhofer Institute for Manufacturing and Advanced Materials (IFAM), Wiener Str. 12, 28359 Bremen, Germany

E-mail: we@ifam.fraunhofer.de

Abstract

Thixocasting of precursor material for aluminium foams is a hybrid process comprising aspects of casting and powder technology and can be used for the production of complex-shaped foamable material. Due to the differences between this process and uni-axial pressing or extrusion both a difference in foaming behaviour and in the resulting foam structures is found. This led to investigations of how to influence the expansion, collapse and drainage behaviour by addition of alloying elements and the use of blowing agent mixtures. In-situ X-ray radioscopy and ex-situ methods were used to quantify these differences. AlSi6Cu4 specimens were thixocast containing 0.8wt% TiH₂ as blowing agent. The alloying elements Fe, Sn, Sb and In were used in contents of 0.5 and 1 wt%. Expandometry showed that Fe, Sn, Sb and especially In additions led to a remarkable increase of the maximum expansion due to the additives and showed that Fe and Sb containing foams are stable for a longer time. Further experiments with mixtures of TiH₂ with ZrH₂ and CaH₂ but unchanged overall content of bonded hydrogen in the precursor showed that CaH₂ leads an increase of the maximum expansion while ZrH₂ does not.

1. Introduction

A combined casting and powder metallurgical process for the production of metal foam precursor material has been developed based on thixocasting of cold-isostatically compacted powder mixtures. Advantages of this process in comparison to other approaches are the possibility to produce precursor components with complex geometries, a more isotropic microstructure and a good adaptation of the temperature ranges of the matrix alloy melting and the blowing agent decomposition. Another advantage to other powder metallurgical approaches is the possibility to substitute the expensive metal powder used for the production by less expensive recycling waste (1). On the other hand first investigations had shown that thixocast precursor material generally has a lower maximum expansion, a higher coalescence activity and a more pronounced collapse behaviour (2). These effects might be explained by a different content, structure and distribution of oxide particles in comparison to extruded or hot-pressed precursor material. Oxides are known to play an important role in the stabilization of metal foams.

Starting with AlSi6Cu4 precursor alloys with the previously (2) determined optimal blowing agent content of 0.8wt% TiH₂, several stabilizing measures to reduce drainage and collapse were investigated. This included the use of blowing agent mixtures and the addition of alloying elements. The use of blowing agent mixtures is known to improve the foaming behaviour of Zn foams (3). It is also known that capillarity drainage is proportional to the capillary pressure difference (4). So it should be possible to attain better foaming results by the addition of relatively low levels of certain solutes which are preferentially adsorbed at the surface. In Al-Si systems, elements like Sb and Sn act like surface active elements thus decreasing surface tension. The viscosity, another essential parameter that governs the rate of drainage, is also markedly affected by the addition of solutes, as e.g. iron. The influence of the blowing agent mixtures and alloying elements upon the foaming behaviour was investigated by conventional and X-ray expandometry and the comparison of the foam structures and densities.

2. Experimental procedure

For the investigation of the influence of the blowing agent composition, CaH_2 and/or ZrH_2 were added to AlSi6Cu4 powder along with TiH₂ powder. It was ensured that in either case the combination of blowing agents contained an equivalent amount of hydrogen as present in 0.8 wt% of TiH₂. This appeared to be a good starting point as this amount was found to be a suitable choice for thixocast AlSi6Cu4 precursor foamed with TiH₂ only (2). The amount of hydrogen present in 0.1 or 0.2 wt% TiH₂ of a total of 0.8 wt% TiH₂ was replaced by an equivalent amount of hydrogen present in CaH₂ and/or ZrH₂ powders. In order to determine the effect of alloying elements on the foamability of Al-Si alloys, Fe, Sb, Sn and In were added at proportions of 0.5% and 1.0% to AlSi6Cu4. The blowing agent content was 0.8 wt% TiH₂ in these cases.

Metal powders and additions were mixed in a tumbling machine for 60 minutes. After this, the material was compacted by cold isostatic pressing (CIP) which is applied to consolidate the powder mixture to slugs with a relative density of about 70 to 80%. CIP was carried out at a compaction pressure of 1500 bar on a machine made by EPSI. The mould used for CIP had an internal diameter of 85 mm and was 245mm long, resulting in slugs with 65 mm diameter and 210 mm length. Three slugs were produced for each variant. Holes were drilled into the slugs at three positions, one in the middle of the precursor and two 15 mm away from each front face. In these holes nickel-chromium-nickel thermocouples could be positioned. To avoid excessive oxidation of the precursor surface during heating the slugs were wrapped into anodized aluminium foils.

Heating to the semi-solid state was carried out by putting the slug into a pre-heated annealing furnace kept at 640°C. After heating the semi-solid slugs were manually transferred into the sleeve of the Bühler SC N/66 horizontal cold chamber high pressure die casting machine and pressed into the die cavity. The locking force of the die casting machine was 6615kN. A mould optimised for conventional thixocasting was used (4). The mould was equipped with pressure sensors in the casting system and in the component cavity. The test component, a connecting rod, demonstrates typical features of castings such as varying wall thickness, cores and shrinkage restrictions. The smallest wall thickness was 5 mm in at distance of approximately 420 mm from the biscuit. The shot weight was 1530 g. The speed of the plunger to press the semi-solid slug into the die casting cavity was 0.3 m/s and the secondary compression was 1520 bar.

For the expandometer tests three specimens of each combination of blowing agent and alloying element were prepared from cast semi-finished components. The temperature of the expandometer was 750°C. The main parameters for the evaluation of the foaming behaviour were the maximum expansion and the degree of collapse after a certain time (30 s).

For the in-situ real-time X-ray analysis the specimens were machined to a size of $20 \times 10 \times 5$ mm³. Samples with different metal additions were foamed freely without any mould. For this they were placed in a X-ray transparent furnace with resistive heating - described elsewhere (5) - and heated up with a constant heating rate of around 133 K/min to the final temperature of 700°C and held at this temperature up to 20 minutes. Herewith a qualitative impression of the foaming behaviour, stability and pore structure development can be achieved. Additionally, 2D expansion perpendicular to the X-ray beam, drainage and foam density can be determined quantitatively. Furthermore, the precursor material was foamed in and without moulds [see also (6)] and eroded to determine the influence of the additions upon the foam structure.

3. Results and Discussion

The heating time for slugs containing a mixture of blowing agents (TiH_2+ZrH_2/CaH_2) ranged from 46 to 52 minutes. As temperature control is not sensitive enough for the determination of the state of the slug in the melting range the slugs were transferred to the sleeve and pressed into the mould after some expansion of the slugs could be observed in the furnace due to the decomposition of the blowing agent in the softening matrix material. CaH₂-containing specimens expanded at higher slug temperatures in comparison to slugs containing solely TiH₂ or mixtures of TiH₂ and ZrH₂. The addition of ZrH₂ led to a faster expansion of the slugs.

The slugs could easily be cast and the castings showed a very good surface quality. No cracks could be observed. The good quality of the castings was confirmed by radioscopic tests in which no cracks or pores were found. However, some demixing effects were observed in the radiographs which became visible due to the different absorption coefficients of the demixed phases and the alloying elements (6).

The maximum expansions observed in the expandometer tests on precursor material containing blowing agent mixtures are presented in **FIGURE 1**. Compared to conventional uni-axially hot-pressed or extruded precursor materials the overall level of expansion is rather low. Use of ZrH_2 decreases maximum expansion, whereas all specimen with CaH₂ show a clearly increase of the maximum. The highest maximum expansion (285%) was obtained using a combination of 0.7wt% TiH₂ with CaH₂.



FIGURE 1: Effect of composition of blowing agent mixtures on maximum expansion of AlSi6Cu4

The reason for the positive influence of CaH_2 might not be its higher decomposition temperature in comparison to TiH_2 as ZrH_2 shows also a high decomposition temperature (7) but decreases the maximum expansion. It might be argued that the introduction of calcium in form of CaH_2 stabilizes the foam structure and improves the general foaming behaviour. However, as the decomposition of the hydrides in close contact to liquid aluminium is not really known no final conclusion can be given for the reason of the observed influence.

FIGURE 2 shows the influence of additions of 0.5 wt% or 1.0 wt% Fe, Sn, Sb and In on the maximum expansion of AlSi6Cu4 precursor material. The solid bars show the mean value, the vertical error bars the maximum and minimum values of 3 expandometer tests. It can be concluded that all additional alloying elements increase the maximum expansion significantly. With the exception of Fe this effect is more pronounced with increasing contents of the additional elements. No definite influence of alloying elements on the degree of collapse 30 s after the maximum expansion peak could be observed. On the other hand, as can be seen in the X-ray measurements below, for Fe and Sb the collapse is notably retarded.



FIGURE 2: Average maximum expansion for precursor AlSi6Cu4-material with different additional alloying elements

Foam expansion can be measured by mechanical or by X-ray methods. As the former measures a volume change, the latter a change in cross section (F/F_0) , the results are not exactly the same but should express the same trend. In practice, foaming conditions such as foaming temperature, heating rate or the usage of a mould are slightly different in these two experiments which can influence e.g. the drainage behaviour, etc. Anyhow the real-time insitu X-ray experiments corroborate the mechanical expandometer results, showing also an increased expansion with the addition of Fe, Sn, Sb and In and a higher foam stability in the case of Fe and Sb (**FIGURE 3**). In the latter two cases the 2D foam expansion after 1200 s is still around 3.5, whereas in the other cases it is reduced to 1.5-2.5 due to collapse. For the alloying elements Fe, Sn and Sb, but not for In, the start of the foaming process is retarded by 80-100 s even if using the same heating parameters. This seems to be a positive effect for the stabilization of the foam in later stages, so that collapse is not so pronounced. For the sample containing Sb foam expansion kinetics is even slower than for the other specimens, but eventually leads to the most stable foam without notable collapse after maximum expansion.



FIGURE 3: Development of cross-section perpendicular to the X-ray beam during foaming of AlSi6Cu4 + 0.8 wt% TiH₂ without and with 1% Fe, Sn, Sb and In additions

In **FIGURE 4** we clearly see the differences in maximum expansion between foams a) without and, b) with 1% Fe addition. Moreover, an increased drainage in the sample without addition compared to the sample with Fe addition can be noticed. Real-time observation shows that in the case of no additions drainage appears immediately after the beginning of foaming while with additions drainage effects occur later.



FIGURE 4: Radioscopic images of AlSi6Cu4 + 0.8 wt% TiH $_2$ foams at maximum expansion, a) without and b) with 1% Fe

The evolution of the vertical foam density profile during foaming is shown in **FIGURE 5**. If we follow the times axis we can first recognize the compacted bulk precursor material (we assume it to be 100% dense) up to the starting point of foaming around 250-300 s after the initiation of heating. At this point foam height increases and foam density decreases. Drainage appears very soon, forming a dense layer on the bottom of several millimetres with 90-100%

of full density. One can find: a) strong drainage which is typical for thixocast precursors, b) a slight reduction of drainage for the precursor containing Sb addition. As well as in **FIGURE 3** we can monitor in **FIGURE 5** the collapse of the foams just by following the height reduction in time. We observe that collapse of the foam without additions (a) continues even 1000 s after the foaming started. On the other hand, with Sb additions (b) the foam is quite stable.



FIGURE 5: Density evolution during foaming for AlSi6Cu4 + 0.8 wt% TiH₂ with different additional alloying elements. Expansion, drainage and collapse can be observed. a) without addition and b) + 1% Sb

The curves in **FIGURE 6** are sections of the density time development shown in **FIGURE 5** for all specimens 300 s after foaming. We can clearly observe that the addition of 1% Fe, Sn, Sb and In leads to a reduction of drainage over the sample height in all cases. In addition, an increased expansion of around 25% at this point of foaming for Fe and Sb can be noticed.



FIGURE 6: Density in dependence of the foam height for AlSi6Cu4 + 0.8 wt% TiH₂ with different additional alloying elements 300 s after foaming

A qualitative comparison of the pore structure of specimen foamed with and without mould showed that iron additions lead to coarse structures and significant drainage but Sb, In and Sn lead to an increased number of pores and a reduction of drainage. **FIGURE 7a** and **7b** show the cross section ($205 \text{ mm} \times 38 \text{ mm}$) of a complex foam component made from AlSi6Cu4 and AlSi6Cu4In1 alloy respectively. The component has varying wall thicknesses leading to pronounced drainage and collapse at the thin-walled left and right ends. Furthermore, increased drainage can be found near the overflow. The reduction of pore size as well as of drainage by the addition of indium can clearly be observed.



FIGURE 7: Cross-sectional foam structure of a) AlSi6Cu4 and b) AlSi6Cu4In1 foam components

4. Conclusions

Foamable precursors of AlSi6Cu4 + 0.8 wt TiH₂ with 0.5-1% Fe, Sn, Sb and In additions were prepared by the thixocast process. By means of ex-situ and in-situ X-ray expandometry it could be shown that in the case of metal additions an increase of the foam maximum expansion up to 40% and of the expansion after 1200 s up to 50% could be reached. Additionally a reduction of drainage and an improvement of pore structure can be achieved. For Fe and Sb also stability could be increased, reducing the collapse effect. Experiments with blowing agent mixtures of TiH₂ with ZrH₂ and CaH₂ showed that CaH₂ leads an increase of the maximum expansion though ZrH₂ does not.

Acknowledgemends

This work was supported by the DFG-Program SPP 1075, grants BA1170/3-2 and WE2840/1-1.

Literature

- (1) J. Weise, H. Stanzick, J. Banhart, Cellular Metals: Manufacture, Properties, Applications, Proc. Int. Conf. MetFoam2003, MIT-Verlag Berlin (2003), p.169.
- (2) F. Garcia-Moreno, N. Babcsan, J. Banhart, M. Haesche, J. Weise, Cellular Metals and Polymers (2004), in press
- (3) K. Stöbener, M. Weigand, G. Rausch, Cellular Metals: Manufacture, Properties, Applications, Proc. Int. Conf. MetFoam2003, MIT-Verlag Berlin (2003), p.161.
- (4) D.T. Wasan, A.D. Nikolov, L.A. Lobo, K. Koczo, D.A. Edwards, Prog. Surf. Sci. 39 1992, 119.
- (5) F. Garcia-Moreno, M. Fromme, J. Banhart, Adv. Eng. Mater., 6, 2004, 416.
- (6) M. Haesche, O. Marchetto, F. Höcker, J. Baumeister, J. Weise, Aluminium, 2005, accepted for publication.
- R.L. Beck, W.M. Mueller, in: Metal Hydrides, eds. W.M.Mueller, J.P. Blackledge, G.G. Libowitz, Academic Press New York 1968, 241.