Influence of Alloying Additions on Foaming Behaviour of Thixocast AlSi11 Precursor

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Precursor for making aluminium foam via powder compact process was made by mixing metal and blowing agent powders, by compacting the mixture by cold-isostatic pressing and by thixocasting the billet to a dense shaped product. The precursor was then foamed by heating to above the melting temperature of the alloy. We chose an AlSi11 alloy and 1 wt. % of the common blowing agent titanium hydride. In order to tailor foaming characteristics 1 wt. % of one of the alloying elements Fe, Sn, Sb and In was added. The foaming behaviour was characterized with a mechanical expandometer. We showed that addition of Sn significantly increased maximum expansion, while Fe, Sb and In only had a minor influence. X-ray radioscopic experiments confirmed this influence on maximum expansion due to the additives and showed that In and Sb containing foams are more stable than Sn for a longer time. This stability effect was also corroborated by the reduced coalescence rate and collapse degree measured for Sb and In. The findings are compared to the slightly different results found for AlSi6Cu4 in a previous study and are explained in terms of the changes in solidification range of these two alloys caused by the additions.

Keywords: *metallic foam, thixocasting, foaming behaviour, X-ray*

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[1,2]. This process offers the possibility to produce precursor components with complex geometries, an isotropic microstructure and a good adaptation of the temperature ranges of matrix alloy melting and blowing agent decomposition. It also allows to substitute expensive metal powder used by less expensive recycling waste (cutting chips)[1,3]. On the other hand thixocast precursor material has a comparably low maximum expansion, a higher coalescence activity and a more pronounced collapse after foaming. This behaviour might be explained by a different content, structure and distribution of oxides on the surface of the compacted and thixocast metal powder particles in comparison to extruded or hot-pressed precursor material. Drainage is even more pronounced for precursor material produced from cutting chips.

These effects led to investigations of how to influence the foaming and especially the drainage behaviour of foaming precursor material based on thixocast powder slugs. There are several measures to improve foam stability and to suppress drainage, e.g. the addition of SiC-particles. On the other hand it is known that the compostion of the matrix alloy itself also has an significant influence upon the foaming behaviour and the resulting pore structure. The aim of the work presented here was therefore to investigate the influence of small additions (0.5wt% and 1wt%) of alloying elements on the foaming behaviour of an AlSi11 alloy. Though with the addition of alloying elements no single but several parameters will be influenced the focus of the investigation was on elements reducing surface tension or increasing viscosity of aluminium melts. Using the data of Altenpohl[4] Sn, Sb and In were used to influence the surface tension, whereas Fe was used to increase the viscosity.

2. Experimental procedure

AlSi11-alloy powder blend was prepared using aluminium and silicon powders. In a first series of experiments TiH_2-powder was added in amounts of 0wt% to 1.5wt% in order to determine the optimal amount of blowing agent. In the subsequent experiments Fe, Sb, Sn and In were added in amounts of 0.5wt% and 1.0wt% to AlSi11 alloy powder mixture together with the determined optimal TiH_2-amount.

The powder blends were treated and compacted to slugs as described by Garcia-Moreno et al.[2,5] (compaction unit, model EPSI, , 1500bar, mould diameter 85mm, mould length 245mm, slug diameter 65mm diameter, slug length 210mm).

Heating to the semi-solid state was carried out by putting the slug into an annealing furnace pre-heated to 640°C and keeping it there for approximately 60-64 minutes at 640ºC. The condition of the slugs in the semi-solid state and their readiness for casting was tested by means of an indention test.

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 (locking force 6615kN) and pressed into the die cavity, a mould optimised for conventional thixocasting[5]. The speed of the plunger to press the semi-solid slug into the die casting cavity was 0.4 m/s and the secondary compression was 1520 bar. The quality of the castings was examined by means of X-ray radioscopy, metallographic sectioning and scanning electron microscopy (SEM Leo 438VP with EDX). Furthermore, DSC measurements were done with a Netzsch STA 409 in order to evaluate the influence of the alloying elements on the melting behaviour.

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). The precursor material was also foamed without moulds and eroded to determine the influence of the additions upon the foam structure.

Specimens of 20x10x5mm^3 were prepared for in-situ real-time X-ray analysis at HMI. They were foamed without any mould in a X-ray transparent furnace by means of resistive heating described elsewhere[5]. The samples were heated up together with the furnace at a constant heating rate of around 1100 K/min to the final temperature of 700°C and held at this temperature for up to 200 seconds. Radioscopic pictures of the foaming process were acquired using a X-ray source voltage of 100 kV. The pictures were taken with a magnification of 4 and a repetition rate of 1 Hz. Analysis of the radioscopic pictures yields a qualitative impression of the foaming behaviour, stability and pore structure development. Additionally, quantitative 2D expansion perpendicular to the X-ray beam, foam density, drainage and coalescence rate can be determined using AXIM, a self developed image analysis program described elsewhere[6].

3. Results

3.1 Thixocast precursor

The temperature of the slugs after the heating into the semi-solid state was 581-582°C. This temperature was reached for all specimens regardless, if alloying elements were used or not. The slugs could easily be cut. Deformation of the slugs due to premature decomposition of the foaming agent was almost not noticeable.

The slugs could be cast easily. The castings had a very good surface and no cracks or other defects. The good quality of the castings was confirmed by radioscopic tests in which no cracks or pores were found. However, in earlier experiments some demixing effects were observed in the radiographs which are visible due to the different absorption coefficients of the demixed phases and the alloying elements[3]. These demixing layers near the surface of the component and parallel to the flowing direction could also be observed in the metallographic section, see figure 1. They are caused by shear rate gradients between a prematurely solidified shell near the mould surface and the still flowing semi-solid material during the filling process. Except for this demixing effect the structure of the components is very homogeneous.

In figure 2 a scanning electron microscopy image of a TiH_2-particle in the as-cast condition of AlSi11-precursor without additions is shown. Obviously, some reaction zones near the particle's surface have formed by interaction with the matrix elements. This was confirmed by line-scan measurements which led to the detection of diffusion of Si and Al into the TiH_2-particle but also of Ti into the aluminium matrix. Furthermore, the light gray phases in figure 2 were identified as intermetallic compounds of silicon and titanium (in large areas of the composition Si:Ti=1:1) with high contents of aluminium.

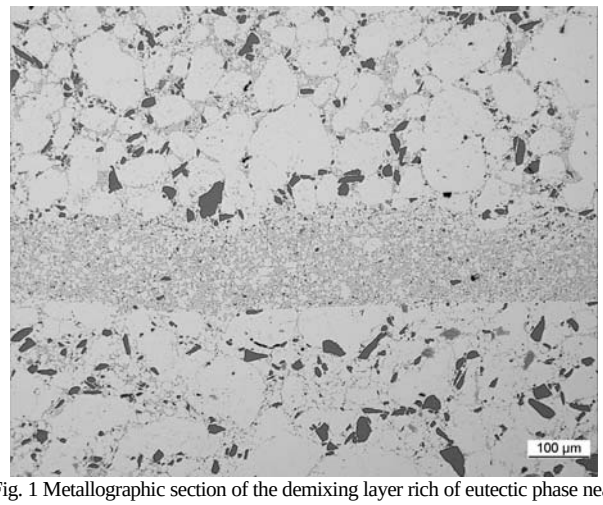


Fig. 1 Metallographic section of the demixing layer rich of eutectic phase near the component surface

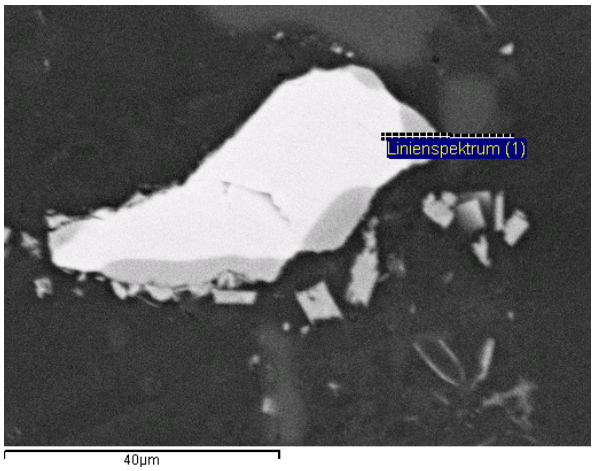


Fig. 2 SEM-figure of a TiH_2-particle in the as-cast condition of AlSi11-precursor without additions

Aluminium contents of up to 6 wt% in Ti already change the stability of the hydride phases[7] though still large amounts of H can be bound. For higher aluminium contents and intermetallic Al-Ti-phases (e.g. $TiAl_3$ and TiAl) the hydrogen storage capacity is drastically reduced[8]. For the interaction of titanium silicides with hydrogen no data could be found.

3.2 Mechanical expansion and collapse

The interaction of the TiH_2-particle with the matrix elements together with the elevated casting temperature lead to a reduction of the hydrogen amount available for the foaming process for thixocast precursor material. Consequently, higher optimal amounts of foaming agent in the precursor are needed as already demonstrated for the alloy AlSi6Cu4. This was confirmed in first experiments where it could be shown that the optimal content of the foaming agent TiH_2 in the thixocast AlSi11-precursor is 1wt%, see figure 3. This optimal content of blowing agent is clearly higher in comparison to conventional extruded or hot-pressed precursor material[9] and is mainly caused by the described hydrogen losses during the heating into the semi-solid state.

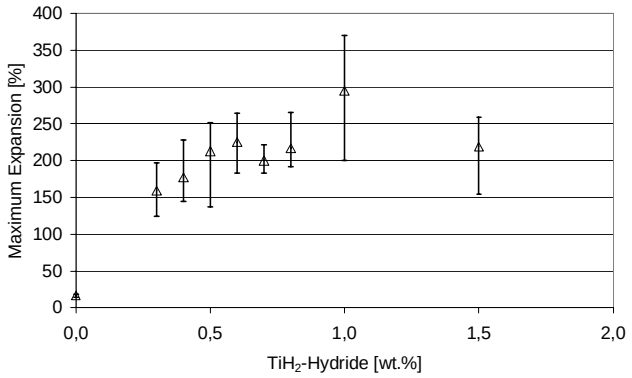


Fig. 3 Maximum expansion of thixocast AlSi11-precursor in dependence of the foaming agent content measured with the mechanical expandometer (minimum, maximum and mean value of 3 specimens), T = 750°C

Subsequent expandometry experiments showed that the maximum expansion of the foam is low in comparison to conventional precursor material but also in comparison to other thixocast precursor alloys. The relatively low expansion of AlSi11-precursor material in comparison to other alloys agrees with own earlier findings of uni-axially pressed precursor material.

Figure 4 shows the influence of additions of 0.5 wt% or 1.0 wt% Fe, Sn, Sb and In on the maximum expansion of the precursor material. The solid bars give 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 but indium increase the maximum expansion. The most positive influence on the maximum expansion has tin which causes a relative increase of the expansion of 40%. No significant difference between specimens with 0.5wt% or 1.0wt% of alloying elements could be found.

The generally positive influence of the alloying elements agrees with the results found with AlSi6Cu4 precursor alloys[2]. However, the improvements achieved by the addition of alloying elements is higher for AlSi6Cu4 than for AlSi11. It is also interesting to note the degree of improvement changed for all elements but tin. The effect caused by the addition of Sn is even increased in AlSi11 in comparison to AlSi6Cu4 though the effect of other elements is reduced and there is almost no effect of In in AlSi11.

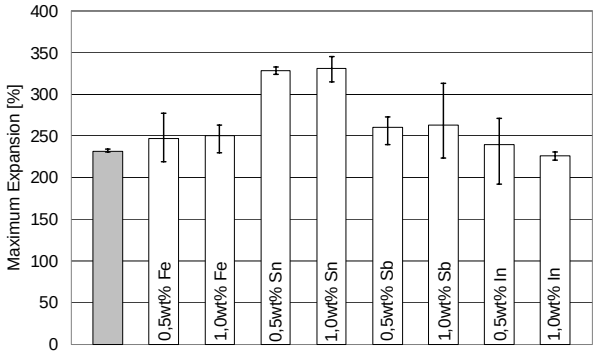


Fig. 4 Average maximum expansion for AlSi11 precursor-material with different additional alloying elements Fe, Sn, Sb and In, T = 750°C

Figure 5 gives the influence of alloying elements on the degree of collapse 30 s after the maximum expansion peak could be observed. With the exception of iron all alloying elements reduce the collapse tendency. This effect is also clearly demonstrated in the sections of foams produced without mould, see figure 6. All elements but iron lead to a more homogeneous structure with an increased number of pores and correspondingly with a smaller mean diameter. Moreover drainage is reduced. These effects are similar to the ones found for AlSi6Cu4[2].

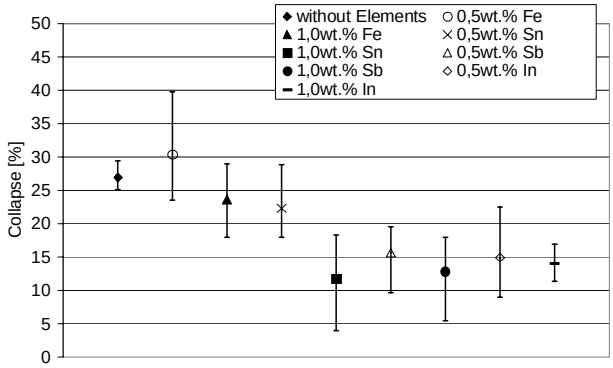


Fig. 5 Influence of alloying elements on the degree of collapse 30 s after the maximum expansion peak, T = 750°C

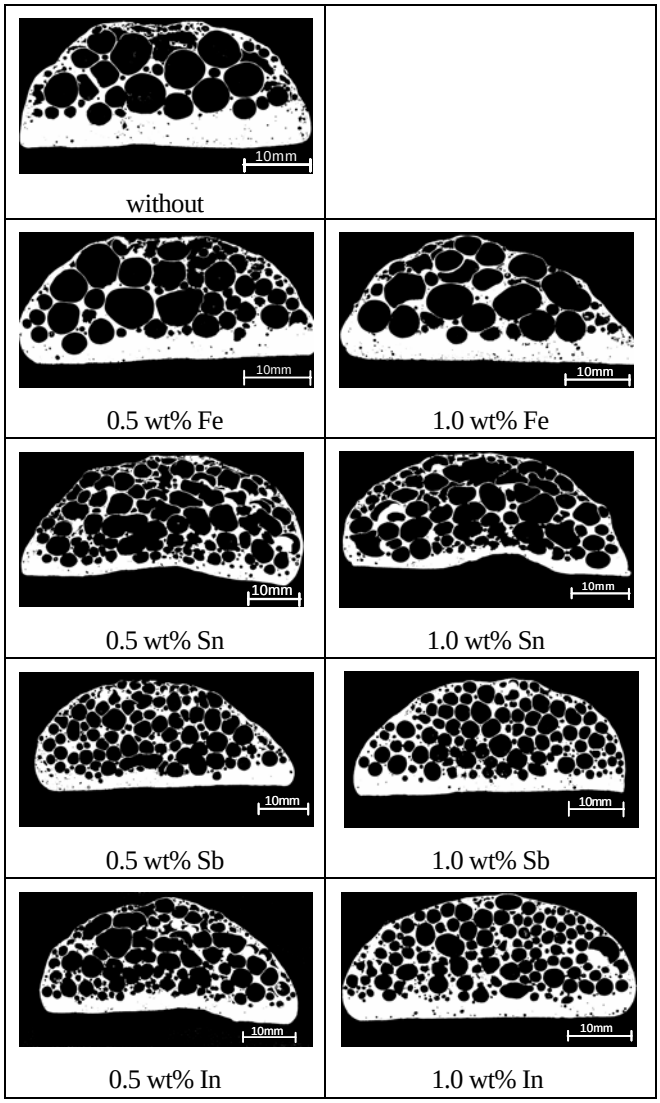


Fig. 6 Pore structure of AlSi11-foams with different additional alloying elements, foamed without mould at T = 750°C.

3.3 X-ray radioscopy

X-ray radioscopy analysis allowed us to follow the expansion kinetics and pore evolution of the foaming process in real-time for the different specimens.

The 2D expansion curves, see figure 7, show that in our set of experiments foaming began around 30s after starting the heating. 70 s later, after around 100s, the main expansion was reached approximately for all specimens. At this point e.g the specimen with Sn addition had the maximal expansion. In the following 100s at a constant temperature of T = 700°C the collapse of Sn-containing specimen was very pronounced, although not as extreme as in the case of Fe. This is the same behaviour as could be also observed in the mechanical expandometer measurements, where Sn had both the largest maximal expansion and the largest degree of collapse together with Fe.

For In and Sb the stability of the foam after 100s was remarkable, no collapse even after 200s at T = 700°C could be observed. For these two elements, also in the expandometer measurements at T = 750°C, the lowest collapse was observed (compare figure 5). Sb and In seem to be the most suitable cell wall stabilisation agents.

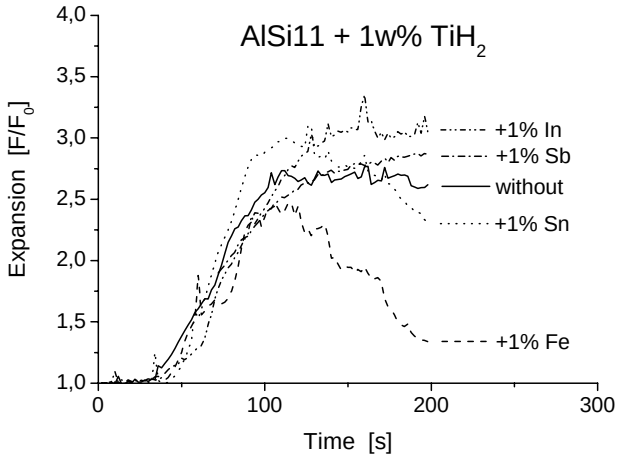


Fig. 7 2D expansion development for AlSi11 precursor material with different additional alloying elements (Fe, Sn, Sb and In), T = 700°C for t > 100s

Figure 8 shows radioscopic images of the free foamed samples in figure 7 after 200s. Obviously, there is a dramatic collapse of the sample with Fe, followed by the one with Sn. On the other hand the samples with Sb and In are more homogeous than the sample without. They have also a reduced pore diameter. Here after 200s no significant differences in drainage between the samples can be found.

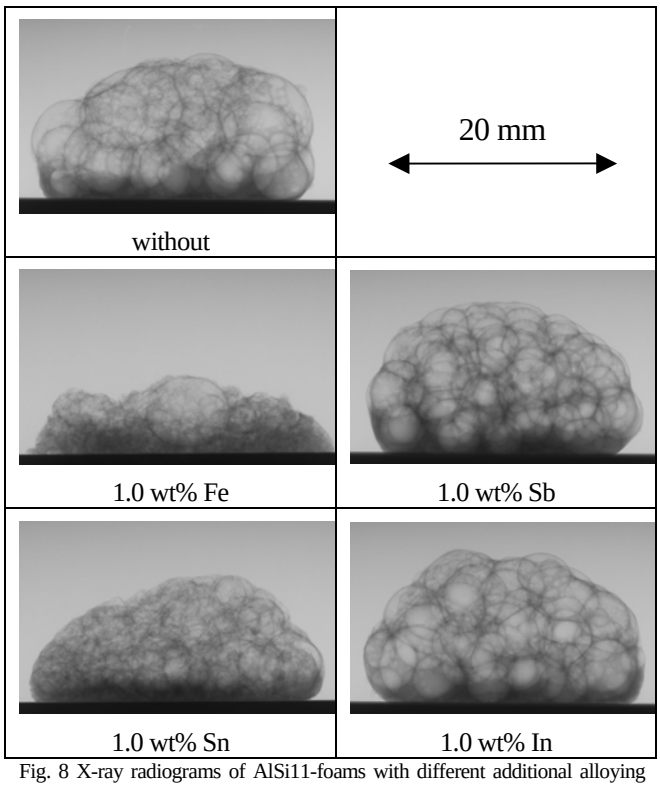
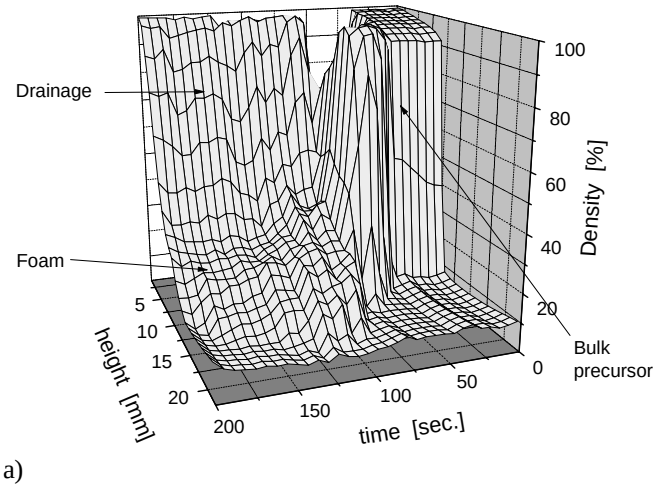


Fig. 8 X-ray radiograms of AlSi11-foams with different additional alloying elements 200s after start of heating, foamed without mould and corresponding to figure 7.

Observing the density evolution as a function of foam height during the entire foaming process (figure 9) for AlSi11 specimens without and with e.g. 1 wt% Sb addition we can notice the increased expansion with Sb addition in terms of an increased foam height. The average foam density is also higher with Sb additions and consequently we find a smaller drainage gradient in this case. At around 50-75s a first stage of drainage with almost 100% density can be found for the sample without any additions, but not for the one with Sb. This early and pronounced drainage typical for thiocast precursor material is also visible in the X-ray radiograms (figure 10). There we can see clearly the difference between AlSi11 and AlSi11Sb1 precursor material 75s after the start of heating, during the main expansion. The height of the drainage zone at the bottom of the sample without Sb takes more than 2/3 of the sample height. This first stage of drainage disappears mostly at the end of the main expansion, at around 100s, as consequence of the high bubble formation rate in this foaming stadium. In contrast to this with Sb addition no early drainage can be found, only the normal drainage during the stabilisation phase (t > 100s).



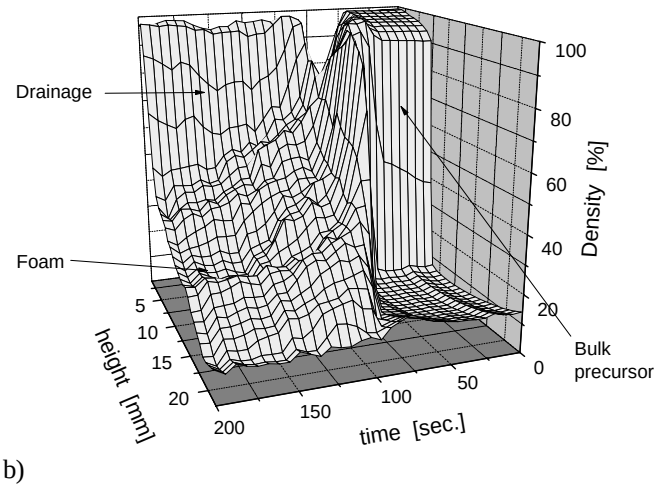


Fig. 9 Foam density evolution for AlSi11 a) without additions and b) with 1 wt% Sb. Bulk precursor, foam expansion and drainage can be distinguished

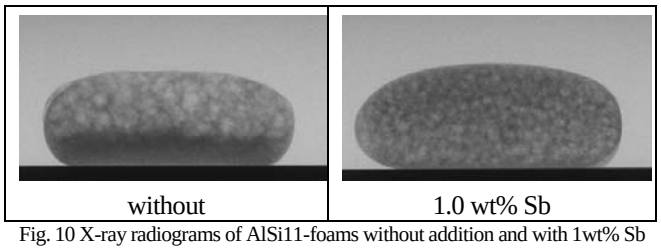


Fig. 10 X-ray radiograms of AlSi11-foams without addition and with 1wt% Sb 75 s after starting heating. Early drainage in the case of no addition can be observed.

From the X-ray images it was possible to quantify the number of cell wall ruptures. Of interest is not the expansion induced coalescence, but the one after the main expansion. That means only the coalescence between 100s and 200s was taken into account. The sum of all cell wall rupture events for AlSi11 without and with additions is plotted in figure 11. Without any and with Fe addition the number of ruptures is similar, although in the case of Fe, due to the quick and extreme foam collapse, the exact number was difficult to quantify. Sn addition produces the highest number of rupture events, which shows that in this case stability was decreased. On the other hand Sb and In show a reduced amount of ruptures, corresponding to the increased stability found in figure 7.

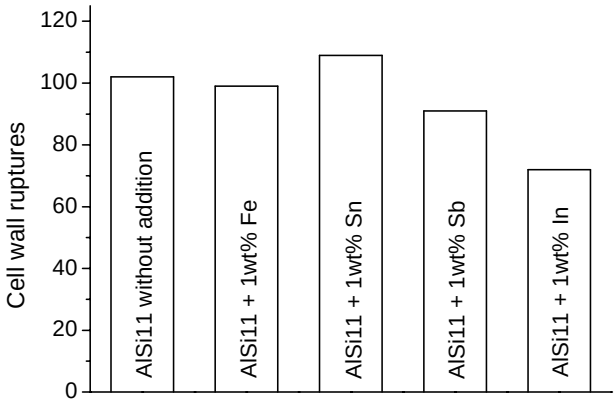


Fig. 11 Coalescence strength quantified by number of cell wall ruptures in fully expanded specimens. Samples correspond to experiment in fig. 7 100s and 200s after the start of heating.

4. Discussion

The identification of the mechanisms which lead to the observed influence of the alloying elements on foaming behaviour is very difficult as the addition of these elements to the matrix change various properties at the same time as e.g. the compaction and flow behaviour, the melting range, interactions of matrix alloy and foaming agent, viscosity and surface tension. Keeping this in mind it is not surprising that the elements can change different aspects of the foaming in different and seemingly contradictory ways. An eminent example is Fe which increases (in the case of AlSi6Cu4[2]) or does not change (AlSi11) the maximum expansion and at the same time deteriorates foam structure and increases drainage. Especially the latter fact demonstrates that the effects caused by an alloying element cannot be considered seperately as Fe is known to increase the viscosity[4] of aluminium melts and is therefore supposed to reduce the drainage in contradiction to the observed behaviour.

Similarly, Sn improves the maximum expansion of AlSi11 significantly but improves the collapse behaviour as well as the pore structure only marginally. The positive role of Sn for the maximum expansion might be explained by the change of the melting range of AlSi11 caused by the addition of Sn. Figure 12 shows the results of DSC measurements of AlSi11 without addition and with 1wt% additions of Sn, Fe, Sb and In. As can be seen, only Sn changes the solidus temperature of the alloy in a larger extent.

In and Sb have in thixocast AlSi11-precursor material as in AlSi6Cu4[2] a positive influence on pore structure, collapse and drainage behaviour. Also maximum expansion is significantly increased. This is in correspondence with the expected behaviour as both elements decrease the surface tension of aluminium melts. However, also for these elements the influence is more complex as e.g. In reduces the collapse in AlSi11-precursor though no large influence can be found in AlSi6Cu4.

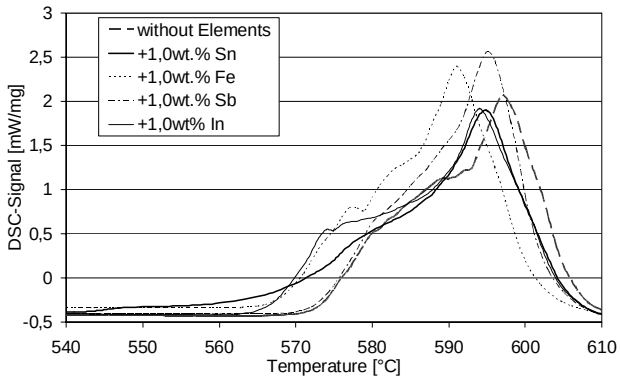


Fig. 12 Influence of the addition of 1wt% of alloying elements on the solidification range (DSC-measurement)

4. Summary and Conclusions

Foamable precursors of AlSi11 + 1.0 wt% TiH_2 with 0.5-1 wt% Fe, Sn, Sb and In additions were prepared by the thixocasting process. The precursor alloy was produced using elemental powders. The specimens were investigated by means of ex-situ and in-situ X-ray expandometry, metallography and DSC. Furthermore, coalescence rates in the fully expanded state were measured.

The optimal content of the foaming agent TiH_2 in the precursor alloy AlSi11 was determined to be 1wt%. It was shown that during heating into the semi-solid state and during thixocasting interactions between the matrix alloy powders and the foaming agent particles occur leading to diffusion of Si and Al into the foaming agent particle and the formation of Ti-Si and Ti-Si-Al-compounds. Also diffusion of titanium into the matrix could be detected.

The mechanical expandometer experiments showed that the addition of small amounts of Sn increases significantly the maximum expansion of AlSi11 but the addition of Sb, In, and Fe changes the expansion only slightly. All elements but Fe improve pore structure and reduce drainage, especially Sb.

By means of in-situ X-ray expandometry it could be shown that AlSi11 with Sn addition shows also the maximal expansion after the first 100s of heating, but leads to an increased collapse 100s later at T = 700°C. This collapse is even more dramatic in the case of Fe additions. In contrast to this samples with Sb and In are very stable under the same conditions. The decreased coalescence rate together with the collapse measurement confirm the stabilisation effect of Sb and In. It was also shown that the typical early drainage of the thixocast process can be avoid with Sb additions.

5. Acknowledments

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

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