Influence of particle additions on the foaming behaviour of AlSi11/TiH$_2$ composites made by semi-solid processing

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Abstract

The influence of Al$_2$O$_3$ and SiC particles on the foaming behaviour of thixocast AlSi11 precursor material was investigated using mechanical and X-ray expandometry, image analysis of X-ray images and optical analysis of metallographic sections. A significant improvement of maximum expansion, collapse behaviour and pore structure could be obtained by addition of 1 to 5 wt.% particles, with higher efficiency of smaller particles. The role of the particles and the observed variations of efficiency are explained by differences in the way of particle agglomeration on or near cell wall surfaces.

Keywords: metallic foam, thixocasting, particles, foam stabilisation

1 Introduction

The mechanisms of metallic foam stabilisation are still not fully understood though the importance of particles for the stabilisation of cell walls is well known [1–3]. In the melt route ('MR') usually 10 to 30 wt.% of ceramic particles such as SiC or Al_2O_3 are added to an aluminium alloy melt to make it amenable to foaming. By sparging gas through this conditioned melt very uniform foams can be produced, but at the price of difficulties with machining and recycling caused by the presence of hard particles. In the powder metallurgical ('PM') route in which pressed mixtures of metal and blowing agent powders are foamed, the metal oxides contained in the powders are found to form networks in the foam, providing enough cell wall stabilisation without the need of additional particles [1,4,5]. It has been found that addition of non-metallic particles to the powder blends used in the PM process can improve stabilisation as long as compaction is not influenced in a negative way [6].

The thixocasting approach for producing foamable precursor material [7,8] is a hybrid of the MR and PM routes, comprising aspects of both: as for the traditional PM method it starts from powder mixtures but processing takes place in the semi-solid state. A foamable precursor is produced and foamed in a second step. Foam stabilisation relies on the presence of oxides on the surfaces of metal powders used as known from the PM process, but there is the possibility to use ceramic particle additions for further foam stabilisation following the ideas of MR processes. The aim of this study is to get to understand the influence of small additions (1 to 5 wt.%) of either SiC or Al_2O_3 particles on the foamability of precursors made by the thixocasting process. The influence of the particle size is of special interest, knowing that very small particles are difficult to include directly into a melt [9].

2 Experimental procedure

2.1 Preparation of precursors

Aluminium, silicon, blowing agent and ceramic powder were mixed, cold-isostatically compacted, heated into the semi-solid state and then thixocast to complex shaped precursor components as described in Ref. 10 in detail.

Aluminium (ECKA Granules, Al99.7%, <160 μm) and silicon powder (O. Rave 98.5%, <150 μm) were mixed in such fractions that a nominal composition AlSi11 was obtained. After this 1 wt.% TiH_2 (Chempur "grade N", purity 98%) and 0, 1, 3 or 5 wt.% of either SiC (SIKA,

black F1200, d_{50}=3.1±0.5 µm, or F500, d_{50}=12.3±1.0 µm) or Al_2O_3 (Martoxid MZS-1 d_{50}=1.5–1.9 µm, Martoxid MN/Y-212 d_{50}=10–15 µm, manufacturer's specifications) were added.

In the next step different powder mixtures were consolidated by means of cold isostatic pressing (CIP) to a cylindrical slug. Consolidation was carried out on a EPSI press (Engineered Pressure Systems International N.V.), with a pressure of 1500 bar (compaction time per slug approx. 2 min). The manufactured powder slugs had a density between 70 and 80% of the theoretical density. After the slugs had been held for a given time at a predefined temperature in a furnace, the then semi-solid slugs were transferred to the sleeve of a high pressure die casting machine (Bühler SC N/66) and were then cast. The speed of the plunger pressing the slug into the die cavity was 0.3 m/s, the compression pressure was 1500 bar.

The thixocast components obtained via this casting process were controlled visually and by X-ray radiography. The microstructure was characterised by metallographic analysis. Specimens for the various foaming experiments were cut out of the thixocast material: cuboids (25×25×15 mm³) for the free foaming tests, cylindrical tablets (diameter: 29 mm, height: 9 mm) for the expandometer tests and cuboids (20×10×5 mm³) for in-situ X-ray radioscopy.

2.2 Foaming and analysis of foams

Foamable precursors were foamed to the maximum volume without using a mould ('free foaming') at 750°C and were sectioned after. Pore macrostructures as well as the microstructure of the cell walls were evaluated. The thickness of the cell walls was measured using a Leica QWin V3 image analysis system. For the determination of the pore number and pore size, an analysis of macroscopic sections of specimens foamed to the maximum volume was carried out. All detected pores above 1 mm diameter were taken into account.

For the determination of the minimum cell wall thickness three very thin cell walls were selected in each sample. The thickness of these cell walls was measured using the image analysis system and the average value from these three measurements was taken as minimum cell wall thickness. Due to this procedure this is a semi-quantitative parameter only.

A mechanical expandometer was used to measure the expansion of the foamable precursors inside a cylindrical mould as a function of time and temperature [11]. The temperature of the expandometer furnace was kept constant at 750°C.

X-ray radioscopy was used to study in-situ the pore formation and foam evolution [8,12]. For this a X-ray transparent resistive heater described in detail in Ref. 13 was used. The samples ($20\times10\times5$ mm^3) were heated up to 650°C in approximately 75 s. This temperature was maintained constant for further 125 s, after which the power of the heater was switched off and the samples were allowed to cool down freely. All through the process X-ray images were taken with a time resolution of 1 s. The apparent cross section A of each sample was determined as a function of time from the X-ray images using an automatised image analysis routine. The relative area expansion A/A_0 with respect to the initial area A_0 was compared with the relative volume expansion V/V_0 obtained with the mechanical expandometer. Rupture of films in the liquid metal foam was detected automatically by analysing the sequence of images using the software 'AXIM' [12].

3   Results

Cold isostatic compaction of powder mixtures, heating to the semi-solid range and the following casting process could be carried out using the parameters given above without encountering any obvious problem. Casting quality as defined by the absence of unwanted porosity was considered very good as only very few pores were observed in radiographic X-ray images of the precursors and the density of the cast material was close to the theoretical density of aluminium. Demixing effects which sometimes can be observed in thixocast components were almost negligible as deduced from radiographic observations and metallographic analysis.

Fig. 1 gives the peak foam expansion of the various precursors as measured in the mechanical expandometer. Three samples were tested for each composition. A significant influence of particle type, particle content and particle size on the maximum expansion is observed. Increased particle content led to higher expansions as well for SiC as for Al_2O_3 with a more pronounced effect for Al_2O_3.

Expandometry showed that the collapse 30 s after maximum expansion was reduced for all specimens which contained particles, as demonstrated by the example in Fig. 2 for a sample containing Al_2O_3. Again, higher contents of particles proved to be more effective, whereas only slight differences between SiC and Al_2O_3 and no clear tendency for the influence of particle size could be found.

Analysis of the sectioned foams showed that the equivalent 2D pore radius was slightly reduced by increased contents of 3 μm Al_2O_3, 3 μm SiC and of 12 μm Al_2O_3 particles even though the maximum expansion was higher in comparison with specimens without particles (Fig. 3). This corresponds to a significant increase of the mean 2D pore numbers of the cross section, see Tab 1. Only increasing the content of 12 μm SiC particles to 5 wt.% led to a slight increase of the equivalent pore radius within the experimental error. Examples of pore structures of foams produced from precursor material with Al_2O_3 particles of different sizes are shown in Fig. 4.

X-ray radioscopic analysis of the foam structure shortly before solidification is shown in Fig. 5. Significant increase of expansion and reduction of pore size can be observed with increased amounts of Al_2O_3 particle addition. A similar effect was observed for SiC particles. A quantitative analysis of the cross-sectional expansion measured by in-situ radioscopy is shown in Fig. 6 for Al_2O_3 particles with a mean particle size of 12 μm. It can be seen that the expansion factor increases with the content of particles contained in the foam. Little collapse is observed in any of the particle-stabilised systems, unlike particle-free foams where the typical partial collapse after the expansion maximum is found. These findings are compatible with those obtained with the mechanical expandometer.

Utilising the possibilities of in-situ X-ray analysis we are able to compare the foam structure and density during the process in the liquid state at a fixed time just 80 s after starting heating (Fig. 7). The sample without particle additions (Fig. 7a) exhibits the typical early drainage frequently occurring in foams made by the thixocasting route [7]. This early drainage is reduced by the increasing amount of particles (Fig. 7b-d).

In Fig. 8 the number of cell wall rupture events up to a given time during foaming of AlSi11 samples containing different amounts of Al_2O_3 particles is shown. The coalescence activity is nearly the same or even higher for foams with particle additions. Image analysis of the minimum cell wall thickness showed that the addition of Al_2O_3 particles as well as SiC particles led to an increase of the wall thickness, see Table 2.

The added particles are predominantly situated at the cell walls, with partially wetting observed for both Al_2O_3 and SiC particles (Fig. 9). Furthermore, an interesting observation was that larger particles were mainly concentrated directly on the cell wall surface, whereas for smaller particles also an enrichment zone was found near the surface.

4   Discussion

Ceramic particle additions – both SiC and Al_2O_3 – to thixocast foamable precursors improve their foaming behaviour significantly. Maximum expansion of the foams could be improved (Fig. 1 and Fig. 6) without notably increasing the mean pore size (Fig. 3) as would happen if the blowing agent content were increased. This can be interpreted in terms of an enhanced stability of the liquid films in the foam which do not rupture so easily during pore inflation. Mass conservation requires that the mass in the individual cell walls and struts of the solidified foams is reduced in higher expanded foams, which is indeed observed. Due to the increased number of pores the accumulated absolute number of cell wall rupture events as shown in Fig. 8 is slightly increased for samples with higher particle contents. This is not a contradiction to the enhanced stability.

Due to the different efficiency of particles of different sizes to create a significant stabilisation effect the addition of >1 wt.% for small particles and >5 wt.% for larger particles is recommended. Such particle contents are rather low compared to the level of particle additions used for foam stabilisation in melt technologies such as the Alcan or Metcomb processes, where typically 10 to 15 vol.% are required [14]. This is not a contradiction since these foams are exclusively stabilised by ceramic particles, whereas the foams studied here owe their stability mainly to the oxide network stemming from the aluminium powder particles used as starting material. The significant influence of the particles on the level of maximum expansion also concurs with results of Kennedy et al. [6] for 3 wt.% of Al_2O_3, SiC and TiB_2 contained in uniaxially compacted aluminium/TiH_2 powder blends.

Nevertheless, the results of the experiments indicate that the addition of Al_2O_3 is more efficient than that of SiC in the present case. This assumption is corroborated by the results of the metallographic sections (Fig. 9) which show that SiC particles were more wetted than Al_2O_3 particles. In reference to the work of Aveyard et al. [2] this fact might explain the higher efficiency of Al_2O_3 in comparison to SiC. Aveyard et al. used spherical glass beads which had been silanised for the evaluation of the influence of the contact angle upon the foaming behaviour of soap foams. They found an optimal contact angle of approximately 90°. The larger influence of smaller particles might be attributed to the higher particle density (for the same wt.%-additions) at the cell wall surfaces.

Drainage in early stages of foaming is typical for precursors manufactured by the semi-solid processing route applied [10]. This is a potential drawback for the method since it creates an

unwanted non-uniform mass distribution in the foam. Particle additions help reducing this effect (Fig. 7) and are therefore beneficial. The strong drainage effect in some thixocast precursors has been explained by possible breaking and reordering effects on the oxide network in the powder compact caused by the shearing of the semi-solid material during thixocasting. Addition of ceramic particles helps to overcome this effect.

The various manufacturing methods for aluminium foams can be classified with respect to their stability: foams made from powder mixtures consolidated in the solid state by hot pressing or extrusion are stable due to the continuous oxide network present in the precursors [1,4]. Additional ceramic particles have an effect [6] but are not really essential. Foams made from precursors prepared by semi-solid processing are less stable and ceramic particle additions can be beneficial or perhaps are even essential. Foams made by injecting gas into liquids owe all their stability almost entirely to ceramic particles since few oxides are present and consequently a high volume fraction of added particles is needed.

For all kinds of investigated particles an increase of the film thickness was observed. This is in agreement with observations and models in the literature [4]. Higher contents of particles led to thicker films. The fact that particle additions result at the same time in larger expansion, increased number of pores and larger film thicknesses seems at the first glance contradictory. However, this observation can be explained by the large size of some of the films in which very small wall thicknesses could be found beside rather thick regions and also by the reduction of the volume of the struts. Foams with increased particle contents possess more angular shaped pores than foams with low particle contents.

5   Summary

Small additions of Al_2O_3 and SiC particles proved to be advantageous for the foaming behaviour and the stability of thixocast AlSi11 precursors. The height of maximum expansion and the number of pores increased, whereas the level of drainage and collapse were reduced. The efficiency of the particle additions increased with higher particle content and smaller particle size. Particles concentrated near or directly on the cell wall surfaces. SiC is more wetted than Al_2O_3, expressed by a deeper immersion of SiC particles into the metallic film. The role of the particles in foam stabilisation cannot be attributed to only one mechanism. Several effects such as the occurrence of thin film thicknesses over large areas and the reduction of drainage are important for the overall foam stabilisation efficiency of the particles.

Acknowledgements

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TABLES

Table 1: Number of pores in 2D sections of AlSi11 foams (maximum expansion) with or without particle additions (averaged over 3 specimens for each composition).

particle type	none	Al_2O_3						SiC					
		12 µm			3 µm			12 µm			3 µm		
%	0	1	3	5	1	3	5	1	3	5	1	3	5
number of pores	76	84	130	128	104	141	185	74	92	77	80	119	165
min.	62	82	120	122	85	134	171	62	81	67	73	113	148
max.	88	89	140	131	115	146	193	80	102	87	91	123	192

Table 2: Minimum cell wall thickness of AlSi11 foams with or without particle additions.

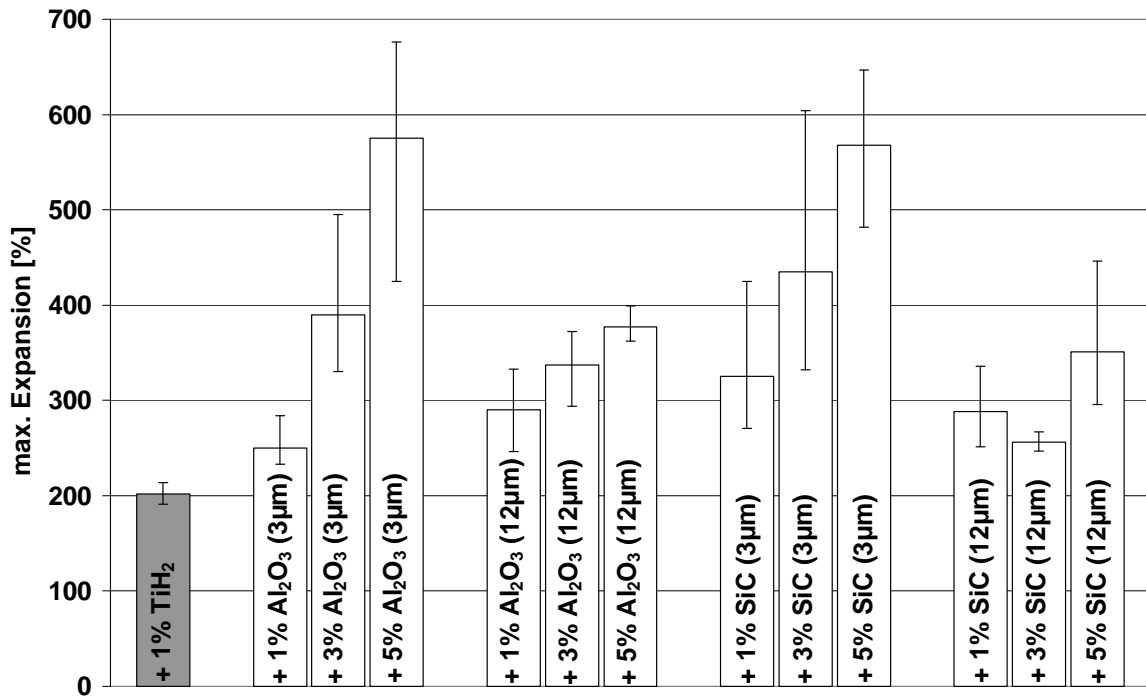|	$AlSi11+1\%TiH_2 +$								
	-	1% Al_2O_3 (12 µm)	5% Al_2O_3 (12 µm)	1% Al_2O_3 (3 µm)	5% Al_2O_3 (3 µm)	1% SiC (12 µm)	5% SiC (12 µm)	1% SiC (3 µm)	5% SiC (3 µm)
min. wall thickness [µm]	49	80	53	54	67	63	64	61	91

FIGURES

Fig. 1: Peak expansion of AlSi11-precursors measured in the mechanical expandometer. Precursors containing different amounts (given in wt.%), types and sizes of ceramic particles were tested and compared to a sample without particles (grey bar). For each composition the minimum, maximum and mean expansion of three individual measurements are given. All precursors contained 1 wt.% TiH_2.
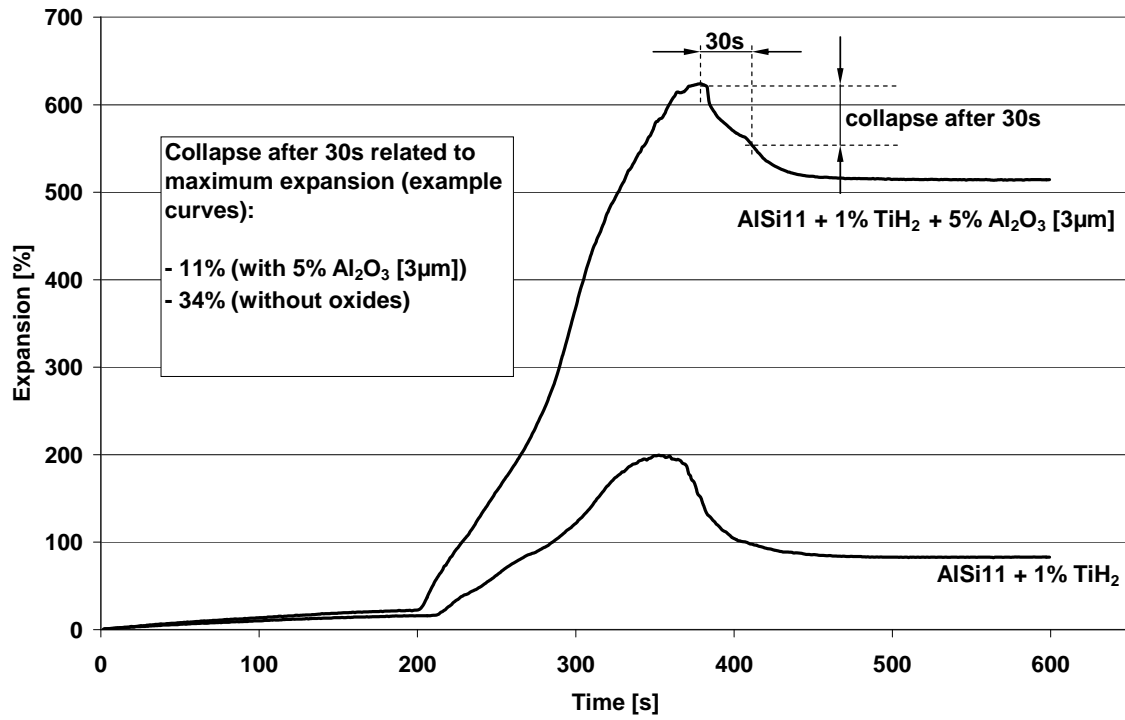
Fig 2: Description of the collapse behaviour of metal foams demonstrated by two expansion curves corresponding to samples with and without additional particles.
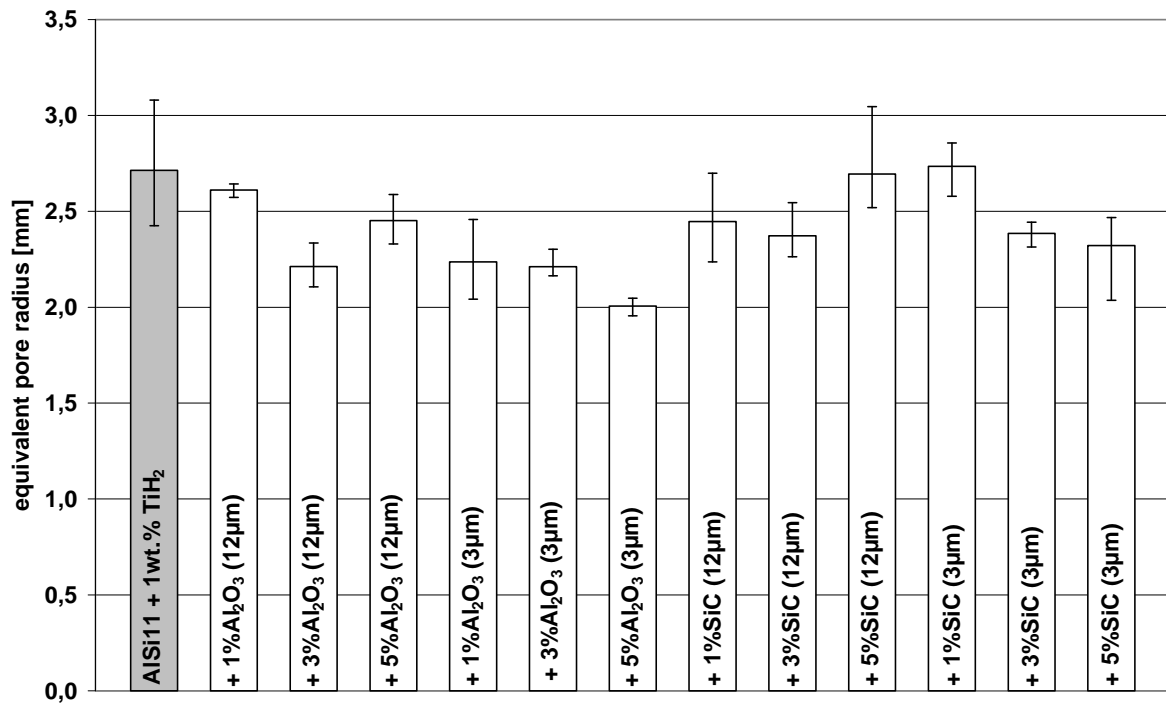
Fig. 3: Equivalent pore radius of foams (maximum expansion) made of AlSi11-precursors containing different amounts, types and sizes of ceramic particles. Gray bar gives data for precursor without particles. For each composition the minimum, maximum and mean expansion of three individual measurements are given. All precursors contained 1 wt.% TiH$_2$.
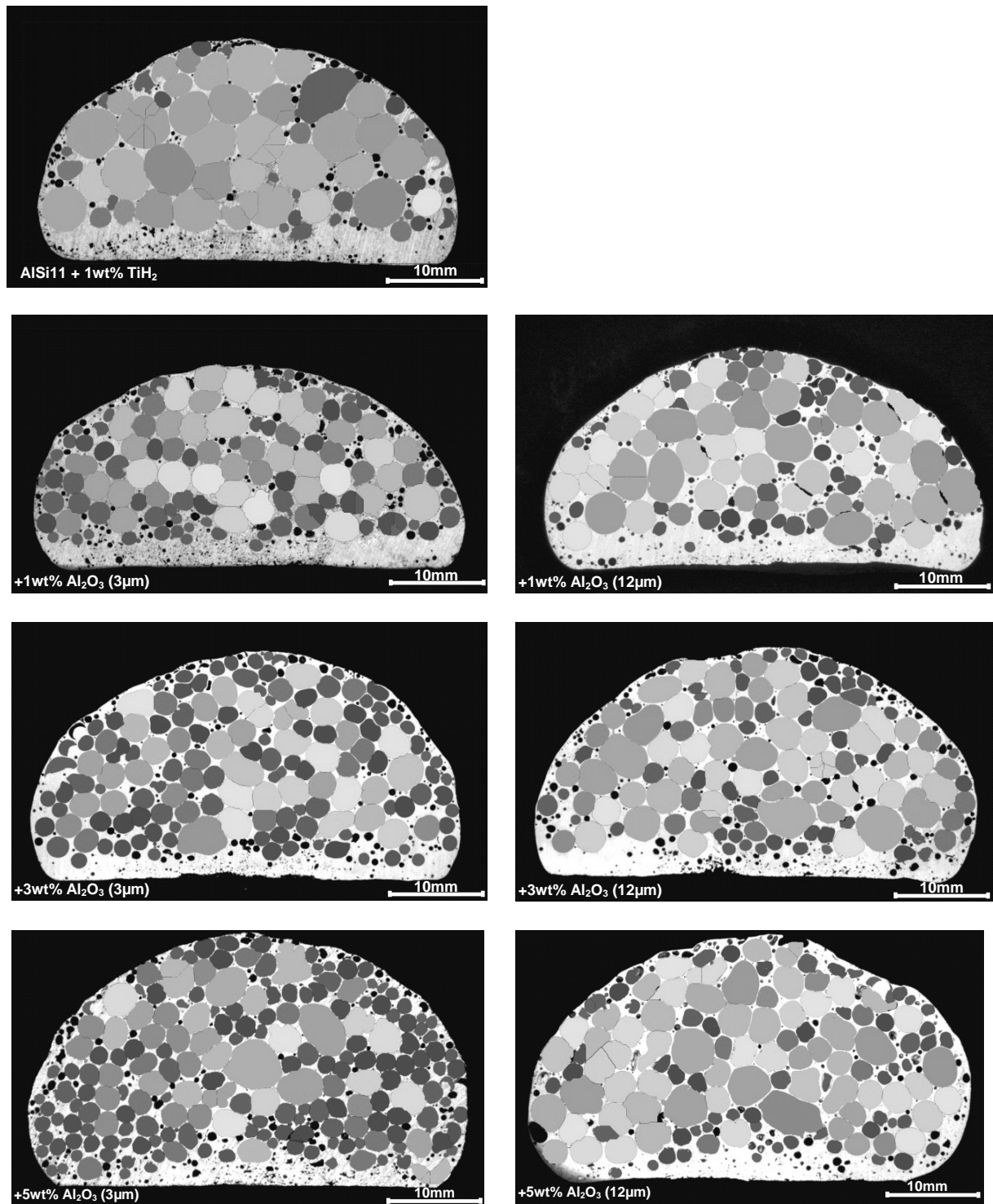
Fig. 4: Comparison of the pore structure of foams made from precursors with and without particles (maximum expansion). Different pore size ranges were labelled by different grey scales by the image analysis system.
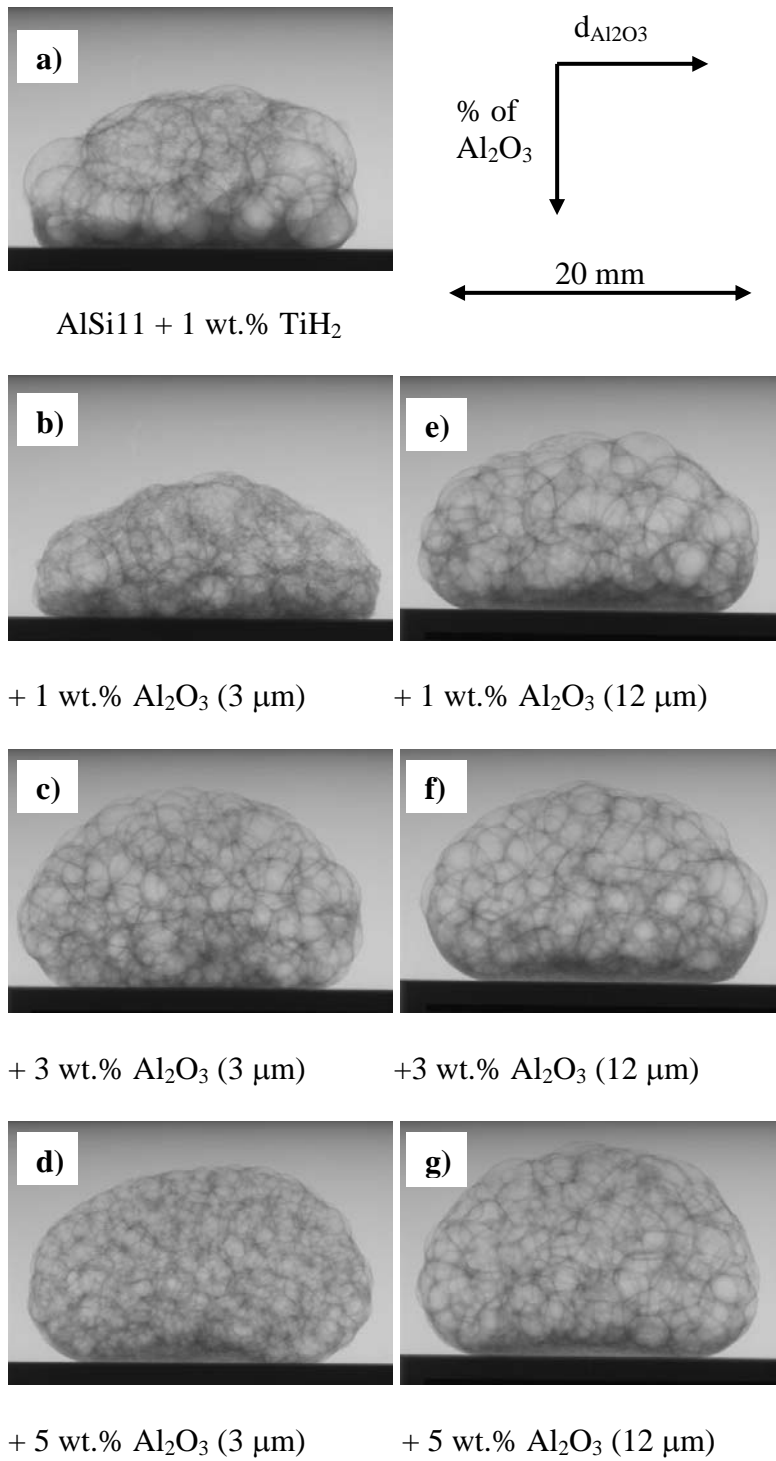
a)

d_{Al2O3}

% of
Al_2O_3

20 mm

AlSi11 + 1 wt.% TiH_2

b)

+ 1 wt.% Al_2O_3 (3 μm)

e)

+ 1 wt.% Al_2O_3 (12 μm)

c)

+ 3 wt.% Al_2O_3 (3 μm)

f)

+3 wt.% Al_2O_3 (12 μm)

d)

+ 5 wt.% Al_2O_3 (3 μm)

g)

+ 5 wt.% Al_2O_3 (12 μm)

Fig. 5: X-ray radioscopic images of AlSi11 + 1 wt.% TiH_2; a) without, b-g) with particle additions. State after 200 s of foaming is shown.
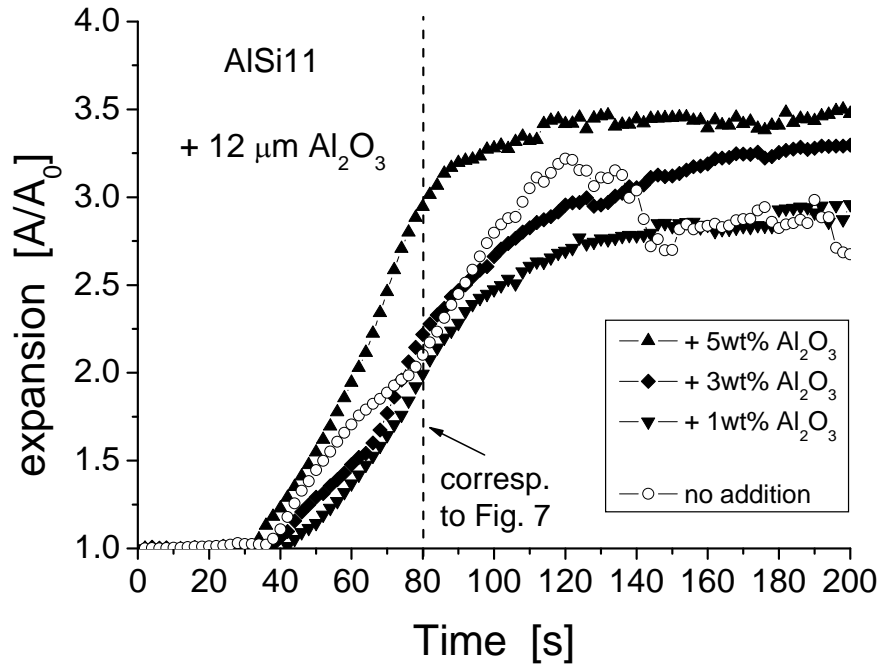
Fig. 6: Time evolution of relative cross section A/A_0 of AlSi11 foams containing different amounts of 12 μm Al_2O_3 particles. Data derived from X-ray radioscopic image sequences.
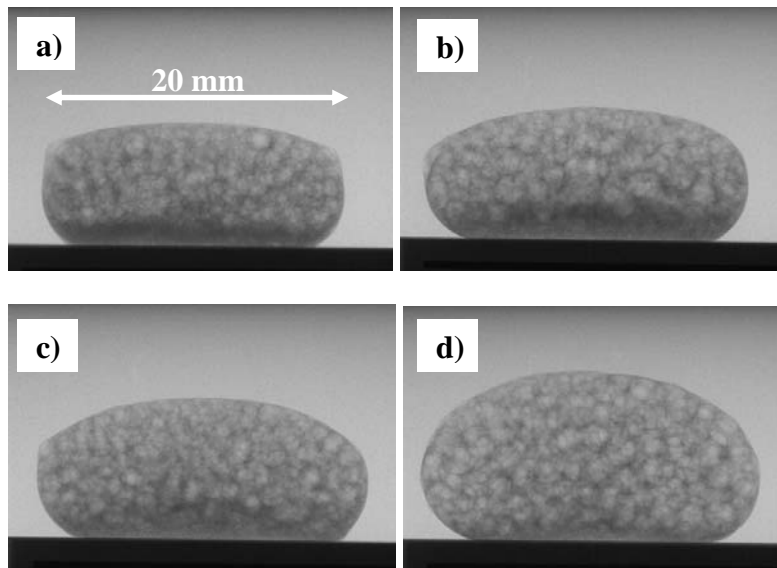
Fig. 7: X-ray radioscopic images of AlSi11 + 1 wt.% TiH_2, a) without particles, b) + 1 wt.%, c) + 3 wt.% and d) + 5 wt.% Al_2O_3 (12 μm). State 80 s after begin of heating is shown.
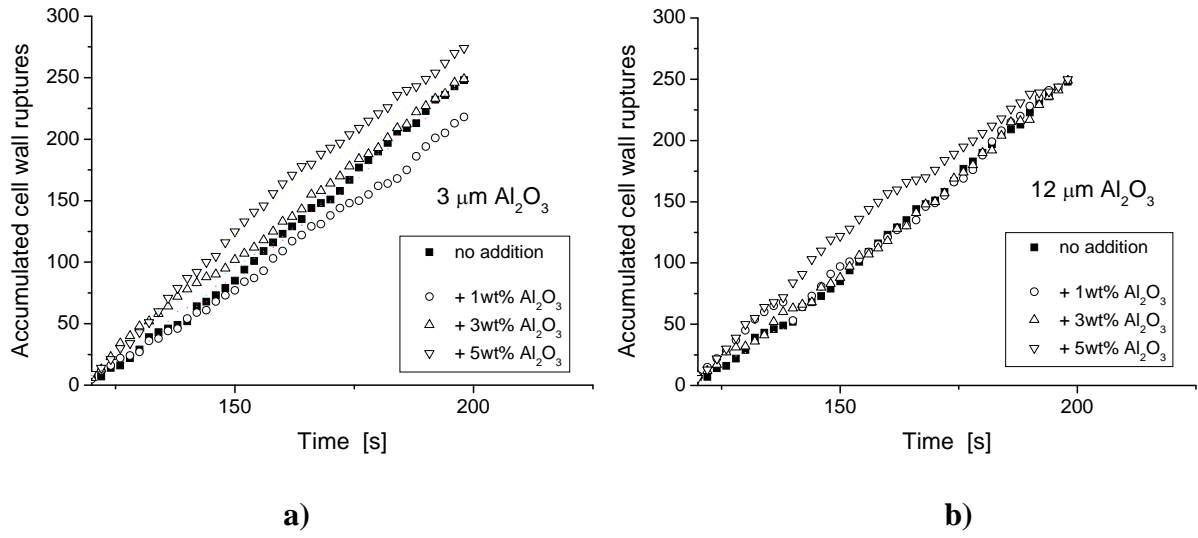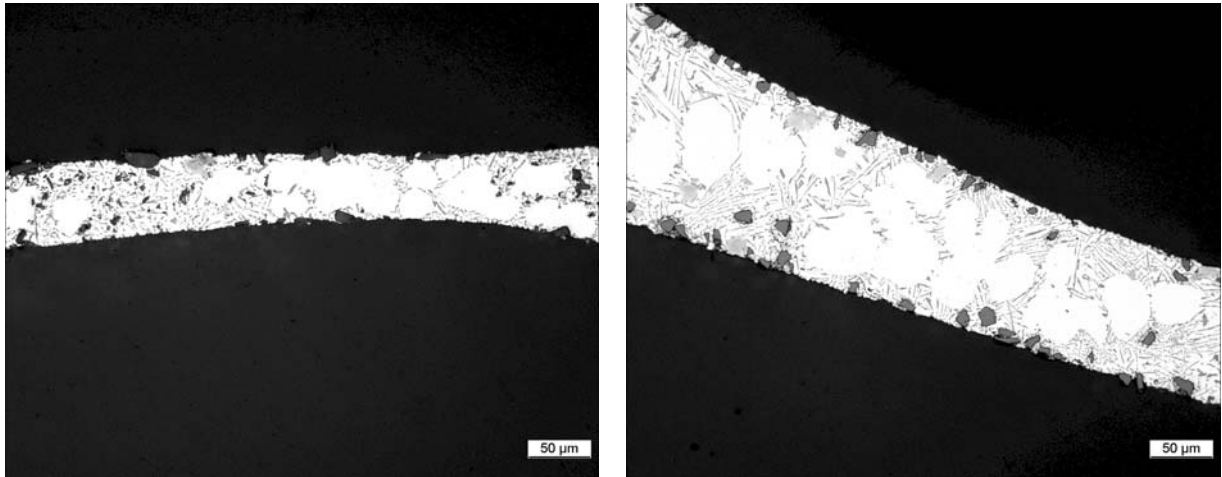
Fig. 8: Time dependence of the number of accumulated cell wall rupture events in AlSi11 samples containing different amounts of Al_2O_3 particles. Particle mean sizes a) 3 μm and b) 12 μm.

a)   b)

Fig. 9: Micrographs of cell walls of AlSi11 foams containing, a) Al$_2$O$_3$ or b) SiC particles (mean particle size 12 μm in both cases).