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INFLUENCE OF THE FOAMING PRECURSOR'S COMPOSITION AND DENSITY ON THE FOAMING EFFICIENCY, MICROSTRUCTURE DEVELOPMENT AND MECHANICAL PROPERTIES OF ALUMINIUM FOAMS

VPLIV SESTAVE IN GOSTOTE PREKURZORJEV ZA PENJENJE NA UČINKOVITOST PENJENJA TER RAZVOJ MIKROSTRUKTURE IN MEHANSKIH LASTNOSTI ALUMINIJSKIH PEN

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In this work, the influence of the composition, density and porosity of foaming precursors on the foaming efficiency, microstructure development and mechanical properties of aluminium foams are presented and discussed. The foams were prepared, starting from precursors made either by powder metallurgy (PM) or by the melt route. Following the PM route, precursors were made by mixing Al powder and 3–10 % of volume fractions of dolomite or calcium carbonate particles of particle size from 20 μm to 120 μm and cold isostatically pressing the mixture at 700 MPa. In the case of the melting route, precursors were made by introducing dolomite or calcium carbonate particles directly into the molten aluminium at 700 °C. After melt stirring, the precursors were prepared by casting the semi-solid slurry into a cylindrical, water-cooled mould.
Finally, aluminium foams were made in all cases by inserting precursors into a cylindrical stainless-steel mould and heating the arrangement at 750 °C for 10 min. After that, the mould was removed from the furnace and the foaming process was stopped by cooling in air to room temperature.
The microstructure of the obtained foams was investigated by optical and scanning electron microscopy (SEM-EDS), while XRD was applied for a detailed identification of phases.
The quality of the precursors was evaluated by determining their mechanical properties (uniaxial room-temperature compression stress-strain curve, compressive strength and energy absorption after a 30 % strain) and the foaming efficiency (the relative density of the foam obtained). The concentration of the foaming agent and the density of precursors were found to have a detrimental influence on the foaming efficiency as well as on the foam's microstructure and mechanical properties. The foaming of precursors with open porosity were inefficient.

Key words: aluminium foams, foaming agents, calcium carbonate, dolomite, characterization

V delu poročamo o vplivu sestave ter gostote oz. poroznosti prekurzorja za penjenje na učinkovitost penjenja ter razvoj mikrostrukture in mehanskih lastnosti aluminijevih pen. Pene smo izdelovali z uporabo prekurzorjev na osnovi Al s homogeno porazdeljenimi delci dolomita ali kalcijevega karbonata. Prekurzorje smo pripravljali po postopku prašne metalurgije (PM) in z litjem taline, kar je cenejše, zagotavlja pa manj homogeno porazdelitev sredstva za penjenje. S PM-postopkom smo prekurzorje za penjenje izdelovali iz zmesi Al prahu in 3–10 % dolomita ali kalcijevega karbonata različne povprečne velikosti (od 20 μm do 120 μm), ki smo jo izostatsko stisnili pri 700 MPa. Z litjem smo prekurzorje pripravljali tako, da smo delce penila uvajali v Al-talino, segreto do največ 700 °C, premešali in nastalo suspenzijo ulili v cilindričen, vodno hlajen jekleni model. Pene smo iz obeh vrst prekurzorjev izdelovali tako, da smo prekurzor vstavili v zaprt jeklen model za penjenje, segrevali pri 750 °C 10 min. ter nato ohlajali na zraku do sobne temperature.
Mikrostrukturo nastalih pen smo preučevali z optično in elektronsko (SEM/EDS) mikroskopijo, in sicer tako, da smo ugotavljali morfologijo in povprečno velikost por, sestavo vsebovanih faz pa z metodo rentgenske (XRD) difrakcije. Gostoto prekurzorjev in pen smo določali na osnovi mase in izračunane prostornine strojno obdelanih vzorcev, učinkovitost penjenja (relativno gostoto dobljene pene) pa na osnovi primerjave dejansko dosežene gostote pene in gostote aluminija. Primerjalno smo gostoto pen določali tudi z Arhimedovo metodo.
Kakovost izdelanih pen smo ocenjevali na osnovi njihovih mehanskih lastnosti (krivulje napetost – deformacija pri sobni temperaturi, tlačne trdnosti in sposobnosti absorpcije energije pri 30-odstotni deformaciji). Ugotovili smo, da na učinkovitost penjenja ter razvoj mikrostrukture in mehanskih lastnosti vplivata predvsem kemijska sestava in gostota prekurzorja za penjenje, pri čemer je bilo prekurzorje z odprto poroznostjo nemogoče peniti.

Ključne besede: aluminijske pene, sredstvo za penjenje, kalcijev karbonat, dolomit, karakterizacija

1 INTRODUCTION

Closed-cell-foamed aluminium is a macro-composite material consisting of an aluminium alloy matrix, usually discontinuously reinforced with various ceramic particulates and closed pores filled with gas distributed throughout the matrix. This unique structure possesses an unusual combination of properties, such as a low density, a high weight-specific stiffness, extraordinary energy absorption and a remarkable vibration attenuation, which are important for a number of engineering applications. Aluminium foams are also non-flammable, ecologically harmless and easily recyclable.

Methods to produce closed-cell aluminium foams have been known for a number of years and can be generally separated into two fundamental groups: (i) foaming liquid metal (casting procedures) and (ii) foaming metallic precursors (powder metallurgy-PM procedures)[1].

Titanium hydride (TiH_2) was mainly applied as a blowing agent for both the casting and powder metallurgical procedures of foaming for aluminium and aluminium alloys. The role of the blowing agent in both – the casting and powder metallurgical procedures of foaming of aluminium – is to release gas, since the metal is transferred in the liquid or the semi-liquid viscous state. However, as discussed in detail by Gergely et al.[2], several factors are involved in the selection of a blowing agent for the foaming of aluminium alloys. These include: (i) the kinetics and thermodynamic characteristics of decomposition reactions and reactions between the blowing agent particles and the molten or semi-molten aluminium alloy; (ii) the wetting of foaming-agent particles with molten metal; (iii) the influence of decomposition products and foaming gas on the stabilisation of foams; and (iv) the availability, cost and user-friendly handling of the powdered agent concerned.

As a foaming agent for the production of aluminium alloy foams by the casting and powder metallurgical routes, TiH_2 has several limitations that significantly influence the mass application of Al foams. The main limitations of the foaming technologies using TiH_2 as a foaming agent are the following:

- TiH_2 is very expensive.
- The decomposition temperature of TiH_2 is very low – starting at about 400 °C for the untreated hydride.
- TiH_2 particles act only as a blowing agent and are not involved in the foam stability.
- The density of TiH_2 (approx. 3.9 g/cm^3) is significantly higher than the density of molten aluminium (approx. 2.7 g/cm^3). Thus, during foaming the settling of TiH_2 particles occurs by gravity, resulting in a non-uniform microstructure of the foam.

The cost-effective and highly promising alternatives to the TiH_2 blowing agent are $CaCO_3$, as a marble powder or synthetic calcium carbonate, and dolomite powder. In contrast to TiH_2, calcium carbonate and dolomite are of low cost, significantly lower than the cost of aluminium, and with a density (2.71 g/cm^3 to 2.84 g/cm^3) almost identical to the density of molten aluminium. Moreover, the $CaCO_3$ and dolomite decomposition temperatures are above the melting point of aluminium, usually in the temperature interval between 660 °C and 930 °C. Therefore, $CaCO_3$ and dolomite are particularly suitable for the melt-route, settling-free production of foamed aluminium-based materials.

The "Foamcarp" process of the indirect foaming of aluminium using $CaCO_3$ particles previously introduced into a solidified precursor and, more recently, an additional indirect foaming procedure of molten aluminium with a $CaCO_3$-containing preform isostatically pressed from a mixture of $CaCO_3$ and AA6061 powders were reported[2,3].

In some early patents[4] and in the recent work of Nakamura et al.[5], the use of $CaCO_3$ was found to be potentially suitable as a foaming agent for direct (melt-route) foam manufacturing. Moreover, Bryat et al.[6] reported that $CaCO_3$ acts in contact with the molten aluminium as a foaming agent and, at the same time, through the decomposition products has a significant effect on the foam stabilisation, enabling the formation of "self-stabilized aluminium foams" (i.e., foams solidified from a foamable suspension created through the controlled decomposition of carbonate powders with molten aluminium). A similar cell face stabilising mechanism operating in carbonate-foamed melts was also reported by Gergely et al.[2].

The additional advantage of $CaCO_3$ in comparison with a TiH_2 blowing agent of the same average particle size is in achieving foams with higher porosity levels and finer cell sizes[5]. Gergely et al.[2] showed that by replacing TiH_2 by $CaCO_3$ as a foaming agent, foams can be produced having appreciably finer cells and more uniform cell structures at a significantly reduced raw-material cost.

On the other hand, Alcoa patented[7] a method of making an aluminum foam product by adding reactive gas producing particles to a molten aluminum alloy at a temperature that is above the decomposition temperature of the reactive-gas producing particles. The reactive-gas producing particles are selected from the group consisting of magnesium carbonate, calcium carbonate, dolomite and mixtures thereof.

Although the applicability of $CaCO_3$ and dolomite powders as a foaming agent in the direct and indirect foaming of aluminium alloys and composites has already been investigated, there is a need for an additional study of the influence of the $CaCO_3$ and dolomite powder morphology (particularly the average particle size and particle size distribution) and the role of $CaCO_3$ and dolomite decomposition products on the achievement of a better foam stability and a higher foam quality.

Hence, in this paper, the performance of synthetic calcium carbonate and natural dolomite powders as

cost-effective foaming agents was investigated by both the powder metallurgical and the melt route of foam preparation. The influence of $CaCO_3$ and dolomite particle morphology and volume fraction on the foaming behaviour and the development of the foam microstructure were also monitored. As a result, a clearer interrelation between the foam morphology and structure, on the one hand, and its mechanical properties, on the other, was established.

2 EXPERIMENTAL

All the foams made in this work were prepared by indirect foaming methods starting from a solid foamable precursor consisting of a metallic matrix containing uniformly dispersed blowing-agent particles. Foamable precursors were made either by: (i) the powder metallurgical route, or (ii) the melt route using the same blowing agent, i.e., $CaCO_3$ powders (types C-A, C-B and C-C) with various average particle sizes ((38, 72 and 120) μm, respectively) and dolomite powders (type D-A, D-B and D-C) with various average particle sizes ((44, 76 and 97) μm, respectively).

Following the powder metallurgical (PM) route, foamable precursors with $CaCO_3$ particles were made by mixing Al powder with an average particle size of 63 μm (purity (mass fractions, *w*: 99.7 %, oxygen content: 0.25 %) and (3, 5, 7 and 12) % of the blowing agent, followed by cold compaction in a lubricated 20-mm diameter die to a pressure of 600 MPa to 900 MPa.

In the case of the melt route, foamable precursors with the same concentration of $CaCO_3$ blowing agent ((3, 5, 7 and 12) %) and the same geometry were prepared by an induction-heated, batch-type, stir-casting method by which the aluminium powder (the same as used for the PM route) was induction melted, followed by the addition of $CaCO_3$ particles, stirring and casting. Once the molten aluminium had reached 750 °C, the power was switched off and the melt stirring was initiated until the temperature of the melt decreased to 700 °C. After that, the blowing agent/aluminium powder mixture (1 : 2 mass ratio) was introduced and the melt was stirred (at approximately 1200 r/min) for an additional 30–90 s. Finally, the foamable precursors were prepared by casting the semi-solid slurry into a room-temperature mould with a diameter of 20 mm.

In the case of the powder metallurgy (P/M) route, the fabrication of foamable precursors with dolomite particles was conducted by mixing Al powder with an average particle size of 63 μm (purity: 99.7 %, oxygen content: 0.25 %), 5 % of SiC particles with an average particle size of 10 μm and (3, 5, 7 and 12) % of blowing agent, followed by cold compaction, in a lubricated 20-mm diameter die to a pressure of 600 MPa to 900 MPa.

Following the melt route, foamable precursors with the same concentration of dolomite blowing agent ((3, 5, 7 and 12) %) and the same geometry were prepared by induction-heated, batch-type, stir-casting in which aluminium powder (the same as used for the P/M route) was induction melted, followed by the addition of dolomite particles, stirring and casting. Once the molten aluminium was heated to 700 °C, the power was switched off and melt stirring was initiated until the temperature of the melt decreased to 685 °C. After that, the blowing agent/aluminium powder mixture (1 : 2 mass ratio) was introduced and the melt was stirred (at approximately 1200 r/min) for an additional 30–90 s. Finally, the foamable precursors were prepared by casting the semi-solid slurry into a room-temperature mould with 20-mm diameter.

The solidified precursors were machined and some of the samples were additionally cold isostatically pressed. The density of the foamable precursors as well as of the foams obtained was calculated from the mass and geometry of the samples and, in addition, measured by Archimedes' method. The distribution of the blowing-agent particles inside the Al matrix was examined by an assessment of the optical and scanning electron micrographs of as-polished bars.

All the precursors were foamed in a conventional batch furnace with air atmosphere circulation under the same experimental conditions (temperature, time, cooling method). Before foaming, the individual precursors were inserted into a cylindrical (40-mm diameter, 70-mm long) stainless-steel mould coated with a boron nitride suspension. The mould dimensions and the precursor size (20-mm diameter and 60-mm long) were selected to allow the expansion of the precursor to a foam with a theoretical density close to 0.6 g/cm^3. The arrangement was placed inside a pre-heated batch furnace at 750 °C for 10 min. After that period of time, the mould was removed from the furnace and the foaming process was stopped by rapid cooling with pressurised air to room temperature. The thermal history of the foam sample was recorded, using a thermocouple located directly in the precursor material.

The porosity of the foam was calculated using equation: 1 – (foam density/ aluminium density). Macro and microstructural examinations were performed on sections obtained by precision wire-cutting across the samples and on samples mounted in epoxy resin using optical and scanning electron microscopy (SEM-EDS).

The average size of the pores in the foams was estimated by an assessment of the optical and scanning electron micrographs of as-polished foam bars using the point-counting method and image-analysis and processing software.

Regarding the mechanical properties of the foams, uniaxial room-temperature compressive tests were carried out on a Zwick 1474 testing machine at a constant 5-mm/min crosshead displacement. Testing was performed on standard prismatic foam specimens of 50 mm × 12 mm × 17 mm and each point of the stress-strain

curve was determined as an average of four individual measurements. Compression was stopped whenever either 80 % strain or 95 kN force (equivalent to 61.9 MPa) was reached. As a result of testing, the uniaxial compression stress-strain curve, compressive strength and energy absorption after a 30 % strain were determined and correlated with the density, the average pore size and microstructure of the foam samples.

3 RESULTS AND DISCUSSION

3.1 Characterisation of the foamable precursors

The measured and calculated densities of the foamable precursors obtained by the PM route, **Table 1a,b**, confirmed that under isostatic pressing with an applied pressure of 700 MPa the precursors prepared by PM possessed a closed porosity and densities above 98 % of theoretical, whereas as-machined precursors obtained by the melt route (**Table 2a,b**) had a significant fraction of open porosity and thus were not suitable for foaming to the desired foam densities (usually about 0.5 g/cm^3 to 0.7 g/cm^3). However, after additional isostatic pressing, the porosity in these precursors was successfully reduced to below 2.0 % (volume fraction φ/%) (**Table 3 a,b**) and foam samples with densities between 0.62 g/cm^3 and 0.80 g/cm^3 were obtained.

It is important to note that very high precursor densities (>99 % of theoretical) were achieved only in the precursors prepared by the powder metallurgical route with 3–7 % of $CaCO_3$ and dolomite particles of Type-C-A or Type D-A (**Table 1 a,b**). With a higher particle content, and by using coarser $CaCO_3$ or dolomite powders of Type-C-B or Type-C-C as well as Type D-B and Type D-C, this could not be achieved and resulted in a lower foaming efficiency, as evident in **Tables 4 a,b–6 a,b**.

The foaming efficiency of the precursors was evaluated from the relative density of the foam obtained, ρ, calculated by dividing the apparent density of the foam, ρ_F, by the density of aluminium, ρ_{Al}. Thus, the foaming efficiency is expressed as:

$$\eta = 1 - \rho = 1 - (\rho_F/\rho_A) \quad (1)$$

which actually corresponds to the volume fraction of pores in the foam samples. The lower the foam density, the higher is the foaming efficiency.

In all cases the experimental results clearly indicate that the porosity measured in foamable precursors and the apparent density achieved in aluminium foam samples are inversely proportional. Generally, foamable precursors with a lower porosity resulted in foam samples with a higher apparent density and a lower foaming efficiency.

Under the same foaming conditions (temperature, time), the average pore size of the foam samples was influenced by the density of foaming precursors and the initial size of the foaming particles. As a rule, in foams made from precursors with high density (≥99 % of theoretical), the average pore size remained below 1.0 mm. On the other hand, in foams made from precursors with a lower density (below 99 % of theoretical), the pores grew to a 20 % to 50 % higher average size pore.

Regarding the initial size of the foaming particles, which also influences the density of the precursor and, hence, the density of the foam samples, the increase of the average particle size of $CaCO_3$ or dolomite foaming agent was observed to have a detrimental influence on the average size of the pores. Coarser $CaCO_3$ and dolomite powders led to the formation of larger bubbles in the foam structure.

Table 1 a: Porosity of $CaCO_3$ particles containing foamable precursors obtained by the PM route

Tabela 1 a: Poroznost prekurzorjev s $CaCO_3$-penilom, izdelanih s postopkom prašne metalurgije

Chemical composition of precursors (w/%)		Porosity (φ/%)	
$CaCO_3$	Al powder	Calculated	Measured
Type C-A			
3	97	0.8 ± 0.08	0.7 ± 0.04
5	95	0.8 ± 0.08	0.8 ± 0.04
7	93	0.9 ± 0.09	1.0 ± 0.05
10	90	1.1 ± 0.11	1.2 ± 0.06
Type C-B			
3	97	1.1 ± 0.10	1.0 ± 0.05
5	95	1.1 ± 0.11	1.1 ± 0.06
7	93	1.3 ± 0.13	1.3 ± 0.07
10	90	1.7 ± 0.17	1.8 ± 0.09
Type C-C			
3	97	1.3 ± 0.11	1.4 ± 0.07
5	95	1.5 ± 0.15	1.4 ± 0.07
7	93	1.6 ± 0.16	1.7 ± 0.09
10	90	1.8 ± 0.18	2.0 ± 0.10

Table 1 b: Porosity of dolomite particles containing foamable precursors obtained by the PM route.

Tabela 1 b: Poroznost prekurzorjev z dolomitom kot penilom, izdelanih po postopku prašne metalurgije

Chemical composition of performs w/%			Porosity ρ/%	
Dolomite	SiC	Al powder	Calculated	Measured
Type D-A				
3	5	92	0.7 ± 0.07	0.7 ± 0.04
5	5	90	0.8 ± 0.08	0.8 ± 0.04
7	5	88	0.9 ± 0.09	1.0 ± 0.05
10	5	85	1.2 ± 0.12	1.3 ± 0.07
Type D-B				
3	5	92	1.0 ± 0.10	1.0 ± 0.05
5	5	90	1.0 ± 0.10	1.1 ± 0.06
7	5	88	1.2 ± 0.12	1.3 ± 0.07
10	5	85	1.6 ± 0.16	1.8 ± 0.09
Type D-C				
3	5	92	1.1 ± 0.11	1.1 ± 0.06
5	5	90	1.2 ± 0.12	1.3 ± 0.06
7	5	88	1.3 ± 0.13	1.4 ± 0.07
10	5	85	1.8 ± 0.18	2.0 ± 0.10

Table 2a: Porosity of as-machined $CaCO_3$ containing foamable precursors prepared by the melt route

Tabela 2a: Poroznost strojno obdelanih prekurzorjev s $CaCO_3$ penilom, izdelanih po livarskem postopku

Chemical composition of precursors (*w*/%)		Porosity (φ/%)	
$CaCO_3$	Al powder	Calculated	Measured
Type C-A			
3	97	4.2 ± 0.42	4.1 ± 0.21
5	95	4.6 ± 0.46	4.4 ± 0.22
7	93	4.9 ± 0.49	4.8 ± 0.24
10	90	5.3 ± 0.53	5.2 ± 0.26
Type C-B			
3	97	4.1 ± 0.41	4.2 ±0.21
5	95	4.7 ± 0.47	4.7 ± 0.24
7	93	5.0 ± 0.50	5.1 ± 0.26
10	90	5.4 ± 0.54	5.5 ± 0.28
Type C-C			
3	97	3.9 ± 0.39	4.1 ± 0.21
5	95	4.2 ± 0.42	4.2 ± 0.21
7	93	4.3 ± 0.43	4.4 ± 0.22
10	90	4.8 ± 0.48	4.9 ± 0.25

Table 2b: Porosity of as-machined dolomite particles containing foamable performs prepared by the melt route.

Tabela 2b: Poroznost strojno obdelanih prekurzorjev z dolomitom kot penilom, izdelanih po livarskem postopku.

Chemical composition of performs *w*/%			Porosity ρ/%	
Dolomite	SiC	Al powder	Calculated	Measured
Type D-A				
3	5	92	4.7 ± 0.47	4.9 ± 0.25
5	5	90	4.9 ± 0.49	5.1 ± 0.26
7	5	88	5.4 ± 0.54	5.8 ± 0.29
10	5	85	6.1 ± 0.61	6.6 ± 0.33
Type D-B				
3	5	92	4.5 ± 0.45	4.7 ± 0.24
5	5	90	4.7 ± 0.47	5.0 ± 0.25
7	5	88	5.0 ± 0.50	5.3 ± 0.27
10	5	85	5.7 ± 0.57	6.2 ± 0.31
Type D-C				
3	5	92	4.4 ± 0.44	4.5 ± 0.23
5	5	90	4.5 ± 0.45	4.7 ± 0.24
7	5	88	4.7 ± 0.47	5.0 ± 0.25
10	5	85	4.9 ± 0.49	5.3 ± 0.27

Table 3a: Porosity of $CaCO_3$ containing foamable precursors obtained by the melt route improved by additional isostatic pressing

Tabela 3a: Poroznost prekurzorjev s $CaCO_3$-penilom, izdelanih po livarskem postopku, izboljšana z dodatnim hladnim izostatskim stiskanjem

Chemical composition of precursors (*w*/%)		Porosity (φ/%)	
$CaCO_3$	Al powder	Calculated	Measured
Type C-A			
3	97	1.3 ± 0.13	1.3 ± 0.06
5	95	1.6 ± 0.16	1.7 ± 0.09
7	93	1.9 ± 0.19	2.0 ± 0.10
10	90	2.0 ± 0.20	2.2 ± 0.11
Type C-B			
3	97	1.1 ± 0.11	1.2 ± 0.06
5	95	1.2 ± 0.12	1.2 ± 0.06
7	93	1.4 ± 0.14	1.5 ± 0.08
10	90	1.7 ± 0.17	1.9 ± 0.09
Type C-C			
3	97	1.0 ± 0.10	1.0 ± 0.10
5	95	1.2 ± 0.12	1.1 ± 0.06
7	93	1.3 ± 0.13	1.4 ± 0.07
10	90	1.6 ± 0.16	1.8 ± 0.09

Table 3b: Porosity of dolomite particles containing foamable performs obtained by the melt route improved by additional isostatic pressing

Tabela 3b: Poroznost prekurzorjev z dolomitom kot penilom, izdelanih po livarskem postopku, izboljšana z dodatnim hladnim izostatskim stiskanjem

Chemical composition of performs *w*/%			Porosity ρ/%	
Dolomite	SiC	Al powder	Calculated	Measured
Type D-A				
3	5	92	0.9 ± 0.09	0.9 ± 0.05
5	5	90	0.9 ± 0.09	0.9 ± 0.05
7	5	88	1.0 ± 0.10	1.1 ± 0.06
10	5	85	1.1 ± 0.11	1.2 ± 0.06
Type D-B				
3	5	92	0.9 ± 0.09	0.9 ± 0.05
5	5	90	0.9 ± 0.09	0.9 ± 0.05
7	5	88	0.9 ± 0.09	1.0 ± 0.05
10	5	85	1.0 ± 0.10	1.1 ± 0.06
Type D-C				
3	5	92	0.8 ± 0.08	0.8 ± 0.04
5	5	90	0.8 ± 0.08	0.8 ± 0.04
7	5	88	0.8 ± 0.08	0.9 ± 0.05
10	5	85	0.9 ± 0.09	1.0 ± 0.05

Table 4a: Density, foaming efficiency and average pore size of aluminium foams prepared by the PM route using $CaCO_3$ foaming agent

Tabela 4a: Gostota, učinkovitost penjenja in povprečna velikost por v aluminijskih penah, izdelanih po postopku prašne metalurgije z uporabo $CaCO_3$ kot sredstva za penjenje

Initial composition of foamable precursors (*w*/%)		Selected properties of foamed samples		
$CaCO_3$	Al powder	Density (g/cm^3)	Foaming efficiency (%)	Average pore size (mm)
Type D-A				
3	97	0.42 ± 0.02	84.4	0.8 ± 0.08
5	95	0.47 ± 0.03	82.6	0.9 ± 0.09
7	93	0.51 ± 0.03	81.1	1.0 ± 0.10
10	90	0.55 ± 0.03	79.6	1.2 ± 0.12
Type D-B				
3	97	0.46 ± 0.03	83.0	0.5 ± 0.05
5	95	0.49 ± 0.03	81.9	0.7 ± 0.07
7	93	0.53 ± 0.03	80.4	0.8 ± 0.08
10	90	0.59 ± 0.03	78.1	0.9 ± 0.09
Type D-C				
3	97	0.53 ± 0.03	80.4	0.5 ± 0.05
5	95	0.55 ± 0.03	79.6	0.5 ± 0.05
7	93	0.59 ± 0.03	78.1	0.6 ± 0.06
10	90	0.61 ± 0.03	77.4	0.8 ± 0.08

Table 4b: Density, foaming efficiency and the average pore size of aluminium foams prepared by the PM route using dolomite as foaming agent

Tabela 4b: Gostota, učinkovitost penjenja in povprečna velikost por v aluminijskih penah, izdelanih po postopku prašne metalurgije z uporabo dolomita kot sredstva za penjenje

Initial composition of foamable performs *w*/%			Selected properties of foamed samples		
Dolomite	SiC	Al powder	Density (g/cm^3)	Foaming efficiency (%)	Average pore size (mm)
Type D-A					
3	5	92	0.56 ± 0.03	79,3	0.9 ± 0.09
5	5	90	0.59 ± 0.03	78,1	0.9 ± 0.09
7	5	88	0.63 ± 0.03	76,7	1.1 ± 0.11
10	5	85	0.69 ± 0.03	74,4	1.3 ± 0.13
Type D-B					
3	5	92	0.51 ± 0.03	81,1	0.6 ± 0.06
5	5	90	0.53 ± 0.03	80,4	0.7 ± 0.07
7	5	88	0.57 ± 0.03	78,9	0.9 ± 0.09
10	5	85	0.59 ± 0.03	78,1	0.9 ± 0.09
Type D-C					
3	5	92	0.50 ± 0.03	81,5	0.6 ± 0.06
5	5	90	0.52 ± 0.03	80,7	0.6 ± 0.06
7	5	88	0.55 ± 0.03	79,6	0.8 ± 0.08
10	5	85	0.56 ± 0.03	79,3	0.9 ± 0.09

Table 5a: Density, foaming efficiency and average pore size of aluminium foams prepared from as-machined foamable preforms fabricated by the melt route using $CaCO_3$ foaming agent

Tabela 5a: Gostota, učinkovitost penjenja in povprečna velikost por v aluminijskih penah, izdelanih iz strojno obdelanih prekurzorjev s $CaCO_3$ kot sredstvom za penjenje, dobljenih po livarskem postopku

Initial composition of foamable precursors (*w*/%)		Selected properties of foamed samples		
$CaCO_3$	Al powder	Density (g/cm^3)	Foaming efficiency (%)	Average pore size (mm)
Type C-A				
3	97	0.89 ± 0.05	67.0	1.1 ± 0.11
5	95	0.92 ± 0.05	65.0	1.3 ± 0.13
7	93	0.97 ± 0.05	64.1	1.4 ± 0.14
10	90	0.99 ± 0.05	63.3	1.4 ± 0.14
Type C-B				
3	97	0.84 ± 0.03	68.9	0.8 ± 0.08
5	95	0.89 ± 0.03	67.0	0.9 ± 0.09
7	93	0.93 ± 0.04	65.6	0.9 ± 0.09
10	90	0.95 ± 0.04	64.8	1.1 ± 0.11
Type C-C				
3	97	0.79 ± 0.04	70.7	0.7 ± 0.07
5	95	0.81 ± 0.04	70.0	0.8 ± 0.08
7	93	0.85 ± 0.04	68.5	1.2 ± 0.12
10	90	0.88 ± 0.04	67.4	1.5 ± 0.15

Table 5b: Density, foaming efficiency and average pore size of aluminium foams prepared from as-machined foamable performs fabricated by the melt route using dolomite as foaming agent

Tabela 5b: Gostota, učinkovitost penjenja in povprečna velikost por v aluminijskih penah, izdelanih iz strojno obdelanih prekurzorjev z dolomitom kot sredstvom za penjenje, dobljenih po livarskem postopku

Initial composition of foamable performs *w*/%			Selected properties of foamed samples		
Dolomite	SiC	Al powder	Density (g/cm^3)	Foaming efficiency (%)	Average pore size (mm)
Type D-A					
3	5	92	0.71 ± 0.04	73,7	1.2 ± 0.12
5	5	90	0.72 ± 0.04	73,3	1.4 ± 0.14
7	5	88	0.75 ± 0.04	72,2	1.4 ± 0.14
10	5	85	0.81 ± 0.04	70,0	1.5 ± 0.15
Type D-B					
3	5	92	0.61 ± 0.03	77,4	0.8 ± 0.08
5	5	90	0.63 ± 0.03	76,6	0.9 ± 0.09
7	5	88	0.66 ± 0.03	75,5	1.0 ± 0.10
10	5	85	0.70 ± 0.04	74,1	1.1 ± 0.11
Type D-C					
3	5	92	0.65 ± 0.03	75,9	0.8 ± 0.08
5	5	90	0.66 ± 0.03	75,5	0.9 ± 0.09
7	5	88	0.68 ± 0.03	74,8	1.1 ± 0.11
10	5	85	0.72 ± 0.04	73,3	1.5 ± 0.15

Table 6a: Density, foaming efficiency and average pore size of aluminium foams prepared by the melt route from as-machined and additionally isostatically pressed foamable precursors with $CaCO_3$ foaming agent

Tabela 6a: Gostota, učinkovitost penjenja in povprečna velikost por v aluminijskih penah, izdelanih iz strojno obdelanih in hladno izostatsko stisnjenih prekurzorjev s $CaCO_3$ kot sredstvom za penjenje, dobljenih po livarskem postopku

Initial composition of foamable precursors (*w*/%)		Selected properties of foamed samples		
$CaCO_3$	Al powder	Density (g/cm^3)	Foaming efficiency (%)	Average pore size (mm)
Type C-A				
3	97	0.69 ± 0.03	74.4	1.1 ± 0.11
5	95	0.72 ± 0.04	73.3	1.2 ± 0.12
7	93	0.76 ± 0.04	71.9	1.3 ± 0.13
10	90	0.80 ± 0.04	70.4	1.6 ± 0.16
Type C-B				
3	97	0.64 ± 0.03	76.3	0.8 ± 0.08
5	95	0.69 ± 0.03	74.4	0.9 ± 0.09
7	93	0.72 ± 0.04	73.3	1.3 ± 0.13
10	90	0.74 ± 0.04	72.6	1.4 ± 0.14
Type C-C				
3	97	0.62 ± 0.03	77.0	0.9 ±0.09
5	95	0.67 ± 0.03	75.2	1.1 ± 0.11
7	93	0.71 ± 0.04	73.7	1.3 ± 0.13
10	90	0.73 ± 0.04	73.0	1.6 ± 0.16

Table 6b: Density, foaming efficiency and average pore size of aluminium foams prepared by the melt route from as-machined and additionally isostatically pressed foamable performs using dolomite as foaming agent

Tabela 6b: Gostota, učinkovitost penjenja in povprečna velikost por v aluminijskih penah, izdelanih iz strojno obdelanih in hladno izostatsko stisnjenih prekurzorjev z dolomitom kot sredstvom za penjenje, dobljenih po livarskem postopku

Initial composition of foamable performs *w*/%			Selected properties of foamed samples		
Dolomite	SiC	Al powder	Density (g/cm^3)	Foaming efficiency (%)	Average pore size (mm)
Type D-A					
3	5	92	0.63 ± 0.03	76,7	1.1 ± 0.11
5	5	90	0.67 ± 0.03	75,2	1.2 ± 0.12
7	5	88	0.71 ± 0.03	73,7	1.3 ± 0.13
10	5	85	0.78 ± 0.03	71,1	1.6 ± 0.16
Type D-B					
3	5	92	0.57 ± 0.03	78,9	0.7 ± 0.07
5	5	90	0.59 ± 0.03	78,1	0.9 ± 0.09
7	5	88	0.62 ± 0.03	77,0	1.1 ± 0.11
10	5	85	0.63 ± 0.03	76,7	1.4 ± 0.14
Type D-C					
3	5	92	0.58 ± 0.03	78,5	0.7 ± 0.07
5	5	90	0.60 ± 0.03	77,8	0.8 ± 0.08
7	5	88	0.64 ± 0.03	76,3	1.0 ± 0.10
10	5	85	0.67 ± 0.03	75,2	1.1 ± 0.11

3.2 Microstructural investigation of aluminium foam samples

A similar cellular structural development with spherical, closed pores was obtained by both the powder metallurgy (**Figure 1**) and the melt processing route (**Figure 2**). However, as is evident in **Figure 1**, the samples obtained by the powder metallurgical route had a more uniform microstructure consisting of well-separated individual cells. On the other hand, the microstructure of the samples obtained by the melt processing route, **Figure 2**, revealed the presence of some individual, non-uniformities created by flow (the movement of bubbles with respect to each other), drainage (flow of liquid metal through the intersection of three foam

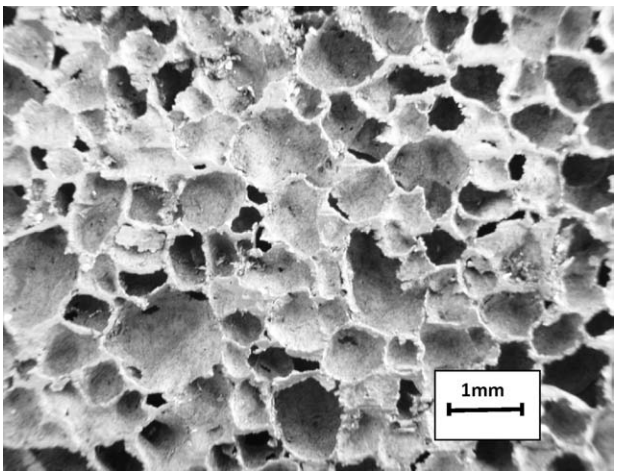

Figure 1: Cross-section of an aluminium foam obtained by the powder metallurgical route with well-separated individual cells and relatively uniform microstructure.

Slika 1: Posnetek prečnega prereza vzorca aluminijske pene, izdelanega po postopku prašne metalurgije, z izraženimi posameznimi porami in relativno enakomerno mikrostrukturo

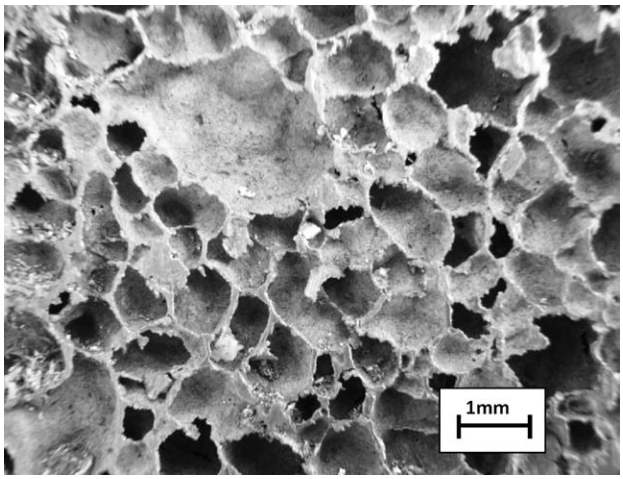

Figure 2: Cross-section of an aluminium foam obtained by the melt route with a characteristic channel network and foam drainage

Slika 2: Posnetek prečnega prereza vzorca aluminijske pene, izdelanega po livarskem postopku, na katerem so poleg posameznih por opazne tudi nehomogenosti, povzročene z zlitjem por in odtekanjem taline skozi značilne kanale v mikrostrukturi

films), coalescence (sudden instability in foam film) and coarsening (slow diffusion of gas from smaller bubbles to larger ones).

The absence of considerable pore coarsening and drainage suggests that there is a cell-face stabilising mechanism operating in the carbonate-foamed melts[2], slowing down the cell-face rupturing process and hence, inhibiting cell coarsening. The mechanism is likely to be a result of the foaming gas (CO_2)/melt or semi-solid slurry reaction during the foaming procedure, as was discussed in detail by Gergely et al.[2].

Concerning the average pore size and the uniformity in cell size distribution, foams made by the powder metallurgical route have finer pores and a more regular morphology than samples made by the melt route, particularly those from as-machined precursors. However, an additional cold isostatic pressing of the as-machined precursor obtained by the melt route was found to help in achieving more uniform foams with a smaller average pore size, similar to those obtained by the powder metallurgical route. The improvement is most probably caused by better compacting of the individual $CaCO_3$ or dolomite particles and the aluminium matrix, resulting in a higher density of the foamable precursor.

3.3 Mechanical properties

Figure 3 shows an example of the stress-strain response of samples foamed from preforms prepared by the PM route in which the compressive strength of the foams was correlated with their density.

Because of the closed cell structure, the compressive foam behaviour in all cases showed a typical stress-strain diagram with a division into three parts: a linear increase in stress mainly caused by elastic deformation, followed by a plateau caused by homogeneous plastic deformation and a final steep increase due to the collapse of the cells. The compressive strength was taken as the initial peak

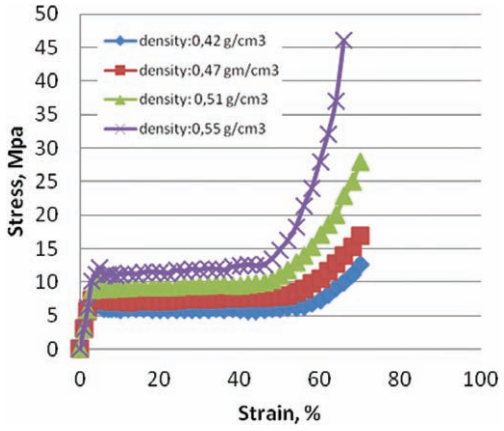

Figure 3: The stress-strain response of various aluminium foam samples from preforms obtained by the PM route using the $CaCO_3$ foaming agent.

Slika 3: Krivulja napetost – deformacija za vzorce aluminijskih pen na osnovi predoblik s $CaCO_3$ penilom, izdelanih po postopku prašne metalurgije

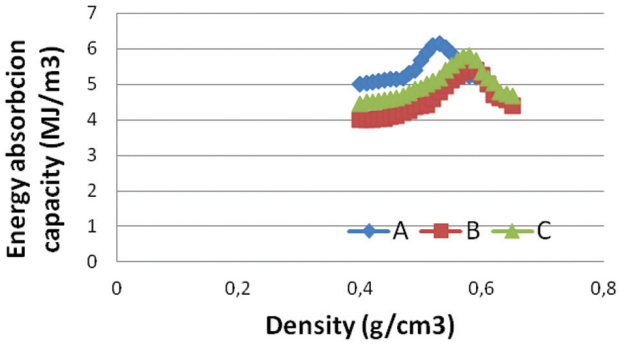

Figure 4: Example of optimization of the aluminium foam density range for the maximum energy absorption capacity: A)-foams obtained by powder metallurgy, B)-foams obtained from as-machined precursors fabricated by the melt route, and C)-foams obtained from as-machined and cold isostatically pressed precursors fabricated by the melt route. The foaming agent was $CaCO_3$.

Slika 4: Primer optimiziranja gostote vzorcev aluminijskih pen za doseganje največje sposobnosti absorpcije energije: A) pene, izdelane po postopku prašne metalurgije; B) pene iz strojno obdelanih prekuzorjev, narejenih po livarskem postopku; C) pene iz strojno obdelanih in hladno izostatsko stisnjenih prekurzorjev, narejenih po livarskem postopku. Kot sredstvo za penjenje je bil uporabljen $CaCO_3$.

stress. Foams made by the PM route possessed the highest compressive strength, while samples foamed from as-machined precursors had significantly lower values. For the interval of foam densities analysed in this work (from 0.42g/cm^3 to 0.55 g/cm^3), it was found that in more dense foam samples the position of the plateau shifted toward higher stress values.

The energy absorbed per unit volume (E-energy absorption capacity), which is one of the most important characteristics of aluminium foams, was determined from the area under the stress-strain plots as follows[8]:

$$E = \int_0^l \sigma(\varepsilon)\mathrm{d}\varepsilon \qquad (2)$$

Where σ is the compressive stress, l is the limit of the strain concerned and ε is the compressive strain. The calculated values of energy-absorption capacity for samples are plotted in **Figure 2** and correlated with the foam density. The typical response was found to be a quasi-Gaussian function with a maximum energy-absorption capacity in a very narrow density range.

The maximum energy-absorption capacity for various foams is summarized in **Figure 3**. For foams made by the PM route, the maximum energy-absorption capacity of 6.14 MJ/m^3 was achieved in foams with a density of 0.53 g/cm^3. On the other hand, in samples foamed from as-machined precursors fabricated by melt route, a maximum energy-absorption capacity of only 5.41 MJ/m^3 was found. The maximum appeared at a foam density of 0.58 g/cm^3. Finally, in the melt route fabricated precursors, additionally isostatically pressed before foaming, an intermediate maximum energy-absorption capacity of 5.82 MJ/m^3 was found in samples with a density of 0.58 g/cm^3.

The foaming process does not materially affect the properties of the cell-wall material. However, it leads to a unique spatial distribution of the aluminium which results in significantly different properties of the foamed component in comparison with the bulk part. It is obvious that the properties of the aluminium foam significantly depend on its porosity, so that a desired property (or combination of properties) can be tailored by selecting the foam density.

The mechanical properties of the foams obtained by applying dolomite powder as a foaming agent are fully comparable with the corresponding properties of foams fabricated using TiH_2.

4 CONCLUSION

The following conclusions can be drawn from this work.

- TiH_2 powder as foaming agent was successfully replaced by commercial $CaCO_3$ or dolomite powders of a different average particle size.
- Foaming precursors with different proportions (φ = 3–10 %) of $CaCO_3$ or dolomite powder particles as a foaming agent were routinely prepared either by the powder metallurgical or melt route.
- Precursors obtained by powder metallurgy had superior homogeneity and densities ≥98% of theoretical. Moreover, in precursors obtained by the PM route containing φ = 3–7 % of $CaCO_3$ or dolomite particles of an average particle size of below 50 μm, densities ≥99 % of theoretical were achieved.
- With greater addition of $CaCO_3$ or dolomite particles and by using $CaCO_3$ or dolomite powders with a higher average particle size (above 70 μm), densities ≥99 % of theoretical could not be achieved.
- The foaming efficiency of experimentally prepared precursors was evaluated based on the relative density of foams obtained (the apparent density of the foam divided by the density of aluminium). The experimental findings showed that the apparent density of the foam samples is inversely proportional to the density of the foaming precursor. Thus, foamable precursors with a higher density resulted in foam samples with a lower apparent density and a higher foaming efficiency. On the other hand, the foaming efficiency and the average pore size of the foamed samples are generally reciprocally dependent. Thus, a higher foaming efficiency results in a foam microstructure with finer pores.
- The mechanical properties (compression strength and energy absorption capacity) of the foamed samples are also strongly influenced by the foaming efficiency. For the range of foam densities analysed, the compression strength, considered as the initial peak stress, was found to be superior (approx. 13 MPa) in samples with increased density (0.55 g/cm^3) and hence, lower foaming efficiency (79.6 %). In contrast to this, the maximum energy-absorption capacity was achieved in foams with the highest foaming efficiency.
- From the experimental findings is obvious that the properties of an aluminium foam significantly depend on its porosity and the desired property (or combination of properties) can be tailored by the foam density.
- The experimental findings confirm that the microstructure, compression strength and energy-absorption capacity of aluminium foams prepared with $CaCO_3$ or dolomite powder as foaming agent are quite comparable with their counterparts foamed by TiH_2.

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