

EFFECT OF A FOAMING AGENT AND ITS MORPHOLOGY ON THE FOAMING BEHAVIOUR, CELL-SIZE DISTRIBUTION AND MICROSTRUCTURAL UNIFORMITY OF CLOSED-CELL ALUMINIUM FOAMS

VPLIV VRSTE IN MORFOLOGIJE SREDSTVA ZA PENJENJE NA PROCES PENJENJA, PORAZDELITEV POR PO VELIKOSTI IN UNIFORMNOST MIKROSTRUKTURE ALUMINIJSKIH PEN Z ZAPRTIMI PORAMI

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A quantitative evaluation of the microstructure of aluminium foams and, particularly, any quantitative comparison is a very demanding and complex issue. In this work, the cell-size distribution (CSD) was proposed as the most efficient approach for their assessment.

The foams were made by the powder metallurgy (P/M) route, by applying titanium hydride and dolomite powders of five different average particle sizes as the foaming agents.

The average size of the pores and the pore-size distribution were estimated by assessing optical and scanning electron micrographs of as-polished foam bars by applying the point-counting method and image-analysis software.

The uniformity of the CSD in the foamed samples with closed cells was studied as a function of the particle size distribution of the foaming agents, the average particle size of the applied AlSi12 powders, the concentration of the foaming agents, the foaming temperature and the foaming time.

Generally, the samples foamed with the dolomite foaming agent had a more uniform cell-size distribution and a lower average bubble size. The most uniform cell-size distribution was achieved in the foam samples foamed with the minimum amount of the mass fraction ($w = 0.5\%$) of dolomite powder grades, having the lowest average particle size and a narrow particle-size distribution. In contrast, in samples made from coarser and less-uniform grades of foaming agents, the cell-size distribution was broader, with a significantly higher fraction of large bubbles. Longer foaming times and higher foaming temperatures also led to foam samples with a less-uniform microstructure.

Based on the experimental findings and theoretical considerations regarding aluminium-foam microstructural development, the preconditions for stable bubble growth into a homogeneous and uniform foam structure were modelled and compared with the experimentally determined values.

Keywords: aluminium foams, comparison of different foaming agents and processing parameters, microstructural characterisation, modelling of microstructural development

Kvantitativna karakterizacija in primerjava mikrostruktur različnih vzorcev aluminijevih pen sta zelo zahtevni ter zapleteni nalogi. Naša raziskovalna skupina se je odločila za način, kjer kvantitativna karakterizacija mikrostrukture aluminijevih pen temelji na določanju porazdelitve por po velikosti (PPV).

Vzorci pen smo izdelovali s postopkom metalurgije prahov. Kot sredstvo za penjenje smo uporabili pet vrst titanhidridnih in dolomitnih prahov z različno porazdelitvijo delcev po velikosti.

Povprečno velikost por in porazdelitev por po velikosti v vzorcih aluminijevih pen smo ugotavljali s slikovno analizo posnetkov njihove mikrostrukture, narejenih s svetlobno in vrstično elektronsko mikroskopijo.

Enakomernost porazdelitve velikosti por v vzorcih pen smo preučevali v odvisnosti od porazdelitve velikosti sredstva za penjenje, povprečne velikosti uporabljenih prahov AlSi12, koncentracije sredstva za penjenje in temperature ter časa penjenja.

Na splošno so imeli vzorci, ki smo jih penili z dolomitom, veliko bolj enakomerno oz. ožjo porazdelitev velikosti por ter manjšo povprečno velikost. Najnižjo porazdelitev por po velikosti smo opazili v vzorcih pen, izdelanih iz najfinejših dolomitnih prahov, z ozko porazdelitvijo delcev po velikosti ter najnižjo masno koncentracijo (0,5 %) sredstva za penjenje. V nasprotju s tem je bila v vzorcih aluminijevih pen, izdelanih iz bolj grobih vrst dolomitnih prahov, s širšo porazdelitvijo delcev po velikosti, tudi porazdelitev por po velikosti širša z naraščajočim deležem velikih por. Poleg tega se je izkazalo, da je stopnja uniformnosti mikrostrukture pen odvisna v veliki meri od temperature in časa penjenja, pri čemer je s podaljšanjem časa in višanjem temperature penjenja porazdelitev velikosti por postajala vse širša.

Eksplozivne rezultate in teoretične ugotovitve o razvoju mikrostrukture aluminijevih pen z zaprti poroznostjo smo strnili v model, ki predpisuje pogoje nastanka homogene in uniformne strukture pen.

Ključne besede: aluminijeve pene, primerjava različnih sredstev za penjenje in procesnih parametrov, karakterizacija mikrostrukture, model razvoja mikrostrukture

1 INTRODUCTION

Closed-cell aluminium foams are a promising class of lightweight structural materials with a unique combination of properties resulting in their cellular structure. However, at the same time, the cellular structure of the aluminium foams also introduces several difficulties, particularly in achieving foams with a constant and uniform quality, which is an important prerequisite for their broad commercialisation in more demanding applications.

The assurance of a constant, uniform and repeatable quality and the properties of aluminium foams are based on the development of a homogeneous microstructure, especially in the cell-size distribution, cell morphology and wall thickness. The development of such a repeatable and homogeneous foam microstructure is influenced by several parameters, which should be carefully and completely controlled.

The following parameters are the most important:

- Selection of the foaming agent (hydrides, carbides, etc.),
- The average size and particle morphology of the foaming agent,
- The chemical composition of the foaming agent's surface (e.g., the level of oxidation, etc.),
- The concentration and homogeneity of the distribution of the foaming agent in the aluminium matrix,
- The homogeneity of the aluminium matrix,
- The temperature and time of foaming,
- The cell coalescence and coarsening.

A quantitative evaluation of the microstructure of aluminium foams and, particularly, their quantitative comparison is a very demanding and complex issue¹. As a rule, and quite independently of the applied foaming procedure, the microstructure of aluminium foams is rather heterogeneous, with a significant amount of irregularities and non-homogeneities affected by the movement of the foam in the liquid and/or semi-solid state.

The cell-size distribution (CSD) is one of the most important determinants of the level of aluminium foam microstructural uniformity. Although limited to just a representative area of the microstructure of the entire foam sample, the CSD is one of the most suitable quantitative parameters of the aluminium foam microstructural investigation. A mutual correlation between the CSD and the aluminium-foam processing parameters has not yet been established.

Hence, the purpose of this work was to investigate the interdependence of the most frequently applied processing parameters (time, temperature, particle size distribution of foaming agent, concentration of foaming agent in foamable mixture, etc.) on the cell-size distribution and the density of aluminium foams made by the powder metallurgy (P/M) route. Recently, the identical methodology was reported for evaluating the foaming

behaviour, cell-size distribution and microstructural uniformity of Al panels made from hot-rolled precursors².

2 EXPERIMENTAL PROCEDURE

2.1 Foaming agents

Titanium hydride (supplier: AG Materials Inc., USA) and dolomite powders (supplier: Granit, d. o. o., Slovenska Bistrica, Slovenia) of five different average particle sizes were applied as foaming agents. The average particle size of the powders used in the experiments is listed in **Table 1**. The particle size distribution of the powdered foaming agents was measured using a laser particle analyser (Malvern Mastersizer 2000). The relative error of the measurement was within $\pm 1\%$.

Table 1: The average particle size and cumulative particle size distribution of the TiH₂ and dolomite powders applied as foaming agents

Tabela 1: Povprečna velikost delcev in porazdelitev delcev po velikosti TiH₂ in dolomitnega prahu, uporabljenih kot sredstvo za penjenje

TiH ₂ powders	TIH-003B	TIH-0420	TIH-3242	TIH-2032	TIH-1020
Average particle size (µm)	3.1	20.4	40.8	60.3	110.4
Cumulative particle size distribution (µm)					
D ₁₀	1.2	13.1	23.4	45.8	80.4
D ₂₅	2.8	17.4	32.5	53.8	97.3
D ₅₀	3.1	20.4	40.8	60.3	110.4
D ₇₅	3.3	23.7	50.4	69.2	129.8
D ₉₀	5.7	41.4	65.9	82.3	151.1
Uniformity of particle size distribution (µm)					
D ₉₀ – D ₁₀	4.5	28.3	42.5	36.5	70.7
Dolomite powders					
	D-1	D-2	D-3	D-4	D-5
Average particle size (µm)	3.4	5.2	10.1	20.8	35.7
Particle size distribution (µm)					
D ₁₀	1.6	3.9	5.6	11.2	28.6
D ₂₅	2.3	4.7	8.6	15.9	33.0
D ₅₀	3.4	5.2	10.1	20.8	35.7
D ₇₅	3.5	5.6	11.8	23.1	36.9
D ₉₀	4.2	6.0	13.7	27.2	39.2
Uniformity of particle size distribution (µm)					
D ₉₀ – D ₁₀	3.0	2.1	8.1	16.0	10.6

In the case of dolomite powder, part of the as-received powder (D-5) with an average particle size of 35 µm was additionally milled in a planetary mill (in acetone with Al₂O₃ balls) for various times (10, 30, 60 and 120) min and laboratory sieved to provide fractions (D-1, D-2, D-3 and D-4) with a lower average particle size.

The applied TiH₂ and dolomite powders had relatively narrow particle size distributions and different average particle sizes. In the case of commercial grades of TiH₂ powders, the average particle size (D₅₀) was within the range of 3 µm to 110 µm, while in the case of

laboratory sieved fractions of the milled dolomite the powders were from 3 μm to 36 μm .

The uniformity of the particle size distribution in the TiH_2 and the dolomite powders applied was different in various grades of powders. The TiH_2 powder grade TIH-003B had a very narrow particle size distribution and excellent uniformity, whereas the other TiH_2 powders were less uniform, especially the grade TIH-1200.

Generally, the uniformity of the particle size distribution in the selected dolomite powders was significantly higher than the TiH_2 . However, also in that case, some grades of the applied dolomite powders (e.g., D-4) had a less uniform particle size distribution.

2.2 Aluminium powders

For most experiments, AlSi12 powder with an average particle size of 80 μm was applied. Some trials were also made with a coarser AlSi12 powder having an average particle size of 350 μm .

2.3 Concentration of foaming agent

The concentration of foaming agents (TiH_2 and dolomite) in the foaming precursors in the mass fraction was $w = 0.5\%$. However, in two separate sets of experiments, the concentration of foaming agents was changed systematically – in the case of TiH_2 from 0.5 % to 1.5 % and in the case of dolomite, from 0.5 % to 3.0 %. In order to reduce the number of experiments, only one grade of each foaming agent was applied – TIH-0420 in precursors with TiH_2 and D-4 in precursors with dolomite. As is evident from **Table 1**, both applied grades had the same average particle size of approx. 20 μm .

2.4 Homogenisation of foaming mixtures

Homogenisation of foaming mixtures was performed in a laboratory turbula device, by applying different homogenisation times. Thus, mixtures of AlSi12 powder and 0.5 % of TiH_2 (grade TIH-0420) or dolomite (D-4), were homogenized using two different regimes: for 10 min or 240 min.

2.5 Preparation of foaming precursors

The foams made in this work were prepared by the indirect foaming method starting from solid, foamable precursors of the AlSi12 matrix containing uniformly dispersed foaming agent particles. The foamable precursors were made using the powder metallurgy route. The homogenized mixtures of selected AlSi12 and TiH_2 or dolomite powders were uniaxially cold pressed under a pressure of 100 MPa and then additionally isostatically pressed under 950 MPa.

2.6 Foaming procedure

The precursors were foamed in a conventional batch electrical furnace with air atmosphere circulation under various experimental conditions (time, temperature) and applying the same cooling method. Before foaming, the

individual precursors were inserted into a cylindrical (40 mm in diameter and 70 mm long) stainless steel mould coated with a boron nitride suspension. The mould dimensions and the precursor size (20 mm in diameter and 30 mm long) were selected to allow the complete expansion of the precursor to foam. The arrangement was placed inside a pre-heated batch furnace at a selected temperature and held for the selected holding time. After that, 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. Precursors with the TiH_2 foaming agent were foamed in the temperature interval of 580 $^{\circ}\text{C}$ to 700 $^{\circ}\text{C}$ and a foaming time of 10 s to 180 s, while precursors with the dolomite foaming agent were foamed at a higher temperature (700 $^{\circ}\text{C}$ to 900 $^{\circ}\text{C}$) for slightly shorter foaming times of 10 s to 120 s.

2.7 Foam density and porosity

The foam density was measured using the Archimedes method. The porosity of laboratory prepared foams was then calculated using equation: $1 - (\text{foam density} / \text{aluminium alloy density})$.

2.8 Microstructural investigation of foamed samples

The macro- and microstructural examinations were performed on sections obtained by precision wire cutting across the samples and on samples mounted in epoxy resin, using light and scanning electron microscopy as well as energy-dispersive x-ray spectrometry (SEM/EDS).

The average size of the pores and the pore size distribution were estimated by analysing the optical and scanning electron micrographs of as-polished foam bars applying the point-counting method and image analysis software.

2.9 A model of foam microstructure development (bubble growth)

The bubble growth can be expressed by applying a simple, stoichiometric model, in which the complete thermal decomposition of an individual TiH_2 or dolomite particle provides the gas phase for bubble nucleation and growth. Based on that simple assumption, the maximum bubble diameter depends on the maximum bubble pressure (p_{max}) determined by the Laplace equation:

$$P_{\text{max}} = (2\sigma_{\text{lg}}/r) + \rho gh + p_0 \quad (1)$$

The maximum bubble pressure, p_{max} , is the sum of the capillary ($2\sigma_{\text{lg}}/r$), hydrostatic (ρgh) and atmospheric (p_0) pressures. The capillary pressure depends on the surface tension, σ_{lg} , at the gas-liquid interface and the bubble radius (r); the hydrostatic pressure is determined by the immersion depth (h) and the density of the molten aluminium alloy (ρ).

The maximum radius (r_{max}) of an isolated bubble immersed in a molten (or semi-solid) aluminium alloy can be calculated by applying the ideal gas equation:

$$P_{max} V = nRT \quad (2)$$

where n corresponds to the number of moles of gas phase inside the bubble, R is the universal gas constant, V is the bubble volume and T is the temperature.

For a spherical bubble by combining Eqs. (1) and (2), we can calculate:

$$[(2\sigma_{ig}/r) + \rho gh + p_0] (4/3)r_{max}^3\pi = nRT \quad (3)$$

In the early stage of bubble growth, only the capillary pressure ($2\sigma_{ig}/r$) needs to be considered, while in the final stage of bubble growth the only important pressure is the atmospheric (p_0). Note that for laboratory conditions, the hydrostatic pressure (ρgh) is always negligible.

In **Tables 2a** and **2b**, the maximum bubble radius, r_{max} , is calculated for various initial particle sizes (D_{50}) of individual TiH₂ and dolomite particles, assuming in all cases a complete chemical conversion without the loss of the gaseous phase.

Under these conditions, the average particle size of the foaming agent is correlated with the number of moles of gas phase using the following expression:

$$n = (d_{50}^3\pi)/(6M) \quad (4)$$

where M is the molar mass of the foaming agent applied. The maximum bubble radius, r_{max} , is finally determined by the formula:

$$r_{max} = d_{50} [RT/(8 M p_0)] \quad (5)$$

Table 2a: Maximum bubble radius for TiH₂ particles with a different initial particle size (D_{50})

Tabela 2a: Maksimalni premer pore iz TiH₂ delcev različne začetne velikosti (D_{50})

The average particle size of TiH ₂ (µm)	3	20	40	75	140
Maximum bubble radius, r_{max} /µm	27	180	404	676	1263

Table 2b: Maximum bubble radius for bubbles created by dolomite particles of different initial particle size (D_{50})

Tabela 2b: Maksimalni premer pore iz delcev dolomita različne začetne velikosti (D_{50})

The average particle size of dolomite (µm)	3	5	10	20	35
Maximum bubble radius, r_{max} /µm	20	34	66	134	234

3 RESULTS AND DISCUSSION

The influence of the type, average particle size and particle size distribution of the foaming agent on the density, average cell size and cell-size distribution in aluminium foam samples is reported in **Tables 3a** and **3b**.

The uniformity of the cell-size distribution in foamed samples was studied as a function of the particle size

Table 3a: Experimentally determined density and cell-size distribution of aluminium foam samples as a function of TiH₂ foaming-agent morphology. Foaming conditions: 700 °C, 120 s.

Tabela 3a: Eksperimentalno izmerjene vrednosti gostote in porazdelitve velikosti por v vzorcih aluminijjskih pen, izdelanih pri različnih koncentracijah delcev TiH₂, uporabljenega kot penila. Pogoji penjenja: 700 °C, 120 s.

Type of foaming agent	TiH ₂				
Powder grade	TIH-003B	TIH-0420	TIH-3242	TIH-2032	TIH-1020
Density of aluminium foam (% of T. D.)	24.2 ± 1.2	25.6 ± 1.3	21.8 ± 1.1	18.9 ± 0.9	17.1 ± 0.9
Cell-size distribution (mm)					
D_{10}	2.6 ± 0.3	2.5 ± 0.3	4.3 ± 0.4	6.2 ± 0.6	8.4 ± 0.8
D_{25}	2.9 ± 0.3	2.6 ± 0.3	4.8 ± 0.4	6.6 ± 0.7	8.7 ± 0.8
D_{50}	3.1 ± 0.3	2.7 ± 0.3	4.9 ± 0.5	6.8 ± 0.7	8.9 ± 0.9
D_{75}	4.0 ± 0.4	3.2 ± 0.3	5.5 ± 0.6	8.0 ± 0.8	10.2 ± 1.0
D_{90}	6.4 ± 0.6	4.9 ± 0.5	7.0 ± 0.7	10.7 ± 1.1	12.5 ± 1.3
Uniformity of cell-size distribution (µm)					
$D_{90} - D_{10}$	3.8 ± 0.4	2.4 ± 0.2	2.7 ± 0.3	4.5 ± 0.5	4.1 ± 0.5

Table 3b: Experimentally determined density and cell-size distribution of aluminium foam samples as a function of the dolomite foaming agent morphology. Foaming conditions: 700 °C, 120 s.

Tabela 3b: Eksperimentalno izmerjene vrednosti gostote in porazdelitve velikosti por v vzorcih aluminijjskih pen, izdelanih pri različnih koncentracijah delcev dolomita, uporabljenega kot penila. Pogoji penjenja: 700 °C, 120 s.

Type of foaming agent	Dolomite				
Powder grade	D-1	D-2	D-3	D-4	D-5
Density of aluminium foam (% of T. D.)	13.7 ± 0.7	14.9 ± 0.7	16.3 ± 0.8	15.4 ± 0.8	13.1 ± 0.7
Cell-size distribution (mm)					
D_{10}	2.6 ± 0.3	2.2 ± 0.3	2.2 ± 0.2	2.2 ± 0.2	2.9 ± 0.3
D_{25}	2.7 ± 0.3	2.3 ± 0.3	2.2 ± 0.2	2.3 ± 0.2	3.0 ± 0.3
D_{50}	2.8 ± 0.3	2.5 ± 0.3	2.2 ± 0.2	2.3 ± 0.2	3.1 ± 0.3
D_{75}	3.2 ± 0.3	2.8 ± 0.3	2.4 ± 0.2	2.6 ± 0.3	3.4 ± 0.3
D_{90}	3.9 ± 0.4	4.2 ± 0.4	4.4 ± 0.4	4.5 ± 0.5	4.6 ± 0.5
Uniformity of cell-size distribution (µm)					
$D_{90} - D_{10}$	1.3 ± 0.1	2.0 ± 0.2	2.2 ± 0.2	2.3 ± 0.2	1.7 ± 0.2

distribution of the foaming agents (**Tables 3a** and **3b**), the average particle size of the applied AlSi12 powders (**Table 4**), the concentration of foaming agents (**Tables 5a** and **5b**), foaming temperature (**Table 6**) and foaming time (**Table 7**).

A typical microstructure of the obtained aluminium foam samples is presented in **Figure 1**.

Generally, the samples foamed with dolomite foaming agent had a more uniform cell-size distribution

Table 4: Experimentally determined correlation between the average size of AlSi12 particles in the foaming precursor and the density and the cell-size distribution in samples of aluminium foam. Foaming conditions: 700 °C, 120 s.

Tabela 4: Eksperimentalno ugotovljena odvisnost gostote vzorcev aluminijjskih pen v odvisnosti od povprečne velikosti delcev AlSi12 prahu v prekurzorju za penjenje. Pogoji penjenja: 700 °C, 120 s.

The average particle size of AlSi12 (µm)	80 ± 10		350 ± 10	
	TIH-0420	D-4	TIH-0420	D-4
Density of Al foam (% T. D.)	24.6 ± 1.2	15.1 ± 0.8	23.3 ± 1.2	12.9 ± 0.6
Cell-size distribution (mm)				
D ₁₀	2.6 ± 0.3	2.1 ± 0.2	3.5 ± 0.4	2.8 ± 0.3
D ₂₅	2.8 ± 0.3	2.3 ± 0.2	3.8 ± 0.4	3.1 ± 0.3
D ₅₀	2.9 ± 0.3	2.4 ± 0.2	3.9 ± 0.4	3.3 ± 0.3
D ₇₅	3.2 ± 0.3	2.7 ± 0.3	4.3 ± 0.4	3.6 ± 0.4
D ₉₀	5.0 ± 0.5	4.6 ± 0.5	7.1 ± 0.7	6.7 ± 0.7
Uniformity of cell-size distribution (µm)				
D ₉₀ – D ₁₀	2.4 ± 0.2	2.3 ± 0.2	3.6 ± 0.4	3.9 ± 0.4

Table 5a: Experimentally determined density and cell-size distribution of aluminium foam samples as a function of the concentration of TiH₂ foaming agent (TIH-0420). Foaming conditions: 700 °C, 120 s.

Tabela 5a: Eksperimentalno ugotovljene vrednosti gostote in porazdelitve velikosti por v vzorcih aluminijjske pene, v odvisnosti od koncentracije TiH₂ kot sredstva za penjenje (TIH-0420). Pogoji penjenja: 700 °C, 120 s.

Concentration of TiH ₂ (w/%)	0.5	1.0	1.5
Density of Al foam (% T. D.)	25.9 ± 1.3	23.8 ± 1.2	21.2 ± 1.1
Cell-size distribution (mm)			
D ₁₀	2.6 ± 0.3	3.2 ± 0.3	3.3 ± 0.3
D ₂₅	2.7 ± 0.3	3.5 ± 0.3	3.8 ± 0.3
D ₅₀	2.8 ± 0.3	3.7 ± 0.4	5.1 ± 0.5
D ₇₅	3.0 ± 0.3	4.2 ± 0.3	5.5 ± 0.6
D ₉₀	4.6 ± 0.5	8.4 ± 0.3	10.4 ± 1.0
Uniformity of cell-size distribution (µm)			
D ₉₀ – D ₁₀	2.0 ± 0.2	5.2 ± 0.6	7.1 ± 0.7

Table 5b: Experimentally determined density and cell-size distribution of aluminium foam samples as a function of the concentration of dolomite foaming agent (D-4). Foaming conditions: 700 °C, 120 s.

Tabela 5b: Eksperimentalno ugotovljene vrednosti gostote in porazdelitve velikosti por v vzorcih aluminijjske pene, v odvisnosti od koncentracije dolomita kot sredstva za penjenje (D-4). Pogoji penjenja: 700 °C, 120 s.

Concentration of dolomite (w/%)	0.5	1.0	1.5
Density of Al foam (% T. D.)	15.4 ± 0.8	12.8 ± 0.6	11.1 ± 0.6
Cell-size distribution (mm)			
D ₁₀	2.0 ± 0.2	2.4 ± 0.2	2.9 ± 0.3
D ₂₅	2.3 ± 0.2	2.9 ± 0.3	3.3 ± 0.3
D ₅₀	2.5 ± 0.3	3.2 ± 0.3	4.7 ± 0.5
D ₇₅	2.7 ± 0.3	4.8 ± 0.6	5.1 ± 0.5
D ₉₀	5.8 ± 0.6	7.6 ± 0.8	9.7 ± 1.0
Uniformity of cell-size distribution (µm)			
D ₉₀ – D ₁₀	3.8 ± 0.4	5.2 ± 0.5	6.8 ± 0.7

and a lower average bubble size. The most uniform cell-size distribution was achieved in foam samples foamed with the minimum amount ($w = 0.5\%$) of dolomite powder grades (D-1, D-2) having the lowest average particle size and narrow particle size distribution. In contrast, in samples made from coarser and less-uniform grades of foaming agents, the cell size distribution was wide-ranging, with a significantly higher fraction of large bubbles. In addition, a longer foaming time and higher foaming temperatures also led to foam samples with a less-uniform microstructure.

The experimentally determined values of the average bubble radius reported in **Tables 2a** and **2b** are at least one order of magnitude higher than those predicted by the model. The reason for this difference is due to the effects limiting the stability of individual bubbles, which are not considered by the model. These effects are bubble flow, drainage, rupture or coalescence, and coarsening.

From the difference between the theoretically predicted and experimentally determined values of the bubble radius, it is possible to estimate the stability of the real foam systems considered in this work. The experimental findings clearly confirm that coarser bubbles are more stable than finer ones. In addition, it is also evident that the stability of bubbles is much higher in foams created by dolomite particles than in the counterparts foamed by TiH₂. However, in both cases the average bubble sizes are proportional to the average initial size of the foaming particles – finer foaming particles create finer bubbles, while coarser particles create larger bubbles, as was predicted by the model.

On the other hand, the density of aluminium foam samples was inversely proportional to the bubble radius: foam samples with finer bubbles (**Tables 3a** and **3b**) had a higher density and, vice versa, foam samples with larger bubbles were specifically lighter. At the same time

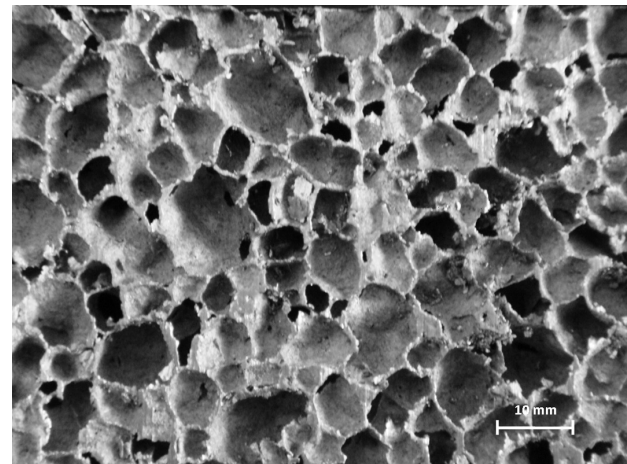


Figure 1: SEI of microstructure of aluminium foam sample foamed by applying TIH-3242 TiH₂ foaming agent. Foaming conditions: 700 °C, 120 s.

Slika 1: SEM-posnetek vzorca aluminijjske pene, izdelane s pomočjo TIH-3242 TiH₂ sredstva za penjenje. Pogoji penjenja: 700 °C, 120 s.

Table 6: Experimentally measured density and cell-size distribution of foam samples at various foaming temperatures. Foaming time: 120 s.**Tabela 6:** Eksperimentalno izmerjene vrednosti gostote in porazdelitve velikosti por v vzorcih aluminijske pene, izdelanih pri različnih temperaturah penjenja. Pogoji penjenja: 120 s.

Type and grade of foaming agent	TiH ₂ (TIH-0420)			Dolomite (D-4)		
	600	650	700	700	800	900
Foaming temperature (°C)	600	650	700	700	800	900
Foam density (% T. D.)	25.9 ±1.3	24.3 ±1.2	23.7 ±1.2	17.2 ±0.9	16.1 ±0.8	15.8 ±0.8
Cell-size distribution (mm)						
<i>D</i> ₀	2.0 ±0.2	2.4 ±0.2	2.7 ±0.3	1.7 ±0.2	1.9 ±0.2	2.0 ±0.2
<i>D</i> ₂₅	2.1 ±0.2	2.5 ±0.3	2.9 ±0.3	1.8 ±0.2	2.0 ±0.2	2.2 ±0.2
<i>D</i> ₅₀	2.6 ±0.3	2.8 ±0.3	3.3 ±0.3	2.0 ±0.2	2.2 ±0.2	2.3 ±0.2
<i>D</i> ₇₅	3.0 ±0.3	3.2 ±0.3	3.7 ±0.4	2.9 ±0.2	3.1 ±0.3	3.4 ±0.3
<i>D</i> ₁₀₀	4.3 ±0.4	4.8 ±0.5	5.4 ±0.5	4.1 ±0.4	4.5 ±0.5	4.9 ±0.5
Uniformity of cell-size distribution (µm)						
<i>D</i> ₉₀ – <i>D</i> ₁₀	2.3 ±0.2	2.4 ±0.2	2.7 ±0.3	2.4 ±0.2	2.6 ±0.3	2.9 ±0.3

Table 7: Experimentally measured density and cell-size distribution of aluminium foam samples at various foaming times. Foaming temperature: 700 °C.**Tabela 7:** Eksperimentalno izmerjene vrednosti gostote in porazdelitve velikosti por v vzorcih aluminjskih pen, izdelanih pri različnih časih penjenja. Pogoji penjenja: 700 °C.

Type and grade of foaming agent	TiH ₂ (TIH-0420)			Dolomite (D-4)		
	10	90	180	10	60	120
Foaming time (s)	10	90	180	10	60	120
Foam density (% T. D.)	31.4 ±1.6	28.7 ±1.4	25.2 ±1.3	23.7 ±1.2	18.1 ±0.9	15.5 ±0.8
Cell-size distribution (mm)						
<i>D</i> ₁₀	1.7 ±0.2	2.2 ±0.2	2.5 ±0.3	1.5 ±0.2	1.9 ±0.2	2.1 ±0.2
<i>D</i> ₂₅	1.8 ±0.2	2.3 ±0.2	2.6 ±0.3	1.6 ±0.2	2.1 ±0.2	2.2 ±0.2
<i>D</i> ₅₀	1.9 ±0.2	2.4 ±0.2	2.8 ±0.3	1.6 ±0.2	2.0 ±0.2	2.3 ±0.2
<i>D</i> ₇₅	2.7 ±0.3	2.9 ±0.3	3.3 ±0.3	2.5 ±0.3	2.8 ±0.3	3.5 ±0.4
<i>D</i> ₉₀	3.7 ±0.4	4.4 ±0.4	4.9 ±0.5	3.5 ±0.4	4.1 ±0.4	5.0 ±0.5
Uniformity of cell-size distribution (µm)						
<i>D</i> ₉₀ – <i>D</i> ₁₀	2.0 ±0.2	2.2 ±0.2	2.4 ±0.2	2.0 ±0.2	2.2 ±0.2	2.9 ±0.3

and under the same foaming conditions (temperature, time), foams made using dolomite had a significantly lower density than samples with a similar cell size foamed by TiH₂.

The foaming of precursors made from two grades of AlSi12 powder (Table 4) resulted in foam samples with a bubble radius proportional to the average size of the AlSi12 powders. Independently of the kind of foaming agent (TiH₂ or dolomite), coarser AlSi12 powder resulted in foams with larger bubble radius.

As evident from the cell-size distribution data listed in Tables 5a and 5b, an increase in the foaming-agent concentration (either TiH₂ or dolomite) led to the formation of foams with larger bubbles and a lower density. However, also in that case, samples foamed with dolomite had smaller bubbles and lower densities.

Finally, an increase in the foaming temperature and time (Tables 6 and 7) also favoured the formation of coarser bubbles. Again, the coarsening tendency was found to be higher in samples foamed by TiH₂.

The experimentally developed foam microstructures were mainly influenced by a slowing down of the level of foam movement (i.e., the foam stability) attained in particular trials. The slowing down of the movement of the foam includes the prevention of flow (the movement of bubbles with respect to each other caused either by external forces or changes in the internal gas pressure during foaming), drainage (flow of liquid metal through the foam), coalescence (sudden instability in a bubble wall leading to its disappearance) and coarsening (slow diffusion of gas from smaller bubbles to bigger bubbles).

4 CONCLUSION

The effects of a foaming agent and its morphology on the foaming behaviour, cell-size distribution and microstructural uniformity of closed-cell aluminium foams were investigated. Furthermore, a model of the microstructural development (bubble growth and stabilisation) was developed and compared with the experimental findings.

According to the experimental findings, samples foamed with the dolomite foaming agent had a more uniform cell-size distribution and a lower average bubble size. The most uniform cell-size distribution was achieved in foam samples foamed with the minimum amount (*w* = 0.5 %) of dolomite powder grades having the lowest average particle size and a narrow particle size distribution. In contrast, in samples made from coarser and less-uniform grades of foaming agents, the cell-size distribution was broader, with a significantly higher fraction of large bubbles. In addition, longer foaming times and higher foaming temperatures also led to foam samples with a less-uniform microstructure.

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5 REFERENCES

- V. Kevorkijan, S. D. Škapin, I. Paulin, B. Šuštaršič, M. Jenko, Mater. Tehnol., 44 (2010) 6, 363–371
- V. Kevorkijan, U. Kovačec, I. Paulin, S. D. Škapin, M. Jenko, Mater. Tehnol., 45 (2011) 6, 537–544