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SYNTHESIS OF ALUMINIUM FOAMS BY THE POWDER-METALLURGY PROCESS: COMPACTING OF PRECURSORS

SINTEZA ALUMINIJEVIH PEN PO POSTOPKU METALURGIJE PRAHOV: STISKANJE PREKURZORJEV

Irena Paulin[1,2], Borivoj Šuštaršič[2], Varužan Kevorkijan[3], Srečo D. Škapin[4], Monika Jenko[2]

[1]TALUM, d. d., Kidričevo, Tovarniška cesta 10, SI-2325 Kidričevo, Slovenia
[2]Institute of Metals and Technology, Lepi pot 11, SI-1000 Ljubljana, Slovenia
[3]Independent Researcher, Betnavska cesta 6, SI-2000 Maribor, Slovenia
[4]Jozef Stefan Institute, Jamova 39, SI-1000 Ljubljana, Slovenia
irena.paulin@imt.si

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Aluminium foams, produced by the powder-metallurgy route, have a good potential for use in weight-sensitive structural parts. The goal of this study was to evaluate the properties and to optimize the preparation of precursors by a powder-compacting process. Various compacting pressures, from 200 MPa to 900 MPa, were used in the double-axial powder-compacting process for two different aluminium powders: pure aluminium and an AlSi12 alloy with the addition of 1 % of TiH_2 as a foaming agent. The green density of the precursors and the distribution of the foaming agent were examined. The powder particles was also characterised. The results of the effective preparation of precursors are shown as the effectiveness of the foaming of the precursors.
The relation between the powder characteristics, the aluminium-alloy properties and the preparation of precursors was studied by SEM/EDS analysis, powder-metallurgy standard testing of metallic powders, granulometry, etc. Different parameters were used for the precursor preparation and foaming. The foaming temperature varied between 680 °C and 770 °C, and the foaming time was from 6 min to 13 min. The relation between the properties and the applied production parameters was studied in detail and is described in this paper.

Keywords: aluminium foam, Al, AlSi12 alloy, powder-metallurgy, powder compacting, sintering

Aluminijeve pene, narejene po postopku metalurgije prahov, imajo velik potencial v uporabi lahkih konstrukcij. Cilj raziskave je ugotoviti lastnosti in parametre za optimizacijo priprave prekurzorja po postopku hladnega stiskanja. Za pripravo prekurzorjev smo uporabili obojestransko stiskanje s tlaki od 200 MPa do 900 MPa. Stiskali smo mešanico prahov tehnično čistega aluminija 99,7 % in zlitine AlSi12, v obeh primerih z dodatkom 1 % TiH_2 kot penila. Določili smo zelene gostote in porazdelitev penila v prekurzorjih. Naredili smo tudi karakterizacijo vseh treh uporabljenih prahov kot vstopnega materiala. Rezultat uspešne priprave prekurzorjev se kaže v uspešnosti penjenja materiala.
Povezavo med lastnostmi prahov, lastnostmi aluminijevih zlitin in priprave prekurzorjev smo raziskovali s SEM/EDS-analizami, s preizkušanjem prahov s standardnimi metodami, uveljavljenimi v metalurgiji prahov, granulometrijo itd. Uporabljeni so bili različni parametri za pripravo in penjenje prekurzorjev. Temperature penjenja so bile med 680 °C in 770 °C, čas penjenja pa med 6 min in 13 min. Povezava med lastnostmi in uporabljenimi parametri za pripravo aluminijevih pen je bila raziskana in je podrobno opisana v tem članku.

Ključne besede: aluminijeve pene, aluminij, zlitina AlSi12, metalurgija prahov, stiskanje prahov, sintranje

1 INTRODUCTION

Aluminium foams are metallic materials with various physical and chemical properties that can be used for several purposes[1,2]. Production methods can be classified into four groups: powder-metallurgy (PM), molten-metal foaming, metallic deposition, and sputter deposition[3]. Each production method gives its own characteristic range of densities, cell sizes and shapes. In this study a method that is adapted to produce complex shapes of metallic foams is discussed, i.e., the powder-metallurgy process.

PM is one of the possible techniques to produce metallic foams. This production process is not as widely used as the less-expensive, molten-metal foaming process, but it also has advantages [4,5]. Its general application in the metal-foaming industry is not very extensive due to the relatively high costs of the input materials. The principle of PM is simple and the process consists of three stages: 1) mixing the metallic powder and the powder of the foaming agent, usually TiH_2, ZrH_2, etc; 2) compacting the powder mixture and; 3) sintering as the final stage of the process. All three steps are important for the quality of the final production and the properties of the aluminium-foam products.

The first step of the PM process is mixing of the metallic powder and the powder of the foaming agent in the proportions needed for the final properties of the product. The higher is the fraction of foaming agent, the greater and/or more numerous are the pores. The addition of the powder ceramic compounds (SiC, CaO) is desired for the stabilization of the foams, but this is not essential [6–8]. The important task of mixing is to achieve a

homogeneous distribution of foaming-agent particles and of the ceramic-powder particles if they are added.

The next important step of the foaming process is the compacting of the powder mixture. The density of the compacted material, the so-called green or theoretical density, is a significant property of precursors and it must be very high (up to 99 %). The shape and size of the metallic-powder particles plays an important role in achieving the highest possible green density of the precursors. However, depending on those properties of the powders, various angles of the repose and bulk densities are achieved. The green density of the precursors also depends on the possible plastic deformation of the metallic powder particles. The lower is the porosity of the precursors, the more of the liberated gas during the sintering is captured in the matrix material.

Sintering is the final stage of the PM production process. During the sintering process at a temperature that is above the temperature of the melting point, and which is specific for each alloy and depends on the foaming agent used, hydrogen is released from the hydride and forms pores in the material. A larger fraction of liberated gases is captured in the matrix material if the molten metal has a higher viscosity [9–11].

The sintering time varies from 3 min up to 10 min, depending on the alloy used for foaming, the foaming agent and the size of the sintered piece. In the decomposition of the TiH_2 used as a foaming agent, the Ti remains and hardens the matrix material while the hydrogen captured in the matrix forms foams with closed-cell structures.

In this study we have focused on the second stage of the PM process. The production of precursors by compacting powder mixtures can be performed in a variety of ways, e.g., by uni-axial, double-axial or isostatic pressing, extrusion, rolling, etc., and all the methods can be hot or cold. Furthermore, the compacting process can be performed in an inert atmosphere, in air or in vacuum [12]. The most economical way is double-axial pressing in air, but the most efficient one is high-temperature extrusion.

2 EXPERIMENTAL WORK

The PM foaming process was applied in our research work. Air-atomized Al powder with a purity of 99.7 % and a D_{50} of 98 μm, air-atomized AlSi12 alloy powder with D_{50} of 42 μm, and TiH_2 powder with a purity of 98 % and a D_{50} of 12.6 μm were used as the starting materials (the purity was specified by manufacturer). The metallic powders were prepared as Ecka granules (Non Ferrum Kranj, d. o. o.), while the TiH_2 powder (325 mesh, 98 %) was purchased from the Sigma-Aldrich Company. In all cases, the metallic powder was mixed with mass fraction 1 % of TiH_2 powder in a turbular mixer (TURBULA WAB Type T2C, 50 Hz, 180 W) for an hour. The characteristics of the powders are shown in **Table 1**. The size and the distribution of the powders were determined by laser granulometry (Alcatel CILAS HR 850-B, isopropanol as analysis medium).

The bulk density, tapped density and free-flowability of the powders were determined. The free-flowability and the bulk density of the powders were determined by the Hall flowmeter funnel that contains a standard flowmeter funnel for metallic powders [13]. A dry test specimen must be carefully loaded into the flowmeter funnel and permitted to run into the density cup through the discharge orifice. In order to determine the free-flowability of the powders it is necessary to measure the time needed for 50 g of powder to pass through the orifice. When the bulk density is determined the powder must completely fill the density cup till it starts to overflow, then the powder is levelled with the top of the cup using a spatula with a blade held perpendicular to the top of cup. Afterwards, the powder is weighed. For a determination of the powder's tapped density a standard mechanical device is used that enables tapping of the graduated cylinder containing powder at a rate of 100 taps per minute [13]. The analyzed powder must be weighed (50 g) and the volume of the powder is read after the tapping.

Powder mixtures were compacted cold, using a double-axial compaction, in a 24-mm-diameter, lubricated, tool-steel die with pressures in the range from 200 MPa to 900 MPa to achieve different green densities [14]. The samples were compacted in INSTRON 1255 equipment with an INSTRON 8800 computer system and the Bluehill2 program. Different pressures were used for both metallic powder mixtures and the green densities were calculated by assuming that the density of the base metal is 2.7 kg/dm^3 when aluminium is used, and 2.65 kg/dm^3 for the AlSi12 alloy. The samples were later sintered at a pre-determined temperature in the air. After various times of sintering, samples were taken out of the retort furnace and quenched into water to solidify the foam structures.

The sintered samples were characterized with a light microscope (LM, Nikon microphot – FXA) and analyzed

Table 1: Characteristics of the used powders
Preglednica 1: Lastnosti uporabljenih prahov

Powders	Manufacturer	Mean particle size d/μm	Purity w/%	Method of manufacturing
Al	Non ferrum, Kranj	106	99.7	Air atomization
AlSi12	Non ferrum, Kranj	47	composition w(Si) = 11.29 %	Air atomization
TiH_2	Sigma-Aldrich	14	98	Ball milling

with the AnalySIS PRO 3.1 program. The size and the distribution of the pores were determined by a standard metallographic method for determining the size and the distribution of the grains in the microstructures [15]. Metallographic samples of the precursors and the foam samples were prepared using a standard metallographic procedure, examined with scanning electron microscope (FE-SEM JEOL JSM-6500F) and analyzed by energy-dispersive electron spectroscopy (EDS, INCA X-SGHT LN2 detector, INCA ENERGY 450 analyzing program) for the elemental analyses. SEM images of the powders were also made.

3 RESULTS AND DISCUSSION

Metallic powders produced in a gas atomizer, in our case in air, have an oblong, quasi-spherical shape with varying size distributions. **Figure 1** shows SEM images of the used powders; (a) Al 99.7 %, (b) AlSi12 and (c) TiH_2. The size and he characteristics of the powders are shown in **Table 1**. The aluminium powder particles (**Figure 1** (**a**)) have a mean size of 106 μm, while the AlSi12 powder particles (**Figure 1** (**b**)) have a mean size of 47 μm, which is half the size of the Al powder particles. However, the morphology of the powders is typical for gas-atomized powders from a liquid metallic melt, and it can be described as a bulk prolonged, semi-spherical shape. On the other hand, the morphology of the TiH_2 powder particles is quite different from that of the metallic powders (**Figure 1** (**c**)). The TiH_2 powder was prepared by the ball milling of titanium in a hydrogen atmosphere under pressure [16–18] and it exhibits an irregular, sharp, polygonal morphology. The mean particle size was 14 μm, significantly smaller than that of the matrix-material powder particles [19].

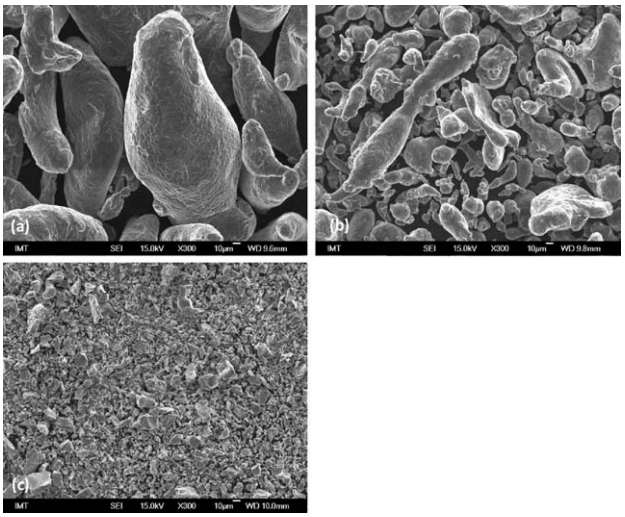

Figure 1: SEM images of as-received powders at the same magnification; (a) Al powder of 99.7 % purity, (b) powder of the AlSi12 alloy, and (c) TiH_2 powder

Slika 1: SEM-slike prahov, narejene pri enaki povečavi; (a) Al prah s čistoto 99,7 %, (b) prah zlitine AlSi12 in (c) prah TiH_2

Table 2: Influence of compacting pressures on the green densities of precursors

Preglednica 2: Vpliv pritiska stiskanja na zeleno gostoto prekurzorjev

	Green density of precursor	
Compacting pressure *p*/MPa	*w*(Al + 1 % TiH_2)/%	*w*(AlSi12 + 1 % TiH_2)/%
200	94.8	75.6
300	97.9	82.4
400	98.1	86.1
500	98.5	89.9
700	98.8	93.2
750	/	94.8
800	/	95.8
900	/	96.2

The surface of all the powder particles was oxidized. The thickness of the oxide layer depended on the affinity of the basic material for oxygen, which was very high for both the aluminium and TiH_2 [20–24]. This oxide layer was examined in our previous work [21,23] and found to be one of the influential parameters for the formation of metallic foams by the powder-metallurgy process. The oxide layer on TiH_2 powders caused a delayed gas evolution, while the oxide layer on the aluminium powder particles increased the viscosity of the melt, which had a favourable influence on the foaming process [11,25].

The free flowability of the Al powder particles was 75 s per 50 g of powder, while the AlSi12 and TiH_2 particles did not flow freely, since they did not have good flowing properties. The bulk density (cumulative volume fraction as a function of the particle size of powder/Al) of the Al powder was 1.23 kg/dm^3, and 1.20 kg/dm^3 for the AlSi12 powder. The TiH_2 powder had a bulk density of 1.34 kg/dm^3. Both powders, the Al and of the AlSi12, had a tapped density of 1.43 kg/dm^3, while the TiH_2 powder had a tapped density of 1.61 kg/dm^3. There was no major difference in the densities of both metallic powders, but there was a difference in the flow properties of the two. Though produced in the same way, with similar tapped densities, the two metallic powders had different final green densities after the compacting process. The characteristics of the TiH_2 powder did not influence

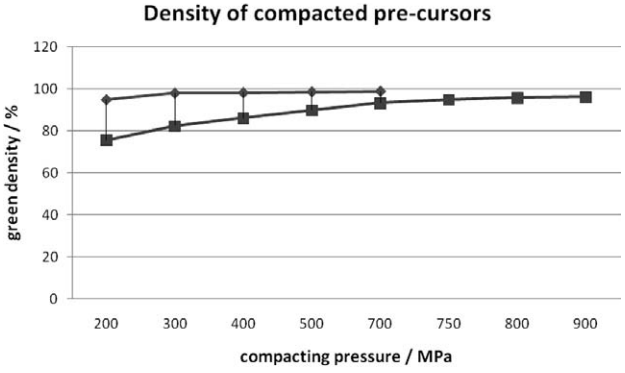

Figure 2: Green density of the compacted precursors

Slika 2: Zelene gostote stisnjenih prekurzorjev

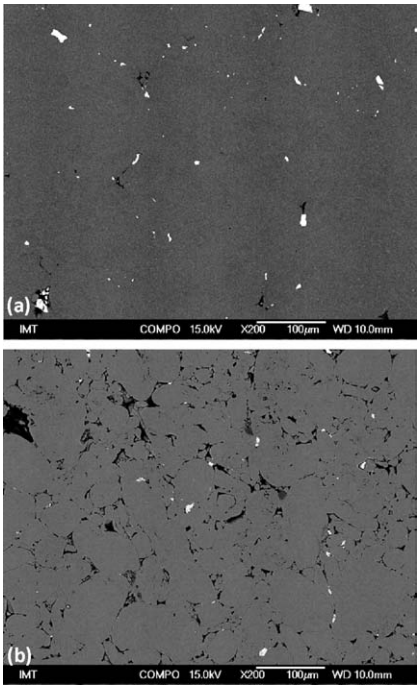

Figure 3: Precursors; double-axially compacted with pressure of 400 MPa, (a) Al precursor of 99.7 % pure Al, determined density was approximately 98.1 %, (b) AlSi12 precursor, determined density was approximately 86.1 %. BE image – bright spots are titanium particles and dark spots the porosity.

Slika 3: Prekurzorji, obojestransko stisnjeni s 400 MPa, (a) Al prekurzor iz Al prahu čistote 99,7 % z zeleno gostoto 98,1 %, (b) AlSi12 prekurzor z zeleno gostoto 86,1 %. BE slike – svetli predeli so titanovi delčki, temni predeli so poroznost.

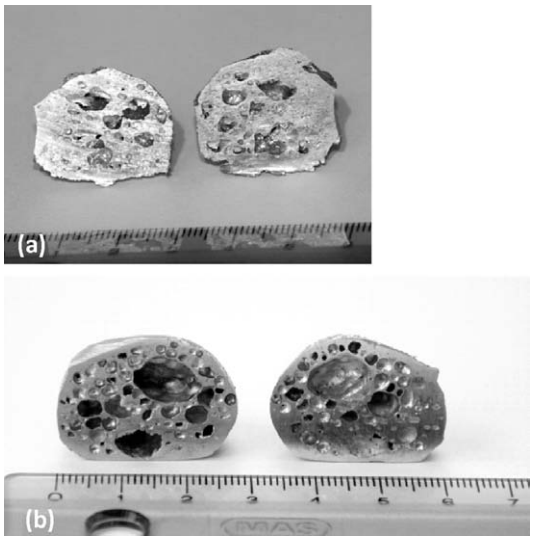

Figure 4: Cross-section of pure Al foams; (a) cold pressed with 200 MPa, sintered at 750 °C for 7 min, (b) cold pressed with 400 MPa, sintered at 750 °C for 6 min 30 s.

Slika 4: Prerez Al-pene iz tehnično čistega aluminija; (a) hladno obojestransko stisnjeno z 200 MPa, sintrano pri 750 °C, 7 min, (b) hladno obojestransko stisnjeno s 400 MPa, sintrano pri 750 °C, 6 min 30 s.

the properties of the precursors, since a relatively small amount of TiH_2 powder was used in the mixture, but its properties did have an influence on the distribution of the TiH_2 powder in the metallic powder matrix. In any case, it was preferable to have a homogenous distribution of TiH_2 particles in the precursor material.

After one hour of mixing the powder mixtures were compacted with various pressures. The influence of the compaction pressures on the green densities of the precursors is presented in **Table 2** and in **Figure 2**. The precursors were examined by SEM and analyzed by EDS. The aluminium precursor that was compacted by the double-axial process with a pressure of 400 MPa is shown in **Figure 3** (**a**). The porosity of the precursor was approximately 2 %. TiH_2, as the foaming agent, was homogenously distributed in the matrix material. On the other hand, the powder of the AlSi12 alloy was also double-axially compacted, with pressures in the range from 200 MPa to 900 MPa. With a pressure of 400 MPa the porosity of the precursors was approximately 14 %. When the compacting pressure was raised up to 900 MPa, the porosity was reduced to only 4 %. Different theoretical densities of the precursors depended on the different qualities of matrix powders, e.g., on the size and the shape of the particles, the bulk and the tapped density. Though the matrix powders had practically the same shape, but different sizes, the compressibility of the powders differed. Compressibility also depended on the bulk and tapped densities of the powders. Double-axial compacting of both, the Al and the AlSi12 powders, at room temperature did not enable us to achieve the same green density through using higher pressures than for the alumosilicates. The limitation of the used compacting pressures was also related to the lubricated tool-steel die that started to stick due to the too high forces in compacting aluminium, while the AlSi12 alloy powder was harder and had a higher compacting resistance. In other words, a larger green density could be achieved with materials that had a better plastic deformability. Though high compacting pressures were applied, the porosity, especially in the AlSi12 precursors, still remained relatively high.

The EDS analysis determined the distribution of Si and Ti in the matrix material. The Si in the AlSi12 matrix was distributed uniformly throughout the whole

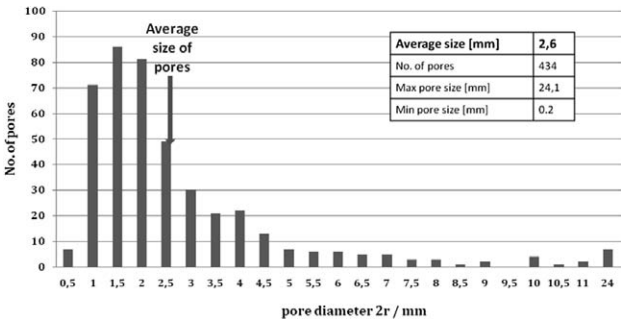

Figure 5: Histogram of pore-size distribution of pure Al foams

Slika 5: Histogram velikosti in porazdelitve por v Al-penah

matrix material, which proved that the AlSi12 powder was manufactured from the AlSi12 aluminium alloy and was not a mixture of Al and Si powder in the demanded ratio. The titanium was randomly but uniformly distributed as individual particles that could be seen in **Figure 3** (**b**) as bright phases in the BE image. On the other hand, only titanium particles were detected in the aluminium matrix, and they were similarly distributed as individual particles in the matrix material, as in the sample of the AlSi12 alloy (**Figure 3** (**a**)). The distribution of TiH_2, with the captured liberated gases around the particles of foaming agent, enabled a homogeneous distribution of pores in the aluminium foams.

The precursors were heat treated in a retort furnace at temperatures that were slightly higher than the melting temperature of the matrix material. All the samples were sintered at temperatures between 680 °C and 770 °C, for various sintering times. The temperatures of the retort in the furnace were controlled with CrNi thermocouple. After sintering at a pre-determined temperature and for a chosen time, the samples were taken out of the retort and quenched in air and/or water. The densities of the samples were then determined with the Archimedes' method.

The samples of pure aluminium that were compacted at lower pressures had fewer pores, and those were not spherical, while the samples that were pressed at 400 MPa or higher pressures contained more pores that were semi-spherical. Cross-sections of the obtained aluminium foams are shown in **Figure 4**.

Light microscopy was employed to determined the size and the distribution of the pores. A diagram of the pore-size distribution in our samples is presented in **Figure 5**. The same comparison was also made with samples of the AlSi12 alloy that were pressed with various forces and sintered at various temperatures and for various times. The cross-sections of the foams are shown in **Figure 6** and a diagram of the pore size distribution is shown in **Figure 7**.

The Al precursors were foamed at 750 °C from 6 min to 10 min. A histogram of the Al foampore size distribution (**Figure 5**) gave evidence that the majority of the pore sizes varied from 1 mm to 3 mm, giving an average

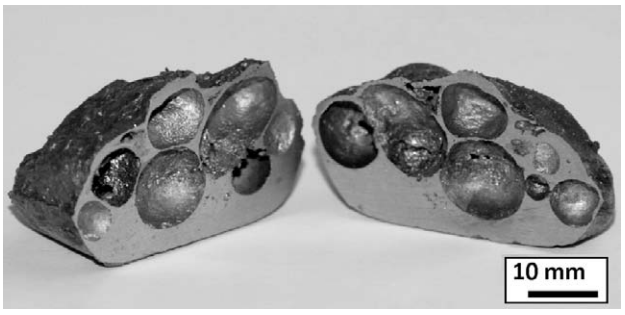

Figure 6: Cross-section of AlSi12 foam, cold pressed with 900 MPa, sintered at 770 °C for 9 min
Slika 6: Prerez pene zlitine AlSi12, hladno obojestransko stisnjeno z 900 MPa, sintrano pri 770 °C, 9 min

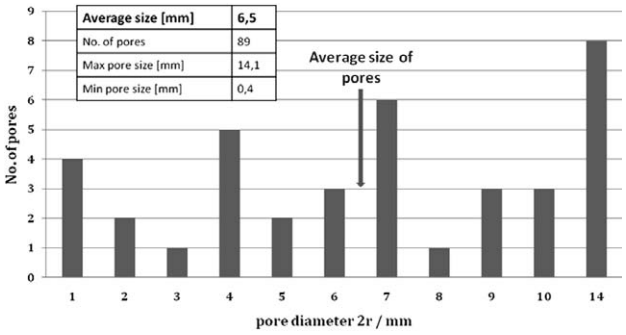

Figure 7: Histogram of pore-size distribution of the AlSi12 foam
Slika 7: Histogram velikosti in porazdelitve por v penah zlitine AlSi12

pore size of 2.6 mm. Only a smaller amount of pores larger than 5 mm was found. On the other hand, the AlSi12 foams were sintered at 770 °C and for times from 6 min to 13 min. A histogram of the pore-size distribution is presented in **Figure 7**. The distribution was very scattered and there was a larger amount of pores with sizes of more than 10 mm.

The difference between the size and the distribution of pores in both foamed materials was due to the green densities of the precursor materials. The compacted material with the higher green density was more easily and uniformly foamed. Therefore, the foaming effectiveness of the AlSi12 precursors was relatively low due to their low green densities (only up to 96 % in our case). The pores in this material were larger due to the prolonged foaming times that were needed to foam the material. This process causes further coarsening of the pores (the large pores were growing at the expense of the small ones).

The density of the foamed material, determined by the Archimedes' method, was from 0.58 kg/dm^3 to 1.2 kg/dm^3 for pure Al foams and from 0.9 kg/dm^3 to 1.6 kg/dm^3 for the AlSi12 foams. The reason for the difference in the densities was in foamability, which depends on the green density of the precursors.

Another difference in the foaming of both aluminium foams was caused by the Si. The AlSi12 alloy is a so-called casting alloy, and according to the Al-Si phase diagram, it is lying near the eutectic point, thus its melting-cooling properties are different from those of pure aluminium. The area between the liquidus and solidus lines is much larger for pure aluminium than for an alloy of the near-to-eutectic composition. Therefore, the temperature range between the liquidus and solidus state in which the pores can be formed is higher with pure aluminium.

4 CONCLUSIONS

It is very important for better foaming results to achieve a green density of at least 98 %. This can be easily achieved with pure aluminium powder. On the

other hand, it is practically impossible to achieve green densities higher than 96 % with AlSi12 powder using only a double-axial compression at pressures even up to 900 MPa at room temperature. For industrial applications it is also important to use as simple industrial machines as possible or to use techniques that are already used in the PM industry for the preparation of different types of complex products. Higher green densities can be achieved by increasing the compacting pressure and/or pre-heating the precursors to increase their compressibility [26,27].

The difference in size of our powder particles does no play such an important role in the difference in the green densities of both precursors, Al and AlSi12, though the bulk density is slightly higher with larger aluminium powder particles. The tapped density is approximately the same for both powders. The most important parameter for achieving higher green densities is the compressibility of powder particles, which depends on the properties of the metallic alloy.

A very important parameter in the foaming process is the foaming temperature. In our study, a temperature of 750 °C was chosen as the most suitable temperature for pure aluminium and 770 °C for the AlSi12 alloy. The results of our study and from the references confirm that the foaming temperature differs from the melting temperature of the matrix material and it cannot be correlated directly from the relation with the melting point of the material. However, while there is no direct correlation between the foaming and the melting temperature, so the foaming temperature should be determined for each matrix material separately, depending on the applied foaming agent. Also, the foaming time has to be chosen in a similar way. It should be determined separately for each alloy and for each foaming temperature.

The most efficient aluminium alloys used for foaming by the PM route are the alloys with higher contents of aluminium and technically pure Al powder. Therefore, so-called aluminium wrought alloys that have a lower content of impurities and of alloying elements (usually less than $w = 1$ %) are more often used for foaming than the casting alloys that have higher amounts of alloying elements. The reason for the selection of wrought alloys is also due to the fact that according to the Al-Si phase diagram the alloys closer to the aluminium corner of the diagram have a higher temperature interval between the liquidus and the solidus temperature in which pores can be formed. In any case, the viscosity of wrought alloys is higher than that of casting alloys, and therefore pores are formed due to the capture of liberated gases from the foaming agents.

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