Al-BASED FOAMS OBTAINED BY POWDER METALLURGY METHOD AND TiH_2 AS BLOWING AGENT. PART I: DETAILS OF FOAMING PROCESS

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Abstract. This article presents a detailed study of the processes which accompany obtaining of aluminum alloy AlSi7Mg3 foam by means of powder metallurgy method. Entire set of the techniques is discussed, including the steps: preliminary powders treatment, powder mixture preparation, compacting of powder mixture, obtaining precursors by extrusion and foaming of the precursors. In order to elucidate processes at each step, the different characteristics of used materials are studied by optical microscopy, Scanning Electron Microscopy (SEM), Energy Dispersive Spectrometer (EDS), X-ray Diffraction (XRD), Differential Scanning Calorimetry (DSC), and X-ray tomography. Main technological parameters and their effect on the structure formation process are discussed. The following conclusions are drawn: (i) The TiH_2 powder should be oxidized at 380 °C for 90 minutes before being used as a foaming agent. Otherwise, its decomposition will start and finish completely for a short period of time before alloying of the powder mixture, and the foaming process will be impossible; (ii) In order to destroy chemically the thin layer of Al2O3 on the Al particles, Mg of about 3 mass.% should be added to the base alloy. The extrusion of the compacted powder mixture should be extruded at a ratio of 11:1, which will ensure a precursor density of not less than 2.6 g/cm^3; (iii) It was found that the foaming process could be realized most effectively at about 670 °C for 10 minutes; (iv) The size of pores and amount of porosity could be controlled by the foaming time.

Keywords: Al-based foams, powder metallurgy method, foaming process, SEM, EDS, XRD, DSC.

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1. INTRODUCTION

Metallic materials with high porosity, which could be considered as biomimetic ones, have increasing application for structural and functional elements in various engineering constructions. In the last three decades, aluminum-based foams have been the focus of many investigations and find more and more industrial applications. The increasing interest to these materials is due to their specific combination of mechanical and physical properties such as high strength-to-weight ratio, large specific surface area, high level of energy and sound absorption capacity, controllable permeability of open-cells materials, considerable vibration reduction ability and others [1–3]. The basic research in this field is directed to design of composition as well as macro- and microstructures of the cellular materials in order to meet some predefined requirements to obtained materials. Large variety of production methods has been developed [4–6]. The man-made metallic porous materials can be classified in respect to their structure as follows:

– foams (conventional and syntactic) in which the pores are closed;

– structures obtained by replication methods in which the pores are open;

– gasars-type obtained by solidification of gas-saturated melts in which the porosity is ordered [7–8].

In general, metallic materials with high porosity are fabricated by means of *in vitro* (case of open cells structures) or *in situ* (case of closed cells structures) formation of a space holder. In both cases, the space holder provides for a void space in the final structure. The *in vitro* process includes preliminary formation of a solid body with high porosity (space holder), which to be infiltrated by liquid metal and then to be removed in order to obtain a metallic skeleton with open porosity [6]. The space holder formed *in situ* is a gas, which in most cases is H_2, and is released after chemical decomposition of some substance, for example TiH_2, respectively called a "foaming agent". Gasar technology also applies H_2 as a space holder, but it is released at the solid/liquid interface during solidification of the metal.

In this paper, our efforts are directed to the elucidation of specific processes accompanying the application of Powder Metallurgical (PM) methods of obtaining high porosity closed-cells aluminum foam. Here, we present original results and pay a special attention to the influence of alloy composition, temperature, preliminary treatment of used powders, and the time and pressure applied to compact the powder mixture. Different characteristics of used materials and specific features of the obtained foams are studied by means of Optical microscopy, Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), Differential Thermal Analysis (DTA), and X-ray tomography. Main

technological parameters and their effect on the structure formation process are also discussed. Such studies are valuable because they could help technologists develop additional processing techniques and effectively find a better alloy composition.

2. EXPERIMENTAL PROCEDURE

The technique for production of closed-sells foam consists of 5 different steps. They could be listed as follows, see Fig. 1:

1. Powders selection and preliminary preparation;
2. Powder mixing;
3. Compaction and precursors formation;
4. Additional mechanical processing – extrusion;
5. Sintering and foaming.

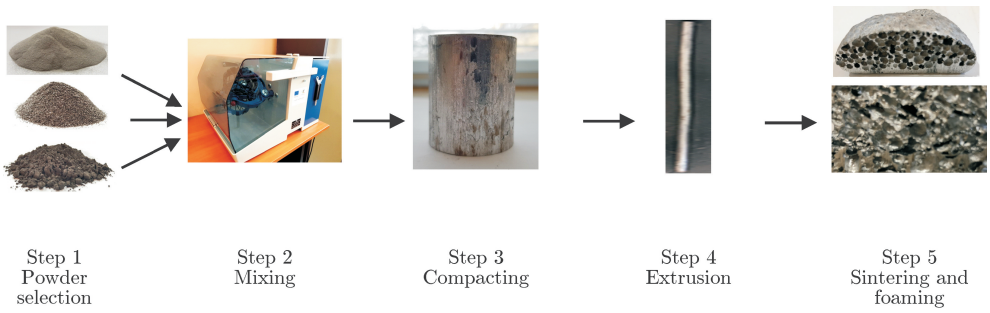


Fig. 1. Steps of PM technique for production of closed-cells metallic foams

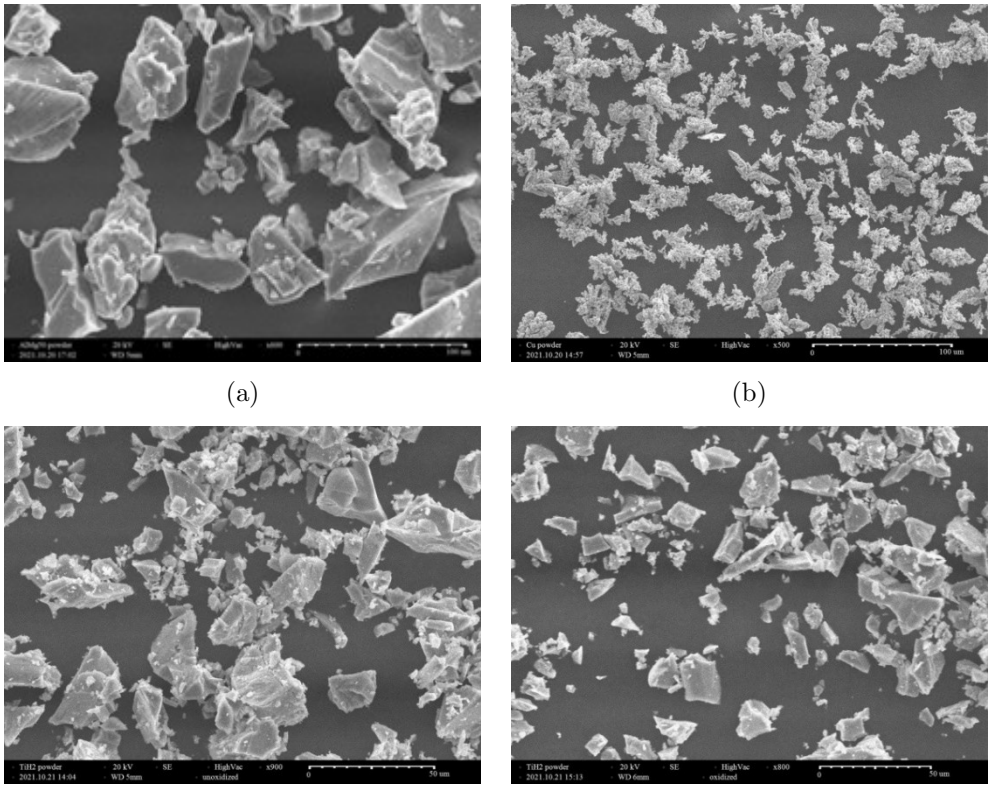
2.1. Materials and preliminary processing

The first step determines the chemical composition of the aluminum alloy we want to use, the amount of the porosity we expect to obtain, and the temperature and time which should be used during thermal treatment in the last step. In this paper we study the alloy AlSi7Mg3Cu. It was obtained from Al, Si, AlMg50 and Cu powders in different percentages. The size of particle was predominantly in the range 35–125 μm. SEM images of these powders are shown in Fig. 2. The Al powder particles are covered by a thin layer of Al_2O_3, which is an insuperable obstacle to their melting and obtaining an integrated body by sintering in Step 4. This oxide layer should be removed by chemical

reaction and/or destroyed mechanically by high pressure. The role of Mg is to reduce Al_2O_3 according to the reaction:

$$Al_2O_3 + Mg \rightarrow MgO + Al. \tag{1}$$

During Step 3 and Step 4, a mechanical destruction of the Al oxide layer takes place and thus melting of the aluminum powder becomes possible.



(a) (b)

(c) (d)

Fig. 2. SEM photos of used powders: (a) AlMg50; (b) Cu; (c) TiH_2 before oxidation; (d) TiH_2 after oxidation

Another specific operation should be carried out before powder mixture preparation. It is related to the decomposition process of TiH_2. This process is more intensive at 560–580 °C. The sintering becomes successful at a temperature in the range 580–620 °C and lasts about 20 minutes, which will provide conditions a large amount of H_2 to leave without causing foaming. In order to slow down decomposition velocity we stimulate the formation of an oxide layer

on the TiH_2 particles by heating at 380 °C for 90 minutes. That preliminary processing does not cause any visual changes, compare Fig. 2 (c and d), but effectively reduces the decomposition velocity.

2.2. Powder mixing, compaction and precursor formation

The homogenization of the powders was accomplished by using 3D powder blender mixer – WAB T2F Turbula Heavy-Duty Shaker-Mixer, in Step 2. The chemical composition of this powder mixture is as follows: Al 87.9%, Si 7.1%, Mg 3.2% and TiH_2 1.8%. Variations of the amount of Al and TiH_2 are tested and the composition was found to be optimal. The obtained powder mixture was compressed in Step 3 at different temperatures and pressure 450 MPa for 60 s in order to obtain cylindrical compacts with a diameter 40 mm and a height 60 mm. Additional mechanical treatment was conducted by extrusion of the obtained precursors in Step 4. The extrusion was carried out at 300 °C and a reduction ratio of 11:1.

2.3. Sintering and foaming

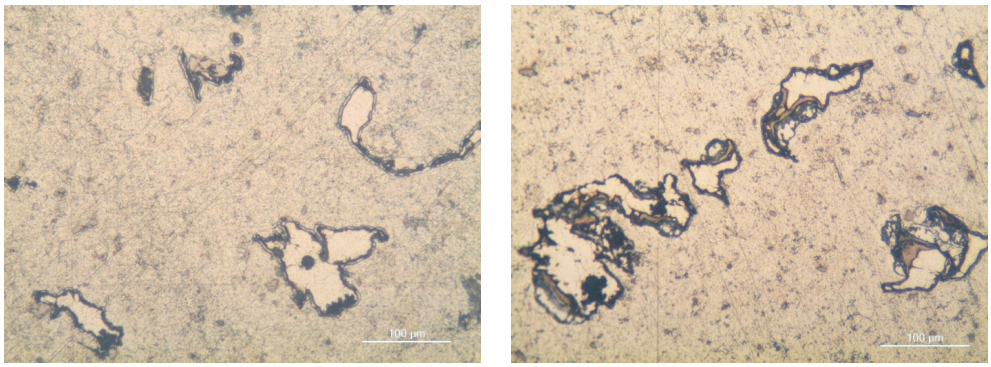
The prepared precursors were ready to be sintered and foamed in Step 5 in order to obtain high porosity materials. The sintering process could provide a liquid phase and obtain an AlSi7Mg3 alloy. In addition, thermal decomposition of TiH_2 and foaming have to take place. The foaming process was realized in a furnace at a temperature 760 °C for a time of about 10 minutes. This time also depends on the thickness of the precursor which should be foamed.

2.4. Applied equipment and methods

Characterization of the obtained materials is carried out by various equipment and methods. Metallographic analysis was performed using a Leader series LM-308BD vertical microscope with a digital camera. SEM observations were carried out on a HIROX SEM SH-5500P (Hirox, Japan) and EDS system QUANTAX 100 Advanced (Bruker, Germany). DTA measurements were conducted on a NETZSCH DSC/DTA machina STA 449 Fx Jupiter. Tomography study was realized on a computer X-ray microtomography SKYSCAN 1272 (Bruker, Germany).

3. RESULTS AND DISCUSSION

The powder compacting process and subsequent extrusion are aimed to provide the highest possible compaction of the powder particles and mechanical destruction of the oxide layer that covers Al particles. The microstructure of the compacts and precursors are shown in Fig. 3. The calculated density of the compact is 2.24 g/cm^3 and the density of the precursor is 2.65 g/cm^3. It can be seen that the precursor density is near 20% higher than the compact density and is in fact equal to the density of aluminum. Both values give us confidence that the precursors are free of porosity and could be successfully foamed. It was found that addition of up to 2 mas.% Cu does not affect foaming process and final foam structure. For this reason, we did not use Cu as an alloying element in our next study.



(a) (b)

Fig. 3. Optical images: (a) microstructure of Al-Si7-Mg3-TiH_2 compact (after compacting); (b) microstructure of Al-Si7-Mg3-TiH_2 precursors (after extrusion)

A view of closed-cells foam obtained after foaming of precursors is shown in Fig. 4. The pore size is predominantly in the range of 3–7 mm, and the pore surface is smooth. The X-ray tomography observation shows that the foamed precursors have homogeneous porosity, which indicates a homogeneous distribution of TiH_2 obtained after powders mixing, see Fig. 5.

An SEM image of the pore surface can be seen in Fig. 6. Microporosity is not observed, and the pore walls could be considered free of pores, which ensure better mechanical strength of the foam obtained.

The EDS analysis of chemical composition on the pore surface is given in Table 1. It can be seen that the composition is the same as in the bulk alloy and there is no segregation of chemical elements during the foaming process.

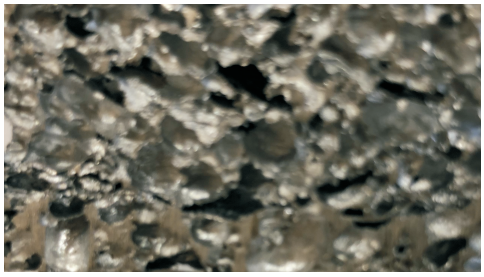


Fig. 4. Pore view in closed-cells aluminum alloy sample

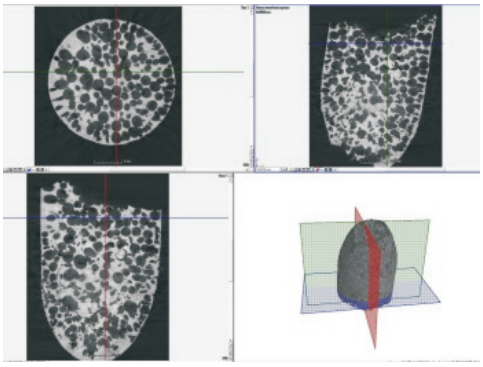


Fig. 5. Tomography images of various cross-sections of test sample

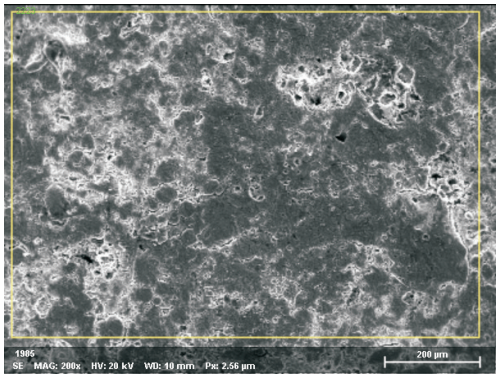


Fig. 6. SEM image of inner pore surface of closed-cells aluminum alloy sample

Table 1. EDS analysis of pore surface, mas.%

	Al	C	O	Mg	Fe	Si	Ti	Cu	Total
Aver., %	73.91	1.73	10.60	6.17	0.57	6.69	0.33	0.24	100.00

In order to have more information about the thermal effects on the foaming process in our precursors, we carried out a DSC analysis. The result is shown in Fig. 7. The main components of the foaming process are melting of Al powder, alloying and decomposition of TiH_2. All these processes are endothermal, which can be seen in the DSC curve in Fig. 7. The beginning of the processes starts at 653.2 °C and ends at 676.9 °C. It can be seen that the most intensive decomposition of TiH_2 is in the temperature range 653–677 °C. This

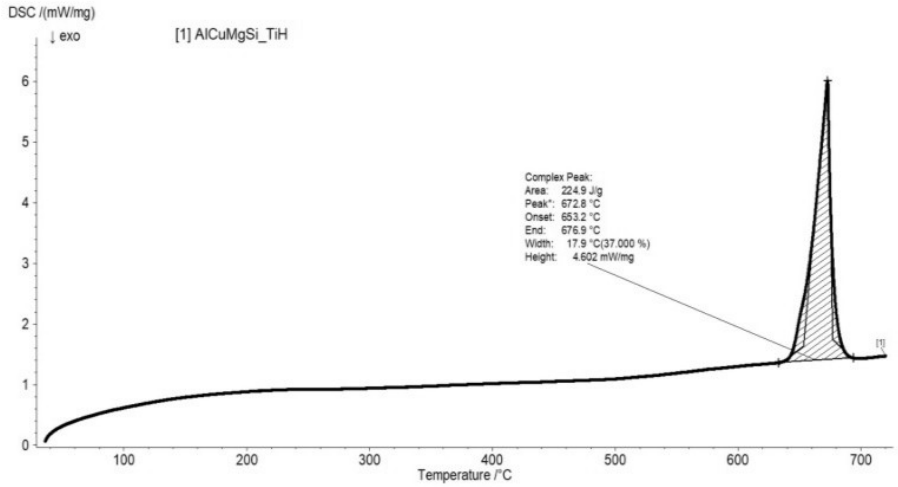


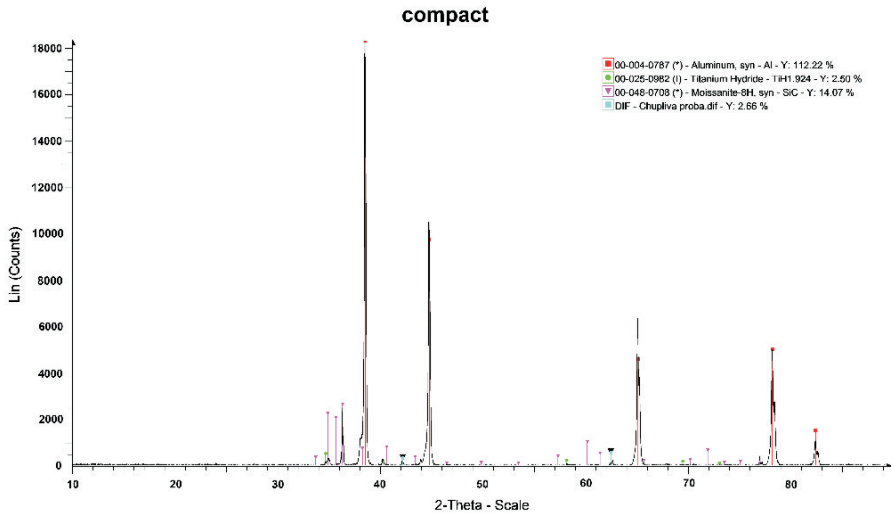
Fig. 7. DSC curve of the precursor mixture. Both processes of melting and TiH_2 decomposition are isothermal, and the combined thermal effect is clearly seen

information is very important for people who want to foam a large amount of precursors in a resistive furnace.

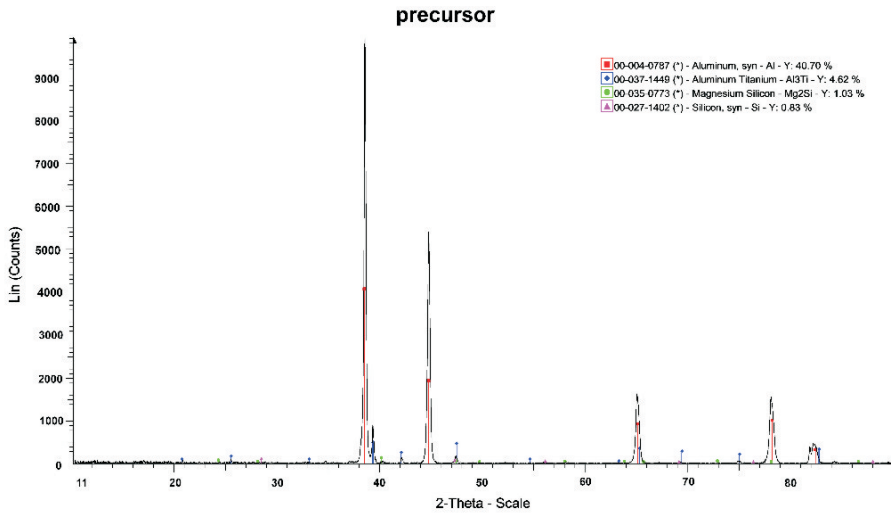
The results of the phase analysis carried out by XRD method are shown in Fig. 8. The phase composition of precursor contains mainly Al, $TiH_{1.924}$, Si and SiC, see Fig. 8(a). The phase composition of the obtained foam consists of Al, Al_3Ti, Mg_2Si and Si, which means that the decomposition of TiH_2 is totally accomplished during foaming. There is no reason the foaming process to last longer than 10 minutes, as the source of H_2 is still exhausted and the foamed liquid will start to reduce uncontrollably its volume. The time period for foaming should be shortened to less than 10 minutes, which will result in a smaller pore size and lower porosity of the material obtained.

4. CONCLUSION

This article presents a detailed study of the processes involved in the preparation of aluminum-based foam (AlSi7Mg3) by methods of powder metallurgy. Entire set of the techniques is considered, including the steps: preliminary powders treatment, powder mixture preparation, compacting powder mixture, obtaining precursors by extrusion and foaming of the precursors. In order to elucidate processes at each step, the different characteristics of used materials and specific features of the foams obtained are studied by means of optical mi-



(a)



(b)

Fig. 8. Results from XRD observation of: (a) precursor material before foaming; (b) foam obtained from the same precursor

croscopy, SEM, EDS, XRD, DSC and X-ray tomography. Main technological parameters and their effect on the structure formation process are discussed. The next conclusions could be drawn:

- The TiH_2 powder should be oxidized at 380 °C for 90 minutes before being used, as foaming agent. Otherwise, its decomposition will start and finish completely for a short period of time before alloying of the powder mixture, and the foaming process will be impossible.
- In order to destroy chemically the thin layer of Al_2O_3 on the Al particles, Mg of about 3 mass.% should be added to the base alloy. The extrusion of the compacted powder mixture should be extruded at a ratio of 11:1, which will ensure the precursor density of not less than 2.6 g/cm^3.
- It was found that the foaming process could be realized most effectively at about 670 °C for 10 minutes.
- The size of pores and amount of porosity could be controlled by the foaming time.

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