

MAKING LARGE CLOSED CELL-METAL FOAM FOR STRUCTURAL APPLICATIONS

Lucian Paunescu¹, Sorin Mircea Axinte² and Adrian Ioana³

¹ Daily Sourcing & Research SRL Bucharest, Romania, *Corresponding author*, ORCID No. 0000-0002-2467-5120
lucianpaunescu16@gmail.com

² National University of Science and Technology POLITEHNICA, Faculty of Applied Chemistry and Materials Science, Bucharest, Romania, sorinaxinte@yahoo.com

³ National University of Science and Technology POLITEHNICA, Faculty of Materials Science and Engineering, Bucharest, Romania, ORCID No. 0000-0002-5993-8891 adyioana@gmail.com

ABSTRACT: Commercially recycled aluminum scrap was processed by an own method of melting under the influence of microwaves and atomizing the melt with nitrogen gas jets. Fine aluminum granules together with titanium hydride as an expanding agent and a subsequent addition of very fine alumina powder as a foam stabilizer constituted the materials used to produce large-cell aluminum foam for light structural applications. The material mixture was heated to 753-768 °C in a microwave oven functionally adapted for high temperatures. The use of microwave irradiation of the material allowed to obtain extremely high heating rates (53.4-91.6 °C·min⁻¹) and very short process times. The aluminum foams made in this way had excellent features regarding thermal insulation properties (low values of density and thermal conductivity), a sufficiently high compressive strength and cell size of the foam structure up to almost 2 mm, being suitable for light structural industrial applications.

KEYWORDS: aluminum foam, own method, titanium hydride, alumina powder, heating rate, structural application.

1. INTRODUCTION

Porous metals or metal foams began to be used in engineering applications only at the beginning of the 20th century. The first reference in the literature to metal foams, produced by expanding a metal precursor to form a foamed structure with high porosity, is a French patent [1]. The commercial exploitation of metal foams was first noted in the late 1950s in the United States. The development of research in this activity field worldwide was only noticed in the early 1990s, but it is still in continuous progress today, metal foams being required in applications with a wide range of properties and structures [2].

The disadvantage of ceramic material fragility and the difficulty of their processing can be overcome by using porous metallic materials due to their unique characteristics. This type of materials has started to be very interesting for different fields (energy, environment, chemistry, medicine, etc.), and their exploitation is already being felt on a large scale [3].

Making metal foams is oriented towards the realization of closed-cell or open-cell foam materials. Closed-cell foams provide better mechanical properties, but have a sealed structure, being used in structural applications. Open-cell foams, having a permeable structure, are mainly intended for functional applications [2].

The main applications of metal foams fall into two categories: structural and functional [4]. Structural

applications rely on the foam low weight and excellent mechanical properties, while functional applications rely on the high free surface area, adequate thermal conductivity for heat dissipation, and very good electrical conductivity for use as a battery electrode.

In general, a metal melt is foamed with a gas, stabilized with non-metallic particles and finally solidified. The gas bubbles can be obtained either by injecting a gas or by decomposing a chemical compound used as an expanding agent inside the melt [5]. Foam stabilization means maintaining them in a state of physical separation from each other and is due to the addition of non-metallic particles, mentioned above [6].

The research areas currently investigated in the world with reference to metal foams are related to foaming and stabilization mechanisms, optimization of their production processes, research for the use of new expansion agents and reduction of production costs.

The paper [7] chose single liquid films to facilitate the understanding of the properties of the cell walls of metal foams and to investigate the foaming and stabilization of these foams in more depth. The stabilization of metal foam is achieved by introducing ceramic particles into the metal melt. These have the ability to adhere to the gas/metal interface during the foaming process, preventing cell coalescence. This work presented the results of

using 10-20 vol. % silicon carbide (SiC) or alumina (Al_2O_3) particles in a liquid matrix composite, in which an expanding gas is injected. The results showed that very regular porous foams are obtained in this way. However, high particle proportions lead to embrittlement of the foam. This negative effect can be removed by replacing relatively larger particles with nanoparticles. Thus, 5 % SiC particles with dimensions below 70 nm used in the paper [7] were satisfactory in the foaming process. In another work [2], 4 % titanium carbide (TiC) particles with sizes between 200-1000 nm added to an aluminum melt favours the formation of stable foams.

Other investigations have focused on the nature of the blowing agent used in the production of metal foams. Titanium hydride (TiH_2) has been adopted since the 1950s, and is still considered to be a very strong and suitable agent for the production of foams from aluminum and magnesium alloys. However, several disadvantages of applying this agent type have been identified: the rather high price that contributes to an inadequate cost for the entire process of manufacturing the metal foam as well as the decomposition temperature of TiH_2 in disagreement with the melting temperature of aluminum and magnesium alloys, which generates the premature release of hydrogen gas. The problems related to the early release of the foaming gas could be improved by affecting the features of TiH_2 by oxidation or by coating its particles with a thin layer of nickel [2]. The problem of the high cost of the agent can be solved by substituting it with a more financially accessible expanding agent, for example with calcium carbonate.

Closed-cell foams are the type of foam on which the current work focused. Two main routes for the fabrication of these foams are known: melt foaming and powder metallurgy foaming [8].

The first commercially produced metal foams by melt foaming were manufactured by Shinko Wire (Japan) in the late 1980s. Calcium was added and stirred into an aluminum melt to stabilize the foam by increasing its viscosity. The traditional foaming agent (TiH_2) in powder form was introduced into the melt and, by stirring, a mixture homogeneity was obtained. The melt processed inside a crucible has foamed. After cooling, the foam is usually cut into slabs. This metal foam manufacturing process is known as Alporas, designed in the United States [9].

Another variant of melt foaming of an aluminum alloy was developed in the early 1990s by Alcan International Ltd. (Canada) and Norsk Hydro (Norway). This process is characterized by preparing the melt with SiC particles or aluminum oxides in

proportions between 5-20 vol. % to stabilize the foam. Then, dispersed air bubbles are injected into the melt. The bubbles, raised to the surface of the melt, are integrated into a liquid foam and begin to solidify after leaving the furnace on a conveyor belt. The method described above is currently applied by Cymat, Mississauga, Canada, producing foam panels [10]. A high-performance method characterized by reducing cell size and increasing the homogeneity of the foam structure, through gas injection and ultrasonic melt treatment, was developed by Aluivent, Miskolc, Hungary [11].

The first information regarding the powder metallurgy foaming method was a patent from 1963 [12]. The method is based on the indirect foaming of solid precursors by heating. The precursor is formed by mixing aluminum powder with appropriate alloying components and an expanding agent (usually 0.5-1.0 wt. % TiH_2). After forming the mixture, it is consolidated and sintered by extrusion, uniaxial compaction or lamination. Heating the mixture until melting begins leads to the nucleation of the gas released by the foaming agent. By increasing the temperature, the amount of released hydrogen increases and diffuses to the nucleated bubbles, causing their volumetric growth and the development of the foam. Oxides in the metal powders (up to 1 %) ensure the stability of the foam during its maintenance in liquid state. When the foam growth is complete, its structure can be preserved by cooling and solidifying the foamed material. A new metal foam product based on the powder metallurgy has been developed by Pohltec Metalfoam, Collogne, Germany in 2012 [13]. This product is an aluminum foam sandwich in the form of a lightweight panel with thicknesses between 8-80 mm, composed of a foam core and two aluminum alloy-based face sheets. The material processing consists of mixing the metal powder and the expanding agent and introducing them into a welded container. Then, the container is sealed and hot-rolled into a compact three-layer precursor. The advantage of this process is the metallurgical bonding between the face sheets and the foam core, resulting in a rigid product with high temperature resistance. The foamed product is also fully recyclable.

Since powder metallurgy foaming involves several processing steps, the idea of integrating them into one was investigated. Thus, a proposed technical solution was high-pressure casting of structural foams. According to [2], the process involves injecting a molten aluminum alloy into a die casting machine together with the expanding agent (MgH_2) and the possibility of controlled foaming of the

components inside the die in which the casting was performed.

Most of techniques for producing closed-cell metal foams have been oriented towards the use of aluminum as a precursor. The reason for this preferential choice is the set of suitable features such as low melting temperature of aluminum, its ease of processing and its favourable mechanical properties. However, the efforts of researchers to manufacture foams with other metals (steel, zinc, magnesium and even some heavy elements) should not be neglected.

Some authors of the current paper have conducted previous research on the experimental fabrication of aluminum foams, bodied in two articles published in the literature between 2019-2023 [14, 15]. In both experiments, the fine aluminum powder was produced by an own melting technique using the microwave radiation and atomization of the resulting melt with nitrogen gas jets. The foaming of the aluminum powder was performed using dolomite and calcium carbonate, respectively. The heating of the powder for foaming was carried out at temperatures between 700-750 °C in a conventional microwave oven, adapted for use at high temperatures. The characteristics of the produced foams were excellent, with density ranging between 1.17-1.19 g·cm⁻³ [14] and 0.88-1.10 g·cm⁻³ [15], respectively, thermal conductivity ranging between 5.71-5.84 W·m⁻¹·K⁻¹ [14] and 3.30-5.65 W·m⁻¹·K⁻¹ [15], respectively, and compressive strength ranging between 6.83-7.01 MPa [14] and 3.9-7.4 MPa [15]. The cell size in the foam structure was within the limits of 0.4-0.9 mm [14] and, respectively, below 0.45 mm [15]. Non-metallic particles for stabilizing the metal foam were not used in either of the two mentioned experiments.

Among the known techniques and mentioned above for producing metal foam, melt foaming is considered the most accessible, leading to the production of relatively inexpensive foams with adequate properties [16, 17].

In the current work presented below, pre-oxidized TiH₂ powder was chosen as a suitable expanding agent for obtaining aluminum foams. The optimal way to oxidize the powder with atmospheric oxygen is to heat it up to 440 °C for 1 hour. The fine particles acting as foam stabilizers chosen for this experiment were alumina particles (Al₂O₃). The original method of using microwaves in processing the fine aluminum powder as well as in the heating/foaming process of this precursor was also preserved in this work.

2. METHODS AND MATERIALS

2.1 Methods

According to [18], the decomposition rate of TiH₂ increases with increasing temperature, influencing the decomposition process of the hydride, which can be grouped into several stages in the temperature range between 620-720 °C. In the first stage (620-680 °C), the decomposition of TiH₂ is violent in the first 10 min after starting, then the decomposition rate becomes slow in the next 10 min, and the process tends to stagnate after 20 min. In the range of 700-720 °C, the decomposition rate of TiH₂ becomes very fast again in the first 6 min after starting, then decelerates in the next 4 min and stagnates thereafter. At the beginning of the decomposition, a less-hydrated titanium is formed, followed by decomposition to almost pure metallic titanium with dissolved hydrogen and only finally, after the temperature of the mixture exceeds the melting point of aluminum, the hydrogen gas is completely released. This controlled release of gas into the matrix of softened/molten aluminum causes the material expansion, forming the foam structure.

The working method corresponding to the experimental process of producing aluminum foam included several steps. First, aluminum powder together with TiH₂ as a foaming agent (in proportions between 0.5-1.7 wt. %), the mixture being slightly moistened, were mixed until a wet suspension was formed. This was poured into a ceramic crucible made in China from silicon carbide and silicon nitride in a mass ratio of 80/20, which is highly microwave absorbent. The ceramic crucible was thermally protected on its outer wall with ceramic fibre mattresses resistant up to 1200 °C. The crucible containing the suspension was placed in a commonly used 800 W-microwave oven, constructively and functionally adapted to withstand high temperatures of over 1100 °C (Figure 1). The recommended heating rate in the process of manufacturing aluminum foam with TiH₂ through the melting foaming process, without stabilizer particles, is extremely high (from about 90 °C·min⁻¹ to values of several hundred °C·min⁻¹) [19]. In the current experiment, the heating rate was very high, but under 100 °C·min⁻¹. Alumina particles were used as a foam stabilizer, being introduced into the mixture melt immediately after reaching the melting point of aluminum by briefly stopping the power supply of the oven and mixing alumina particles into the melt.

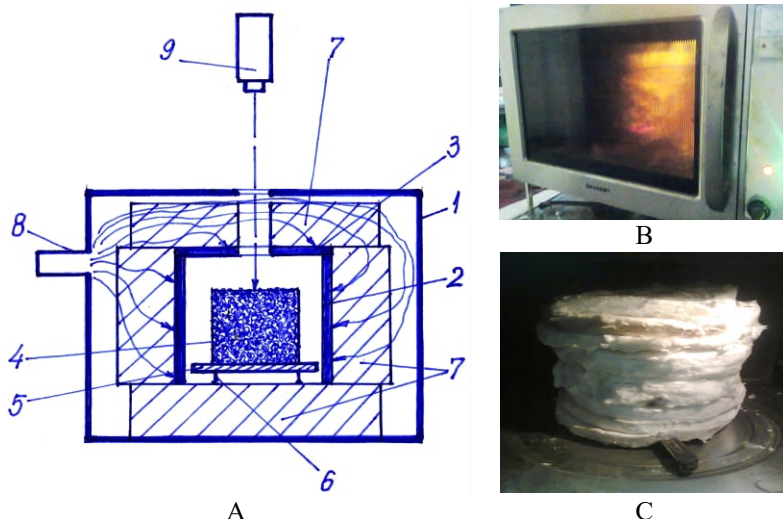


Figure 1. Experimental microwave equipment

A – Construction and functional scheme of the equipment: 1 – microwave oven; 2 – ceramic tube; 3 – ceramic lid; 4 – pressed sample; 5 – metal plate; 6 – support; 7 – ceramic protection; 8 p waveguide; 9 – radiation pyrometer; B – image of the oven; C – ceramic protection.

2.2 Materials

The materials chosen for this experiment were: aluminum powder, titanium hydride (TiH_2) and very fine alumina ceramic particles (Al_2O_3).

Aluminum powder with particle sizes below $30\ \mu\text{m}$ was produced by the own method of nitrogen gas atomization of the molten aluminum jet obtained by microwave irradiation of recycled aluminum waste.

Titanium hydride powder according to ASTM B348 with particle size below $25\ \mu\text{m}$ and apparent density of $2.8\ \text{g}\cdot\text{cm}^{-3}$, was the adopted expanding agent. The choice of this type of agent was justified by the research objective of obtaining larger cells of the metal foam, despite the higher price compared to the more economical solutions of using calcium carbonate or dolomite, but which lead to considerably finer metal foam structures.

Alumina powder with very high purity (over 99.90 %) was chosen as an aluminum foam stabilizer. The size of alumina particles varies between nano-size and $500\ \mu\text{m}$. For the current experiment, a powder with dimensions below $1\ \mu\text{m}$ was commercially purchased.

2.3 Investigation methods to determine the metal foam features

The apparent density was measured using the gravimetric method [20]. The porosity was calculated as a percentage of the difference between the estimated density of the non-porous aluminum foam and the measured apparent density including

the pore volume, reported to the density of the non-porous foam, according to NE 012-99. Determining the heat conductivity was performed with the HFM 446 Lambda apparatus based on the heat-flow method (SR EN 1946-3:2004). The compressive resistance was measured using a 2000 kN hydraulic axial press (EN 826:2013). The microstructural features of the foamed samples were examined with the Smartphone microscope type ASONA 100X Zoom.

3. RESULTS AND DISCUSSION

3.1 Results

The components of the mixture for the production of aluminum foam were dosed into four groups, according to Table 1, aiming to maintain a constant total quantity for each group. The weight proportion of titanium hydride was progressively increased from 0.5 wt. % in variant 1, to 1.7 wt. % in variant 4, while the proportion of alumina particles was kept constant at 3.0 wt. %. By default, the weight of aluminum powder suffered a slight decrease from 96.5 wt. % to 95.3 wt. % between the four groups.

Table 1. Composition of materials used for producing the foam

| Material (wt. %) | Variant 1 | Variant 2 | Variant 3 | Variant 4 |
|-------------------|-----------|-----------|-----------|-----------|
| Aluminum powder | 96.5 | 96.1 | 95.7 | 95.3 |
| Titanium hydride | 0.5 | 0.9 | 1.3 | 1.7 |
| Alumina particles | 3.0 | 3.0 | 3.0 | 3.0 |

| | | | | |
|-------|-----|-----|-----|-----|
| Total | 100 | 100 | 100 | 100 |
|-------|-----|-----|-----|-----|

The operational parameters of the microwave heating/foaming process are shown in Table 2. The final process temperature increased from 753 to 768 °C, while the very high heating rate decreased from 91.6 to 53.4 °C·min⁻¹ and the process duration was very short (between 8-14 min).

Table 2. Operational parameters of the heating/foaming process

| Parameter | Variant 1 | Variant 2 | Variant 3 | Variant 4 |
|---|-----------|-----------|-----------|-----------|
| Mixture amount (g) | 230 | 230 | 230 | 230 |
| Heating/foaming temperature (°C) | 753 | 758 | 765 | 768 |
| Process time (min) | 8 | 9 | 11 | 14 |
| Heating rate (°C·min ⁻¹) | 91.6 | 82.0 | 67.7 | 53.4 |
| Foam amount (g) | 216 | 217 | 215 | 216 |
| Specific energy consumption (kWh·kg ⁻¹) | 0.386 | 0.432 | 0.533 | 0.675 |

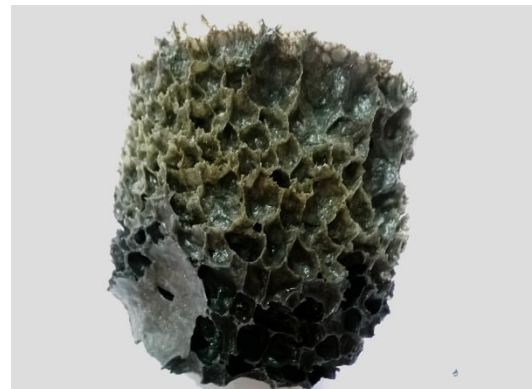
The physical, thermal, mechanical and morphological features of the aluminum foam samples made in this experiment are exposed in Table 3.

Table 3. Features of aluminum foam samples

| Feature | Variant 1 | Variant 2 | Variant 3 | Variant 4 |
|------------------|-----------|-----------|-----------|-----------|
| Apparent density | 0.89 | 0.72 | 0.61 | 0.49 |



A



B

| (g·cm ⁻³) | | | | |
|---|---------|---------|---------|---------|
| Porosity (%) | 68.9 | 70.8 | 73.0 | 76.4 |
| Heat conductivity (W·m ⁻¹ ·K ⁻¹) | 4.93 | 4.12 | 3.65 | 3.04 |
| Compressive strength (MPa) | 6.5 | 5.4 | 4.1 | 3.4 |
| Cell size (mm) | 0.2-0.8 | 0.3-0.9 | 0.5-1.0 | 0.9-1.9 |

The data in Table 3 reflect the ability of the aluminum foam samples, using TiH₂ as the expanding agent and the melt foaming method, to exhibit excellent thermal insulation properties. Thus, the low values of apparent density (0.49-0.61 g·cm⁻³) and thermal conductivity (3.04-3.65 W·m⁻¹·K⁻¹), especially highlighted in the case of samples C and D made by experimental variants 3 and 4, are perfectly suitable for light structural applications at the qualitative level of similar products manufactured by conventional heating methods. The foam cell size has higher values compared to foams made using cheaper expanding agents (calcium carbonate or dolomite) tested by the authors in previous experiments [14, 15]. On the other hand, the compressive resistance of samples C and D is slightly affected by the manufacturing recipe applied in their case compared to samples A and B. Thus, the resistance value decreased to 3.4-4.1 MPa, but is still sufficiently high considering the low density of the foamed products.

Images of the aluminum foam samples made by the four experimental variants are exhibited in Figure 2. Also, the microstructural appearance of samples is presented in Figure 3.



Figure 2. Images of aluminum foam samples
A – variant 1; B – variant 2; C – variant 3; D – variant 4.

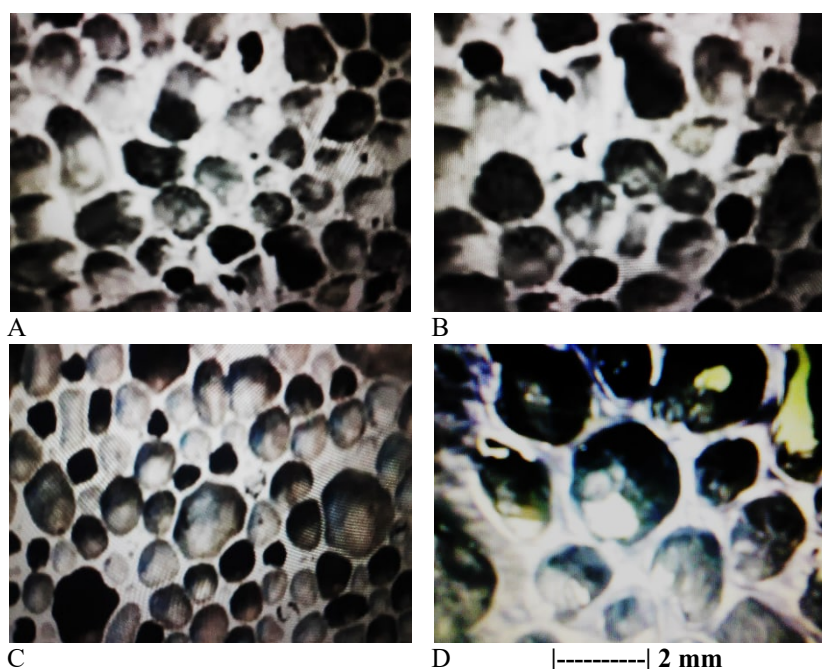


Figure 3. Microstructural appearance of aluminum foam samples
A – variant 1; B – variant 2; C – variant 3; D – variant 4.

3.2 Discussion

The analysis of the results regarding the physico-thermal and mechanical characteristics of the aluminum foam samples, including also their microstructural peculiarities, led to the conclusion that the optimal foam manufacturing option should be chosen between samples C and D. The reason for this choice is to meet the requirements of the initially adopted objective.

The main discussion regarding the current work is the choice of TiH_2 , given that the current global trend is to replace this foaming agent, considered quite expensive, with other cheaper solutions such as calcium carbonate or dolomite. Previously, some authors of the present work have performed experiments based on the manufacture of aluminum foam with $CaCO_3$ or dolomite and the results

showed that these agents are adequate for producing foams with fine cell sizes.

4. CONCLUSION

According to the work objective, a large-cell aluminum foam made by melt foaming using TiH_2 as an expanding agent for structural applications was produced. The thermal insulation properties (low density and heat conductivity values) were excellent, while the strength was sufficiently high. The work originality consisted in using the microwave effect both for rapid heating of the mix and for processing the fine aluminum powder by an own technique.

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