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FABRICATION OF OPEN CELL Fe-10%Al FOAM BY SPACE-HOLDER TECHNIQUE

WYTWARZANIE PIANY Fe-10%Al O OTWARTEJ POROWATOŚCI METODĄ WYPEŁNIACZA PRZESTRZENI

In this paper, fabrication of open-celled porous Fe-10%Al foam by space-holder technique in which NaCl powder and polyester resin were utilized as space holder and binder respectively to generate 30-80% porosities was studied. FeAl solid solution was prepared via mechanical alloying method after 25 h high energy milling process. The results indicate that the size and shapes of the pores are determined by the size and shapes of NaCl particles. Also, when compaction pressure was in the range of 70-150 MPa and the sintering process was performed at temperature around 1100°C, the samples had the best quality. Microstructure observations show the pore size was in the range of 200-500 μm.

Keywords: Open-celled foam, Fe-Al alloy, Mechanical alloying, Metal foam, Space-holder

W niniejszej pracy badano wytwarzanie techniką wypełniacza przestrzeni piany Fe-10%Al o otwartej porowatości, w której proszek NaCl i Żywicę poliestrową wykorzystano odpowiednio w roli wypełniacza porów i spoiwa do wytworzenia piany o 30-80% porowatości. Roztwór stały FeAl został wytworzony poprzez mechaniczne stopowanie przez 25 h w trakcie wysokoenergetycznego mielenia. Wyniki wskazują, że rozmiar i kształt porów determinowany jest przez wielkość i kształt cząstek NaCl. Ponadto, gdy ciśnienie prasowania było w zakresie 70-150 MPa a proces spiekania prowadzono w temperaturze około 1100°C, próbki miały najwyższą jakość. Obserwacje mikrostruktury wykazały, że rozmiar porów mieścił się w zakresie 200-500 μm.

1. Introduction

Open cell metal foams are identified as a new class of materials of great interest due to their unique combination of properties such as air and water permeability, good electrical insulating properties, impact energy absorption capacity, high stiffness and very low specific weight [1-6]. These remarkable properties extend the application of metal foams in various fields such as automotive parts, filters, cushions, insulators and biomedical implants [5-7].

The production methods utilized for the fabrication of metallic foams are divided mainly into two major groups: liquid state and solid state methods (usually in powdered form) [8]. The liquid state techniques have been extensively used for the production of Al-foams due to simplicity of the processing because of low reactivity and low melting point (660°C) of aluminum [9]. These processes are very effective in continuously producing large size foams, but it is difficult to control the process to obtain a uniform structure [4]. Use of liquid processing for some metals, such as Fe and Ti is very difficult due to high melting point and extreme chemical reactivity with oxygen and nitrogen [6, 8]. Powder-based techniques (solid-state) such as powder metallurgy and space holder methods are developing due to their considerable advantages. These techniques can fabricate porous metal parts at much lower tem-

peratures and under less severe chemical reactivity constraints with more precise control of process variables and pore size [8].

Recently, a new powder-based technique using space holder materials has been developed by its advantages like adjustable porosity amount, pore shape, and pore size distribution [10]. In this method, metal powders and appropriate space holder material are mixed and the mixture is compacted either uniaxially or isostatically to form a green compact and give sufficient mechanical strength. Then, the space holder is generally removed by thermal treatment, leaching or by the use of an aqueous solvent. Using chloride sodium (NaCl) as space-holder has numerous advantages such as low cost, fast dissolution in water, reduced corrosive attack of metal during dissolution and free of toxicity [2].

Fe (Al) solid solution is a very attractive compound for structural applications at elevated temperatures due to its physicochemical properties: low density, high heat resistance, high oxidation and corrosion resistance, relatively high strength as well as low cost [11]. As a result, if Fe (Al) foams with high porosity can be fabricated, then, they are much superior to aluminum foam. High melting point (~1500) and chemical reactivity of the element with atmospheric gases are some restricting factors for production of Fe (Al) foams through liq-

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uid process. Low melting and decomposition temperatures of space holders in comparison with high melting metals lead to collapse or corrosion of the porous metal structure before strong bonding between the metal particles is formed during sintering process. Moreover, removing space holder via leaching process before sintering is difficult due to low raw strength obtained by only compaction process.

To overcome these problems, authors suggest using NaCl as water-soluble space holder and choosing polyester resin as water- insoluble binder which can provide adequate strength to prevent deformation during leaching process. In particular, this process consists of two steps: firstly, preparing Fe (Al) solid solution powder via mechanical alloying method (MA). MA is a very convenient and versatile method to produce intermetallic and solid solution powders. MA is described as a high energy milling process in which powder particles are subjected to repeated cold welding, fracturing, and rewelding [12]. Secondly, the foam preparation process starts by mixing Fe (Al) powders obtained from MA process with NaCl and polyester resin, compressing them into a mould, leaching compact specimens in water to achieve open porosities and finally sintering the compact in an electrical furnace to obtain enough strength.

In view of the above, we have fabricated high-porosity Fe (Al) foam by using solid state space holder method. The effect of process parameters, such as quantity of the space holder, compaction pressure and sintering temperature, on the microstucture of foams was investigated. Also, the final morphological features were described by scanning electron microscopy (SEM).

2. Experimental procedure

2.1. Fabrication of the Fe (Al) powder

High purity Fe (purity >99%, Merck) and Al (purity>99%, Merck) powders, with an average particle size of 10 and $12\mu m$, respectively, were mixed in a proportion of 10 wt. % Al in milling vial and then mechanically alloyed under argon atmosphere, using a FP2, for 25 h of milling time. The vial and balls made from hardened chromium steel, and the ball-to-powder weight ratio (BPR) was 20:1.

2.2. Fabrication of foam specimen

Fig. 1 shows the steps for fabrication of Fe (Al) foams via space holder technique. The NaCl powders as space-holder (99.0% purity from Alfa Aesar, MA) was sieved in the range of 200-500 μm, and had cubical shape (Fig. 2).

In general, the quantity of polyester resin (as a binder) added to powders mixture depends on the amount of NaCl. As the volume percent of NaCl powder in mixture increases, the amount of resin needed to cover all the powder surfaces also increases. Therefore, in the present study, the weight percentage of polyester resin was about 10 wt. % of NaCl value. Increase in the value of resin more than 10 wt. % causes to prevent removing embedded NaCl particles.

In order to obtain high porosity and less trapped NaCl in the final structure, different values of NaCl powder (in the range 30-70 wt. %) and proportional amount of polyester resin

were added to the mixture of Fe (Al) obtained from MA. To ensure that the powders were completely mixed, the powders were mixed for more than 30 minutes in a porcelain mortar.

The next step involved powder compaction to obtain green compact by applying pressure in the range of 70-150 MPa. The powders were uniaxially pressed into the cylindrical shapes in dimensions of Ô10 mm×15 mm.

Complete removal of crystalline NaCl powder from the green compact can be achieved in two steps: Firstly, the green samples were placed in hot water (50°C) for 5 days to remove most of the embedded NaCl particles. Secondly, the samples were put in an ultrasonic washer machine to ensure that all NaCl particles were leached. The Sodium sulfate (Na_2SO_4) was used as an inhibitor to prevent corrosion of Fe (Al) foam in water.

Based on TGA results, elimination of resin can be started at 220°C. Thus, before the final sintering, the sample was heated at 400°C for 40 minutes to ensure complete elimination of resin. Finally, the sintering process of Fe (Al) powder was carried out at 1100°C for 2 hours. All compacts were heated to sintering temperature under argon atmosphere in tube furnace with heating rate of 10°/min and then cooled down to room temperature.
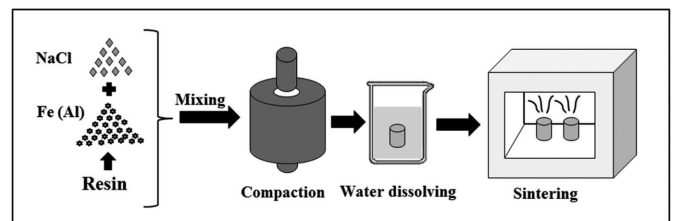

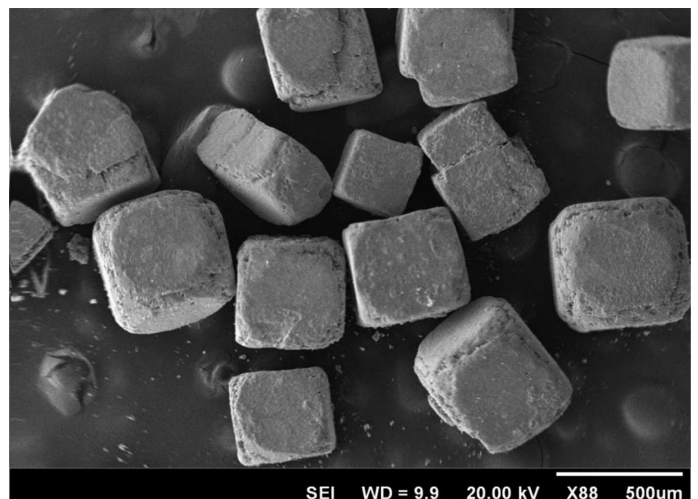Fig. 1. Schematic representation of Fe (Al) foam fabrication steps

Fig. 2. Typical morphology of NaCl powder

2.3. Sample Characterization

XRD analysis (Philips X-ray diffractometer) was carried out using Cu K_α radiation to identify different phases present in the starting powders and in mechanically alloyed powders. The diffractometer was operated at 40 kV and 30 mA. Scans were performed between 10° <2θ <90°. Porosity measurements were performed according to ASTM standard

C373-88. The morphology of powders and pores (size and shape) was examined by scanning electron microscopy (SEM, Seron technology AIS-2100, 15KV) and optical microscope (Leica DMIRM).

3. Result and discussion

3.1. FeAl powder fabrication process

Fig. 3(a) exhibits XRD patterns for the starting powders containing the characteristic peaks for Fe and Al phases. As can be seen, after 25 hours of milling, three new sharp peaks at $2\theta = 44.828°$, $2\theta = 65.263°$ and $2\theta = 82.662°$ appear to emerge, corresponding to formation of new phase that is similar to Fe pattern with bcc structure. But, as can be understood, Fe diffraction peaks shift to the lowangle side. This fact, coupled with disappearing of all the Al peaks can reveal that Al atoms were dissolved in bcc Fe lattice and formed Fe (Al) solid solution.

Fig. 3(b) illustrates the morphology of powder particles after 25 hours of ball milling. By increasing milling time to 25 hours, reduction of the particle size and increasing in the number of defects areas created new additional unstable surface [13]. Thus, the agglomeration phenomenon occurred to diminish surfaces and to decrease the energy.
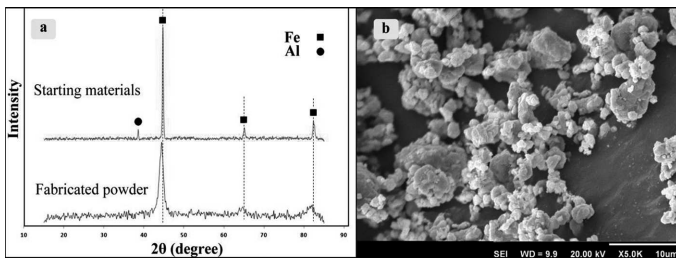

Fig. 3. (a) XRD patterns of starting materials and fabricated powder (b) morphology of powder particles

3.2. Characterization of porosities and pores

Fig. 4 indicates the morphologies of open cell Fe (Al) foam with 65% NaCl and sintered at 1100°C for 2 hours. Homogenous distribution of interconnected pores (average pore size of ~0.2mm) in Fe (Al) matrix can be obviously observed.
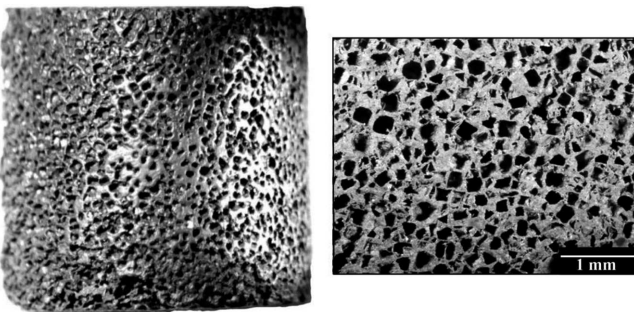

Fig. 4. Morphologies of open cell Fe (Al) foam with 65% NaCl

Fig. 5 shows typical morphologies of the sample during steps of preparation process. The cylindrical shape of foam specimen which was shown in Fig. 5(a), was prepared under 100 MPa compaction pressure with resin and NaCl powder. The resultant pores after leaching process are shown in Fig. 5(b). Similarity in both the shape and size of NaCl particles and pores reveals that the use of polyester resin can provide adequate strength to prevent deformation and collapse of the compacts during leaching process. Additionally, corrosion and dissolving of the base metal can be significantly decreased by using resin due to formation of a layer of resin and reduction of direct contact between Fe (Al) and water during leaching. The appearance of pores after sintering process (Fig. 5c) represents the particle characteristics of the original NaCl which demonstrated the final morphology of pores can be directly operated by selecting types of space holder with variety of size. But, by comparing Fig. 5(b) and Fig. 5(c), it can be clearly seen that the final pore size was larger than that of the size of the NaCl particles. The reason for this can be related to elimination of the resin layer during the sintering process. It should be mentioned that vacancies of dissolved NaCl after leaching process can facilitate elimination of resin and prevent breaking the mechanical bonds of the metal matrix during the sintering.
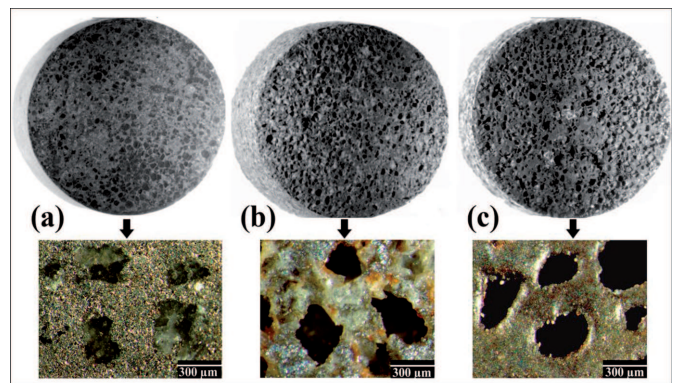

Fig. 5. Typical morphologies of the sample during preparation process

3.3. Space holder (NaCl) value

In this work, the samples with porosity from 35% to 78% were prepared by changing weight fraction of NaCl (30 to 70%) via space holder method. Total porosity of the foams can be formed from two sources: dissolving of NaCl as space holder during leaching process and removal of polyester resin as binder during sintering. Porosity of Fe (Al) specimens as the function of NaCl additive amount before and after sintering process is shown in Fig. 6. As can be seen, for all the samples before sintering, porosity increases linearly with the increase in the amount of NaCl under the same compaction pressure. The difference between value of final porosity of specimens after leaching and sintering process was attributed to the elimination of resin (about 10 wt. % of NaCl value) during sintering. As expected, by comparison with before sintering samples, the slope of after sintering line is slightly lower. It can be occurred due to the shrinkage of the specimen, which causes pore shrinkage during sintering after the evaporation of resin. When the porosity is above 60%NaCl, the change in the slope of the line can be observed. The reason is that in

addition to porosity obtained thorough thermal treatment or water soluble, excess amount of porosity can be resulted from partial sintering of Fe (Al) powders. Moreover, experimental works demonstrated that there is a limiting porosity or critical NaCl content, which determines whether the compact will collapse or not. This critical volume percentage of NaCl was found more than 70%.
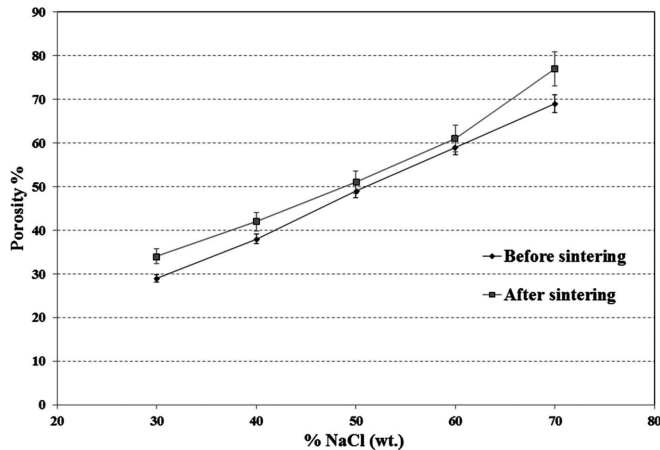

Fig. 6. Porosity of Fe (Al) specimens as the function of NaCl additive amount before and after sintering process

Fig. 7 shows SEM micrograph of a sample with 75% NaCl after sintering process. As can be seen, increase in the amount of NaCl causes the imperfection of cell walls due to insufficient sintering. Based on these results, we conclude that porosity which is between 35 to 80% can be obtained by space holder process.
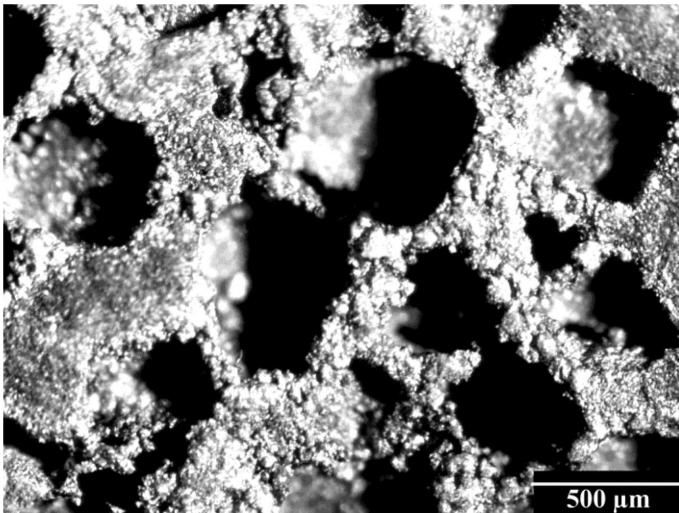

Fig. 7. SEM micrograph of sample with 75% NaCl after sintering process

3.4. Compaction pressure

Usually the first stage of densification processes is compaction powder at room temperature or cold compaction to prepare a green body. Final properties of sintered samples are widely affected by cold compaction due to available surface contacts necessary for sintering increase with compaction pressure and a decrease in diffusion distances occurs [14].

Further densification of compacts may be achieved through the use of smaller particles, powders with wide range of particle size distribution and higher compaction pressure. Although providing higher green density and better final microstructure needs higher pressure, under too high a pressure (>150 MPa) NaCl particles can be crushed. Additionally, lower compaction pressures (<70 MPa) caused subsequent collapse and spall of the Fe (Al) powder during leaching process.

In our work, polyester resin was used as a binder and lubricant to improve pressure transmissions and to reduce the friction between the powders and powder/die surface. Moreover, smaller particles obtained by MA process and wide range of particle size distribution achieved by using resin can provide sufficient strength at lower pressure. These results coupled with experimental observations suggested that the compaction pressure should fall into the range of 100-150 MPa.

3.5. Sintering temperature

Generally, solid sintering process of metal powder comprises three basic stages: (a) primary bonding between particles (neck formation) (b) pore channel closer and neck growth (c) densification and shrinkage [14]. The main driving force for solid state sintering is the excess surface free energy that accompanies the reduction in porosity. In this case, sintering behavior of Fe (Al) powder was influenced by high energy ball milling process. The onset sintering temperature was decreased due to mechanical activation and high specific surface resulted from reduction in size of powders during milling process [15]. Fig. 8 shows microscopic morphologies of pores and cell walls of sample with sintering temperature 1100°C for 2 hours. Generally, the foams contained mainly two types of pores: the interconnected macropores obtained by the elimination of space holder particles and, micropores which may be obtained by incomplete sintering of the base metal [6, 16, 17]. In this sample, there are macropores with the size ranging from 100 μm to 300 μm that are mainly manipulated by the space holder (NaCl) particles in green samples. Higher amplification pictures of this sample reveals that metal walls are perfect and no micropores can be seen in it.
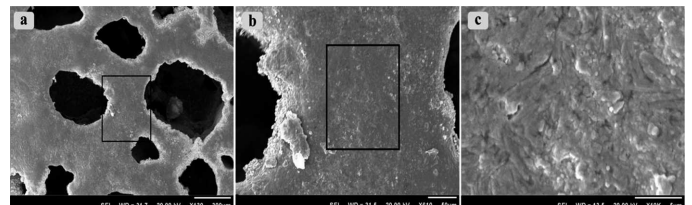

Fig. 8. SEM morphologies of pores and cell walls of sample with sintering temperature 1100°C for 2 h

When the sintering temperature was lower than 1100°C, there are also some micropores in the cell walls of the structure (Fig. 9a). They are the result of partial sintering and incomplete bonding between the Fe (Al) particles. Sintering at higher temperature should be avoided because of the increase in tendency of partial melt and liquid phase formation in cell walls (Fig. 9b). Therefore, sintering process at lower temperature can assist to maintain ideal foam shape.
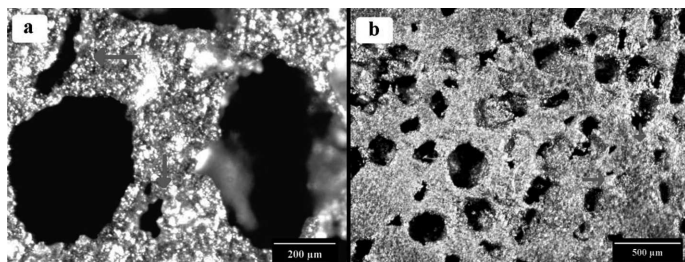
Fig. 9. Morphologies of Fe (Al) foam samples sintered at (a): lower and (b): higher 1100°C

4. Conclusion

An open-celled porous Fe-10%Al foam near to 80% porosity (in the range 200-500 μm) has been successfully fabricated by replication of NaCl space-holders. Fe (Al) solid solution was prepared via mechanical alloying method after 25 hours milling process. The results of this research work can be summarized as follows:

1. Polyester resin could be successfully used as water- insoluble resin which can provide adequate strength to prevent deformation during leaching process.
2. Total controllable porosity of the foams can arise from two sources: dissolving of NaCl and removal of polyester resin. The porosity increases linearly with increase in the amount of NaCl under the same compaction pressure. When the porosity was above 65%, partial sintering of Fe (Al) powders occurred; so; higher porosity than the utilized NaCl and resin contents was obtained.
3. When compaction pressure is in the range of 100-150 MPa and the sintering temperature about 1100°C, better quality of the samples with about 75% porosity can be achieved.

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