High mechanical performance alumina-reinforced aluminum nanocomposite metal foam produced by powder metallurgy: fabrication, microstructure characterization, and mechanical properties

Hamid Oveisi and Toktam Geramipour
Department of Materials and Polymer Engineering, Hakim Sabzevari University, Sabzevar 961796487, Iran
E-mail: hamid.oveisi@hsu.ac.ir
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Abstract
In this research, alumina-reinforced aluminum nanocomposite foams with high compressive performance were produced using the powder compact melting (PCM) process and CaCO₃ foaming agent. The content of Al₂O₃ nanoparticles, with sizes smaller than 80 nm, varied between 0–15 wt%. Aluminum, Al₂O₃, and CaCO₃ powders were mixed and compacted to dense precursors with 318 MPa stress. Finally, foam samples were fabricated by heating up the precursors at 1000 °C for 10 min. A range of porosity values was achieved between 34% to 43% for different combinations of aluminum matrix and alumina reinforcement phase. The crystallite size of the alumina reinforcement phase in Al-3 wt% alumina and Al-15 wt% alumina composite foam was estimated as 23.51 nm and 23.24 nm while the crystallite size of the aluminum matrix was determined as 15.72 nm and 15.44 nm for the same samples. The Field Emission Scanning Electron Microscope (FESEM) images indicated micro-size pores, which were connected via channels. The composite foam with 3 wt% of Al₂O₃ nanoparticles had a more uniform microstructure and more channels than other samples. The Vickers hardness values of the composites foam increased with an increase in wt% of alumina particles. This amounts to an approximately 93% increase in hardness when 15 wt% alumina particles are added to the aluminum foam. The sample with 3 wt% Al₂O₃ nanoparticles showed enhanced compressive strain up to 50% with high compressive strength up to 45 MPa and a uniform microstructure.

1. Introduction

Porous materials have attracted a lot of interest both in academia and industry because of their exceptional mechanical and physical properties [1–4]. In the recent decades, much attention has been paid to aluminum foams in different industries, especially in the automobile, aerospace and building industries, mainly due to their unique properties such as low density, high strength-to-weight ratio, high energy absorption at compression, sound absorption and heat resistance [5–9]. In general, pure aluminum foams demonstrate poor mechanical properties, depending on their production method, composition, microstructure, and density [10].

Various techniques have been developed that enhance mechanical performance and broadening the applications of these materials. Some examples include the application of Al alloys with higher strength, heat treatment of foams for age-hardening and precipitation-hardening [11–14], the addition of alloying elements to promote the formation of intermetallic compounds and incorporation of ceramic particles [15–30] or carbon nanotubes [9, 31, 32].

The effects of ceramic particles, especially SiC and Al₂O₃, on liquid foam stability, cell microstructure and mechanical properties of aluminum foams have been extensively studied in powder metallurgy and casting routes [15–23]. M Haesche et al [18] showed that adding 1%–5% wt% SiC and Al₂O₃ particles improve the maximum
expansion, collapse behavior and cell microstructure of aluminum foams. They also reported that smaller ceramic particles resulted in higher efficiencies.

A R Kennedy et al [21] demonstrated that the addition of 3 vol% of Al₂O₃, SiC and TiB₂ particles to aluminum foams resulted in an increase in foam expansion by reducing the critical cell wall thickness before the foam ruptures. A study by W Deqing et al [22] showed that the cell size and a wall thickness of aluminum foams can be increased by raising the amounts and particle size of SiC and Al₂O₃. Mahmutyazicioglu et al [23] investigated the effects of Al₂O₃ content on hardenability, microstructure and compressive behavior of 6061 aluminum foams. According to their results, the cell structure and drain reduction were improved by the addition of no more than 5 vol% of Al₂O₃ particles [23].

The nano-size alumina particles compare to micro-size ones provide a higher surface-area-to-volume ratio, reduce the brittleness of foams and, consequently, smaller volume fractions are required to improve the desired properties [33, 34]. However, the reports concerning the effects of ceramic nanoparticles on the properties of aluminum foams are very scarce [34]. B Prabhu et al [34] showed that the addition of SiC nanoparticles to aluminum foams decreased the cell sizes by 50.4% and improved the cell structure, yield stress, and energy absorption capacity.

In order to reinforce the aluminum foams, Al₂O₃ nanoparticles can be suitable candidates due to their unique characteristics, including (a) high capability for strengthening the cell walls because of their hardness and strength; (b) the absence of chemical interfacial reactions between molten aluminum and alumina particles, which results in the loss of structural integrity. Herein, the aim of our study is to investigate the physical, microstructural and compressive properties of aluminum nanocomposite foams reinforced with Al₂O₃ nanoparticles with sizes smaller than 80 nm, produced via a powder metallurgy method and low-cost CaCO₃ foaming agent.

2. Material and methods

2.1. Materials
Aluminum (Merck, Germany, >99% Purity, ≤150 nm), Al₂O₃ (Research Nanomaterials, US, >99% Purity, ≤80 nm) and CaCO₃ (Farzan Powder, Iran, >99.5% Purity, <50 μm) powders were used as aluminum and alumina sources, and a foaming agent, respectively.

The morphology and particle size of the aluminum powder and alumina nanoparticles are shown in the FESEM images of figures 1(a), (b). The high magnification image in figure 1(a) exhibits the homogeneous distribution of semi-spherical aluminum powder particles with an average size of ≤130 nm. The α-alumina nanoparticles with the size of ≤80 nm and spherical morphology can be clearly seen in figure 1(b).

2.2. Samples fabrication
Following the ‘powder compact melting’ method, aluminum, Al₂O₃ (0–15 wt%) and CaCO₃ (15 wt%) powders were mixed together for 30 min to obtain homogeneous mixtures. The mixed powders were then compacted using uniaxial cold pressing with 318 MPa pressure to form dense cylindrical components called ‘foamable precursors’ with a diameter of 20 mm, the height of 19.5 mm and relative density of 77%–80% (figure 2). In order to form the primary connections between aluminum and Al₂O₃ particles, precursors were preheated at 450 °C for 30 min under air atmosphere. In the next step, the precursors were placed in a cylindrical steel mold.
(20 mm in inner diameter, 100 mm in height) which was open only at the top. Foaming was done in a preheated furnace at 1000 °C for 10 min under air atmosphere. Since the precursors and the mold had similar diameters, expansions occurred only in the height direction. After the foaming process, the samples were removed from the furnace and foams were solidified through natural cooling in the air.

2.3. Characterization
The foam’s density ($\rho_s$) was calculated from their mass and geometry (ASTM-B962–17 standard test methods for the density of compacted or sintered (PM) products). Furthermore, the relative density of the samples ($\rho^*$) was calculated by dividing their density by the theoretical density of bulk aluminum (2.7 g cm$^{-3}$) as follows:

$$\rho^* = \left(\frac{\rho_s}{2.7}\right) \times 100$$

ASTM E92 standard test methods for Vickers hardness and knoop hardness of metallic materials were utilized for produced composite foams. The hardness distribution in the foams was examined by the micro-indentation technique. To this end, a micro-hardness machine, Qualitest QV-1000DM, equipped with a tetrahedral diamond indenter with tip angle 68° was used. Micro-Vickers hardness was measured under a 25 g load and a 15 s dwell time. At least five micro-hardness measurements were performed on each sample.

In order to investigate the compressive behavior of the materials under the ASTM C365–05-compression test standard, the samples were cut into cylindrical parts, 20 mm in diameter with a height-to-diameter ratio between 1.5 and 2. Therefore, the minimum dimension of the specimen was at least seven times the cell size to avoid the size effects [6]. The compression tests were performed at room temperature using a Zwick/Roell Z250 testing machine, at a constant ram speed of 6 mm min$^{-1}$. Furthermore, samples were sectioned by a wire-cutting machine to investigate the microstructure and elemental analysis using Field Emission Scanning Electron Microscope (FESEM), TESCAN Mira 3-XMU with 15.0 kV accelerating voltage.

3. Results and discussion

3.1. Measurement of relative density
The relative density of the nanocomposite foams as a function of Al$_2$O$_3$ content is plotted in figure 3. A range of porosity values was achieved between 34% to 43% for different combinations of aluminum matrix and alumina reinforcement phase. The highest value of relative density (∼65%) was obtained using 3 wt% of Al$_2$O$_3$ nanoparticles. In fact, the uniform distribution of Al$_2$O$_3$ nanoparticles inhibited the growth of cells throughout the precursor by affecting the viscosity and surface tension between CO$_2$ bubbles and melted aluminum particles [19]. Moreover, the addition of 15 wt% of Al$_2$O$_3$ nanoparticles increased the density due to the agglomeration of CaCO$_3$ and Al$_2$O$_3$ particles and diminished the foaming agent in the aluminum matrix.

3.2. X-ray diffraction characterization
In order to identify the phase of the materials, x-ray diffraction (XRD) characterization was carried out on the aluminum powder, alumina nanoparticles and composite foams with 3 wt% and 5 wt% of Al$_2$O$_3$ nanoparticles. Figure 4 shows the XRD pattern of alumina nanoparticles and aluminum powder before densification. The XRD results of alumina nanoparticles showed the presence of $\alpha$-alumina peaks located at 20 (Miller indices) = 35.197° (104), 37.804° (110), 43.381° (113), 52.588° (024), 57.538° (116), 59.782° (211), 61.333°
The intensity of the XRD peaks indicated the distribution of high purity crystalline $\alpha$-alumina nanostructure phase throughout the sample. The crystal system of $\alpha$-alumina nanoparticles was hexagonal and had a density of $3980$ kgm$^{-3}$ and lattice parameters of $a = b = 4.758$ Å and $c = 12.991$ Å. The peaks located at $2\theta = 38.93^\circ$ (111), $45.03^\circ$ (200), $65.41^\circ$ (220), $78.51^\circ$ (311) in XRD pattern of aluminum powder correspond to the FCC crystal lattice of pure aluminum. Notably, no additional peaks indicative of impurities in aluminum powder are detected.

The XRD pattern of the reinforced foam materials is also shown in Figure 4. The patterns only reveal the aluminum and $\alpha$-alumina phase, even in the sample containing 15 wt% alumina nanoparticles. These patterns evaluate the high stability of the alumina reinforcement phase after the foaming process. On the other hand, the maximum intensity of the (113), (116), and (104) of alumina crystal planes are clearly increased by the addition of a reinforcement agent to the nanocomposite materials. This is believed to be due to the low content of the 3 wt% alumina nanoparticles in aluminum matrix compared to the 15 wt% alumina nanoparticles in the aluminum matrix.

The crystallite size of the Al matrix ((111) planes) and alumina reinforcement agent ((113) planes) in the composite foams can be calculated from the full width at half maximum intensity (FWHM) measured on the diffraction profile. Mean crystallite size of alumina reinforcement phase in Al-3 wt% alumina and Al-15 wt% alumina composite foam was estimated (accuracy $\pm 30\%$) as 23.51 nm and 23.24 nm while the crystallite size of the aluminum matrix was determined as 15.72 nm and 15.44 nm for the same samples.
3.3. Investigation of microstructure

Figures 5(a)–(d) shows the FESEM images of nanocomposite aluminum foams with 0–15 wt% Al₂O₃ nanoparticles. For non-reinforced foam (figure 5(a)) a uniform distribution of cells, connected via channels, was observed. Interestingly, adjusting the critical fabrication parameters in this study resulted in semi open-cell aluminum foams with highly distributed pores while all the previous reports on aluminum foam fabrication using this method showed the closed-cell structure [35–43]. This uniform microstructure was due to CaCO₃ decomposition product (i.e. CaO), the oxide layer on the surface of aluminum particles and further oxidation of aluminum during foaming, which stabilized the foam by enhancing the viscosity [35, 44–47]. The cells were irregularly shaped and smaller than 100 microns, due to very fine aluminum particles [48].

The small pores between primary aluminum particles, which is prominent in the inset image of figure 5(a) was due to the powder compact melting process. The formation of these pores was a consequence of the presence of an oxide layer on the surface of aluminum particles, which can be deteriorated the sufficient diffusion and complete bonding of particles. PM pores are likely the sites for crack initiation in mechanical tests [49].

As evidenced in figure 5(b), the sample with 3 wt% of Al₂O₃ nanoparticles has a more uniform microstructure and more channels than other samples. The Al₂O₃ nanoparticles can help the stabilization of aluminum foam by affecting the viscosity and the surface tension of CO₂ gas/aluminum melt. However, increasing the Al₂O₃ content to 6 wt% decreased the uniformity of the microstructure (figure 5(c)). Moreover, the nanocomposite foams with a high content of Al₂O₃ nanoparticles showed pore coarsening, unusually large pores and non-uniform microstructure (figure 5(d)). These undesirable effects were caused by the

![Figure 5. FESEM images of aluminum nanocomposite foams with 0 wt% (a), 3 wt% (b), 6 wt% (c) and 15 wt% (d) Al₂O₃ nanoparticles. Circle in the inset image (a) shows the PM pores. The arrows indicate the channels between individual cells.](image-url)
Figure 6. Cross-sectional image of aluminum nanocomposite foam with 15 wt% Al₂O₃ nanoparticles (a). The white dots in (a) are agglomerated Al₂O₃ and CaCO₃ particles. The FESEM image (b) and EDX pattern (c) of agglomerated regions. The large and small particles in (b) are CaCO₃ and Al₂O₃, respectively. EDX mapping (d), (e), and (f) from the same region as in (b) indicating the uniform and spatial distribution of Al, Ca, and O elements.
agglomeration of the particles (white dots in figure 6(a)). Considering the white color of these regions, the agglomerated particles would be Al2O3 and/or CaCO3. Considering the geometry of the foaming mold, which was open only at the top, the foaming process began from the top of the precursors. Consequently, the bottom of the precursors remained relatively dense. Furthermore, higher stresses in the upper and lower zones of the precursor during compaction resulted in the formation of shear bands and large sectional cracks in the samples (figure 6(a)).

The FESEM image and related energy dispersive x-ray (EDX) analysis of the agglomerated regions is shown in figures 6(b) and (c), respectively. The Aluminum, oxygen, calcium, and carbon were detected by EDX analysis (figure 6(c)), which were assigned to the CaCO3 and Al2O3 particles. It was found that Al2O3 nanoparticles are homogeneously distributed inside the aluminum matrix.

### 3.4. Micro-hardness of composite foams

The micro-indentation technique was used to evaluate the distribution of alumina nanoparticles in the composite foams since it was impossible to detect and distinguish Al2O3 nanoparticles in the aluminum matrix by EDX analysis. Figure 7 and table 1 show the micro-hardness values for the foams containing different levels of Al2O3 nanoparticles.

The inset image of figure 7 shows a representative optical image of the micro-indentation test. The similar indentation diameter demonstrated the mechanical isotropy of the aluminum matrix. It can be expected that the measured values of micro-hardness were less than the actual hardness values of bulk aluminum which are arised from PM pores in the foams matrix.

Moreover, the micro-indentation data gives additional information on the uniformity of the alumina particles distribution. The micro-hardness data of the reinforced foams with 3 wt% of Al2O3 displayed a narrow range of hardness values, suggesting that the alumina nanoparticles were uniformly distributed in this sample. However, a wide range of micro-hardness values was observed for samples with larger amounts of Al2O3 nanoparticles (>3 wt%). This shows a nonuniform distribution of Al2O3 nanoparticles in the foams, which can be confirmed by the agglomerated regions in nanocomposite foam with 15 wt% Al2O3 (figure 6(a)).

#### Table 1. Mechanical properties of alumina-reinforced aluminum nanocomposite foam.

| Sample          | Compressive strength (MPa) | Elastic modulus (GPa) | Densification strain (εD) (%) | Vickers hardness (MPa) |
|-----------------|---------------------------|-----------------------|-------------------------------|------------------------|
| Al–0 wt% Al2O3  | 43 ± 5                    | 0.1 ± 0.01            | 45 ± 5                        | 4.3 ± 0.7              |
| Al–3 wt% Al2O3  | 45 ± 5                    | 0.1 ± 0.01            | 50 ± 4                        | 5.5 ± 1.3              |
| Al–6 wt% Al2O3  | 40 ± 7                    | 0.2 ± 0.02            | 33 ± 2                        | 6.0 ± 1.8              |
| Al–9 wt% Al2O3  | 28 ± 4                    | 0.1 ± 0.01            | 34 ± 4                        | 8.2 ± 3.0              |
| Al–12 wt% Al2O3 | 28 ± 6                    | 0.1 ± 0.01            | 34 ± 2                        | 8.2 ± 3.4              |
| Al–15 wt% Al2O3 | 22 ± 4                    | 0.1 ± 0.02            | 33 ± 3                        | 8.3 ± 3.4              |

Figure 7. The micro-hardness values of alumina reinforced aluminum foams as a function of Al2O3 nanoparticles content. The inset image is the optical image of the micro-indentation.
It was clearly observed that compared to the unreinforced aluminum foam, the Vickers hardness values of the composites foam reinforced with alumina nanoparticles, increased with an increase in wt% of alumina nanoparticles. This amounts to an approximately 93% increase in hardness when 15 wt% alumina nanoparticles are added to the aluminum matrix. Increasing hardness is due to the higher volume fraction of alumina nanoparticles in the aluminum matrix.

3.5. Compressive properties

Figure 8 and table 1 show the experimental stress-strain data obtained by the compression test of aluminum composite foams with 0–15 wt% Al₂O₃ nanoparticles. All of the compressive curves in figure 8 can be divided into three stages: (1) elastic deformation, (2) plateau region and (3) close contact of cell walls and collapsing. In all samples, the stress increased considerably with the strain in the plateau region. This response is due to various possible sites for the crack initiation, including PM pores, undecomposed CaCO₃, CaO (product of CaCO₃ decomposition), the broken part of the oxide layer on the surface of aluminum particles and the Al₂O₃ nanoparticles as a reinforcing agent. The interfaces between these brittle solid particles, which have low plasticity, and the aluminum matrix are the appropriate sites for the crack nucleation.

Comparing the curves in figure 8 and table 1 clearly demonstrated that the addition of 3 wt% of Al₂O₃ nanoparticles enhanced the densification strain of the foams (from ~45% to ~50%) as a result of the improved microstructure. This would be mainly because of the small pores formed between primary aluminum particles during the compacting, the higher cell walls strength, as well as uniformity of cell microstructures improved by alumina nanoparticles.

Moreover, increasing the amount of alumina reinforcement agent to more than 3 wt% reduced the plateau length and compressive strength of the samples due to the pore coarsening and unusual pores (figures 5(c) and (d)). The advantages of the nanocomposite foam with 3 wt% of Al₂O₃ nanoparticles will be best identified when compared with the results of non-reinforced aluminum foams and those reinforced with micro-size ceramic particles.

Despite the higher relative density (~66% compare with <30%), the fabricated nanocomposite foams showed comparable strain densification (~50%) and high compressive strength (~45 MPa compare with <50 MPa) than aluminum foams reported by other researchers [23, 35, 51, 55]. However, the compressive response of the fabricated nanocomposite foams was similar to those aluminum foams reinforced with SiC nanoparticles [34]. This confirms the significant role of ceramic nanoparticles in enhancing the compressive behavior of aluminum foams.

4. Conclusion

Here we report for the first time, that adjusting the parameters of the ‘powder compact melting’ process using alumina reinforcement nanoparticles and CaCO₃ foaming agent resulted in semi open-cell aluminum nanocomposite foams. To obtain desired physical and mechanical properties like high hardness, high strength, and high stiffness, Al foam was reinforced with different content of alumina nanoparticles. The results showed an increase in relative density (~66%) and uniform microstructure by the addition of 3 wt% of Al₂O₃.
nanoparticles. Adjusting the fabrication parameters in this study resulted in semi open-cell aluminum foams with highly distributed pores. This uniform microstructure was due to CaCO₃ decomposition product (i.e. CaO), the oxide layer on the surface of aluminum particles and further oxidation of aluminum during foaming, which stabilized the foam by enhancing the viscosity. The cells were irregularly shaped and smaller than 100 microns. The sample with 3 wt% of Al₂O₃ nanoparticles had a more uniform microstructure and more channels than other samples. Increasing the Al₂O₃ content to 6 wt% decreased the uniformity of the microstructure. Moreover, the nanocomposite foams with a high content of Al₂O₃ nanoparticles showed pore coarsening, unusually large pores and non-uniform microstructure. The addition of 3 wt% alumina nanoparticles to the aluminum matrix resulted in a compressive strain of ~50% and high compressive strength up to 45 MPa. These are due to the effective roles of Al₂O₃ nanoparticles on foam viscosity and surface tension between CO₂ bubbles and molten aluminum. However, agglomeration of Al₂O₃ and CaCO₃ particles was observed for large amounts of Al₂O₃ nanoparticles (>3 wt%). The pore coarsening, non-uniformity of the microstructure and deteriorated compressive properties were observed due to decomposition products of CaCO₃ and Al₂O₃ nanoparticles in the aluminum matrix. Due to a semi open-cell microstructure and high compressive strain and stress of aluminum-alumina nanocomposite foams, these materials can be considered as promising candidates for both structural and functional applications in the near future.

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Conflict of interest

The authors declare that they have no conflict of interest.

ORCID iDs

Hamid Oveis i https://orcid.org/0000-0002-0996-7811

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Mater. Res. Express 6 (2019) 1250c2

H Oveis and T Geramipour

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