Fabrication and Characterization of SiC and Si₃N₄ Reinforced Aluminum Matrix Composites

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Abstract In this study, Si₃N₄ (varying from 0 to 12wt.%) or SiC (varying from 0 to 30wt.%) reinforced aluminum matrix composites were fabricated by powder metallurgy method. The effects of reinforcement element (SiC or Si₃N₄) addition on the mechanical properties of aluminum composites were newly examined for large-scale distribution. Microstructures and crystal structures of composites were analyzed via scanning electron microscope (SEM) and X-ray diffractometry (XRD), respectively. Density, hardness and compressive strength of composites were tested by density meter, micro Vickers hardness tester and universal test machine. From the experimental results, it was observed that the hardness value of pure aluminum was obtained 30 HV. Also, the highest hardness values were obtained at Al-9%Si₃N₄ (57 HV) and Al-30%SiC (79 HV) composite. Similarly, the highest yield and ultimate compressive strength were determined at Al-30%SiC and Al-9%Si₃N₄ composite. Pure Al, Al-30%SiC and Al-9%Si₃N₄ were measured as 82, 138, 198 MPa and 113, 224, 292 MPa, respectively. Increased to weight percentage of Si₃N₄ from 9% to 12% in Al-Si₃N₄ composites, the apparent density, Vickers hardness and compressive strength of Al-12 wt.%Si₃N₄ composite sharply decreased due to the agglomeration of Si₃N₄. As a result, it has been observed that SiC or Si₃N₄ addition makes a positive contribution to the mechanical and microstructure of aluminum composites. These composites should be used for the wear application of aluminum.

Keywords Aluminum, Silicon Carbide, Silicon Nitride, Powder Metallurgy, Microstructure

1. Introduction

Nowadays, the performances expected from the engineering materials (high strength, high electrical properties etc.) are not sufficient while industrialization has been increasing. For this reason, research and development activities have been growing to develop the functional,

high value-added and high technological materials in recent years. A new generation of composite materials has begun to be produced in many areas into human life [1-3].

Composite materials are widely used in aerospace, aircraft, automotive, nuclear applications, defense industry and sporting goods due to their superior properties such as high strength, wear, fatigue and corrosion resistance, toughness, high-temperature performance, lightness and aesthetic appearance. Composite materials consist of the mixture of two or more elements to combine their superior properties in a single material. They are classified as metal, ceramic and polymer matrix composites according to matrix materials [4-7].

In metal matrix composites, many engineering materials can be used as the matrix material such as Mg, Al, Ti, Cu, Mo, Fe, Co and Ni and their alloys. Also, some reinforcing elements (Al₂O₃, SiC, Si₃N₄, B₄C, TiB₂, TiC, WC, W, C and MgO etc.) may be used in metal matrix composites [8]. The most commonly used metal matrix composites are aluminum matrix composites. Aluminum matrix composites are frequently used for electronic, automotive, aeronautical and aerospace industries due to their important performance including high ductility, good workability, electrical and thermal conductivity, high strength to weight ratio and superior corrosion strength [9-11]. As a result, different particles have been reinforced in aluminum and its alloys including metallic oxides [12], borides [13], carbides [14] and nitrides [15].

There are many studies in the literature related with examining the mechanical properties of SiC or Si₃N₄ reinforced aluminum matrix composites. Pakseresht et al. (2016) fabricated Al-SiC nanocomposites by powder metallurgy method. The effects of SiC content and milling time on mechanical properties and microstructure of Al-SiC composites were investigated. The Vickers hardness value of Al-SiC composites increased with increasing ball milling time and SiC content. [16]. Gallardo et al. (2014) investigated the effects of SiC addition (varying from 0 to 5wt.%) on the microstructure, hardness, yield strength and ultimate tensile strength of AA2024-SiC nanocomposites. Hardness, yield strength and compressive

strength of nanostructured AA2024 composites increased with increasing SiC content [17]. Sharma et. al. (2015) examined the production of aluminum (AA6082-T6) matrix composites reinforced with the various weight percentages of silicon nitride particles by conventional stir casting route. The mechanical properties such as ultimate tensile strength and hardness improved at the cost of the reduction in ductility with the increase in weight percentage of silicon nitride particulates (varying 0 to 12wt.%) in the aluminum metal matrix [18]. Kumar et al. (2015) fabricated aluminum matrix composites (AA2618) reinforced by AlN, Si₃N₄ and ZrB₂ particles (0, 2, 4, 6, 8wt.%) by stir casting. Mechanical properties of aluminum composites such as hardness, tensile and compressive strength increased by increasing AlN, Si₃N₄ and ZrB₂ content [19]. Most of the existing studies concentrate on the mechanical properties of the Al-ceramic composites just narrow content. To our knowledge, there has been a few publications on the effect of large-scale SiC and Si₃N₄ amount on mechanical properties and microstructure of Al composites.

In the present study, pure Al/SiC and pure Al/Si₃N₄ composites were fabricated in various proportions using powder metallurgy method. This study aims to investigate and compare the effects of SiC or Si₃N₄ addition on density, microhardness, compressive strength and microstructure of aluminum composites.

2. Material and Method

2.1. Material

In this study, the aluminum powder was used as matrix material which was supplied by Alfa Aesar (USA), with 99.5% purity, average particle size of ~10 µm and density of 2.7 g/cm³. Si₃N₄ (99% purity, the density of 3.2 g/cm³ and average particle size of 800 nm) and SiC (99% purity, the density of 3.25 g/cm³, average particle size of 40 µm). Si₃N₄ and SiC powders were purchased from Ube Industries (Japan).

2.2. Method

Fig. 1 shows the detailed schematic diagram of Al-SiC or Al-Si₃N₄ composite fabrication via powder metallurgy method. In this method, aluminum and SiC (1, 3, 6, 9, 12, 15, 18, 21, 25, 30wt.%) or Si₃N₄ (1, 3, 6, 9, 12wt.%) powders were mixed in ethanol medium using a mechanical agitator. SiC-ethanol or Si₃N₄-ethanol slurry was added dropwise into the aluminum-ethanol slurry. Mixing process continued until obtaining the homogeneous mixture. The mechanically agitated mixture was ground by ball milling during 6 hours, and then the mixture was filtered. The filtered mixture was dried under vacuum overnight to obtain the composite powder. The dried composite powder was compacted in a stainless steel mold with 13 mm diameter at room temperature under 600 MPa to obtain green billets. After compacting, the green billets were sintered under vacuum in the tube furnace at a certain sintering temperature 630 °C and sintering time 180 min.

The cross-sections of sintered samples were ground and polished for Vickers hardness analysis. On the other hand, the sintered samples were immersed into the liquid nitrogen, and broken for fracture surface analysis by scanning electron microscope and X-Ray diffractometry. The microstructure and morphology analysis of composites was carried out by scanning electron microscope (SEM, Jeol JSM-7001F). X-ray diffraction analysis (XRD, Rigaku Smartlab) was used to investigate the phase analysis of powders and fabricated composites. The particle size distribution of Al, SiC and Si₃N₄ powders were measured by particle size analyzer (Malvern Mastersizer 3000). The apparent density was detected with density meter by using Archimedes principle. The Vickers hardness tester (HV-1000B) was used to measure the micro Vickers hardness value using a specific load of 200 g with the dwell time of 15 s, and the mean values of at least five measurements conducted on different areas of each sample were considered. Compressive tests were performed with the universal testing machine (Mares Test-10 tons).

Figure 1. Schematic diagram of Al-SiC or Al-Si₃N₄ composites fabrication via powder metallurgy method [20]

3. Results and Discussion

3.1. Characterization of Powders

Fig. 2 presents the morphology of the aluminum (a), SiC (b) and Si₃N₄ (c) powders. It may be seen from Fig. 2(a) and (c) that the morphology of aluminum powder is nearly spherical. The particle size distributions of Al, SiC and Si₃N₄ powders are shown in Fig. 2(d), 2(e) and 2(f). From

these analyses, the average sizes of Al, SiC and Si₃N₄ particles is approximately 10 μm, 40 μm and 0.8 μm, respectively.

XRD patterns of powders are given in Fig. 3. In this figure, aluminum (2θ=39°, 45°, 65°, 78°), α-Si₃N₄ (2θ=43°, 47°, 48°, 58°, 59°, 73°) and SiC (2θ=35°, 38°, 60°, 72°) peaks were exposed.

Figure 2. SEM micrograph of aluminum (a), SiC (b), Si₃N₄ (c) powders, grain size distribution of aluminum (d), SiC (e), Si₃N₄ (f) powders

Figure 3. XRD patterns of pure Al, SiC and Si₃N₄ powders

Figure 4. XRD patterns of Al-SiC (a) and Al-Si₃N₄ (b) composites

3.2. Characterization of Fabricated Composites

The X-ray diffraction patterns of SiC or Si₃N₄ reinforced aluminum composites are given in Fig. 4a and 4b, respectively. As seen Fig. 4a and 4b, all reflections belong to Al, SiC and Al, α -Si₃N₄, respectively. Moreover, undesired second phases such as aluminum carbide (Al₄C₃) or aluminum nitride (AlN) peaks are not observed for all Al-SiC and Al-Si₃N₄ composites.

The images of Al-SiC composites are represented in Fig. 5(a-c). As shown in the figure, SiC particles were uniformly distributed within the aluminum matrix. Also, aluminum powder and SiC particles were combined a good bonding with each other. It can be concluded that increasing the SiC content decreases the grain growth that acts as barriers against the tendency of grain growth.

Fig. 6 shows the images of pure Al and Si₃N₄ reinforced aluminum composites with SEM. The SEM images reveal the presence of Si₃N₄ particles in the aluminum matrix. In Al-12%Si₃N₄ composite structure, agglomeration of Si₃N₄ particles was observed. This agglomeration causes to reduce of mechanical properties in Al-12wt.%Si₃N₄ composite.

Energy Dispersive X-Ray (EDX) analyses were used from the fracture surfaces to confirm the distribution of Si₃N₄ and SiC in aluminum matrix. As shown in Fig. 7a, Si and N are shown in same region which are distributed homogeneously. Similarly, SiC particles are clearly seen due to its high size in Fig. 7b. The purple and red color mappings confirm the Si and C distributions in composite structure.

Figure 5. The images of 15% (a), 21% (b) and 30% (c) SiC reinforced aluminum composites with scanning electron microscope

Figure 6. The images of pure Al (a), 9% (b) and 12% (c) Si₃N₄ reinforced aluminum composites with scanning electron microscope

Figure 7. The images of Al-9%Si₃N₄ (a) and Al-30%SiC (b) with SEM-EDX mapping

3.3. Density, Hardness and Compressive Strength Analysis of Composites

The apparent density variation of Al-SiC and Al-Si₃N₄ composites with SiC/Si₃N₄ content is given in Fig. 8. As seen from Fig. 8a, it was found that the apparent density of pure aluminum is nearly 2.51 ± 0.01 g/cm³. On the other hand, the apparent density of Al-30wt.%SiC composite was increased to 2.66 ± 0.01 g/cm³ due to the high theoretical density of SiC ($\rho_{\text{SiC}} = 3.25$ g/cm³). It was clear that the apparent densities of fabricated Al-SiC composites were increased by sintering effect and SiC addition. Similarly, the apparent densities of Al-Si₃N₄ composites were increased by increasing Si₃N₄ content up to 9wt.%Si₃N₄ addition (Fig. 8b). Compared to pure aluminum, the apparent density of Al-9wt.%Si₃N₄ composites was increased from 2.51 ± 0.01 g/cm³ to 2.55 ± 0.01 g/cm³ owing to the high theoretical density of Si₃N₄ particles ($\rho_{\text{Si}_3\text{N}_4} = 3.25$ g/cm³). In Al-12wt.%Si₃N₄ composite structure, it was seen that the apparent density was decreased to 2.33 ± 0.01 g/cm³ due to the agglomeration of Si₃N₄ particles.

Fig. 9 demonstrates the Vickers hardness variation of Al-SiC and Al-Si₃N₄ composites with SiC/Si₃N₄ content. As given in Fig. 9a, the Vickers hardness of fabricated Al-30wt.%SiC composite was increased from 28 ± 2 HV to 79 ± 1 HV. The Vickers hardness of Al-SiC composite was increased with increasing content of SiC particles. Similarly, the effect of Si₃N₄ content on the Vickers hardness of Al-Si₃N₄ composites can be seen more clearly in Fig. 9b. The Vickers hardness of Al-9wt.%Si₃N₄ composite increased from 28 ± 2 HV to 79 ± 1 HV when compared to pure aluminum. It appears that increased in Si₃N₄ content up to 9wt.% increased the Vickers hardness of Al-Si₃N₄ composites. After 9wt.%Si₃N₄ content, it was observed a drop (54 ± 2 HV) in Vickers hardness of Al-Si₃N₄ composite. This reduction is due to the agglomeration tendency of powders and nonhomogeneous distributions. This approach was confirmed by SEM images. The hardness is related with the dislocation density. These powders are located at the grain boundaries. Therefore, they suppress the grain growth during heat treatment and restrict dislocation movement during plastic deformation [20].

Figure 8. The apparent density variation of Al-SiC (a) and Al-Si₃N₄ (b) composites with SiC/Si₃N₄ content

Figure 9. The Vickers hardness variation of Al-SiC (a) and Al-Si₃N₄ (b) composites with SiC/Si₃N₄ content

Figure 10. Yield (a) and ultimate (b) compressive strength of pure Al and ceramic-reinforced Al composites

Results of compressive test for the best hardness compositions (Al-30%SiC and Al-9%Si₃N₄) are given in the form of bar charts in Fig. 10. It is clearly shown that both SiC and Si₃N₄ have a positive effect on the yield compressive strength and ultimate compressive strength. The Si₃N₄ composition has a higher compressive strength when compared SiC reinforcement. The compressive strength of Si₃N₄ is higher than the SiC. Therefore, it increases the compressive strength of Al composites even more. The increase in hardness and compressive strength of composite is directly related strengthening mechanisms such as dislocation density, load transfer and crack deflection. The reinforcement element acts as a barrier during sintering which causes to grain size refinement and increase in dislocation density at the matrix-reinforcement element. The load transfer to ceramic in composites has a critical role during plastic deformation. Most of the load transfers the ceramic materials in composites due to its highest compressive strength which enhances the mechanical properties of Al composites. During plastic deformation, the cracks initiate and propagate with increasing load. The ceramics provide a barrier to propagate the cracks. When the cracks propagate to the ceramic particle, it prefers the deflection. The crack propagation energy is not enough to break the ceramic particles.

4. Conclusions

Al/SiC and Al/Si₃N₄ composites were fabricated by powder metallurgy method with various SiC and Si₃N₄ content. From this research work, the following conclusions are derived.

- Fabrication of Al-Si₃N₄ and Al-SiC composites was performed successfully by powder metallurgy method.
- SEM images and XRD analysis revealed the presence of Si₃N₄ and SiC reinforcement particles in the fabricated aluminum matrix composites. Also, bonding between the particles and a good neck formation were observed at Al-9%Si₃N₄ and Al-30%SiC composites. SiC and Si₃N₄ particles

were detected along the aluminum grain boundaries. This approach is confirmed by SEM-EDX analysis.

- Density of the fabricated Al-30wt.%SiC composite was increased from 2.51 to 2.66 g/cm³ compared to pure aluminum. Similarly, the apparent density of Al-9wt.%Si₃N₄ composite was increased from 2.51 to 2.55 g/cm³ compared to pure aluminum. Increased to weight percentage of Si₃N₄ from 9% to 12% in Al-Si₃N₄ composites, the apparent density of Al-12 wt.%Si₃N₄ composite sharply decreased to 2.33 g/cm³ due to the agglomeration of Si₃N₄ particles.
- Compared to pure aluminum, the Vickers hardness of fabricated Al-30wt.%SiC composite was increased from 28 HV to 79 HV. In addition, the Vickers hardness of Al-9wt.%Si₃N₄ composite increased from 28 HV to 57 HV with respect to the addition of weight percentage of Si₃N₄ particles (i.e. from 0wt.% to 9wt.%). After 9wt.%Si₃N₄ addition to the aluminum matrix, the Vickers hardness of Al-12wt.%Si₃N₄ composites is decreased to 54 HV due to the agglomeration of Si₃N₄ particles. Microstructure images confirmed this agglomeration.
- Yield and ultimate compressive strength of Al-30wt.%SiC were measured as 138 and 224 MPa, respectively. These values were 198 and 292 MPa for Al-9wt.%Si₃N₄. When compared pure Al (82 MPa and 113 MPa), SiC and Si₃N₄ have a positive effect on the compressive strength of aluminum matrix composites.
- SiC particles were more effective reinforcement material than Si₃N₄ particles for hardness results. On the other hand, Si₃N₄ particles had a critical role on the compressive strength of composites because of its higher compressive strength.

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REFERENCES

- [1] Şenel, M. C., Gürbüz, M., Koç, E., New generation composites with graphene reinforced aluminum matrix, *Engineer & Machine*, vol.56, no.669, pp.36-47, October 2015 (in Turkish).
- [2] Khan, M., Amjad, M., Khan, A., Microstructural evolution, mechanical profile, and fracture morphology of aluminum matrix composites containing graphene nanoplatelets, vol. 32, issue. 11, p. 2055-2066, June 2017.
- [3] Şahin, İ., A Review of Drilling for Aluminium matrix composite materials, *Engineer & Machine*, vol.55, no.649, pp.9-16, February 2014 (in Turkish).
- [4] Erdoğan, M. The Production of steel fibre reinforced aluminium composite material and investigation of its mechanical properties experimentally, The Degree of Master Science, Institute of Science, Dumlupınar University, Kütahya, Turkey, 2005 (in Turkish).
- [5] Işık, E., The production of Al-12Si matrix composite reinforced with SiC particles by powder metallurgy and analysis of the wear behaviour, The Degree of Master Science, Institute of Science, Gazi University, Ankara, Turkey, 2004 (in Turkish).
- [6] Bağcı, C., Production of silicon nitride (Si_3N_4) type technological ceramic material from sepiolite and its characterization, Philosophy of Doctorate Degree, Institute of Science, Gazi University, Ankara, Turkey, 2007 (in Turkish).
- [7] Mindivan, H., The investigation of tribological behavior in silicon carbide (SiC) reinforced aluminum matrix composites, Philosophy of Doctorate Degree, Institute of Science, Istanbul Technical University, Istanbul, Turkey, 2007 (in Turkish).
- [8] Şahin, Y., Introduction to composite materials, Seckin Publisher, 2nd press, Ankara, Turkey, 2006 (in Turkish).
- [9] Yan, S. J., Dai, S. L., Zhang, X. Y., Yang, C., Hong, Q. H., Chen, J. Z., Lin, Z. M., Investigation aluminum alloy reinforced by graphene nanoflakes, *Materials Science & Engineering A*, vol.612, pp.440-444, August 2014.
- [10] Latief, F. H., Sherif, E. S.M., Effects of sintering temperature and graphite addition on the mechanical properties of aluminum, *Journal of Industrial and Engineering Chemistry*, vol. 18, pp. 2129-2134, November 2012.
- [11] Li, J. L., Xiong, Y. C., Wang, X. D., Yan, S. J., Yang, C., He, W. W., Chen, J. Z., Wang, S. Q., Microstructure and tensile properties of bulk nanostructured aluminum/graphene composites prepared via cryomilling, *Materials Science & Engineering A*, vol.626, pp.400-405, February 2015.
- [12] Hanabe, M.R., Aswath, P., $\text{Al}_2\text{O}_3/\text{Al}$ particle-reinforced aluminum matrix composite by displacement reaction, *Journal of Materials Research*, vol.11, issue.6, 1562-1569, July 1996.
- [13] Michael Rajan, H.B., Ramabalan, S., Dinaharan, I., Vijay, S.J., Synthesis and characterization of in situ formed titanium diboride particulate reinforced AA7075 aluminum alloy cast composites, *Materials & Design*, vol. 44, pp. 438-445, February 2013.
- [14] S. Hao, S., Xie, J., Tensile properties and strengthening mechanisms of SiCp-reinforced aluminum matrix composites as a function of relative particle size ratio, *Journal of Materials Research*, vol.28, issue.15, 2047-2055, August 2013.
- [15] Lloyd, D., Particle reinforced aluminium and magnesium matrix composites, *International Materials Reviews*, vol.39, issue.1, pp.1-23, July 2013.
- [16] Pakseresht, A.H., Baghbaderani, H.A., Rad, R.Y., Role of different fractions of nano-size SiC and milling time on the microstructure and mechanical of Al-SiC nanocomposites, *Transactions of the Indian Institute of Metals*, vol.69, no.5, pp.1007-1014, July 2016.
- [17] Gallardo, C.C., Guel, I.E., Melendez, C.L., Sanchez, R.M., Dispersion of silicon carbide nanoparticles in a AA2024 aluminum alloy by a high-energy ball mill, *Journal of Alloys and Compounds*, vol.586, supplement.1, pp.68-S72, February 2014.
- [18] Sharma, P., Sharma, S., Khanduja, D., Production and some properties of Si_3N_4 reinforced aluminium alloy composites, *Journal of Asian Ceramic Societies*, vol.3, pp.352-359, September 2015.
- [19] Kumar, N.M., Kumaran, S.S., Kumaraswamidhas, L.A., An investigation of mechanical properties and corrosion resistance of Al2618 alloy reinforced with Si_3N_4 , AlN and ZrB_2 composites, *Journal of Alloys and Compounds*, vol. 652, pp.244-249, December 2015.
- [20] Gürbüz, M., Şenel, M.C., Koç, E., The effect of sintering time, temperature and graphene addition on the hardness and microstructure of aluminum composites, *Journal of Composite Materials*, doi: 10.1177/0021998317740200 (in Press).