

Properties and Characterization of an Aluminum-silicon Nitride Fiber Powder Metal Composite

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ABSTRACT

There is an increasing demand for new light-weighting materials to improve fuel-efficiency in the automobile and aerospace industries. This has led to an increase in the use of aluminum over traditional metals, such as steel, to increase both efficiency and range. Powder metallurgy is a key enabling technology for the mass production of parts for these industries. Aluminum is known for having excellent specific strength, stiffness and thermal properties, but also is cost-effective for mass production. It suffers, however, from poor wear resistance which lowers its usefulness in applications involving critical moving parts. The inclusion of ceramic reinforcements to produce metal matrix composites (MMCs) can improve both the specific mechanical properties and improve the tribological properties of aluminium. Silicon nitride is a ceramic material with high strength, stiffness and low coefficient of thermal expansion. It also has high toughness for a ceramic material which combines to give it extremely high thermal shock resistance. Fibrous/1D materials provide enhanced material properties in composites (e.g. carbon/glass/polyaramid fibers, carbon nanotubes). Fibrous materials can also enhance the properties of MMCs through dispersion-strengthening and grain boundary-strengthening. The silicon nitride fibers used in this study are sub-micron-diameter, single-crystal fibers with a length 5-24 μm . This is smaller than metal powders allowing the ceramic to coat the metal powder prior to consolidation rather than forming agglomerations that lead to pores and poor fatigue properties. In this study, we have prepared a composite powder using from 2XXX aluminium pre-mix powder and low vol% silicon nitride fibre. This powder was uniaxially pressed and sintered under nitrogen atmosphere. Hardness, tensile properties and elastic modulus data is reported. The mechanical properties are significantly improved even at low loadings. The microstructural analysis clearly shows silicon nitride present in the sintered grain boundaries using EDS to characterise the reinforcement.

Keywords: aluminum, powder metal, silicon nitride, fiber, microstructure

1 INTRODUCTION

In the past few years, the development of aluminium base metal matrix composites (MMCs) has attracted significant attention due to a combination of their low

density, good thermal and electrical conductivities, high hardness and enhanced mechanical strength. This creates potential for numerous applications such as in the automobile, aerospace and defence industries.[1-3] The properties can be tailored through the addition of selected ceramic reinforcements such as metal oxides, metal carbides and metal nitrides.[2-6] The most commonly used reinforcements are in the form of particles, continuous and discontinuous whisker or fibers, with sizes ranging from 10 nm up to 500 μm . [1, 7, 8, 9] Among them, short fiber have been attracting growing interest.[10-12] Short fiber reinforced MMCs have been found that have better mechanical properties than that of particles reinforced MMCs.[10]

Silicon nitride (Si_3N_4) is a material with high mechanical strength and hardness, good thermal and chemical stability, and good wear and corrosion resistance. It is of current interest as reinforcement in the manufacturing of MMCs.[13-16] Several works have been reported on fabrication and mechanical characterization of reinforced with Si_3N_4 particles. AA6082 matrix composites reinforced with Si_3N_4 manufactured by stir-casting process showed an increase in density, hardness and tensile strength with increased content of Si_3N_4 . [13] Al/ Si_3N_4 composite manufactured by pressure infiltration improved the bending strength compared to conventional 2024Al alloy.[14] Al/ Si_3N_4 composite manufactured by powder metallurgy technique showed improved hardness and transverse rupture strength by increasing the volume fraction of Si_3N_4 . [15]

In this paper, we investigate the effect of the addition of 0.5 vol% Si_3N_4 (dominant α -phase) fibers to Alumix 123 to study the impact of high-aspect ratio Si_3N_4 for applications in powder metallurgy (PM). The microstructures and mechanical properties of the as-fabricated samples were evaluated through density tests, hardness tests, tensile tests and microstructure examinations.

2 EXPERIMENTAL PROCEDURE

The base powder used was the commercial Alumix 123 powder (Al-4.5Cu-0.7Si-0.5Mg-1.5 acrawax, Ecka Granules, Germany). The Si_3N_4 fiber (>95% Si_3N_4 , >80 vol% fiber) was manufactured at Nuenz Limited. The morphology of the fiber was investigated and reported in our previous paper.[17] The size distribution of the fibers is given in Table 1.

Reference samples (Al-Ref) and 0.5 vol% Si₃N₄/Al composite samples were used in this study. Green pellets were formed by uniaxially pressing in a 25 mm diameter die at a pressure of 300 MPa. The samples were annealed in a horizontal tube furnace under flowing nitrogen (dew point below -60 °C) at 600 °C for 1 h.

The density of the sintered samples was measured by the Archimedes method. Microstructures of Si₃N₄/Al composites was characterized by scanning electron microscopy (FEI Nova NanoSEM 450) with energy dispersive X-ray Spectroscopy (EDS). HRB of samples were carried out as per ASTM E18. Ultrasonic modulus was measured and calculated according to ASTM E494. Ultimate tensile strength (UTS) measurements were carried out using a specimen as per ASTM E8, B925 (Fig. 19 in ASTM E8) and tested as per ASTM E8. A picture of as-fabricated tensile bar is shown in Fig.1. All mechanical properties were measured in the T1 condition. At least five samples were used for each of the tests.

Table 1: Size distribution of Si₃N₄ fibers.

Si ₃ N ₄ (80 vol% fiber and 20 vol% particles)	D (μm)			L (μm)		
	d ₁₀	d ₅₀	d ₉₀	d ₁₀	d ₅₀	d ₉₀
Fiber size	0.12	0.36	0.66	3	14.5	27.6
Particulate size	0.06	0.15	0.28	-	-	-



Fig. 1. Image of sintered tensile bar.

3 RESULTS AND DISCUSSION

3.1 Green and sintered density

The green and sintered densities of Al-ref and Si₃N₄/Al composites are shown in Table 2. The reference densities were comparable with other investigations and the manufacturer’s specifications.[18] The composite densities were similar to the reference densities which indicated that the fibers did not significantly impact on compaction or densification. The difference between them was within the experimental margin of error.

Table 2: Green and sintered density of samples.

Sample	Green density g/cm ³	Sintered density g/cm ³
Al-ref	2.62 ± 0.01	2.76 ± 0.01
Si ₃ N ₄ /Al composites	2.62 ± 0.01	2.75 ± 0.01

3.2 Mechanical properties

Hardness

The average hardness of Al-ref and Si₃N₄/Al composites were measured to be 28 ±3 HRB and 36 ±3 HRB, respectively. This is a significant increase in hardness. Hardness is a combination of many mechanical properties, including toughness, strength and stiffness. Hardness can be a good indication of wear resistance but is itself, application specific. Specific wear properties were not measured in this investigation but were identified as a part of future, application specific research.

Elastic Modulus

The elastic modulus of Al-ref and Si₃N₄/Al composites were measured as 68.5 ± 1.7 Gpa and 77.1 ±1.1 Gpa, respectively. Models for improved modulus are well known for fiber reinforcement of matrices. The improvement here is beyond the rule of mixtures if a modulus for Si₃N₄ is assumed to be 310 GPa – though the fibers are single-crystalline and this value is often derived from sintered, polycrystalline Si₃N₄. The 13% increase in modulus from a 0.5 vol% addition indicates additional stiffening mechanisms or a significantly higher stiffness in the fibers than in bulk, sintered Si₃N₄.

Tensile Properties

Fig. 2 presents a representative tensile curves of Al-ref and Si₃N₄/Al composites. The average UTS of the reference and composites were 199 ± 11 Mpa and 212 ± 10Mpa, respectively. The yield strength (YS) of the composites was also increased from 124 ± 17 Mpa 132 ± 18 Mpa. The elongation of the composites slightly decreased from 3.1 ± 1.0% to 2.8 ± 0.4%. This demonstrates that the addition of hard Si₃N₄ reinforcement increases the strength of the Al matrix through increased resistance to tensile stresses,[16] with a minor reduction in ductility.

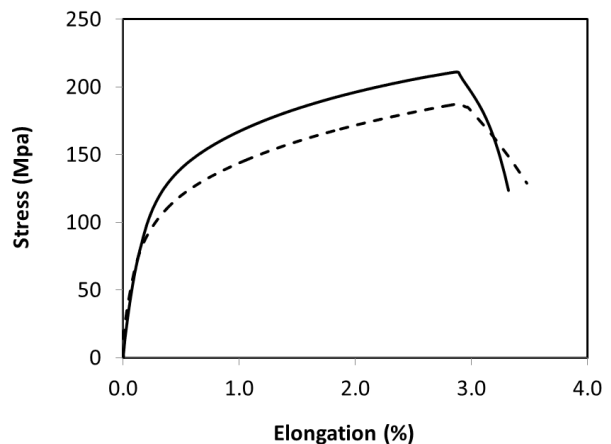


Fig. 2. Typical tensile stress-strain curves of Al-ref (---) and Si₃N₄/Al (—) composites.

3.3 Microstructure

Fig. 3A-B shows optical microscope images of reference and composite samples, respectively, (etched using a corrosive NaF/NaOH etch). There was no notable difference between the arrangement of the grains. Both samples show good densification which is consistent with the density results. Fig. 4A-B shows SEM images of the grain boundary aggressively etched using Weck's reagent. This caused digestion of MgAl species in the grain boundary but left CuAl_x and Si_3N_4 for observation. The digestion of material away from the boundary is clearly evident in the high-magnification images. In this image a Si_3N_4 fiber can be observed lying inside and along the grain boundary. It is also clear that there has been minimal reaction between the matrix and the fiber as the fiber has clean, straight morphology and does have a 'digested' appearance. It is known that the moderate interfacial reaction and enhanced interfacial bonding between matrix and reinforcement results in improved bulk mechanical properties.[19-20] Yet, a reaction is required to intimately bind the reinforcement to the matrix. The improved hardness, modulus and UTS clearly indicate that the fibers have bonded with the matrix to form the strong interphase required for load transfer without too much reaction to cause loss of ductility through formation of undesirable by-products. Furthermore, good density has been achieved – evident through the density analysis and the low-magnification images – which is critical for achieving good mechanical properties considering pressureless sintering was used in this study.

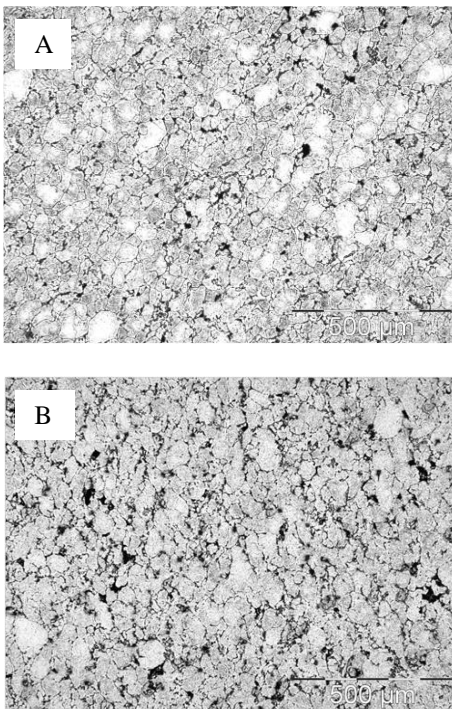


Fig. 3. optical microscope images of reference (A) and composite (B) samples.

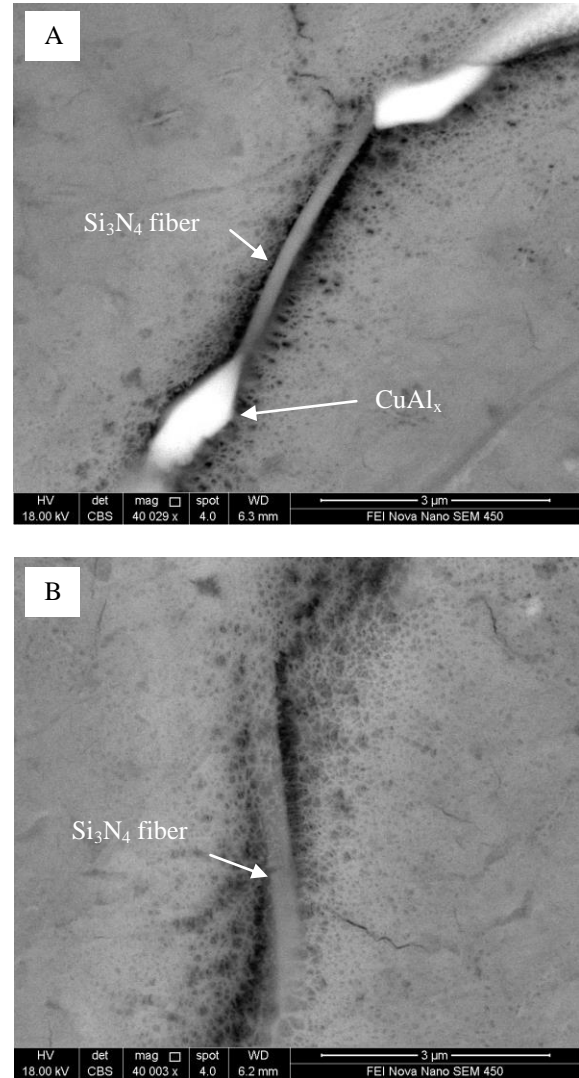


Fig. 4. The SEM images of the grain boundary of composite sample, (Etching Weck)

4 CONCLUSION

In this study, 0.5 vol% Si_3N_4 fiber-reinforced Alumix 123 matrix composites have been successfully fabricated using pressureless sintering under dry nitrogen. The as-fabricated composites exhibit higher hardness (36 HRB), elastic modulus (77.1 Gpa), UTS (212 Mpa) and YS (132 Mpa) and only slightly lower elongation (2.8%), compared with the Al reference. The enhanced mechanical properties of 0.5 vol% Si_3N_4 /Al composite can be attributed to the effective load transfer from the Alumix 123 matrix to the hard Si_3N_4 fiber via good interface bonding. Microstructural analysis reveals easily recognised Si_3N_4 fibers were lying at the grain boundaries. The results obtained above suggest that Si_3N_4 fibers are expected to play a promising reinforcement role for MMCs.

REFERENCES

- [1] Kumar, D., Agnihotri, G., & Purohit, R., "Advanced Aluminium Matrix Composites: The Critical Need of Automotive and Aerospace Engineering Fields", *Materials Today: Proceedings*, 2(4–5), 3032–3041, 2015.
- [2] Rahimian, M., Ehsani, N., Parvin, N., & Baharvandi, H. R., "The effect of particle size, sintering temperature and sintering time on the properties of Al–Al₂O₃ composites, made by powder metallurgy", *Journal of Materials Processing Technology*, 209(14), 5387–5393, 2009.
- [3] Boostani, a F., Tahamtan, S., Jiang, Z. Y., Wei, D., Yazdani, S., Khosroshahi, R. A., Gong, D., "Enhanced tensile properties of aluminium matrix composites reinforced with graphene encapsulated SiC nanoparticles", *Composites: Part A*, 68, 155–163, 2015.
- [4] Pramanik, A., "Effects of reinforcement on wear resistance of aluminum matrix composites", *Transactions of Nonferrous Metals Society of China*, 26(2), 348–358, 2016.
- [5] Jiru, W. G., Sankar, M. R., & Dixit, U. S., "Investigation of microstructure and microhardness in laser surface alloyed aluminium with TiO₂ and SiC powders", *Materials Today: Proceedings*, 4(2), 717–724, 2017.
- [6] Prasad Reddy, A., Vamsi Krishna, P., Narasimha Rao, R., & Murthy, N. V., "Silicon Carbide Reinforced Aluminium Metal Matrix Nano Composites-A Review", *Materials Today: Proceedings*, 4(2), 3959–3971, 2017.
- [7] Chou, T. W., Kelly, A., & Okurat, A., "Fibre-reinforced metal-matrix composites", *Composites*, 16(3), 187, 1985.
- [8] Yashpal, Sumankant, Jawalkar, C. S., Verma, A. S., & Suri, N. M., "Fabrication of Aluminium Metal Matrix Composites with Particulate Reinforcement: A Review" *Materials Today: Proceedings*, 4(2), 2927–2936, 2017.
- [9] Kumar, L., Umasankar, V., Elamathi, P., & Chandrasekar, G., "Synthesis and characterization of nano hexagonal boron nitride powder and evaluating the influence on aluminium alloy matrix", *Materials Today: Proceedings*, 3, 2018–2026, 2016.
- [10] J. M. Papazian, P. N. Adler, "Tensile properties of short fiber-reinforced SiC/Al composites" *Metall. Metallurgical Transactions A*, 21, 401, 1990
- [11] Y. T. Zhu, W. R. Blumenthal, T. C. Loew, "The tensile strength of short fibre-reinforced composites", *Journal of Materials Science*, 32, 2037, 1997.
- [12] Tian, W., Qi, L., Su, C., Liang, J., & Zhou, J. "Numerical evaluation on mechanical properties of short-fiber-reinforced metal matrix composites: Two-step mean-field homogenization procedure", *Composite Structures*, 139, 96–103, 2016.
- [13] Sharma, P., Sharma, S., & Khanduja, D., "Production and some properties of Si₃N₄ reinforced aluminium alloy composites", *Journal of Asian Ceramic Societies*, 3(3), 2–9, 2015.
- [14] Xiu, Z., Chen, G., Wu, G., Yang, W., & Liu, Y., "Effect of volume fraction on microstructure and mechanical properties of Si₃N₄/Al composites", *Transactions of Nonferrous Metals Society of China*, 21, s285–s289, 2011.
- [15] Arik, H., "Effect of mechanical alloying process on mechanical properties of alpha-Si₃N₄ reinforced aluminum-based composite materials", *Materials and Design*, 29(9), 1856–1861, 2008.
- [16] Ramesh, C. S., Keshavamurthy, R., & Madhusudhan, J., "Fatigue Behavior of Ni-P Coated Si₃N₄ Reinforced Al6061 Composites", *Procedia Materials Science*, 6(Icmpc), 1444–1454, 2014.
- [17] T. Dougherty, Y. Xu & A. Hanizan, "Mechanical properties and microstructure of PM Ti-Si₃N₄ discontinuous fibre composite", *TMS 2016*, 721–728, 2016.
- [18] Judge, W. D., "Corrosion behaviour of Alumix 123 P/M and AA2014-T6 in 3.5wt% NaCl", *Dalhousie University, Halifax, Nova Scotia, Canada*, 2015.
- [19] Ramesh, C. S., Keshavamurthy, R., Channabasappa, B. H., & Ahmed, A. (). "Microstructure and mechanical properties of Ni-P coated Si₃N₄ reinforced Al6061 composites", *Materials Science and Engineering: A*, 502(1–2), 99–106, 2009
- [20] Zi-yang, X. I. U., & Yan-mei, L. I. U., "Effects of extrusion deformation on mechanical properties of sub-micron Si₃N₄p/2024 composite", *Transactions of Nonferrous Metals Society of China*, 19, S373–377, 2009.