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# Effect of volume fraction on microstructure and mechanical properties of $Si_3N_4/Al$ composites

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**Abstract:** Si<sub>3</sub>N<sub>4</sub> particles reinforced aluminium matrix composites (Si<sub>3</sub>N<sub>4</sub>/Al) with different particle volume fractions (45%, 50%, and 55%) were fabricated by pressure infiltration method. The effects of Si<sub>3</sub>N<sub>4</sub> volume fraction and T6 treatment on microstructure and mechanical properties of Si<sub>3</sub>N<sub>4</sub>/Al composite were investigated. The results show that Si<sub>3</sub>N<sub>4</sub>/Al composites are well infiltrated with good particles dispersion and no apparent porosity or significant casting defects are observed. High density of dislocations in Al matrix around Si<sub>3</sub>N<sub>4</sub> particles is observed. The bending strength of Si<sub>3</sub>N<sub>4</sub>/Al composites decreases with an increase in Si<sub>3</sub>N<sub>4</sub> volume fraction, and can be greatly improved by T6 treatment. Elastic modulus of composites increases linearly with Si<sub>3</sub>N<sub>4</sub> volume fraction. At a lower Si<sub>3</sub>N<sub>4</sub> volume fraction, more tearing ridge and dimples with elongation are observed. T6 heat treatment shows minor effect on the fracture surface of composite.

Key words: Si<sub>3</sub>N<sub>4</sub>/Al composite; microstructure; bending properties; fracture surface

# **1** Introduction

Ceramic particles reinforced light metal (Al, Mg and Ti) composites, which possess low density, high mechanical properties and good wear resistance [1], have been widely investigated. They have been applied in civil fields in America and Japan, such as piston-connecting rod of automobile and brake disc [2], and show prospective application in aerospace, automotive engine and other industry. Silicon nitride (Si<sub>3</sub>N<sub>4</sub>) has a good thermal and chemical stability, high mechanical strength and hardness, and good wear, creep, and corrosion resistance [3]. These properties make Si<sub>3</sub>N<sub>4</sub> very attractive as reinforcement. Theoretically, Si<sub>3</sub>N<sub>4</sub> particles reinforced aluminium matrix composites (Si<sub>3</sub>N<sub>4</sub>/Al) present several advantages: would low thermal expansion, high specific strength and thermal conductivity, and good dimensional stability [4].

Several methods have been used to fabricate  $Si_3N_4/Al$  composite. ARIK [5] prepared Al matrix composites reinforced with  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> particles (5%, 10% and 15%, mass fraction) by powder metallurgy technique. Al6061 matrix composites reinforced with nickel coated

 $Si_3N_4$  particles (4%–10%, mass fraction) were manufactured by liquid metallurgy route by RAMESH et al [6]. However, it is very difficult to fabricate composites with high reinforcement content by powder metallurgy technique and liquid metallurgy route. Pressureless infiltration method was also reported to fabricate Si<sub>3</sub>N<sub>4</sub>/Al composites [7-8]. However, due to poor wettability between Si<sub>3</sub>N<sub>4</sub> particles and liquid aluminium [9], Mg should be used to improve infiltration process. Even though, fabrication temperature was still around 1 200 °C, and infiltration height was less than 6 mm [7]. Moreover, reaction between Al-Mg alloy and Si<sub>3</sub>N<sub>4</sub> is necessary for Al infiltration in pressureless infiltration method, which restricts the selection of Al allov.

Pressure infiltration method was also used to fabricate  $Si_3N_4/Al$  composites since it is an effective technique to fabricate poor-wetting system [10]. About 50% (volume fraction, the same below if not mentioned)  $Si_3N_4/Al$  composites were successfully fabricated by pressure infiltration method by PENG et al [11]. YANG et al [12] fabricated 36%  $Si_3N_4/2024Al$  by pressure infiltration method and investigated the thermal conductivity properties of the composite. XIU et al [13]

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fabricated Al matrix composites reinforced with 45%  $Si_3N_4$  particles with average particle size of 1.5 µm by pressure infiltration method. LU et al [14] prepared co-continuous  $Si_3N_4/Al$  composites by pressure infiltration method and concluded that reinforcement volume fraction should be around 50% to obtain balanced properties. However, the effect of particles content on high volume fraction of  $Si_3N_4$  particles reinforced Al matrix composites was rarely reported.

Therefore, in the present work, 45%, 50% and 55%  $Si_3N_4/Al$  composites were fabricated by pressure infiltration method. The effects of  $Si_3N_4$  particle volume fraction and T6 treatment on microstructure and mechanical properties were discussed.

# 2 Experimental

The matrix alloy was the commercially available 2024Al alloy, whose compositions (mass fraction, %) were 4.79% Cu, 1.49% Mg, 0.611% Mn, 0.245% Fe, 0.618% Si, 0.068% Zn, 0.046% Ti, 0.013% Ni, and Al balance. The reinforcement was the high pure Si<sub>3</sub>N<sub>4</sub> ceramic particles with nominal diameter of 5 µm, as shown in Fig. 1. The Si<sub>3</sub>N<sub>4</sub>/Al composites with different  $Si_3N_4$  particle volume fractions (45%, 50% and 55%) were fabricated by pressure infiltration method. The preheating temperatures for Si<sub>3</sub>N<sub>4</sub> particle perform and squeeze casting dies were 500 and 740-750 °C, respectively. The matrix alloy was melted under protection of argon and melted salts, and poured into dies at 740-750 °C under argon. Specimens were solution treated at 495 °C in KNO3 salt-bath furnace for 1 h and water quenched at room temperature. After solution treatment, specimens were aged at 160 °C for 8 h and cooled in air.



Fig. 1 Morphology of Si<sub>3</sub>N<sub>4</sub> particles

Microstructures of  $Si_3N_4/Al$  composites were observed by OLYMPUS PMG3 optical microscope (OM). Morphology of particles and fractography of bending specimens were observed on a S-2570 scanning electron microscope (SEM). The further observation was conducted by Philips CM12 transmission electron microscope (TEM) with an accelerate voltage of 120 kV.

The mechanical properties of  $Si_3N_4/Al$  composites were evaluated by three-point bending tests on an Instron5569 universal electron tension testing system. The bending strength was determined on 36 mm× 4 mm×3 mm specimens with a span of 30 mm and a cross-head speed of 0.1 mm/min. All testing have been performed at least five samples, in order to improve statistical significance of the results.

# **3 Results and discussion**

#### 3.1 Microstructure

Figures 2(a), (b) and (c) reveal the morphologies of as-cast  $Si_3N_4/Al$  composites with different reinforcement volume fractions of 45%, 50% and 55%, respectively.  $Si_3N_4$  particles are distributed uniformly in the composite, without any particle clustering. As a result of the high pressure employed during composite fabrication, the molten Al alloy infiltrated the particle preform entirely. Consequently, the composites appear to be free of porosity. The dense microstructure could improve their strength, stiffness as well as dimensional stability, which leads to an improvement in service life [15].

TEM observation reveals high density of dislocations in Al matrix around  $Si_3N_4$  particles, as shown in Fig. 3. Generally, high density of dislocations is always found in Al matrix composites reinforced with particles because of thermal mismatch stress generated due to large difference of coefficient of thermal expansion (CTE) between the ceramic particles and matrix [16].

According to Orowan dislocation theory, the smaller the distance between dispersoids, the larger the curvature radius of dislocation bypass the dispersoids, leading to a higher resistance to dislocation movement. The dislocations would be pinned by  $Si_3N_4$  particles.

#### **3.2 Mechanical properties**

Figure 4(a) shows the effect of  $Si_3N_4$  volume fraction and T6 treatment on bending strength of  $Si_3N_4/Al$  composites. Bending strength of  $Si_3N_4/Al$ composites decreases with an increase in  $Si_3N_4$  volume fraction regardless of heat treatment. The addition of hard  $Si_3N_4$  particles can strengthen the Al matrix alloy and improve its mechanical properties. However, as the volume fraction of the  $Si_3N_4$  particles is much higher than that of the matrix alloy, the ductile deformation ability of the Al alloy would decrease seriously and cause brittleness of the composite. Meanwhile, the defects of  $Si_3N_4$  particles also increase with its volume fraction.



Fig. 2 Optical microstructures of as-cast  $Si_3N_4/Al$  composites with different particle volume fractions: (a) 45%; (b) 50%; (c) 55%



Fig. 3 TEM image of  $Si_3N_4$ /Al composite

These defects may also lead to the failure of  $Si_3N_4/Al$  composites.

The bending strengths of 45%, 50% and 55%



**Fig. 4** Effect of  $Si_3N_4$  volume fractions and T6 treatment on bending properties of  $Si_3N_4/Al$  composites: (a) Bending strength; (b) Bending modulus

Si<sub>3</sub>N<sub>4</sub>/Al composites are increased to 786.5, 728.6 and 668.8 MPa (increased by 40.7%, 35.7% and 26.9%) after T6 treatment, respectively. It should be attributed to the precipitation strengthening effect after aging treatment. Generally, the mechanical properties of composite are determined by matrix, reinforcement and their interface [17]. The interface plays an important role in load transferring, and effective stress transfer is the most important factor which contributes to the strength of two-phase composite materials. If the interface bond is too weak, the stress transfer at the reinforcement/matrix interface is inefficient [18]. Discontinuity in the form of debonding exists due to non-adherence of reinforcement and matrix. Therefore, the reinforcement cannot carry expected stress and the composite strength is low. However, if the interface bond is too strong, the stress concentration at interface would be very high, which may not be sustained by matrix. It will generate initial cracks, which leads to the failure of composite [19]. In this state, the composite would also present low mechanical strength. For composites with medium interface bond, once microcrack is nucleated under stress, it will deflect at the interface due to interface debonding and sliding. Thus, microcrack is stopped at interfaces or

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in the ductile matrix [20]. The plastic deformation of matrix may absorb part of the fracture energy and hinder the propagation of cracks, leading to high mechanical performance of composite.

Figure 4(b) shows the effect of  $Si_3N_4$  volume fraction and T6 treatment on bending modulus of  $Si_3N_4$ /Al composites. It can be seen that elastic modulus of  $Si_3N_4$ /Al composites increase slightly with an increase in  $Si_3N_4$  volume fraction. Moreover, the T6 treatment shows little effect on bending modulus of  $Si_3N_4$ /Al composites.

Figure 5 shows the SEM fractographs of  $Si_3N_4/Al$  composites. Larger  $Si_3N_4$  particles are broken. At a lower  $Si_3N_4$  volume fraction, more tearing ridge and dimples with elongation are observed. T6 heat treatment shows

#### minor effect on the fracture surface of composite.

### 4 Conclusions

1)  $Si_3N_4/Al$  composites are well infiltrated with good particle dispersion and no apparent porosity or significant casting defects are observed. High density of dislocations in Al matrix around  $Si_3N_4$  particles is observed.

2) Bending strength of  $Si_3N_4/Al$  composites is decreased with an increase in  $Si_3N_4$  volume fraction regardless of heat treatment. Bending strength of  $Si_3N_4/Al$  composites could be further improved by T6 treatment. Elastic modulus of composite is increased linearly with an increase in  $Si_3N_4$  particle volume



Fig. 5 SEM fractographs of  $Si_3N_4$ /Al composites: (a) 45%, as-cast; (b) 50%, as-cast; (c) 55%, as-cast; (d) 45%, T6 treatment; (e) 50%, T6 treatment; (f) 55%, T6 treatment

fraction. At a lower  $Si_3N_4$  volume fraction, more tearing ridge and dimples with elongation are observed. T6 heat treatment shows minor effect on the fracture surface of composite.

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# Si<sub>3</sub>N<sub>4</sub>颗粒体积分数对 Si<sub>3</sub>N<sub>4</sub>/Al 复合材料微观组织和 力学性能的影响

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摘 要:采用压力浸渗法制备 Si<sub>3</sub>N<sub>4</sub>体积分数分别为 45%、50%和 55%的颗粒增强铝基复合材料(Si<sub>3</sub>N<sub>4</sub>/Al)。研究 Si<sub>3</sub>N<sub>4</sub>体积分数和 T6 热处理对 Si<sub>3</sub>N<sub>4</sub>/Al 复合材料微观组织和力学性能的影响。结果表明: Si<sub>3</sub>N<sub>4</sub>颗粒分散均匀, Si<sub>3</sub>N<sub>4</sub>/Al 复合材料浸渗良好,没有明显的孔洞和铸造缺陷;在 Si<sub>3</sub>N<sub>4</sub>颗粒附近的铝基体中,可以观察到高密度位错; Si<sub>3</sub>N<sub>4</sub>/Al 复合材料的弯曲强度随着 Si<sub>3</sub>N<sub>4</sub>体积分数的增大而降低; T6 热处理能提高复合材料的强度; 复合材料的弹性模量随着 Si<sub>3</sub>N<sub>4</sub>体积分数的增加而线性增加;在低 Si<sub>3</sub>N<sub>4</sub>体积分数时,可以观察到更多的撕裂棱和韧窝; T6 热处理对断口形貌的影响较小。

关键词: Si<sub>3</sub>N<sub>4</sub>/Al 复合材料; 微观组织; 弯曲性能; 断口