

FRACTURE TOUGHNESS OF *IN SITU* SYNTHESISED TiNp-AlNp/Al COMPOSITE

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SUMMARY: The effect of particle size, particle volume fraction, and matrix microstructure on the fracture initiation toughness of *in situ* TiNp-AlNp/Al composite was examined. The composites were Al matrix reinforced with 7.8 to 19.6 vol pct of TiN and AlN particles produced *in situ* by S-V-L reaction synthetics. The average particle diameters of TiN and AlN were 3.5 and 0.9 μm respectively, which were distributed in Al matrix dispersedly. The room-temperature plane-strain toughness measured using three point bending specimens ranged from 12.7 to 38.5 $\text{MPa}\sqrt{\text{m}}$. Toughness was adversely affected by increase in TiNp-AlNp volume fraction. Fractography revealed that these composite failed in a ductile manner, with voids initiating at the *in situ* reinforcing TiN and AlN particles. The experimentally measured plane-strain toughness properties of in situ TiNp-AlNp/Al composite agree with the Rice and Johnson model.

KEYWORDS: reaction synthetics, toughness, plane-strain, *in situ* composite

INTRODUCTION

Research during the past two decades has examined discontinuously reinforced aluminum composites as potential materials for use in structural applications. These composites have superior stiffness and wear characteristics in comparison with continuously reinforced composites (DRAs) have the advantage of being amenable to conventional metalworking techniques while exhibiting isotropic properties.

While many investigations^[1,2,3,4,5,6] have examined the tensile behavior of discontinuously reinforced composites including *in situ* metal matrix composites, less attention has been paid to fracture properties. Studies have shown that the plane-strain fracture toughness (K_{IC}) of aluminum alloy matrices reinforced with various ceramic reinforcements typically falls into the range of 8.1 to 38 $\text{MPa}\sqrt{\text{m}}$ ^[2,5,7,9] important reactors influencing the toughness of DRA materials are reinforcement type size, volume fraction and distribution, and matrix properties. It is difficult to make generalizations about the effects of these factors on DRA toughness because available information has been gathered on composites with different aluminum alloy matrices and tempers, different reinforcement types, different reinforcement sizes, and materials with different particle distribution. A recent review by Mortensen^[5] and a research by Back Tan^[12] have shown that it is inappropriate to compare the dependence of the toughness of DRA materials on a given parameter without holding the other constant.

A few studies have been published in which the effects of various parameters on composite toughness are investigated systematically. Stephens et al.^[8] Compared the fracture toughness

of cast Al-7 wt pct Si materials reinforced with either SiC or B₄C and found an effect of reinforcement type; the latter material showed superior toughness. Several authors have found an inverse relationship between reinforcement volume fraction and fracture toughness.^[5-8] The relationship between particle size and fracture toughness is presently not clear. Some researcher working on DRAs have reported increased toughness with increasing particle size, while others found toughness to be insensitive to particle size^[10]. Indication that composite toughness is adversely affected by a nonuniform reinforcement distribution has been documented; i.e., propagating cracks showed an attraction toward particles in areas of high local volume fraction, and damage was accumulated in these areas.^[10,11] The effects of matrix properties on the fracture toughness of DRAs have been less widely studied. A recent study on Al-4Cu-1.5Mg/TiB₂ composite addressed the effects of matrix temper and showed that for over and underaged composites at similar strength and ductility levels, underaged composites exhibited superior toughness properties.^[5,12] No information is currently available comparing the toughness of DRAs with identical reinforcement characteristics but varying matrix chemistry.

The composites under investigation in this work are Al matrix reinforced with up to 19.6 vol pct of discontinuous TiN and AlN particles formed by in situ reaction. These composites were produced by the S-L-V reaction synthesis. As a matter of fact, these in situ composites are a type of DRAs materials. In situ 19.6% vol TiN-AlN/Al composite exhibit up to 52 pct increase in modulus and 280 pct improvement in yield stress at room temperature relative to the unreinforced Al matrix because of TiN-AlN reinforcements, reaching a yield strength of nearly 400 MPa in the T8 temper. Their fracture properties are degraded by the addition of volume fraction in reinforcements, as expected. It is necessary that the factors influencing toughness in the composite be understood in order to achieve an optimum combination of strength and toughness. In the current investigation, the room-temperature fracture toughness of these composites is evaluated as a function of reinforcement size, reinforcement volume fraction, and matrix microstructure by systematically varying each of these parameters in turn. It was found that these variables are uncoupled-- the response of these composites to changes in matrix microstructure is dependent on the volume fraction of the reinforcement. Spacing and distribution of particles and matrix temper are found to be the most important factors governing the toughness of in situ TiN-AlN/Al composite.

EXPERIMENTAL

Eight *in situ* TiNp-AlNp/Al composite were produced with particle volume fraction between 7.8 and 19.6 pct and eight different particle sizes. The composites were produced using the S-V-L reaction synthesis developed at Key lab. of state for MMCs in Shanghai Jiaotong University^[4]. Al-6.4Ti-3.0Mg was melted in an induction furnace in a temperature range between 1273K and 1473K, and subsequently mixed gases, which contained N elements was introduced into the molten aluminum alloy in a form of turning gas-mixing, so the titanium and aluminum atoms reacted with N elements forming TiN and AlN crystal nuclei. Owing to the turning gas and electromagnetic mixing, the reaction alloy melt exhibited a homogeneous distribution of fine *in situ* nuclei which moved with the mixing alloy liquid. When enough volume fraction reinforcements were produced, the treatment of eliminating gas was carried out, and then the reacted alloy melt was allowed to solidify, so *in situ* TiNp-AlNp/Al composite was produced. The microstructures of cast composite are shown in fig.1. The as-cast billets were extruded at 743K with 80% reduction in area, and then the samples were machined for the properties of fracture toughness.

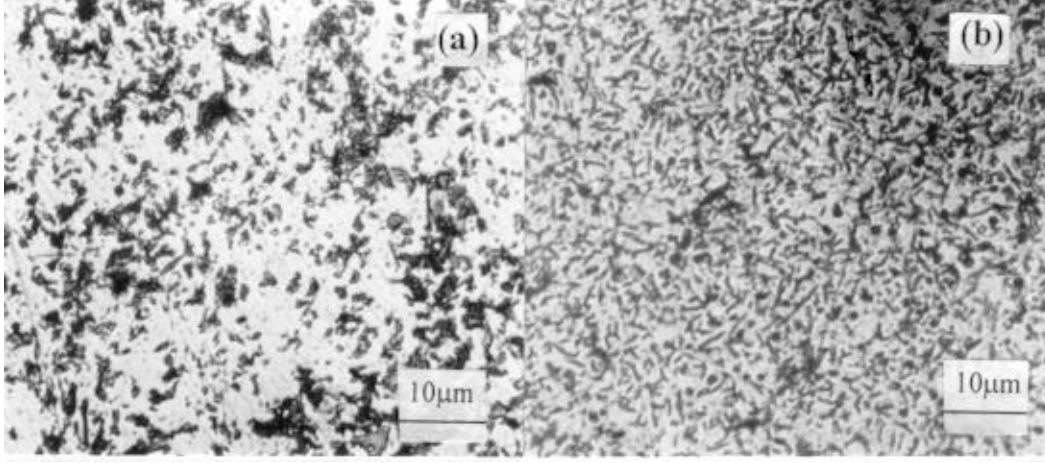


Fig. 1 Microstructure of as cast TiNp-AlNp/Al composite
(a) vol.12.0% TiN-AlNp/Al composite (b) vol.19.6% TiNp-AlNp/Al composite

RESULTS

Fracture toughness tests were performed in The Measurement Standard of K_{IC} in China^[18]. Three point bending samples with thickness of 29 mm were used. All samples were fatigue precracked, and none failed during this process.

One sample was tested for each particle size and volume fraction. The relatively large amount of material required for each measurement prohibited repetition of tests. Of the 40 tests, only a few produced K_{IC} values that were valid within the restrictions imposed by The Measurement Standard of K_{IC} in China. In order to ensure that the tests are conducted under plane -strain conditions, the sample thickness (B) must be such that

$$B \geq 2.5 \left(\frac{K_Q}{\sigma_y} \right)^2 \quad (1)$$

Where K_Q is the measured stress intensity factor and σ_y is 0.2 pct offset yield strength. Because sample thickness was restricted by the dimensions of the extrusion, most of the sample tested did not meet the thickness requirement.

In order to obtain some meaningful relative values of toughness, the methods described in ASTM E992 for determination of an equivalent-energy fracture toughness (K_{ee}) were employed.^[12] This is a standardized method for determining the equivalent-energy fracture toughness that has been used successfully for steels. The Basic difference between E992 and The Measurement Standard of K_{IC} in China is in the choice of load used in the calculation of K_Q . The load chosen for the stress-intensity calculation according to The Measurement Standard of K_{IC} in china, P_Q , is the load at the intersection of the line having a slope 95 pct of the slope in the linear region on the load-displacement curve. In the equivalent-energy method, the energy under the load-displacement curve up to the maximum load is calculated, and a perfectly elastic load triangle of equivalent-energy is constructed. The peak of this triangle, P_e , is the load used to calculate the stress-intensity factor, designated K_{ee} (Figure 2). The K_{ee} values obtained using ASTM E992 do not provide an absolute measure of the plane-strain fracture toughness among the composite materials and trends to be identified.

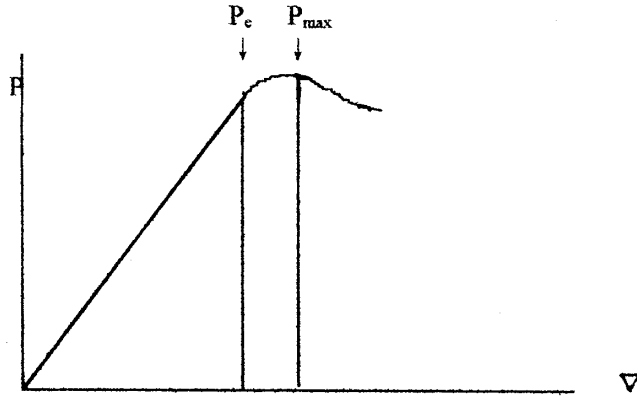


Fig. 2 determination of the load P_e for calculation of equivalent-energy toughness according to ASTM E992

Fractographs were taken using a Philips S515 scanning electron microscopy. Prior to examination, fracture surfaces were cleaned ultrasonically in acetone for approximately 20 seconds. Values of interparticle spacing were obtained by applying a random-line intercept method to scanning electron microscopy images of polished metallographic sections. The volume fraction of TiN-AlN/Al composites were determined by corrosion of Al matrix and weighting of reinforcements.

RESULTS

Values for equivalent-energy fracture toughness, yield strength, modulus, and particle spacing for all composites, in the peak-aged condition (180 °C/10-hour temper), are listed in table 1. As shown in figure 3, fracture toughness decreases with increasing volume fraction of reinforcements, a trend which is consistent regardless of matrix temper. The reduction in toughness per increment of reinforcement volume fraction is the greatest at low volume fractions and then decreases less quickly at higher volume fractions; i.e., there is not a significant toughness penalty for increasing TiN-AlN particle content above 19.6 pct. At a given volume fraction, there is no obvious dependence of fracture toughness on in situ particle size.

Though most fracture toughness tests were invalid according to ASTM E399 because of sample thickness restrictions, a few valid K_{IC} values were obtained from the experiments. Tests which met the validity criterion were those from the higher volume fraction composites in the peak and near peak-aged conditions, which were shown in table 2. Values obtained from the tests were in the range of 9.1 to 18.5 $\text{MPa}\sqrt{\text{m}}$, which is similar to DRAs materials having similar yield strength^[5] and is also similar to values reported for 2XXX matrix composites with SiC reinforcements.^[6,7] Unfortunately, there were not enough valid K_{IC} 's reported to identify any trends in toughness of these composites with volume fraction, particle size, or matrix microstructure. The K_{IC} values are included in table 2 for comparison with K_{IC} values, showing that K_{IC} overestimates the stress-intensity factor.

Table 1. Room-temperature mechanical and physical properties of *in situ* TiC-AlN/Al composites aged at 180 °C for 10 hours

| Particle size (μm) | | Volume Fraction | Interparticle Spacing (μm) | Yield Strength (MPa) | Elastic Modulus (GPa) | Tensile Elongation (pct) | K_{ee} ($\text{Mpa m}^{1/2}$) |
|------------------------------------|------|-----------------|--|-------------------------|--------------------------|-----------------------------|--------------------------------------|
| TiC | AlN | | | | | | |
| 2.35 | 1.37 | 0.078 | 8.2 | 171.6 | 83.3 | 5.3 | 38.2 |

| | | | | | | | |
|------|------|-------|-----|-------|-------|------|------|
| 2.69 | 1.57 | 0.12 | 6.3 | 292.9 | 93.8 | 1.2 | 32.1 |
| 3.12 | 1.61 | 0.145 | 6.1 | 352.3 | 95.5 | 1.0 | 28.8 |
| 3.35 | 1.79 | 0.168 | 4.2 | 361.2 | 97.3 | 0.97 | 22.3 |
| 3.66 | 1.80 | 0.184 | 3.3 | 387.0 | 100.8 | 0.94 | 18.6 |
| 3.80 | 1.83 | 0.196 | 2.7 | 406.3 | 107.9 | 0.87 | 14.8 |

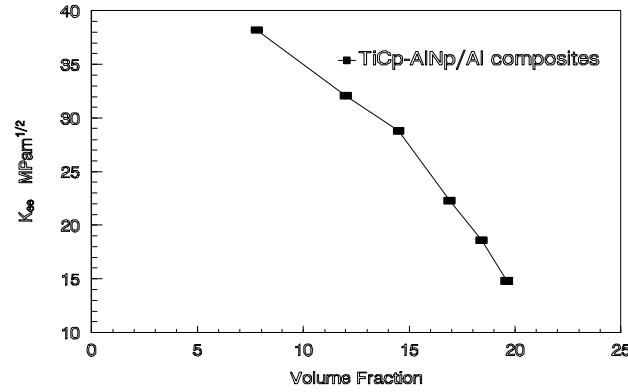


Fig. 3 Variation in equivalent-energy fracture toughness with volume fraction of TiC-AlN particles. The matrix of the composite is in 10 h -180°C aged condition

Table 2. Room-temperature Fracture Toughness of Selected TiC-AlN/Al Composites

| Volume Fraction | Aging Time at 180°C (h) | K_{IC} (Mpa \sqrt{m}) | K_{ee} (MPa \sqrt{m}) | Calculated K_{IC}^* (MPa \sqrt{m}) |
|-----------------|-------------------------|----------------------------|----------------------------|---|
| 0.12 | 10 | 28.2 | 32.1 | 18.6 to 30.6 |
| 0.12 | 15 | 25.8 | 30.2 | 18.2 to 29.9 |
| 0.12 | 20 | 24.5 | 29.3 | 17.8 to 29.2 |
| 0.196 | 10 | 13.7 | 14.8 | 15.4 to 25.3 |
| 0.196 | 15 | 13.0 | 13.9 | 15.1 to 24.6 |
| 0.196 | 20 | 12.7 | 13.8 | 14.5 to 23.8 |

***Calculated K_{IC} show range of values obtained using Rice and Johnson's model**

Fractographs, shown in Figure 4, reveal the dimple morphology characteristic of ductile failure by void nucleation and growth. Void nucleation at the primary ceramic particles is evident in the unreinforced alloy. Density of voids increases and size of voids decreases with increasing in situ TiN-AlN content, indicating that nucleation is connected with the in situ reinforcements. Most of TiN and AlN particles separate from the matrix by decohesion, regardless of matrix temper, though some larger particles are cracked. This is in contrast to void nucleation behavior in DRAs with SiC or Al₂O₃, for which particle cracking dominates.^[2-4,9,10] The main difference in void initiation mechanism between in situ TiN-AlN/Al and traditional SiCp/Al composites may be a result of particle size, particle/matrix interfacial strength, or intrinsic particle toughness. For TiN-AlN/Al composite, the sizes of TiNp and AlNp are 2~5 μ m and 0.2~1.8 μ m respectively. For a larger particle, there is a higher tendency for cracking because of the greater probability of finding a critically sized flaw within them. Several authors have noticed a propensity for cracking in large in SiC

particles for SiC/Al composites.^[6,10,12]

DISCUSSION

Several authors have tried to predicate of DRAs materials and composites based on the analysis of Rice and Johnson.^[11] This model assumes that crack growth will occur when the extent of the heavily deformed region ahead of a crack tip is of comparable size to that of the unbroken ligament separation void initiation sites.^[14] Rice and Johnson's model predicts that the crack opening displacement at ligament fracture will be in the range of

$$\delta \cong 1.0 X_0 \text{ to } 2.7 X_0 \quad (2)$$

With the lower bound applicable to composites of high ductility. Here X_0 is the characteristics dimension of failure, which for the present RDSs is assumed to be represented by the interparticle spacing, λ . The heavily deformed region was shown to extend a distance comparable to the crack opening displacement, given by^[14,15]

$$\delta \cong K^2/2E\sigma_y \quad (3)$$

Where K is the stress-intensity factor, E is Young's modulus, and σ_y is yield strength. The term K can be predicted from Eq.[2] and [3], in the manner of Hahn and Rosenfield^[16]

$$K_{IC} \cong K(E\sigma_y\lambda)^{1/2} \quad (4)$$

Where

$$\sqrt{2} \leq K \leq \sqrt{5.4}$$

Several authors indicated that this model may be applicable to DRAs composites. Folm and Arsenault^[17] suggested that their results on SiCp/Al composites were in general agreement with this type of model, and Kamat et al.^[2] found a correlation with a Rice and Johnson model similar to that described by Eq.[4] for Al_2O_3 -reinforced 2XXX alloys in which the interparticle spacing was less than 25 μm .

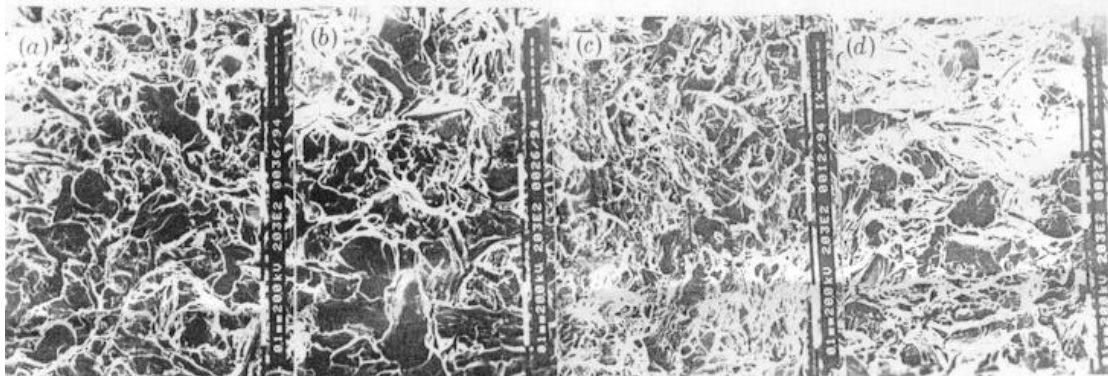


Fig. 4 Fracture surfaces of in situ TiN-AlN/Al composites, showing ductile failure and void nucleation at in situ particulate: (a) unreinforced, (b) 7.8 vol pct, (c) 12.0 vol pct, and (d) 19.6 vol pct TiN-AlN. Matrix artificially 10 h at 180 °C.

Recently, Back Tan, Manhattan and Lewandowski^[12,17] compared the predictions from these models to experimental data and found reasonable agreement between measured and calculated values of toughness (with $K = \sqrt{2}$) and between measured values of δ and calculated values of λ . For 7XXX-SiC and Al-4Cu-1.5Mg/TiB₂ composites.

Upper and lower values of K_{IC} calculated using Eq. [4] for the TiN-AlN/Al samples of this study that met the ASTM E399 Validity criterion are shown in table 2. The measured values of K_{IC} for the composites with many different particle size. All fall into the predicted range.

The measured K_{IC} values for all the *in situ* TiN-AlN/Al composites either at or above the large ductility limit of the model ($K = \sqrt{5.4}$). It is not too surprising that the model fails for all the TiN-AlN/Al composites, the average value of particle spacing used in the calculation is a very poor representation of the actual value of particle spacing because of nonuniform particle distribution in these composites. Some authors have indicated that particle clustering is a detrimental to toughness.^[3,6,10] However, the composites in question has the fields of severe particle clustering alternation with the fields of very low particle volume fraction along the direction of crack propagation. The propagating crack will encounter easy fracture regions, where the volume fraction of particles is high and void initiation sites are plentiful, alternating with regions which are nearly particles free, where the fracture is being controlled by the ductile matrix. The slower crack propagation in the ductile matrix regions may be the controlling step in failure. In this case, the nonuniformity of the microstructure may be the controlling step in failure. In this case, the nonuniformity of the microstructure may actually improve the toughness measured on the macroscale, masking the toughness loss that has been associated with decreasing particle size in previous studies.

The K_{ee} values used to represent stress-intensity parameters for the majority of the composites cannot be directly compared to calculation for K_{IC} . The equivalent-energy parameter should, however, show the same dependence on spacing as the stress-intensity factor.

CONCLUSIONS

1. The fracture toughness of TiNp-AlNp/Al composite is a function of reinforcement volume fraction and size, and matrix microstructure.
2. The room-temperature plane-strain toughness measured from 12.7 to 37.5 MPa \sqrt{m} . Toughness was adversely affected by increase in TiNp-AlNp volume fraction. No conclusion could be made concerning the effect of increasing particle size on toughness, because particle size was fixed while volume fraction is the same for this kind of *in situ* composites.
3. fractography revealed that TiNp-AlNp/Al composites failed a ductile manner with voids initiating at the *in situ* reinforcing TiN and AlN particles.
4. The experimentally tested K_{IC} values of *in situ* TiNp-AlNp/Al composites agree with the Rice and Johnson model.

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