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Original Article

Tribological behavior of α^1/β^1-SiAlON-TiN composites

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ABSTRACT

There are limited studies on the tribological properties of SiAlON-TiN composites. Therefore, in the present study, tribology tests were conducted on a number of α^1/β^1-SiAlON-TiN composites with different α^1/β^1-SiAlON phase ratio and TiN content, fabricated with unique compositional design. The influence of α^1/β^1-SiAlON phase ratios, microstructure, mechanical properties and TiN content on friction and wear behavior was investigated and wear mechanisms were explained. Tribology tests were conducted on computer controlled tribometer under dry unlubricated ambient conditions with a linear reciprocating movement in a ball-on-disc sliding wear configuration and test parameters kept as constant. It was observed that TiN addition (17 wt.%) did not change the friction (CoF) of SiAlON and wear rate and wear volume were observed to increase. Wear mechanisms showed differences with α^1:β^1-SiAlON phase ratio. Fracture toughness had very pronounced effect on wear resistance.

1. Introduction

Alumina based (Al_2O_3-SiC_w) and silicon nitride based (Si_3N_4-SiC/TiC/TiN) ceramic matrix composites have been used for cast iron and superalloy machining. SiAlONs are structured in the fundamental unit of Si_3N_4 and they are basically defined as solid solution between the Si_3N_4 and Al_2O_3. They possess many inherent advantageous over Si_3N_4 such as easy sintering, wide compositional design (100%wt.α to 100% wt. β), different intergranular phase chemistry, microstructure and hence wide range of mechanical and physical properties [1]. SiAlON ceramics have been also used for super alloy machining [2]. However their performance is not as effective as Al_2O_3-SiC_w cutting tools especially at higher cutting speeds. On the other hand Al_2O_3-SiC_w cutting tools have many disadvantages over SiAlON ceramics such as costly production method and SiC whiskers are dangerous to human health. Therefore if the performance of SiAlON ceramics is improved, they will be good competitors to Al_2O_3-SiC_w cutting tools with cost advantage.

Insufficient chemical durability of SiAlON ceramics inhibited wide use of it for superalloys machining. The chemical durability of SiAlONs can be improved in two possible ways: proper compositional design with optimum z (solid solution) value, crystalline intergranular phase (IGP), good coalescence behavior of IGP and addition of second phases. Increasing z value (~1) leads to longer tool life because of the reduced chemical wear during machining [3]. However with the increase of z value to ~1, mechanical properties like fracture toughness and hardness will deteriorate [4,5]. Therefore the z value should be optimized and the incorporation of second phases in order to improve chemical resistance is more feasible. TiN is a very good candidate to improve chemical wear resistance and mechanical properties of SiAlON ceramics [6–11]. On the other hand compositional design is very crucial that effect chemical wear resistance of α^1/β^1-SiAlONs, especially, at high temperatures. The SiAlON samples should have high z value (0.5 <) and crystalline intergranular phase chemistry and good coalescence behavior to resist the high temperatures during superalloy machining. In our previous studies, the effect of cation type, intergranular phase (IGP) amount and cation mole ratios on z value and intergranular phase crystallization of α^1/β^1-SiAlON-TiN composites and the effect of z value on the crystallization and coalescence of IGP were investigated [12–14]. It was found that crystallization tendency of Er and Yb cations were higher than that of Y cation. The highest fracture toughness 7.4 $MPam^{1/2}$ and moderate hardness values (15.37 GPa) was obtained with Er containing composition. It was concluded that, z value was an effective parameter on intergranular phase chemistry and crystallization after sintering. Post sintering heat treatment improved the crystallization and consequent coalescence behavior of IGP.

Since the friction and wear are the important factors during machining, the investigation of the tribological behavior of α^1/β^1-SiAlON-TiN composites is crucial. Studies on tribological behavior of Si_3N_4-TiN composites have been widely reported [15–19]. It was reported that incorporation of TiN phase increase wear resistance and with the formation of titanium oxide solid lubricant film reduce friction [20–23]. However, there are limited studies on the tribological properties of SiAlON-TiN composites [24]. Therefore, in the present study, tribology tests were conducted on a number of

α^1/β^1-SiAlON and α^1/β^1-SiAlON-TiN composites with different α^1/β^1-SiAlON phase ratio and TiN content, fabricated with unique

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Table 1
Specifications of prepared SiAlON-TiN composites.

Sample Code	TiN content (wt%)	α′: β′ -SiAlON Phase Ratios	Bulk Density (g/cm^3)	Theoretical Density (g/cm^3)	Theoretical Density %
T0035	0	35 α′ : 65 β′	3.3592	3.3693	99.70
T1735	17	35 α′ : 65 β′	3.5796	3.5885	99.75
T2510	25	10 α′ : 90 β′	3.6854	3.6939	99.77
T2575	25	75 α′ : 25 β′	3.6851	3.6921	99.81

compositional design. The influence of α′:β′-SiAlON phase ratios, microstructure, mechanical properties and TiN content on friction and wear behavior were investigated and wear mechanisms were explained.

2. Experimental procedures

α′:β′-SiAlON-TiN composites were developed with different α′:β′-SiAlON phase ratios (10α′:90β′, 35α′:65β′ and 75α′:25β′) and containing various amounts of TiN particles (17 and 25wt%). α′:β′-SiAlON compositions were designed with 9Er:0.5Sm:0.5Ca cation system and 0.6 of z value (see Table 1). In our previous study [14], crystalline and good coalescence behavior of IGP, high aspect ratio grains over 7 and hence the highest fracture toughness 7.4 MPam$^{1/2}$ and moderate hardness values (15.37GPa) was obtained with 9Er:05:Sm:0.5Ca cation system. If the material has this kind of composition, it should exhibit better mechanical and chemical properties during superalloy machining. The coding for the compositions is as follows: the first three figures represent the TiN content. T17 means 17 wt.% TiN content. The next two figures represent the α′-SiAlON phase ratios.

UBE-E10 grade α-Si_3N_4 powder (1.4 wt% O content, UBE Co. Ltd., Japan), high purity AlN powder (1.6 wt% O, H Type, Tokuyama Corp. Japan), Al_2O_3 (Alcoa A16-SG, Pittsburgh, USA), Er_2O_3 (> 99.99%, Treibacher, Austria), Sm_2O_3 (> 99.9%, Stanford Materials Corp., USA), $CaCO_3$ (> 99.75%, Reidel-de Haen, Germany) and TiN powder with average particle size of 1–2 μm (> 99% pure, H.C. Starck, Grade C, Berlin, Germany) were used in order to produce α′β′-SiAlON-TiN composites. The designed compositions were prepared by planetary milling for 90 min at 300 rpm in isopropyl alcohol using Si_3N_4 balls. The milling conditions of nitride based powders would affect the subsequent microstructure and mechanical properties, and these milling conditions were specifically choosen [25]. TiN was introduced into the starting powder mixture in order to provide homogeneous dispersion of TiN particles in the SiAlON matrix. Rotary evaporator was used for drying slurries and then the powders were dry sieved with a mesh size of 150 μm. The powders were uniaxially pressed under 25 MPa, and subsequently cold isostatically pressed at 300 MPa. The pellets were sintered using a two-step gas pressure sintering cycle. The first step (pre-sintering) was carried out at 1890 °C for 60 min at 0.5 MPa nitrogen gas pressure followed by a sintering step at 1940 °C for 60 min at 2.2 MPa nitrogen gas pressure and then the furnace was allowed to cool at a rate of 5 °C/min in order to obtain crystalline IGP.

Archimedes principle was used to measure the bulk density of the samples after sintering by using the following equation.

$$\text{Bulk Density} = \frac{W_1}{W_3 - W_2} x\rho_{water} \quad (1)$$

where, W_1 is dry weight, W_2 is wet weight suspended in water, W_3 is wet weight, B.D. is bulk density. Theoretical density of the samples was found by He gas picnometer (Micromeritics Accupyc II 1340 model) and theoretical density% values were calculated with the following equation.

$$\%\ \text{Theoretical Density} = (\text{Bulk density} / \text{Theoretical density}) * 100 \quad (2)$$

The α′:β′-SiAlON phase ratios and intergranular phase chemistry were determined by X-ray diffraction methods (XRD-Panalytical, Empyrean with Cu-Kα radiation). The α′:β′-SiAlON phase ratios were found by quantitative estimation from the XRD patterns using the integrated intensities of the (102) and (210) reflections of α′-SiAlON and the (101) and (210) reflections of β′-SiAlON by the following equation:

$$\frac{I_\beta}{I_\beta + I_\alpha} = \frac{1}{1 + K[(1/w_\beta) - 1]} \quad (3)$$

where I_α *and* I_β are observed intensities of α′ and β′-SiAlON peaks, respectively, w_β is the relative weight fraction of β′-SiAlON, and K is the combined proportionality constant resulting from the constants in the two equations:

$$I_\beta = K_\beta * W_\beta \quad (4)$$

$$I_\alpha = K_\alpha * W_\alpha \quad (5)$$

K was taken as 0518 for β (101) – α (102) reflections and 0544 for β (210) – α (210) reflections [26].

The cell parameters of β′-SiAlON were measured with silicon powders as the internal standard. The z-value of the β′-SiAlON phase was obtained from the mean of z_a and z_c values given by the following equations:

$$z_a = \frac{a - 7.6044}{0.031} \quad (6)$$

$$z_c = \frac{c - 2.9075}{0.026} \quad (7)$$

where *a* and *c* are the calculated unit cell dimensions of β′-SiAlON: JCPDS card 33–1160 was used as a reference for β-Si_3N_4 where a = 7.6044(2)Å and c = 2.9075(1)Å. z value of all of the developed α′:β′-SiAlON-TiN composites were found between 0.6-0.62. Scanning electron microscopy (SEM) analysis was conducted for microstructural investigations (SEM-ZEISS Supra 40VP) with back-scattered electron imaging mode.

Vickers hardness tests were conducted under 98 N load. The Vickers hardness (HV) was calculated by the following equation (Evans and Charles): [27]

$$HV_{10} = 0.47P/a2 \quad (8)$$

where, HV_{10} is the Vickers hardness, P is load applied and a is half the length of the diagonal of the indentation produced by the indenter. Indentation fracture toughness (K_{1C}) was calculated by the hardness tests using the formula proposed by Niihara et al for median cracks: [28]

$$K_{IC} = 0018 * HV * a^{0,5} * (E/HV)^{0,4} * (c/a - 1)^{-0,5} (\text{for } c/a < 3.5 \text{ and } l/a < 2.5) \quad (9)$$

where 2a is the average indent diagonal length (μm), 2c is the crack length (from one crack tip to another), E is the elastic modulus (GPa) which is taken as a constant equivalent to 320 GPa for all the samples and H is the measured hardness (GPa). The crack length and indent diagonal were measured from optical images of the indented surfaces. 3 samples tested for each composition with 5 indents and the standard deviation was calculated.

Before tribology tests, the sample surfaces was polished and cleaned with acetone in an ultrasonic bath for 10 min and then the samples dried at room temperature. MarSurf PS1 (Mahr GmbH, Gottingen, Germany) model surface profilometer was used to determine surface roughness. Tribological behavior of samples was conducted on computer controlled tribometer (TRD Engineering, Sakarya, Turkey) under dry unlubricated ambient conditions with a linear reciprocating movement in a ball-on-disc sliding wear configuration. The samples were placed on a translation plate, which oscillated at the required stroke length and desired frequency. The coefficient of friction was