THE INFLUENCE OF MICROSTRUCTURE AND MECHANICAL PROPERTIES ON THE FRICTION AND WEAR BEHAVIOUR OF Si₃N₄ CERAMICS

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SUMMARY
The influence of microstructure and mechanical properties on the friction and wear behaviour of Si₃N₄ ceramics was investigated by sliding contact tests without lubricant. The microstructures and mechanical properties of the Si₃N₄ ceramics were controlled in the fabrication process by varying the sintering time and amounts of sintering additives. A clear relationship between the mechanical and tribological properties of the Si₃N₄ ceramics could not be determined since the wear mechanism was different depending on grain boundary content and composition.

Keywords: silicon nitride, wear, sintering additive, mechanical properties, microstructure

1 INTRODUCTION
Ceramic materials such as SiC, Si₃N₄ and Al₂O₃ are serious contenders for use as tribological components. Si₃N₄ ceramics, in particular have excellent mechanical properties. In addition, Si₃N₄ ceramics have low chemical reactivity, low density and high young’s modulus compared to metals. These features are maintained at high temperature, so it is expected that Si₃N₄ may be employed in applications such as high temperature gas turbines, thermal insulated engine parts and fuel injection pump nozzles [1,2]. It is well known that the properties of ceramics depend on micro structural features such as grain size, micro pore distribution, grain boundary phase, grain morphology etc. The intergranular glassy phase in Si₃N₄ ceramics, which is usually about 10 or more weight percent, is considered to control not only the sintering behaviour but also the mechanical properties. However, the relationship between grain boundary phase and the mechanical and wear properties has not been sufficiently clarified. In wear tests with low normal load, the relationship between microstructure and wear properties is unclear because the wear behaviour depends on the intrinsic properties of the material, such as chemical stability. In this study, the influence of microstructure and mechanical properties on the friction and wear properties of various Si₃N₄ ceramics was investigated during sliding contact without lubrication in air.

2 EXPERIMENTAL PROCEDURE
2.1 Sample Preparation
Silicon nitride powder (Ube industrial Co. Ltd., Grade E10, >94% α phase) of size 0.3 μm was mixed with Y₂O₃ (Shin-etsu chemical Co. Ltd., RU-P Grade) and Al₂O₃ (Sumitomo Kagaku Co. Ltd., AKP-50) in a polyethylene bottle with polyethylene coated iron balls and ethanol for 24 h. The composition of starting powders is shown in Table 1. The slurry was dried in a rotating condenser and subsequently passed through a 150-mesh screen to pulverize aggregates. The mixed powders were hot-pressed in a furnace (High Multi 10000, Fuji Dempa Co. Ltd.) at temperatures ranging from 1750 - 1950 °C at a pressure of 30 MPa in a nitrogen atmosphere.

<table>
<thead>
<tr>
<th>Si₃N₄</th>
<th>Al₂O₃</th>
<th>Y₂O₃</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.5A0.5Y</td>
<td>99</td>
<td>0.5</td>
</tr>
<tr>
<td>1A1Y</td>
<td>98</td>
<td>1</td>
</tr>
<tr>
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<td>96.5</td>
<td>1.75</td>
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<tr>
<td>2.5A2.5Y</td>
<td>95</td>
<td>2.5</td>
</tr>
<tr>
<td>3.34A3.34Y</td>
<td>93.2</td>
<td>3.34</td>
</tr>
<tr>
<td>5A5Y</td>
<td>90</td>
<td>5</td>
</tr>
</tbody>
</table>

Table 1: Composition of the starting powders (wt%)
ding test was conducted at a loading rate of 0.5 mm min\(^{-1}\) on a multi-testing machine (Sintech 10/GL, MTS corporation).

The surfaces of the specimens were polished to surface roughness of Ra = 0.005 µm. Fracture toughness data were obtained by both the indentation fracture method (IF), with an indentation load of 196 N, and single edge precracked beam (SEPB) method. The fracture toughness values were calculated employing the equation given in JIS R1607 [4].

Vickers hardness values were obtained by indenting polished surfaces at 98 N (HV10) and calculated according to the JISR1610 [5].

2.4 Wear Properties Evaluation

The samples for wear analysis were machined into plates of dimensions 30 x 27 x 5 mm, and polished to surface roughness of Ra = 0.005 µm. Unlubricated ball-on-disk experiments were carried out against a commercial Si\(_3\)N\(_4\) ball (Nihon ceratec Co. Ltd.) with diameter of 5 mm. Wear test conditions were as follows: sliding speed = 0.18 m s\(^{-1}\); normal load = 49 N; test duration = 600 s (108 m); atmosphere = air; temperature = 22 - 28 °C; relative humidity during testing = 20 - 30 %RH. Wear volumes were evaluated using a surface profilometer and calculated according to the method of JIS R1613 [6]. Worn surfaces were also observed by SEM.

3 RESULTS

Figure 1 shows the microstructures of the fabricated Si\(_3\)N\(_4\) samples sintered at 1950 °C for 2 h. The microstructure of all of the Si\(_3\)N\(_4\) material fabricated in this study was typically bimodal in structure. All samples exhibited grain growth. This behaviour was enhanced with increasing amount of sintering additives.

Figure 2 shows X-ray diffraction spectra of selected samples sintered at 1950 °C. For all the samples, only the Si\(_3\)N\(_4\) peaks were identified. At a sintering temperature of 1750 °C both α and β-Si\(_3\)N\(_4\) peaks were observed, but samples sintered over 1850 °C consisted of β-Si\(_3\)N\(_4\) only.

Figure 3 shows an SEM image of a typical worn surface following the wear test. All samples exhibited surfaces showing typical abrasive wear with particulate debris and rough surface. This kind of surface appearance indicates that, under these test conditions, the wear mechanism was predominantly one of microstructure fracture.

Figure 4 shows the relationship between the amount of sintering additives and specific wear rate. The amount of sintering additives was shown to have a significant effect on the wear behaviour. Specific wear rates decreased as the sintering additive amount was reduced from 10 to 5 wt%. However, decreasing the content further led to an increase in wear rate.

Figure 5 shows the mechanical properties plotted against specific wear rate for all the samples. There was a general trend of decreasing fracture toughness with increasing specific wear rate. However both bending strength and Vickers hardness showed no apparent correlation with specific wear rate.
DISCUSSION

A clear relationship between mechanical properties and specific wear rate could not be determined from these results. The fact that fracture strength showed little correlation with wear properties is probably because the wear occurred over very small regions. Amongst the mechanical properties, fracture toughness showed a better correlation, but it is difficult to determine a relationship between mechanical properties and wear properties because of the number of possible contributing factors. In general, the samples with lowest fracture toughness were those with lower sintering additives. When considering the relationship between wear properties and sintering additives, the wear rate was lowest at an additive content of around 5 wt%. However, the wear rate increased for samples with both lower and higher additive contents, and it is thought that the wear mechanism in these two cases may well be different.

The fracture toughness of Si₃N₄ ceramics, in particular, depends on factors such as aspect ratio, grain size and bonding strength between grain and grain boundary. This latter factor is especially important. Raw Si₃N₄ contains around 2.8 wt% SiO₂ on the powder surface [7], which forms a eutectic liquid phase with the oxide additives during sintering.

For the higher additive samples, the Si content in the grain boundary phase is therefore relatively lower, which indicates weaker bonding between the grains and grain boundary phase [8]. Under these conditions, the cracks induced during the wear test may proceed along the grain / grain boundary interface resulting in fracture over a much smaller scale. This may lead to dropping of individual grains and an increase in the wear rate. As the additive content is decreased, the Si content in the glass becomes relatively higher, and the bonding strength increases, which may prevent micro fracture in the region of the grain and grain boundary leading to less dropping of individual grains, and a reduction in wear rate. However, further strengthening of the grain boundary interface by reducing the additive content further, means that the path of the progressing crack is not deflected, resulting in intragranular fracture, and an increase in wear rate. There is therefore an optimum content of additives that strengthens the grain boundary interface sufficiently to prevent grain dropping, whilst retaining the ability to allow crack deflection.

Figure 4: The relationship between specific wear rate and amount of sintering additives

Figure 5: The relationship between specific wear rate and Mechanical properties
5 CONCLUSIONS

A clear relationship between mechanical properties and tribological properties of Si$_3$N$_4$ ceramics could not be obtained because the wear mechanism was thought to be different depending on composition. For Si$_3$N$_4$ with the same composition of sintering additives, changing the sintering additive content changes the grain boundary composition and results in differences in bonding characteristics between grains and grain boundary phase.

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7 REFERENCES


