

## A comparative study of SiAlON ceramics

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Received 20 January 2012; received in revised form 6 April 2012; accepted 6 April 2012

Available online 15 April 2012

### Abstract

Two different varieties of  $\text{Si}_3\text{N}_4$  powders were used to prepare SiAlON ceramics. 100%  $\beta\text{-Si}_3\text{N}_4$  was used from refractory grade powders (B1) and another purer 98%  $\alpha\text{-Si}_3\text{N}_4$  (50A) powder was used to prepare the SiAlON samples. Since SiC + SiAlON composites reportedly perform better, batches were prepared with 15% SiC addition to the refractory powders (B1) and 17.5% SiC was added to the other SiAlON composition (50A). The samples were gas pressure sintered at 1840 °C and at 22 bar with 1 h dwelling time. Thereby, we could achieve 97–98% theoretical density. The hardness was recorded 14–17 GPa while fracture toughness varied from 4.3 to 5 MPa m<sup>1/2</sup>. Fretting experiments showed initial running-in period of 300 cycles for all the tribo-couples. After which, the steady state coefficient of frictions (COF) were achieved. Steel ball of 10 mm diameter, fretting against 50A composition, showed 0.6 average steady state COF while the same composition while fretting against alumina ball of the same diameter, showed 0.57 average steady state COF. Results have been compared with SiAlON composition derived from refractory powder (B1) and found that the 50A composition performs better under identical test conditions. Moreover, cytocompatibility study also suggests that the investigated 50A composition can be used as substrate to support cell adhesion and proliferation of L929 mouse fibroblast cell lines whereas B1 composition derived from refractory powders are toxic in nature.

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**Keywords:** C. Friction; C. Mechanical properties; D. SiAlON; Biocompatibility

### 1. Introduction:

Silicon nitride ( $\text{Si}_3\text{N}_4$ ) ceramics and silicon-aluminium-oxinitride (SiAlON) ceramics are very important class of engineering ceramics. Traditionally alumina has been in use for structural engineering applications but alumina has high hardness and low fracture toughness, whereas silicon nitride ceramics have comparable hardness but very high in situ fracture toughness. Due to higher crack propagation resistance property of nitride ceramics, they are preferred over alumina ceramics in tribological application, specially fretting wear, where a relative cyclic motion between two surfaces, having a non-uniform distribution of local relative displacement at their

contact is involved [1,2]. It is reported that silicon nitride ceramics will perform better under fretting wear contact [3–7]. Fretting contact between femoral stem and ceramic femoral ball at the neck region is a potential biotribological application of this class of ceramics. Attempt has been made to use silicon nitride and silicon carbide class of non-oxide ceramics for total hip arthroplasty (THA) applications [8]. This class of materials have been found to be bio-compatible [9–13], but their derivative oxinitrides or SiAlON + SiC composites have not extensively been investigated for biotribological load bearing applications [14–16].

Moreover, the cost of synthesising phase pure silicon nitride ceramics is high due to low diffusion constant. Here comes the development of new class of ceramic alloy, SiAlON ceramics, where Si atoms are replaced by Al and N atoms are replaced by O atoms. SiAlONs have higher chemical resistance, higher oxidation resistance in compare to  $\text{Si}_3\text{N}_4$ . Presence of Al reduces the eutectic temperature by 200 °C, forming grain

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boundary glasses more easily and so SiAlONs are easy to sinter. They are having equivalent properties to that of pure silicon nitride ceramics [17–26].

Traditionally alumina ceramics are dominating the structural engineering market (more than 90%); whereas due to high fabrication cost of silicon nitride ceramics, although they perform better than alumina, their application is restricted [27]. But if we can reduce the development cost of nitride/oxinitride class of materials, they have the potential of replacing alumina ceramics in structural engineering applications. The aim of the study was the development of cost effective SiAlON ceramics production technologies to use these materials for biotribological applications. There are many different Si<sub>3</sub>N<sub>4</sub> starting powders available in the market. The prices of them vary from 4 to 6 USD/kg for poor quality refractory powders to over 100 Euro/kg for fine and pure quality powders. Starting compositions play an important role in determining the amount of replacement of  $m(\text{Si-N})$  bonds by  $m(\text{Al-N})$  bonds and  $n(\text{Si-N})$  bonds by  $n(\text{Al-O})$  bonds; and these two variants namely  $\alpha$ -SiAlON and  $\beta$ -SiAlON have quite distinct properties.

Microstructural characteristics of SiAlON ceramics are affected by the nature of starting Si<sub>3</sub>N<sub>4</sub> powder, sintering additives and obviously by processing conditions. Generally, two forms of silicon nitride exist as starting powder:  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> and  $\beta$ -Si<sub>3</sub>N<sub>4</sub>.  $\alpha$ -Phase has higher reactivity and is preferred over  $\beta$ -Si<sub>3</sub>N<sub>4</sub> as the former has solution re-precipitation tendency into forming elongated  $\beta$ -Si<sub>3</sub>N<sub>4</sub> morphology after  $\alpha \rightarrow \beta$  phase transformation (1410 °C) during sintering [28–30]. Whereas, elongated grain formation and densification are rather difficult, when  $\beta$ -Si<sub>3</sub>N<sub>4</sub> powder is used as starting powder [31]. Influence of sintering additive and sintering conditions on microstructural evolution, especially on grain growth have been studied in detail [32,33]. In situ composite microstructures with high fracture toughness can be obtained by gas-pressure sintering of Si<sub>3</sub>N<sub>4</sub> ceramics at higher temperatures (>1800 °C) or by heat treating the sintered sample at high temperatures [34–37]. Ekstrom et al. [38] used five different Si<sub>3</sub>N<sub>4</sub> powders, containing up to 15%  $\beta$ -Si<sub>3</sub>N<sub>4</sub> and Li et al. [39] used three different Si<sub>3</sub>N<sub>4</sub> starting powders with similar particle size, but different  $\alpha$ : $\beta$  phase ratios (100 $\alpha$ , 50 $\alpha$ :50 $\beta$  and 100 $\beta$ ). Rosenflanz [40] observed that coarse  $\beta$ -Si<sub>3</sub>N<sub>4</sub> powders ( $d_{50}$ :  $\sim 3 \mu\text{m}$ ) delayed transformation to  $\alpha$ -SiAlON. Acikbas et al. [41] evaluated the effect of using different starting powder with varying particle sizes on the microstructure and phase development as well as to correlate these with the mechanical properties. The starting  $\beta$ -Si<sub>3</sub>N<sub>4</sub> powder (Beijing Chanlian-Dacheng Trade Co., Ltd., China) was produced by combustion synthesis and consisted of 100%  $\beta$ -Si<sub>3</sub>N<sub>4</sub> phase. For comparative purpose, an  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> powder with about 2.1  $\mu\text{m}$  particle size (Grade S, HC-Starck) was also used by them. They

observed that marked differences in microstructures can be obtained by starting with different particle sizes of  $\beta$ -Si<sub>3</sub>N<sub>4</sub> powders. Initial particle size was of primary importance to achieve bimodal microstructure and hence, improved fracture toughness. A good combination of hardness of around 12 GPa and indentation toughness of 6.4 MPa m<sup>1/2</sup> could be obtained in ceramics sintered from finest ( $\leq 0.5 \mu\text{m}$ )  $\beta$ -Si<sub>3</sub>N<sub>4</sub> powders [7,41,42]. In the present study we continue to work on with the same starting  $\beta$ -Si<sub>3</sub>N<sub>4</sub> refractory powders (B1) and have compared this B1 with another SiAlON composition 50A, derived from purer source of UBE powders ( $\alpha$ -Si<sub>3</sub>N<sub>4</sub>). Both the starting Si<sub>3</sub>N<sub>4</sub> powders, namely B1 and 50A, were mixed with some SiC addition to enhance their properties. Here, we have tried to compare the fretting wear performance, cell adhesion behaviour and cytotoxicity of these compositions.

## 2. Materials and methods

### 2.1. Sample preparation

Two different varieties of Si<sub>3</sub>N<sub>4</sub> powders were used to prepare SiAlON ceramics. 100%  $\beta$ -Si<sub>3</sub>N<sub>4</sub> (B1) was used from refractory grade powders and another purer source of powder with 98%  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> (50A) was used to prepare the SiAlON samples. Since SiC + SiAlON composites reportedly perform better [14], batches were prepared with 15% SiC addition to refractory powders (B1) and 17.5% SiC was added to the other SiAlON composition (50A). These powders are having varying amount of impurities. Tables 1 and 2 describe the nomenclatures for starting Si<sub>3</sub>N<sub>4</sub> powders. As the as-received refractory powders (B) were having bigger grain sizes, attrition milling was done to reduce the grain sizes of the starting Si<sub>3</sub>N<sub>4</sub> powders to about 1  $\mu\text{m}$  level. Table 3 describes the nomenclature after attrition milling. The numbers in the nomenclature indicates average grain sizes.

Phase analysis was done using Cu K $\alpha$  radiation by Rigaku Rint 2000 X-ray device. Average  $\alpha$ - $\beta$  proportions are obtained from the intensities of (1 0 2) and (2 1 0) planes for  $\alpha$ -SiAlON, and for  $\beta$ -SiAlON, proportions are obtained from intensities of (1 0 1) and (2 1 0) according to the formula:

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

In this formula  $I_{\alpha}$  and  $I_{\beta}$  show the intensities of  $\alpha$ -SiAlON, and  $\beta$ -SiAlON,  $w_{\beta}$  is the weight fraction of  $\beta$ -SiAlON,  $K$  is the constant of [0.518  $\beta$  (1 0 1) –  $\alpha$  (1 0 2) and 0.544  $\beta$  (210) –  $\alpha$  (210)].

Pure SiC powder from Saint Gobin was added in varying amount (15%, 17.5%) to the UBE powders (50A) and to the

Table 1  
Powder suppliers specification.

Powder	Code	$\alpha$ : $\beta$ phase ratio	$d_{50}$ ( $\mu\text{m}$ ) as-received	Production method
UBE E10	50A	98 $\alpha$ :2 $\beta$	0.5	Diimide
Refractory powder	B1	100 $\beta$	4.7	Combustion synthesis

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