

## Fabrication and Properties of Nano-structured Ceramics

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### Abstract

*Nano-structured ceramics, which consist of structural elements with nanometer-size crystallites, are expected to show various unusual properties. We developed the novel nano-structured ceramics which consists of  $\text{Si}_3\text{N}_4$  and TiN and a self-lubricant material. The ceramics was fabricated by powder metallurgy process using mechano-chemical grinding process and short-time sintering process. Each grain size of matrix and the self-lubricant particle was under about 50 nm and a few nanometer. It showed high wear resistance and low friction coefficient by controlling of microstructure.*

**Keywords :** Non-oxide ceramics, mechano-chemical grinding, self-lubricant, low friction, high wear resistance

### 1. Introduction

The purpose of this work is the development of fundamental processing technologies for a wear resistant material under lubrication free condition.

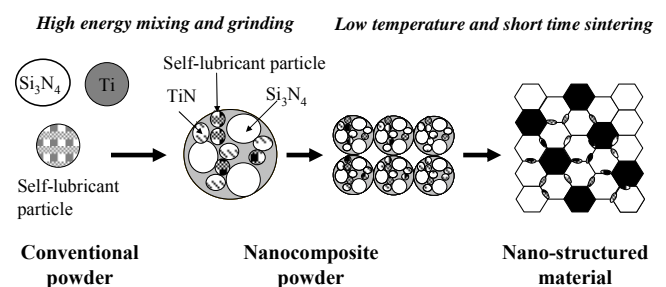
To achieve this purpose, we developed novel nano-structured  $\text{Si}_3\text{N}_4$ -based ceramics containing nano-sized electric conductivity particles and a self-lubricant particle. The obtained material showed high wear resistance and electric machinability, and also, demonstrated to its use as a precious deformation mold die.

### 2. Experimental Procedure and Results

Figure 1 shows the fabrication process of nano-structured ceramics called as mechano-chemical grinding (MCG) process. High purity  $\alpha\text{-Si}_3\text{N}_4$ , titanium and self-lubricant powder were used as the starting materials and they were mixed using a high-energy ball mill. The obtained powder mixture was sintered at 1300-1600 °C for 10 min. in vacuum atmosphere, using a pulse electric current sintering system (PECS) (1-3). Thus, the nano-structured material with  $\text{Si}_3\text{N}_4$ , TiN and a self-lubricant grain was fabricated. Commercial  $\text{Si}_3\text{N}_4$  microcomposites were fabricated using commercial  $\text{Si}_3\text{N}_4$  powder by hot-pressing.

The milled powder and sintered bodies were characterized using TEM and SEM. The material (nano-structured ceramics) was tested using a ball-on-disk arrangement in humid air at 25-28 °C, and polished  $\text{Si}_3\text{N}_4$  balls (AS6.0-01, diameter of 6.0 mm) were used as counterbodies.

Furthermore, we investigated that applicability of the material as the mold die for glass forming at high temperature, such as over 700 °C.



**Fig. 1. Mechano-chemical grinding (MCG) process.**

Figure 2 shows the microstructure of the nano-structured material and commercial  $\text{Si}_3\text{N}_4$ . The grain size of the material was below 50 nm, far less than commercial  $\text{Si}_3\text{N}_4$ .

The TEM photograph is shown in Fig. 3. The obtained material consisted of matrix grains with a size of less than 50 nm and nanometer size self-lubricant dispersions.

Figure 4 shows the friction coefficient of the obtained material and commercial  $\text{Si}_3\text{N}_4$ . The material showed a lower friction coefficient than commercial  $\text{Si}_3\text{N}_4$  due to the nano-sized self-lubricant particle. The specific wear rate of this material was  $6.0 \times 10^{-9}$  ( $\text{mm}^2/\text{N}$ ).

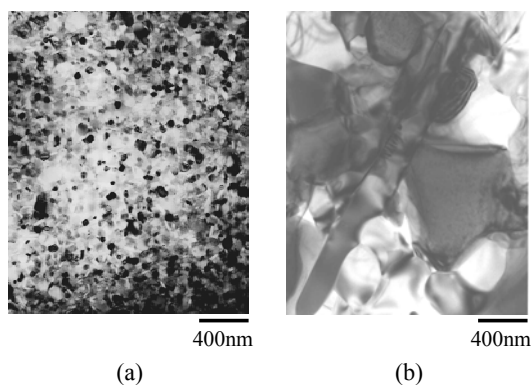


Fig. 2. TEM photograph of (a) nano-structured ceramics and (b) commercial  $\text{Si}_3\text{N}_4$ .

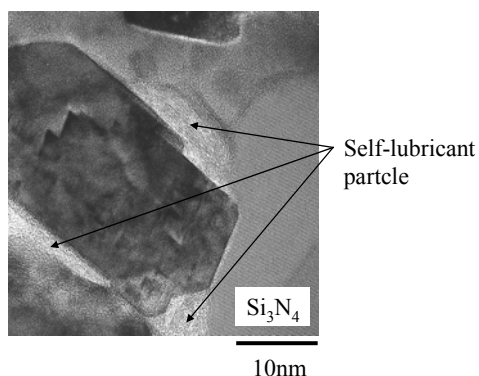


Fig. 3. TEM photograph of nano-structured ceramics.

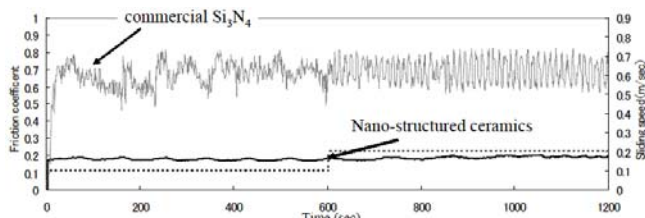


Fig. 4. Friction coefficient of Nano-structured ceramics and commercial  $\text{Si}_3\text{N}_4$ .

In order to apply nano-structured ceramics, a precious deformation mold die was fabricated by this material (4). Nano-structured ceramics die is shown in Fig. 6 and the glass parts produced by this die are shown in Fig. 7. The obtained glass parts had good transfer accuracy which can be applied for various products. Then, it was able to control the thermal expansion of nano-structured ceramics in controlling a mixture amount of TiN loading which have larger thermal expansion coefficient than  $\text{Si}_3\text{N}_4$ . It is important to control the thermal expansion between nano-structured material and glass parts to improve mold release characteristics.

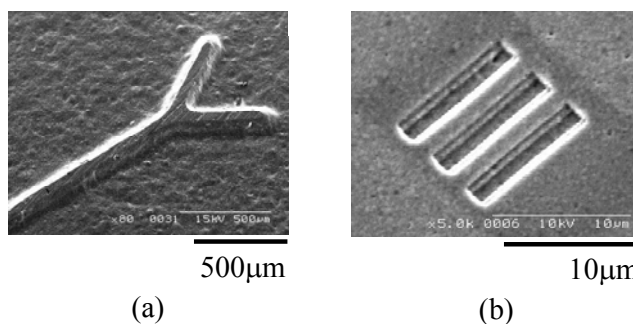


Fig. 6. SEM photograph of nano-structured ceramics die (a) emboss of Y-shape and (b) dent of rectangle-shape.

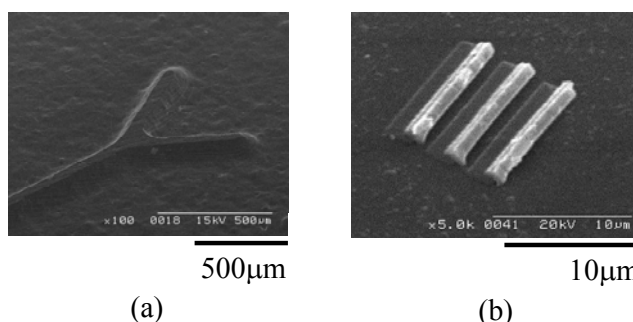


Fig. 7. SEM photograph of glass parts produced die shown in Fig. 6. (a) dent of Y-shape (b) emboss of rectangle-shape.

### 3. Summary

$\text{Si}_3\text{N}_4$ -based nano-structured ceramics with high wear resistance and machinability was successfully fabricated by the combination of the MCG method and short time sintering process. Powder compound was prepared using the MCG process and the obtained nanocomposite powder was consolidated using PECS. The obtained material was consisted of matrix grains with a size of less than 50 nm and nanometer size self-lubricant dispersions. It showed a lower friction coefficient and higher wear resistance than commercial  $\text{Si}_3\text{N}_4$  due to a fine microstructure and self-lubricant particle. The obtained material can also be used as a precious deformation mold die.

### 4. References

1. M.Yoshimura, J.Horea Powder Metallurgy Institute, 210, 8 (2001).
2. M.Yoshimura, O.Komura, A.Yamakawa, Proc. 2000 Powder Metallurgy World Congress, 1532-35 (2000).
3. M.Yoshiomura, O.Komura, A.Yamakawa, Scripta Materialia (2001).
4. M.Yoshimura, 8th International Symposium On Synergy Ceramics (2004).