OURNALOF

Ceramic Processing Research

Fabrication and properties of machinable SiC/h-BN Nano-composites

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SiC/BN ceramic nano-composites powders were synthesized by an in-situ recation method and the composites were sintered by hot pressing in a N_2 atmosphere. The microstructure, secondary phase and grain boundaries were characterized using a range of analytical techniques including TEM, SEM and XRD. The mechanical properties were determined, when the BN content is increased because the nano-sized h-BN crystals were homogeneously dispersed around the SiC grains of the matrix, the fracture strength of the nano-composites decreased more slowly than that of the SiC/h-BN micro-composites, but the hardness of the composites decreased sharply and the machinability properites were improved noticeably.

Key words: SiC; h-BN, Nano-composites, Mechanical properties, Microstructure.

Introduction

Silicon carbide (SiC) ceramics are recognized to be one group of materials with high temperature mechanical properties, excellent thermal, electronical and semiconductor characteristics and thus, they have been used in various fields. However, because SiC ceramics are difficult to machine, it also becomes difficult to process complex and precision components out of them. Therefore research on the machinability of SiC is very important. Hexagonal boron nitride (h-BN) has a crystal structure similar to that of graphite, it is a widely used ceramic material with excellent properties such as high thermal conduction, excellent machinability and high thermal shock resistance. There has been some research on improving the machinability of Si₃N₄, ZrO, Al₂O₃ and AlN etc. with different sized h-BN phas e [1-6]. Although the machinability of SiC ceramics can be improved by adding some µm h-BN, the mechanical properties decrease markedly, in particular the fracture strength decreases noticeably, which limits the use of SiC-BN ceramic composites in many fields.

In this study, SiC particles coated with nano-BN were synthesized by an in-situ reaction method, and the SiC/h-BN ceramic nano-composites with both excellent machinability and good mechanical properties were fabricated by hot pressing.

Experimental Procedures

Preparation of materials

The starting SiC powder for the monolithic and composites

were a commercially available α -SiC powder with a mean particle size of 1 µm. To obtain high-density bodies at lower temperatures, Al₂O₃ and Y₂O₃(5 wt%) were added into each samples as sintering aids.

For comparison, 3 series of specimens were prepared. The contents of BN and sintering aids $(Y_2O_3 \text{ and } Al_2O_3)$ of each series of specimen were as follows:

SC: Monolithic α -SiC ceramics.

SBn: α -SiC/ h-BN nano-composites. The contents of BN were 10 wt% (SBn10), 20 wt% (SBn20) and 30 wt% (SBn30). (The contents of BN of SBn were calculated contents)

SBm: α-SiC/ h-BN micro-composites. The contents of BN were 10 wt% (SBm10), 20 wt% (SBm20) and 30 wt% (SBm30).

The SC and SBm series specimens were obtained by mechanically by milling a mixture of powders of SiC(1 μ m) and BN, and sintered in a hot preaaing system (Multi5000, Fuji Denpa Ltd., Japan) at 1,850 °C for 1 hour under a uniaxial pressure of 30 MPa in a nitrogen atmosphere.

For the SBn series, boric acid (H₃BO₃, analytically pure), and urea $(CO(NH_2)_2)$ $(H_3BO_3 : CO(NH_2)_2 = 1:3 \text{ mol})$ were selected to produce the h-BN precursor coating film on the α -SiC powder. The α -SiC powder, H₃BO₃, and CO(NH₂)₂ were mixed by ball milling to ensure homogeneity of the mixture, and dried. The dried mixtures were put into a graphite-heater furnace and kept at 850 °C (temperature ramp: 2-5 K.minute⁻¹) for 21 hours in a nitrogen atmosphere, and capsule-like t-BN-coated α -SiC was produced. Then the temperature was raised to 1,100 °C and held at this temperature for 6 hours for the BN phase transformation. Al₂O₃ and $Y_2O_3(5 \text{ wt\%})$ were added into each sample as a sintering aid. The mixtures were put into a graphite mould and sintered in a hot pressing system at 1,850 °C with 1 hour holding time under a uniaxial pressure of 30 MPa in a nitrogen atmosphere

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at a pressure of 0.93 MPa. [7, 8]

3 series specimens were obtained by milling and polishing to $\Phi 40 \times 4$ mm wafers.

Observation of microstructures and measurement of properties

The crystalline phases of the composite powders and hot pressed specimens were identified by X-ray diffraction (XRD) analysis. The microstructures were characterized by a scanning electron microscope (SEM, Model JSM 6460, JEOL Co. Ltd., Tokyo, Japan), a transmission electron microscope (TEM, Model JEM 200CV, JEOL Co. Ltd., Tokyo, Japan) and high resolution transmission electron microscopy (HRTEM, Model JEM 2110F, JEOL Co. Ltd., Tokyo, Japan).

The density (ρ) was measured by the Archimedes immersion technique in distilled water.

The bending strengths (σ) were determined by three-point bend tests (sample size 3 mm(b) × 4 mm(h) × 30 mm(l) at room temperature; span 20 mm; load speed 0.5 mm · minute⁻¹) in a NSTRON-1195, with the tensile surface of the specimens perpendicular to the pressing direction of the PAS. All surfaces of specimens were finally ground on an 800 grit diamond wheel, and the edges were beveled.

The Vickers (H_V) hardness was measured by the Vickers indentation method.

The drilling velocity (V) was calculated using the following equation:

$$V = \frac{H}{t} \tag{1}$$

Here, H is the drilling depth, and t is the drilling time. Each test was done five times under the same conditions; the drilling velocity was given by the average value.

Results and Discussion

Phase compositions and microstructures

Fig. 1 shows the XRD pattern for the original mixture of SBn20 (SiC, boric acid and urea), 20 wt.% BN-coated SiC powder synthesized at 850 °C and the composite with an 1,850 °C hot pressing. Peaks of SiC and weak/ wide peaks arising from amorphous BN (abound 26°) are seen. Fig. 1 also shows that the amorphous BN is transformed to h-BN after hot pressing, and the sintering aids at the boundary of the SiC grains changes to a glass phase $Y_3Al_5O_{12}$ under the reaction of Al_2O_3 and Y_2O_3 . This of course promotes the sintering process [6].

Fig. 2 shows a TEM micrograph and the SAD pattern of the BN-coated SiC powder. It shows that the SiC grains are coated with nano-sized BN that has been created from boric acid (H_3BO_3) and urea $(CO(NH_2)_2)$. It also shows the SAD pattern of area (A): the pattern is a set of concentric circles which means amorphous BN.

Fig. 3(a) shows the TEM micrographs of the BN coating, it



Fig. 1. XRD Pattern for 20 wt% BN-coated SiC powder and composite.



Fig. 2. TEM Micrograph of 20 wt% BN Nano-composite powder.

is seen that the growth of SiC particle is restrained by the BN coating. Fig. 3(b) shows an HRTEM micrograph of a lattice image of neighboring grain in 30 wt% h-BN nano-composite (SBn30), it is seen that there are SiC and BN ((002) crystal face, interplanardistance: 0.33 nm) and YAG crystals on the grain boundary.

Fig. 4(a) and (b) show the microstructure of fractures surfaces of a 30 wt.% h-BN nano-composite (SBn30) and a 30 wt% h-BN micro-composite (SBm30). For the series SBn, due to the homogeneous dispersion of pin-leaf-like nano-sized h-BN at SiC grain boundaries, the growth of SiC grains and the BN crystals are resisted, they grow very slowly, so smaller SiC grains coated with the nano-sized BN crystals can be observed in Fig. 4(a). For the series SBm, larger SiC grains and larger pin-leaf-like micro-sized h-BN particles are observed in Fig. 4(b).

Properties

Table 1 shows the density, bending strength, Vicker



Fig. 3. TEM Micrographs of 30 wt% BN Nano-composite BN coating; (b) HRTEM micrograph of a grain boundary.



Fig. 4. SEM Micrographs of fracture surfaces of composites (a)30 wt% BN Nano-composite; (b) 30 wt% BN micro-composite.

Hardness and drilling speed of 3 series. The densities of the micro-composites (series SBm) decrease with an increase in the h-BN content, on the other hand, for the nano-composites (series SBn), there is no large variety of the density when the BN content is less than 10 wt%, and the densities also

Table 1. Properties of SIC/n-BIN composites				
No.	$\rho/g \cdot cm^{-3}$	σ_b/MPa	Hv/ GPa	$\frac{V_{drill}}{mm \cdot minute^{-1}}$
SC	3.24	720	24.33	0.0
SBm10	3.12	450.48	14.19	0.0
SBm20	2.95	390.93	7.88	6.3
SBm30	2.8	275.59	5.43	10.2
SBn10	3.11	650.35	13.91	0.0
SBn20	2.95	550.15	6.71	9.7
SBn30	2.82	426.4	4.95	11.5

 Table 1. Properties of SiC/h-BN composites

decrease with an increase in the h-BN content when it is larger than 20 wt%.

The table also shows that both the bending strength and the Vickers hardness decrease with an increase in the h-BN content. But for the nano-composites (series SBn), the Vickers hardness decreases more rapidly than does the bending strength. The strength of h-BN is very low, and so the bending strength of SiC/h-BN micro composites should decrease rapidly. However, for the SiC/h-BN nano-composites, the nano-sized h-BN crystals are homogeneously dispersed around the matrix grains of SiC during sintering, the SiC grain growth is resisted, and the matrix grain size remains small. According to the Hall-Petch Equation ($\sigma = \sigma_0 + kd^{-1/2}$), the smaller the grain size the higher fracture strength and therefore, the bending strength of the nano-composites decreases more slowly than it does for the micro-composites.

The drilling velocity of a machine tool acting on the specimens also increases with an increase in the h-BN content. The explanation is that h-BN has a layered structure like that of graphite. The shear force caused by the machine tool deforms the h-BN particles coating the SiC matrix, separates them from the boundaries of the matrix, and causes splits along the h-BN layers. As a result, the SiC/h-BN composites are easier to machine. For the SiC/ BN nano-composites, because nano-sized h-BN crystals are homogeneously dispersed around the SiC grains of the matrix, either the specific surface area of the h-BN weak phase is larger or the total surface area of the weak phases is larger, and so the machinability of the SiC/nano-sized h-BN composites is improved more noticeably than that of the SiC/h-BN micro-composites.

Conclusions

Core shell structure particles with micrometre sized SiC particles coated with nanometre sized h-BN particles were synthesized at 850 °C in a nitrogen atmosphere by an in-situ recation method. Machinable SiC/h-BN nano-composites were fabricated by hot pressing.

The density and the bending strength of SiC/h-BN nanocomposites decrease with an increase in the BN content, but the bending strength of SiC/h-BN nano-composites decreases more slowly than that of the micro-composites. With an increase in the h-BN content, the hardness of both microcomposites and nano-composites decreased enormously, and the machinability of the composites also improved noticeably. Therefore machinable SiC/h-BN nano-composites have both excellent machinability and good mechanical properties.

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