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Piezoresistance of pressureless sintered silicon carbide

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Silicon carbide ceramics has been prepared by pressureless sintering with 6 wt% Al_2O_3 and 4 wt% Y_2O_3 as sintering aids. When the compact body was sintered at 1950 °C for 5 min, almost all the β phase transformed to α phase and the resultant sintered body had relative density of 95%. The pressureless sintered SiC clearly exhibited a piezoresistance effect, while considerable amount of the second phase formed. The piezoresistance coefficient of the pressureless sintered SiC was practically equivalent to that of the hot pressed SiC.

Key words: SiC ceramics, pressureless sintering, piezoresistance effect, fracture foreseeing

1. INTRODUTION

Ceramics is said to have a potential to be used in severe condition such as high temperature and corrosive environment. However, before it is used as mechanical part, reliability improvement is necessary, which is usually accomplished by improving the mechanical strength and by narrowing the strength distribution. On the other hand, to maintain the reliability it is desired to foresee a catastrophic fracture of a ceramic body during the operation, which should be based on the precise detection of fracture controlling flaw and strain. For these purposes, electric response could be utilized.

This paper deals with silicon carbide (SiC) ceramics, which has been studied as a potentially important structural ceramics due to its excellent high temperature strength, thermal shock resistance, high wear resistance, high resistance to oxidation as well as corrosion and high thermal conductivity. Moreover, it attracts much attention as electronic material, because it possesses a wide band gap of about 2~3eV and high electronic mobility. Furthermore, from the viewpoint of succession to silicon technology, SiC is thought to be easily applied for the high temperature electronic devise.

In the meanwhile, it is reported that the single crystal of SiC exhibits the piezoresistance effect [1] or linear increase in electric resistance proportional to the applied tensile strain. We have already reported a piezoresistance effect on commercial SiC ceramics fabricated by hot press and a real time monitoring of applied strain [2,3]. A fracture foreseeing system is proposed for SiC ceramics in which initial crack monitoring without stress application and bending strain monitoring utilizing the piezoresistance effect are combined[2].

It is well known that SiC ceramics is a difficult to sinter because of the covalent nature of its chemical bond. In recent studies on the sintering of SiC, suitable sintering aids have been searched and several methods have been tried, pressureless sintering [4, 5], hot pressing, hot isostatic pressing and reaction sintering. Above all, pressureless sintering is advantageous because it does not need a huge pressing equipment and can fabricate complicated ceramics parts. However, its pressureless sintering usually employs huge amount of sintering aids and the influence of such sintering aids on the piezoresistance effect of SiC ceramics has not been examined. The objectives of this study are to fabricate a SiC ceramics by pressureless sintering with Al_2O_3-Y_2O_3 as sintering aids [4] and to evaluate its piezoresistance effect.

2. EXPERIMENTS

Submicrometer size powders of α-SiC (Grade-UF, Ibiden Co., Ltd., Nagoya, Japan), with a mean particle size of 0.29 μm and the specific surface areas of 19.9 m^2 / g, Al_2O_3 (AKP-50, Sumitomo Chemicals, Tokyo, Japan) and Y_2O_3 (High-Purity Chemical Co., Japan; 99.99 % pure) were used as starting powders. The powder mixtures of SiC containing 6 wt% Al_2O_3 and 4 wt% Y_2O_3 as sintering aids were ball-milled in ethanol with zirconia balls for 6 hours. The milled slurry was dried and subsequently sieved through a 75 μm mesh. This powder was uniaxially pressed in a ϕ10 mm die under a pressure of 30 MPa. This treatment was followed by cold isostatic pressing under 300 MPa, using evacuated polyethylene bags to obtain pellets with a green density of near 65 % theoretical. The theoretical density was estimated on the basis of the rule of mixtures. The pellets were sintered at several temperatures (1700 °C to 1950 °C) for 5 min in zirconia furnace under a flow of Ar gas at ambient pressure. Heating was performed at a rate of 2.0 $°Cmin^{-1}$ from room temperature to the sintering temperature and thereafter cooled at the rate of 2.0 $°Cmin^{-1}$ to 1200 °C then 1.5 $°Cmin^{-1}$ to room temperature. The bulk densities of the sintered specimens were measured by the Archimedes method using deionized water as the immersion medium. The phases of the sintered SiC were analyzed by X-ray diffraction (XRD) with CuKα radiation, and the SiC polytypes were analyzed according to the method of Ruska et al [6]. The fracture toughness was estimated by measuring crack length generated by a Vickers indenter with 196N.

The sintered bodies were cut into rectangular bars of 3.8 × 3.8 × 2.2 mm^3 with precision diamond cutter, followed by surface polishing with #2000-SiC abrasive paper. Resultant test pieces were subjected to measurement of the effect of applied stress on the electronic resistance of SiC ceramics. The electronic resistance was measured by a two-probe direct-current (dc) method using a digital high resistance meter (Model R8340A, Advantest Co., Ltd., Tokyo, Japan) with a constant voltage (0.5 V) supply. Silver paste was attached on two of the parallel 3.8 × 2.2 mm^2 planes to form electrodes. Such electrodes test pieces were placed on the mechanical test machine (Model EZTest-500N, Shimadzu Co., Ltd., Kyoto, Japan), and the compressive stress was increased at a constant rate parallel to the 3.8 × 3.8 mm^2 planes. During this process, an electric current change corresponding to the stress perpendicular to the electric field was measured. From the effect of compressive stress perpendicular to the electric field on electric current change, piezoresistance coefficient with perpendicular stress application was calculated. A commercial α-SiC ceramics (JIS; Japanese Industrial Standard, Nihon Gaishi, Co., Ltd., Japan) was employed to compare the piezoresistance coefficient with the pressureless sintered SiC fabricated in the present study.

3. RESUITS and DISCUSSIONS

The characteristics of both the commercial and the fabricated SiC are summarized in Table I. No significant difference can be seen in hardness and fracture toughness between the two kinds of samples. Figure 1 illustrates the X-ray diffraction patterns of β-SiC with sintering aids of 6 wt% Al_2O_3 and 4 wt% Y_2O_3 sintered at 1900 °C (a) and 1950 °C (b) for 5 min. The phase transformation from β to α is observed between 1900 °C and 1950 °C. In 1900 °C sintered sample, there still existed a substantial fraction of β-SiC. But when the powder compacts of β-SiC was sintered at 1950 °C, most of β-SiC transformed to α-SiC which probably consists of several polytypes such as 4H, 6H and 15R. In the latter case, the volume fraction of α-SiC were calculated to be about 90 % based on the method proposed by Ruska[6]. It is widely known that the transformation temperature of pure SiC from β to α is above 2200 °C, but it decreases lower than 1950 °C in the pressureless sintered SiC and this facilitated densification process is thought to be a result of the liquid phase which is said to consist of Al_2O_3 and Y_2O_3 with this composition[4]. It is assumed that sintering aids give effect to decrease the transformation temperature.

The time-electric current and time-strain curves, which is obtained on the present pressureless sintered SiC, is illustrated in Fig. 2. The electric current and strain increase simultaneously. In addition, the piezoresistance effect is apparent and reproducible in this specimen. From this experimental result piezoresistance coefficient perpendicular to strain is calculated to be 9.8×10^{-9} (cm^2 / dyne), whereas similarly calculated the piezoresistance coefficient of commercial hot pressed SiC is $5.24\times10^{-9}cm^2$/ dyne.

When the thermal electromotive force was measured in the pressureless sintered SiC containing 6 wt% Al_2O_3 and 4 wt% Y_2O_3 as sintering aids, the higher temperature side showed positive, meaning the positive semiconductor. It is the reason why this sample exhibits the property of negative semiconductor that added aluminum and yttrium are tivarent cations, so that these sintering aids form the acceptor levels.

Table I Characteristics of the commercial and fabricated SiC

	Hardness (GPa)	Fracture Toughness ($MPam^{1/2}$)	Phases Major	Phases Minor
Commercial-SiC	22	2.7	α-SiC ′	
Fabricated-SiC¶	20	2.8	α-SiC	YAG§, Y_2O_3

¶ Sintering aids : Al_2O_3:Y_2O_3=6:4 in weight ratio
§ $Al_5Y_3O_{12}$(yttrium aluminum garnet)

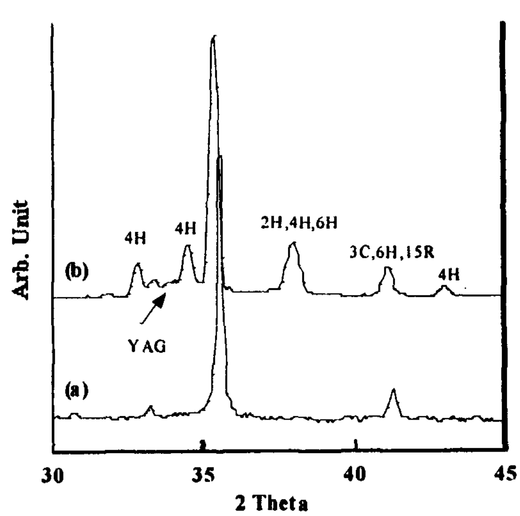

Fig. 1 X-ray diffraction patterns of SiC samples with sintering aids (Al_2O_3+Y_2O_3), which were sintered at 1900 °C (a) and 1950 °C (b)

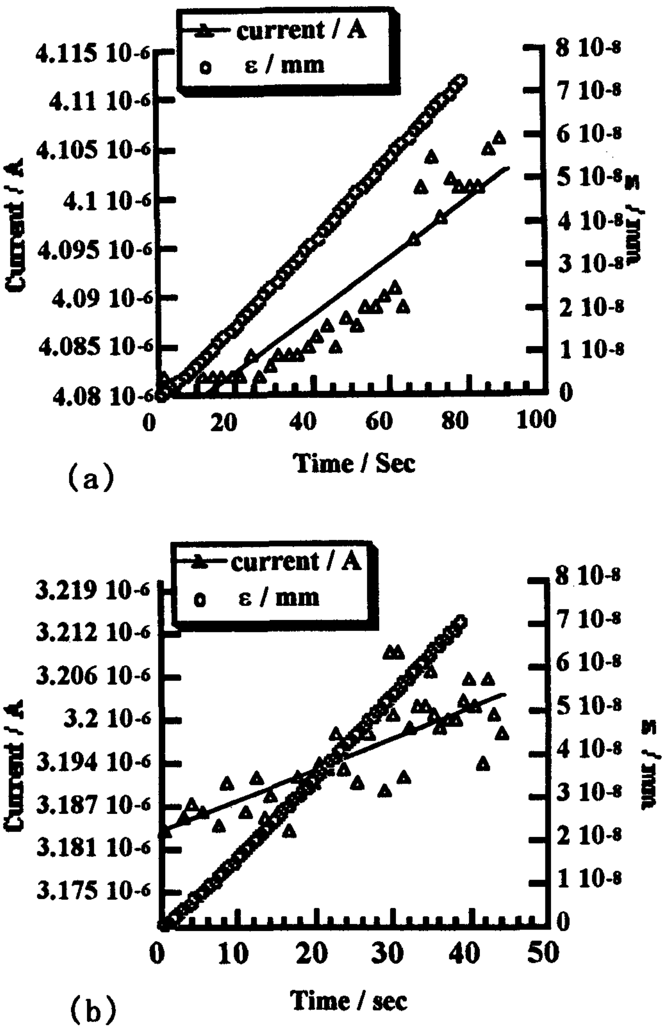

Fig.2 Current, strain-time curve under compressive stress for the commercial (a) and fabricated (b) SiC

The piezoresistive coefficient of pressureless sintered specimen in this paper is equivalent to that of commercial hot-pressed specimen, which is thought to be due to the formation of acceptor levels analogous to the former added with only Al_2O_3. At present, details are under investigation.

4. CONCULUSIONS

It has been shown that the SiC ceramics fabricated by the pressureless sintering can be sintered at lower temperature than by solid phase sintering. The piezoresistance effect is examined for the pressureless sintered SiC which has more seconds phases than a hot pressed SiC ceramics. That is, the pressureless sintered SiC which can fabricate a complicated structural ceramics exhibits the piezoresistance effect to applied strain, so that, the SiC ceramics has a superior property not only as structural ceramics but also to sense a strain itself.

5. REFERENCES

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