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Mechanical properties of electrically conductive silicon carbide ceramics

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Abstract

The mechanical properties of electrically conductive SiC ceramics sintered with 2 vol% Y_2O_3 were characterized. A submicron SiC powder and a mixture of submicron SiC powder and polycarbosilane (PCS) were hot-pressed with 2 vol% Y_2O_3 at 2000 °C for 6 h under an applied pressure of 40 MPa in a nitrogen atmosphere. The SiC without PCS had a finer microstructure consisting of equiaxed grains, whereas the SiC with PCS had a coarser microstructure with equiaxed grains. The coarser microstructure was beneficial in lowering the electrical resistivity but resulted in deteriorated mechanical properties. The typical flexural strength, micro-hardness, fracture toughness, and electrical resistivity values of the SiC ceramics without PCS were 678 MPa, 30 GPa, 5.2 MPa m$^{1/2}$, and 7.8×10^{-2} Ω cm at room temperature, respectively.

Keywords: B. Microstructure-final; C. Mechanical properties; C. Electrical properties; D. SiC

1. Introduction

Silicon carbide (SiC) is one of the most promising structural materials because of the unique combination of its good mechanical properties at room and high temperatures, high thermal conductivity, good chemical inertness, and excellent wear and corrosion resistances [1–10]. Thus, SiC ceramics are used in many industrial applications such as fusion reactor parts, turbine components, hot-gas filters, optical mirrors, structural parts, diesel particulate filters, and components for semiconductor processing equipment [11–14]. Recently, highly electrically conductive polycrystalline SiC ceramics sintered with AlN–RE_2O_3 (RE=Sc, Nd, Dy, Gd, Ho, Er) [15], yttrium nitrate tetrahydrate ($Y(NO_3)_3 \cdot 4H_2O$) [16], or Y_2O_3 [17] as sintering additives have been reported. The reported electrical resistivity of the SiC ceramics was 10^{-2} Ω cm for SiC ceramics sintered with AlN–RE_2O_3 (RE = Sc, Nd, Dy, Gd, Ho, Er) [15], 10^{-3} Ω cm for SiC ceramics sintered with yttrium nitrate [16], and 10^{-3} Ω cm for SiC ceramics sintered with Y_2O_3 [17]. In these electrically conductive SiC ceramics, the high electrical conductivity has been achieved by N-doping into SiC grains as a result of the grain growth of SiC via the solution–reprecipitation mechanism during sintering [15–18]. Electrically conductive polycrystalline SiC can be processed by electrodischarge machining [19], which is a cost-effective process that enables machining of complex parts with high hardness values.

Typical flexural strength values of the pressureless sintered SiC ceramics, gas pressure sintered SiC ceramics, and hot-pressed SiC ceramics were 320–470 MPa [20,21], 450–550 MPa [22,23], and 550–720 MPa [24–26], respectively.

Pre-ceramic polymers as a precursor of SiC have attracted considerable attention due to the expected improvement of the properties of fine-grained materials and the potential of cost-effective polymer processing [27–29]. Furthermore, a higher affinity of nitrogen to precursor-derived nano-sized SiC powders than submicron-sized SiC powders was reported [18]. The effect of polycarbosilane (PCS) addition on the electrical properties of hot-pressed SiC ceramics has not been reported and the mechanical properties of electrically conductive SiC ceramics have not been investigated to date.

In this study, PCS-derived nano-sized SiC was introduced into submicron-sized SiC powders to examine its effect on the electrical and mechanical properties of SiC ceramics sintered

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with 2 vol% Y_2O_3. SiC ceramic without the addition of PCS was also fabricated with 2 vol% Y_2O_3 as a baseline material and its electrical and mechanical properties were characterized.

2. Experimental procedure

To prepare the powder mixture without PCS (designated as SC), a mixture of 97.020 wt% SiC (~0.5 μm, Grade BF-17, H. C. Starck, Berlin, Germany) and 2.980 wt% Y_2O_3 (99.99% pure, Kojundo Chemical Lab Co. Ltd., Sakado-shi, Japan) was ball milled using SiC balls in a polypropylene jar containing ethanol for 24 h. To prepare the powder mixture containing PCS-derived nano-sized SiC (designated as SC-PCS), a mixture of 69.367 wt% SiC, 27.970 wt% PCS (Type S, Nippon Carbon Co. Ltd., Yokohama, Japan), and 2.663 wt% Y_2O_3 was ball milled using the same balls and a jar containing ethanol for 24 h. The yield of the nano-sized SiC from PCS was 62 wt% [30]. The submicron-sized SiC:PCS-derived nano-sized SiC ratio was 8:2 by weight. The mixture without PCS was dried, sieved (60 mesh), pressed uniaxially, and hot-pressed at 2000 °C for 6 h under 40 MPa in a nitrogen atmosphere. The mixture containing PCS was dried, sieved, pressed uniaxially, and heat-treated at 200 °C for 30 min in air to cross-link the PCS in the mixture. The compact was heat-treated at 1000 °C for 1 h to pyrolyze PCS and subsequently hot-pressed at 2000 °C for 6 h under 40 MPa in a nitrogen atmosphere.

The densities of the hot-pressed specimens were determined using the Archimedes method. X-ray diffraction (XRD, D8 Discover, Bruker AXS GmbH, Karlsruhe, Germany) using CuKα radiation was performed on the hot-pressed specimens. The hot-pressed specimens were polished and etched by CF_4 plasma containing 10% oxygen. The etched microstructure and fracture surface were observed by scanning electron microscopy (SEM, S4300, Hitachi Ltd., Hitachi, Japan).

The hot-pressed specimens were cut into $3 \times 2.5 \times 25$ mm^3 bars whose surfaces and edges were polished with an 800-grit diamond wheel for the strength measurements. Bend testing was performed at room temperature on seven specimens for each condition using a four-point method with inner and outer spans of 10 and 20 mm, respectively. The specimens were loaded at a constant speed of 0.5 mm/min. The hardness of the polished specimens was measured by Vickers indentation testing with a 9.8 N load and a dwell time of 10 s. The fracture toughness was estimated by measuring the crack lengths that were generated by the Vickers indenter with a load of 98 N and a dwell time of 10 s.

Hall effect measurements were performed on the specimens (10 mm × 10 mm × 1 mm) at room temperature using the van der Pauw technique. The electrical resistivity of the specimens was calculated from the carrier density and carrier mobility obtained from the Hall effect measurements.

3. Results and discussion

After hot-pressing, relative densities of 98.9% and 99.7% were achieved for the SiC ceramics without and with PCS, respectively. The addition of 20 wt% PCS-derived nano-sized SiC enhanced the densification of SiC when a small amount of additive (2 vol% Y_2O_3) was added. The enhanced densification in the SC-PCS specimen is due to the enhanced driving force of the powder compacts due to the high surface area of nano-sized SiC [18]. The crystallite size of the PCS-derived SiC powder ranges from 2 to 100 nm, depending on the pyrolysis temperature [27,30].

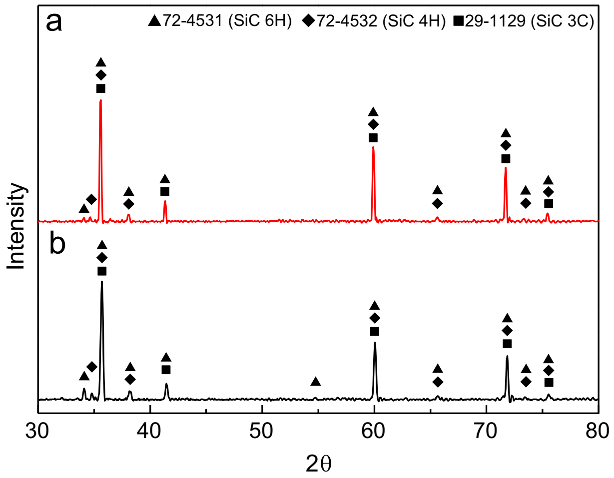

Fig. 1. X-ray diffraction patterns of the SiC ceramics sintered with 2 vol% Y_2O_3: (a) SiC without polycarbosilane (SC) and (b) SiC with polycarbosilane (SC-PCS).

In Fig. 1, the XRD analysis reveals the presence of β-SiC (3C) as a major phase and α-SiC (6H and 4H) as a trace phase in both specimens. The starting SiC powder (BF-17) consisted of 3C (β-SiC) as a major phase and 6H (α-SiC) as a minor phase [31]. During hot-pressing, the Y_2O_3 additive reacted with SiO_2, which is always present on the surface of the SiC particles, to form a Y–Si oxide melt and, with increasing temperature, a Y–Si oxycarbonitride (OCN) melt by the dissolution of SiC and nitrogen present in the sintering atmosphere. The liquid phase is responsible for the densification of the specimens via liquid-phase sintering. It is well documented that oxynitride or oxycarbonitride glasses stabilize β-SiC [16] or suppress the $\beta \rightarrow \alpha$ phase transformation in SiC at high temperatures [32]. Thus, the major phase of both specimens is β-SiC, although they were hot-pressed at 2050 °C for 6 h.

Fig. 2 shows the typical microstructures of the hot-pressed SiC specimens. Some grains showed a core–rim structure, indicating that the grain growth took place by solution–reprecipitation. Both specimens consisted of equiaxed grains where the grain sizes of the SC and SC-PCS specimens were 1.8 μm and 4.1 μm, respectively. The larger grain size of the SC-PCS specimen compared to the SC specimen suggests that the addition of PCS enhanced the grain growth and densification of the submicron-sized SiC powders. The enhanced grain growth is caused by the enhanced driving force for the solution–reprecipitation of SiC grains due to the larger difference of the particle sizes between the PCS-derived nano-sized SiC and submicron-sized SiC particles [33].

The indentation toughness of the SC and SC-PCS specimens were 5.2 ± 0.2 MPa m$^{1/2}$ and 4.5 ± 0.4 MPa m$^{1/2}$, respectively. The improved toughness of the SC specimen is due to the increased tendency of intergranular fracture in the specimen