



Material properties of tungsten carbide–alumina composites fabricated by spark plasma sintering

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Abstract

The densification behavior of WC–Al₂O₃ composites prepared via spark plasma sintering (SPS) was investigated. The initial materials were fabricated using a metal-organic chemical vapor deposition process in which W(CO)₆ was used as a precursor and Al₂O₃ powder was used as the matrix in a spouted bed. The decomposed W(CO)₆ produced W species that coated Al₂O₃. Then, carburization, using a mixture of CH₄–H₂ gas, was used to form tungsten carbide–alumina composite powder. This powder was sintered via SPS in the temperature range of 1200–1350 °C, which produced several secondary phases, namely W, WC, and W₂C. At the highest sintering temperature (1350 °C), the intermediate phase WC decomposed to form W₂C. The material properties of the SPS-treated samples, including density, hardness, and electrical resistivity, were investigated. The hardness and electrical resistivity of WC–Al₂O₃ composites were found to be approximately 22.4 GPa and 4.9 × 10⁹ Ω cm, respectively.

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1. Introduction

Alumina (Al₂O₃) is an important structural ceramic material that is widely used in industry for various applications due to its excellent material properties, such as high mechanical performance, good chemical stability, and high temperature characteristics (e.g., high temperature resistance). However, its intrinsic brittleness and relatively poor reliability make the toughening of alumina ceramics important and challenging [1]. Researchers have adopted several approaches, such as sol–gel [2] and chemical vapor deposition (CVD) [3], to obtain a secondary phase coating on the Al₂O₃ matrix to improve its toughness. CVD, in which metal-organic precursors can be

used as the source of a secondary phase, is capable of producing a uniform coating on the matrix [3].

Tungsten carbide is an ideal secondary phase candidate due to its high melting point and high fracture toughness, which are helpful for mechanical and tribological applications [4–6]. Research on metal-organic compounds has revealed that when a W precursor is used in the CVD process, the deposited W species contain carbon and oxygen [7]. However, the carbon content is insufficient to transform W into pure tungsten carbide [7]. Carburization treatment is thus commonly employed to transform W into WC. Two carburization methods are generally used to obtain tungsten carbide: (1) the addition of carbon to tungsten followed by sintering at high temperature and (2) annealing W in the presence of a methane atmosphere [8]. Annealing W under a methane atmosphere is preferred, as the powder grain size remains constant and no agglomeration occurs during the process.

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Spark plasma sintering (SPS) is a novel method that enables shorter sintering durations and relatively high sintering pressure. This process utilizes a heating rate of a few hundred °C/min, much higher than that generally used in conventional hot press sintering (10 °C/min). SPS has been found to be suitable for consolidating ceramic materials. In recent years, WC-containing composites, such as WC–Co [9], WC–ZrO₂ [10], WC–TiC–Cr₃C₂ [11], and WC–Si₃N₄ [12], have been consolidated via SPS.

In the present work, W(CO)₆ is used as a precursor in the metal-organic chemical vapor deposition (MOCVD) process. During pyrolysis, this precursor decomposes and coats the alumina matrix in a spouted bed. The W-species-Al₂O₃ composite powders are carbonized in a CH₄–H₂ mixed gas to obtain WC–Al₂O₃ composites. Finally, the WC–Al₂O₃ composites are densified via SPS. The microstructure evolution and the mechanical properties of the SPS-treated disks are investigated in detail.

2. Experimental details

2.1. Preparation of composite powder

α-Alumina (α-Al₂O₃, 99.99%, Taimei Chemicals Co. Ltd., Japan) with an average grain size of 150 nm was used as the matrix powder in this study. Tungsten hexacarbonyl (W(CO)₆, 99%, Acros Organics Co., USA) was used as the precursor of tungsten carbide in the MOCVD process. The precursor vapor was carried by He gas (99.9% pure), which was introduced into the spouted bed reactor for the MOCVD process. The reactor vacuum was maintained at 1.3 kPa. The precursor container was kept at 90 °C to vaporize the precursor. The vaporized tungsten hexacarbonyl was decomposed in a low-temperature chamber (300 °C). The decomposed species were then deposited on the spouted alumina powder. The deposition time was fixed at 60 min. After decomposition, the composite powder was treated under a CH₄–H₂ (1:9) atmosphere at 800 °C for 1 h to obtain pure WC–Al₂O₃. The WC–Al₂O₃ composites were analyzed by X-ray diffraction (XRD; Rigaku MultiFlex 2 kW, Japan) and transmission electron microscopy (TEM, JEOL JEM-2100F CS STEM, Japan) equipped with energy-dispersive X-ray spectroscopy (EDS) to identify the phase composition and surface morphology.

2.2. Densification

The WC–Al₂O₃ composite powders were densified via SPS (SPS-515S, Sumitomo, Japan). During SPS, a pulsed direct-current of up to 1500 A was provided by the power supply at a voltage of up to 20 V through the electrodes to the tool set-up that contained the specimen. A pulse-current of 12/2 ms (on/off time) was used for all of the specimens. In this process, 2.1 g of each powder was placed in a graphite mold with a 15.5-mm diameter, and a uniaxial pressure of 50 MPa was imposed on the powder. The pressure of the vacuum was maintained at 6 Pa. The specimens were heated at a heating rate of 200 °C/min from room temperature to 600 °C and at

100 °C/min from 600 °C to the desired temperature (1200–1350 °C), followed by a holding time of 10 min at the desired temperature. All of the specimens were smoothly polished using a diamond paste, ultrasonically cleaned, and finally thermally etched in an Ar atmosphere at 1150 °C with a 15-min holding time.

2.3. Characterization

The density of all of the specimens was measured in water using Archimedes' principle. The phases of the composites were analyzed by XRD. The microstructures and composition of the specimens were analyzed by an ultra-high-resolution scanning electron microscope (UHR-SEM, Carl Zeiss Microscopy, LLC North America) equipped with an EDS detector. Microhardness tests were carried out using a microVickers tester (MXT-70, Matsuzawa Seiki. Ltd., Japan) under an applied 500-g load with an indentation time of 10 s. The magnitude of the Vickers hardness was determined as follows:

$$Hv = 1.854 \frac{P}{d^2} \quad (1)$$

where p is the applied load (in N) and d is the diagonal length (in m). A semiconductor parameter analyzer (Hewlett Packard 4140B, USA) was used for resistance measurements to determine the electrical resistivity of the specimens.

3. Results and discussion

3.1. Density and microstructure evolution of SPS-treated composites

TEM micrographs of the WC/Al₂O₃ nanocomposite powder are shown in Fig. 1(a). Nanoparticles with a size of approximately 5–10 nm are well dispersed on the Al₂O₃ matrix. Fig. 1(b) shows the XRD pattern of the WC/Al₂O₃ nanocomposite powder, indicating the presence of a secondary WC phase along with the Al₂O₃ phase. Fig. 1(c) shows the EDS spectrum of the coating particle “p” shown in Fig. 1(a), which clearly reveals the composition of C, W, Al, and O elements. The Cu signal in the EDS spectrum was contributed by the Cu grid.

Fig. 2 shows the apparent density and porosity of specimens sintered via SPS in the temperature range of 1200–1350 °C. The apparent density mostly increases with increasing sintering temperature. The increase in density is expected due to the existence of WC in the Al₂O₃ matrix because the theoretical density of WC (15.89 g/cm³) is substantially greater than that of Al₂O₃ (3.96 g/cm³). The densities of SPS-treated samples sintered at 1200 °C and 1250 °C are close to 3.98 g/cm³, which is higher than that of pure alumina. When the temperature was increased to 1300 °C and 1350 °C, the driving force greatly increased, densifying the discs. The decrease in porosity with increasing sintering temperature confirms that the composite powder was becoming fully densified.

Fig. 3 shows the shrinkage line of the composite powder sintered at 1350 °C, in which the shrinkage starts at 950 °C and stops at 1330 °C. When the temperature was below

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