Research Article

Effect of Particle Morphology on Sinterability of SiC-ZrO₂ in Microwave

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The effect of particle morphology on the sinterability of microwave-sintered SiC-ZrO₂ was evaluated in this paper. A comparison was also made against the electric furnace and resulted in faster heating rate because of the difference of heat transfer in the sintering processes. High-energy mechanical milling process has been applied to obtain the homogeneous particle morphology and finer particles size. The microstructure of finer particles of SiC-ZrO₂ after milling was better in the region sintered by microwave. The microstructure in the sintered region was smaller for the coarser particles which were obtained by heterogeneous nonmilled powder mixture. The result of the Vickers hardness test was higher for the powder homogeneously dispersed SiC-ZrO₂ after milling and sintering by microwave. These are influenced by the difference of individual electric properties of the initial SiC and ZrO₂ which have an effect on the absorption of the microwave energy.

1. Introduction

The principal advantages of ceramics are high hardness, extremely high melting point, high compressive strength and stiffness, and wear resistance [1, 2]. Silicon-carbide-(SiC-) based ceramics are also very promising high-temperature structural materials owing to their excellent thermal and mechanical properties, such as hardness and flexural strength [3–7]. One of the applications of SiC is in gas turbines. Mechanical properties of SiC, especially fracture toughness, can be improved by the addition of other phases into a material matrix to form a composite structure [8–12]. SiC-based ceramics are generally produced by cold die compaction with subsequent sintering or by hot isostatic pressing and finishing [13]. Among these procedures, the usage of sintering is the most important process for fabricating ceramics. One of the sintering technologies is microwave, which was initially used only in food production but now is also used in the powder processing. However, monolithic SiC is a highly covalently bonded silicon and carbon compound that is difficult to sinter without additives by any method [14] including microwaves [15]. Besides, SiC-based ceramics

have low-dielectric-loss factor, that is, the materials with such properties are not easy to heat up by microwave, especially at lower temperature, as reported by Agrawal et al. [16, 17].

Dispersion of zirconia (ZrO₂) particles in silicon carbide and other low dielectric loss ceramics is an effective method to enhance the absorption of energy in microwave sintering in room temperature. The preparation of ultrafine ZrO₂-based ceramic particles by microwave sintering has been studied extensively [18, 19]. Dispersing of ultrafine particles is expected to improve the sintering ability in ceramics. This is because the specific surface area of the particles provides additional driving force for densification during sintering. Ultra-fine initial size of the particles normally results in higher densities, and in some cases may lead to lower firing temperatures due to faster sintering kinetics [20]. Ultra-fine ZrO₂-based ceramic particles are usually known to contain agglomerates. One of most important factors in sintering is the agglomerate size. In microwave sintering, when the powders are milled and refined to nanometer-sized particles, the microwave absorption properties may be changed. Ruan et al. compared the microwave attenuation between the particles of different sizes of 5 μ m and 65 nm and showed that

the dielectric loss of nanometer-sized particles is larger than that of micrometer-sized particles [21].

Although SiC and ZrO₂ have been sintered individually by microwave by many researchers, the formation of the particles morphology and its effect on the microstructure and mechanical properties have not been previously reported. Thus, the objective of this paper is to evaluate the two different formations of the particle morphology on the microstructure of SiC-ZrO₂. The differences in the formation of the particles morphology were obtained by mechanical milling and compacted by microwave or electric furnace sintering. The hardness tests of SiC-ZrO₂ with different particles morphology were also measured.

2. Experimental Procedure

2.1. Mechanical Milling. In this study, a fine α -SiC of 2~3 μ m and an ultra-fine ZrO₂ of 30 nm were used, as shown in Figure 1(a). Both ceramics were manually mixed with a weight ratio of 1:1. In another set, both ceramics were subjected to vibratory ball milling (Super Misuni NEV-MA-8, Nishin Gikken, Japan). Dense tungsten carbide-cobalt (WC-Co) of 5 mm in diameter was used as a grinding media and of 60 mm in diameter was used as a grinding pot. Figure 1(b) shows the illustration of vibration ball mill. A constant vibration frequency of 12.5 Hz and a total time of 144 ks were used to mill SiC and ZrO₂ with the same ratio, 1:1. The milling time was chosen to achieve different morphology of the particles. The ratio of the grinding ball to milled powder was 5:1 by weight. After mixing and milling, the SiC-ZrO₂ was precompact in a 10 mm diameter stainless steel die under 50 MPa pressure, and then consolidated by nonconventional microwave and conventional electric furnace sintering.

2.2. Sintering. The schematic illustration of microwave sintering is shown in Figures 2(a) and 2(b) (IDX, MSS-TE0004, Japan) with a single-mode cavity and linked to a magnetron that delivers a variable forward power of up to 2 kW at a frequency of 2.45 GHz. Figure 2(a) shows the temperature measurement of the microwave sintering process. It was continuously measured by an infrared radiation thermometer camera cold air intake (Chino, IR-CAI). The microwave sintering was carried out in nitrogen atmosphere at a temperature of 1273 K for 600 s. The microwave heating was generated by 800 W power. The specimen was set upon a silica plate inside a silica tube for thermal insulation, as shown in the front view in Figure 2(b). An alumina stage was used as the susceptor for the purpose of thermally heating the low-dielectric-loss ceramic specimens to a certain temperature. The ceramic specimens directly absorb microwave energy to self-heat to the desired temperature at high heating rates, and the main function of the susceptor is to maintain a uniform temperature. The specimen was heated in the electric- (E-) field, which was controlled via a three-stub tuner. The maximum setting of the specimens on the E-field plunger was set to 142 mm.

For the conventional electric furnace sintering, the temperature of SiC-ZrO₂ was controlled to the same value as the microwave, 1273 K. The sintering temperature was measured using an R-type thermocouple with a constant soak time of 600 s, which was generated by applying a nominal power of 2000 W.

2.3. Characterizations. The impurities of the powders, before and after mechanical milling, were investigated by X-ray diffraction analysis (XRD, RINT-2000, Japan). The morphology of the particles was characterized using a field-emission scanning electron microscope (FE-SEM S-4800, Hitachi, Japan). The sintered samples were cut in half and their cross-sections carefully polished. These were used for microstructure characterization by means of FE-SEM. The mechanical properties were examined using hardness testing. The hardness was measured with an HMV-1 (Shimadzu Corp., Japan) using the Vickers indents obtained by applying a 98.1 N load for 5 s. The measurement was conducted at 30 random points on each specimen.

3. Results and Discussion

3.1. Characterization of Particle Morphology. Figure 3 shows FE-SEM images of SiC-ZrO₂. As shown in the non-milled powder mixing in Figure 3(a), the shape of the SiC was irregular and the ZrO₂ was agglomerate, which is similar to the initial one (Figure 1(a)). Before mixing, the amount of ZrO₂ particles that attach on the surface of the SiC powders is very limited. As a result of mixture, there is no change in the appearance of both SiC and ZrO₂. From this image, it appears that the particle morphology is heterogeneous.

After mechanical milling, the surface of the SiC powders becomes finer and almost fully covered with ZrO₂ particles, as shown in Figure 3(b). In order to achieve homogeneous particle morphology, control of the milling time was very important. If the milling time was too short, the particle morphology of ZrO₂ on the surface of SiC would remain heterogeneous. The homogeneous particle morphology could be fully achieved for a milling time of 144 ks. The size of the SiC powders was not significantly reduced, and their shape became somewhat spherical. In the case of ZrO₂, the size was refined and the shape of the particles was deformed. These morphological changes are attributed to a fragmentation of the particle agglomerates which was caused by the high-energy mechanical milling.

The refinement of the particle size is consistent with the broadening of X-ray diffraction peaks in Figure 4. In the case of the nonmilled powder mixture (heterogeneous particle morphology), the diffraction peaks were slightly narrower than for the milled powders (homogeneous particle morphology). According to Scherrer's formula,

$$\tau = \frac{K\lambda}{\beta \cos \theta}, \quad (1)$$

where K is the shape factor, λ is the X-ray wavelength of the radiation used (CuK_α), β is the line broadening at half the maximum intensity (FWHM) in radians, θ is

FIGURE 1: (a) Appearance of initial silicon carbide powders with the size 2~3 μm and zirconia particles with the size 30 nm (b) Illustration of Super Misumi vibration ball mill with dense WC-Co of 5 mm in diameter used as a grinding media and of 60 mm in diameter used as a grinding pot.

FIGURE 2: Schematic of (a) side and (b) front view of single-mode microwave sintering. The microwave sintering was carried out in nitrogen atmosphere at a temperature of 1273 K for 600 s and was generated by 800 W power.

FIGURE 3: FE-SEM micrographs of particles morphology appearance of SiC-ZrO₂ with (a) nonmilled powder mixture (heterogeneous) with the amount of ZrO₂ particles that attach on the surface of the SiC powders is very limited (b) milled powder (homogeneous) particle morphology with the surface of the SiC powders becoming finer and almost fully covered with ZrO₂ particles.

FIGURE 4: X-ray diffraction pattern of initial SiC and ZrO₂, for the nonmilled powder mixture (heterogeneous); the diffraction peaks were slightly narrower than those for the milled powders (homogeneous distribution).

the Bragg angle, and τ is the mean size of the crystalline domains, which may be smaller or equal to the particle size; the broadening of diffraction peaks indicates a reduction in crystalline size [22]. No phase transformation or new phase formation for both SiC-ZrO₂ was found in the heterogeneous and homogeneous powder mixtures such that SiC and ZrO₂ maintain their rhombohedral and monoclinic structures, respectively.

3.2. Sintering Behaviors. The microwave heating profiles of SiC-ZrO₂ separated into electric and magnetic fields are shown in Figure 5(a). The plot shows different heating results in microwave fields. In the E-field, SiC-ZrO₂ powders could be sintered, unlike in the H-field. The temperature increased to around 40 s. To achieve the desired temperature of 1273 K, the time required in microwave heating is less than 60 s. It means that the heating rate in the microwave sintering is very rapid, 25 K/s on average.

In case of electric furnace sintering, the temperature remained almost constant at room temperature from 1 s to 30 s time. Then, the temperature increased until around 160 s, as shown in the heating profile in Figure 5(b). The heating rate in the electric furnace is slower than in microwave sintering, which is only 8 K/s on average. To achieve the temperature of 1273 K, the time required is as much as 200 s.

3.3. Sinterability and Microstructure of SiC-ZrO₂. Figure 6 shows FE-SEM images of SiC-ZrO₂ specimens sintered by microwave and electric-furnace sintering at the constant temperature of 1273 K. In low magnification of microwave

sintering microstructures, there is an obvious difference in the microstructure of samples derived from heterogeneous and homogeneous particle morphology. It is clearly showed that the sintering ability of heterogeneous particle of SiC-ZrO₂ in microwave is lower than that of the homogeneous one. In heterogeneous particle morphology microstructure, only a few scattered sintered regions are present, as shown in Figure 6(a). After mechanical milling for 144 ks, the particles morphology was changed, the particle morphology was homogeneous, and the average grain size was refined, as shown in Figure 6(b), the better homogeneity of ZrO₂ particle on SiC surface, improve the sintering region in microwave. The grain size of SiC-ZrO₂ is clearly shown in the FE-SEM micrographs at higher magnification in Figures 6(c) and 6(d). In the non-milled powder mixture (heterogeneous), the SiC size is bigger and coarser than the milled powder (homogeneous particle morphology).

The microstructures of the SiC-ZrO₂ at 1273 K temperature, sintered by the conventional electric furnace, are shown in Figures 6(e) and 6(f). In this case, a very different appearance, as compared to the microstructure of microwave sintered powder, is shown for all electric furnace sintered specimens. The SiC-ZrO₂ shows a coarse-grained aspect with high porosity. From the micrographs, there is almost no difference in the heterogeneous and homogeneous particles morphology of the SiC-ZrO₂ after sintering by electric furnace.

3.4. Vickers Hardness. SiC-ZrO₂ specimens sintered by microwave and electric furnace were measured by the Vickers hardness. Those specimens were tested on random points from top to bottom section. In the case of electric

FIGURE 5: Heating profile of (a) microwave and (b) electric furnace sintering. The heating rate in the microwave sintering is 25 K/s on average and in the electric furnace is 8 K/s on average.

furnace sintering, both the heterogeneous and homogeneous particles morphology specimens showed very similar results, as shown in Figure 7. This result indicates that the sintering ability of SiC-ZrO₂ in electric furnace is very low, regardless of grain size and/or particle morphology.

In the case of microwave sintering, the Vickers hardness of the homogeneous is higher than that of the heterogeneous particle morphology specimens. This is consistent with the microstructures of heterogeneous particle morphology of SiC-ZrO₂ in microwave-sintered compacts which shows lower sintering ability than homogeneous particles morphology specimens.

3.5. Discussion. In microwave sintering, the 800 W applied power and E-field were chosen evaluation. In the E-field, SiC and ZrO₂ ceramic powders could be sintered unlike the magnetic H-field; this indicates that the initial ceramics such as SiC and ZrO₂ do not have the magnetic properties. Therefore, the E-field can be used to evaluate the effect of particle morphology of SiC-ZrO₂ in microwave.

The heating profiles of microwave and electric furnace sintering show that for conventional electric furnace sintering, the heating rate required is 8 K/s on average from room temperature to 1273 K. For microwave sintering, the total time was only 50 s on average to reach 1273 K temperature. During electric furnace sintering, the heating rate is limited by the capability of the machine to achieve fast heating rates because of the slow resistive heating of the heating elements and heat transfer via thermal radiation to the material and also to prevent large thermal variation within the compacts

to avoid cracking [23]. The slower heating rate, the need for holding at intermittent temperature to reduce thermal variation, and long soaking time for sintering increase the total processing time of the compacts [23]. For microwave, heating coupled with susceptors, rapid heating rates in excess of 25 K/s on average can be easily achieved since the powder compact can absorb microwave energy directly and can be heated rapidly from within. The susceptors provide radiant heating to the samples externally, thereby reducing the thermal variation in the compacts [23]. The rapid heating rate in microwave will activate the ion exchange and chemical reaction as material transport, thereby reducing the activation energy [24]. Rapid heating minimizes grain growth and enhances the mechanical properties of materials. Those rapid heating rates in microwave resulted in larger sintered regions of microstructure than in the case of electric furnace-sintered compact specimens.

The microwave sintering response in microstructure is not only caused by the rapid heating rates. The better response in microwave for mechanical milling powder is also due to ultrafine ZrO₂ particles dispersed homogeneously on the SiC surface. It is known that smaller particle can enhance the sintering kinetics. Another important factor in the microstructure of SiC-ZrO₂ is the absorption of microwave energy in each material. The absorption of ultrafine ZrO₂ particles is better than that of fine SiC powders, considering the dielectric loss factor of ZrO₂ is higher than that of SiC. As reported by Ruan et al., the dielectric loss of nanometer-sized particles is larger than that of micrometer-sized particles [21]. When the ultra-fine ZrO₂ particles are dispersed homogeneously on the SiC surface, the absorption of the

FIGURE 6: (a) and (b) are low magnification and (c) and (d) are high magnification of FE-SEM micrographs of nonmilled powder mixture (heterogeneous) and milled powder (homogeneous) particle morphology of SiC-ZrO₂ sintered by microwave sintering. (e) and (f) are FE-SEM micrographs of SiC-ZrO₂ sintered by conventional electric furnace with heterogeneous and homogeneous particle morphology and homogeneous particle morphology.

microwave energy is further increased. The homogeneous particle morphology and finer crystalline size that were obtained by high-energy mechanical milling leads to higher dielectric loss compared with the non-milled powder mixture (heterogeneous particle morphology). Therefore, larger sintered regions by microwave sintering could be obtained as an effect of the homogeneous particle morphology of SiC-ZrO₂ ceramic composites.

Although the effects of the particle size and morphology of SiC-ZrO₂ on the microwave sintering response were clearly mentioned, we still have a problem with microwave sintering that was performed in single-mode, 2.45 GHz microwave with small chamber, easy to maintain and to use. The drawback of these single-mode chambers is that they are relatively small, which only allows the heating of one small specimen at a time. Unfortunately, this method has a critical

drawback since the electro magnetic field in the chamber is not uniform [25], resulting in inhomogeneous heating and the unavoidable formation of hot spots. One approach to achieve a uniform electro-magnetic field distribution in a single-mode chamber is to design a chamber with at least one of the major dimensions 100 times greater than the wavelength. A higher frequency is more convenient than using a huge chamber to enlarge the uniform field region. For this reason, higher microwave frequencies such as 28 GHz and 80 GHz could be used [26–28].

In the present study, specimens were sintered by microwave single-mode furnace with a small chamber, in which the field distribution at the center of the chamber is not completely uniform and specimens sintered using the alumina susceptor show regional microstructures. The susceptor can also have a compensating or correcting effect

FIGURE 7: The vickers hardness of non-milled powder mixture (heterogeneous) and milled powder (homogeneous) particle morphology of SiC-ZrO₂ sintered by microwave and conventional electric furnace. The lower hardness of specimen sintered by conventional electric furnace compared with that sintered by microwave indicates that the sintering ability of SiC-ZrO₂ in electric furnace is also very low, regardless of grain size and/or particle morphology.

with respect to the inhomogeneous electric field. In our experiments, the response of microwave sintering of SiC-ZrO₂ with alumina susceptor shows excellent repeatability in microstructure and hardness test results, while sintering without susceptor resulted in differences in the repeatability of microstructure and hardness test results because of an extreme sensitivity to the materials response in the chamber.

4. Summaries

The effects of particles morphology of SiC-ZrO₂ have been evaluated using microwave and electric furnace sintering. Several summaries obtained are as follows.

- (1) The heating rate obtained in the microwave furnace was faster compared with the electric furnace sintering.
- (2) The ZrO₂ particles were refined and the morphology, of the ZrO₂ particles was homogenized on the SiC surface by mechanical milling.
- (3) The homogeneous particle morphology resulted in larger sintered regions in microstructure and higher hardness values after sintering by microwave, compared with the nonmilled powder mixture (different particle morphology).

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