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Effect of Residual Porosity and Pore Structure on the Mechanical Strength of $SiC–Al_2O_3–Y_2O_3$

Dean-Mo Liu & Chen-Tsu Fu

Materials Research Laboratories, Industrial Technology Research Institute, Chutung, Hsinchu 31015, Taiwan

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Abstract: Effect of residual porosity on the flexural strength of sintered α-SiC ceramic with Al_2O_3 and Y_2O_3 as sintering aids was investigated. The flexural strength (σ)–porosity (p) behaviour of the SiC ceramics was experimentally derived based on the minimum solid area approach. Both the data from the present work and those available in the literature can be simply and well-expressed by an exponential function at a correlation coefficient as good as $r^2 \simeq 0.90$:

$$\sigma = 915\exp(-9.0p)\ \text{MPa}$$

The value of the exponent constant (9.0) agrees reasonably with that theoretically predicted by using the approach for an extreme of idealized oblate spheroids. The pore structure appeared to have little or no influence on the exponential σ–p behaviour, except for the presence of surface pores which act as fracture flaws leading to a major deterioration of mechanical strength compared to the predicted one.

1 INTRODUCTION

The sintering of silicon carbide (SiC) via a pressureless-sintered method has received wide attention over the last two decades.[1–5] The first work on pressureless-sintering of SiC ceramic was achieved by Prochazka[1] who used boron and carbon as sintering aids and usually resulted in a dense SiC body with moderate mechanical strength. Also, a number of sintering aids, such as Al, B, C, AlN, oxides, etc., together with some of their combinations have been extensively studied.[6,7] A successful densification of SiC using Al_2O_3 and Y_2O_3 as sintering aids was first achieved by Omori and Takei[3] and a number of recent reports have demonstrated that a significant improvement in mechanical properties,[8–10] particularly fracture toughness (having a value of as high as 8.3 $MPa{\cdot}m^{1/2}$),[8] of the SiC ceramic was attainable in the system of $SiC–Al_2O_3–Y_2O_3$ and was comparable to that in silicon nitride ceramics.[11] A recent investigation on the high-temperature strength of the $SiC–Al_2O_3–Y_2O_3$ system demonstrated a promising feature of strength retention (above ~90% of room-temperature strength) to 1200°C.[12] These findings strongly suggest the use of $SiC–Al_2O_3–Y_2O_3$ ceramic as a state-of-the-art material for modern advanced applications.

Pore phase is frequently present in the ceramic body, particularly within those obtained by means of the pressureless-sintering method. In spite of small pore fractions (e.g. <5 vol%), the mechanical properties, such as strength, modulus and hardness, can usually be deteriorated in a fashion depending on a variety of pore factors such as pore size, pore distribution/location, etc.[13,14] Although the influence of porosity on the mechanical properties of ceramic materials has been an important subject both experimentally and theoretically over a range of porosity fractions,[15] the relevant reports on the $SiC–Al_2O_3–Y_2O_3$ systems have not been found in literature, particularly in sintered bodies containing limited porosity. This communication describes the influence of residual porosity on the flexural strength of sintered α-SiC ceramics with

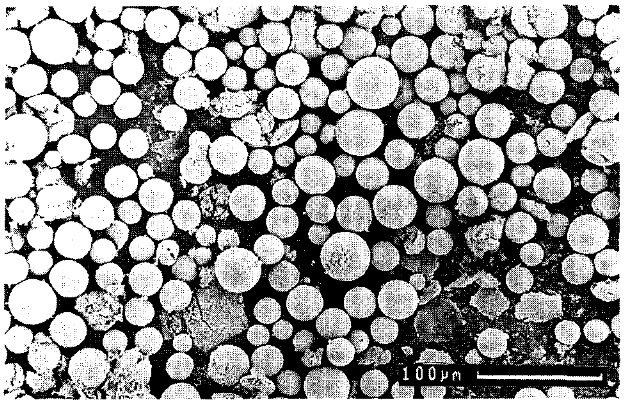

Fig. 1. Spray-dried α-SiC powders.

Al_2O_3 + Y_2O_3 as sintering aids. A porosity dependence of the flexural strength, together with those published data, was empirically derived.

2 EXPERIMENTAL PROCEDURES

Spray-dried alpha SiC powders (Showa Denko, Japan, containing 6.2 wt% Al_2O_3 and 3.8 wt% Y_2O_3), shown in Fig. 1 (having a mean raw particle size of 0.48 μm), were consolidated into green compacts using a conventional single-acting die-pressing method of varying compaction pressures from 6.8 to 155 MPa, corresponding to different green densities. The green compacts were sintered at 1900°C for 2 h followed by increasing temperature to 2000°C for 30 min in 1 atm Ar atmosphere. The density of the sintered SiC bodies was measured by using Archimedes' principle. The sintered SiC bodies were machined to form bending bars with dimensions of 2 mm × 3 mm × 25 mm. The surface of the specimens was ground with 600-grit diamond wheel. The three-point flexural strength of the SiC ceramics was measured using an Instron tester (Model 1361) at a crosshead speed of 0.5 mm/min. Five to seven specimens were employed for the determination of the flexural strength. Microstructure of the sintered SiC body was examined using scanning electron microscopy (Cambridge Instruments, S-360).

3 RESULTS AND DISCUSSION

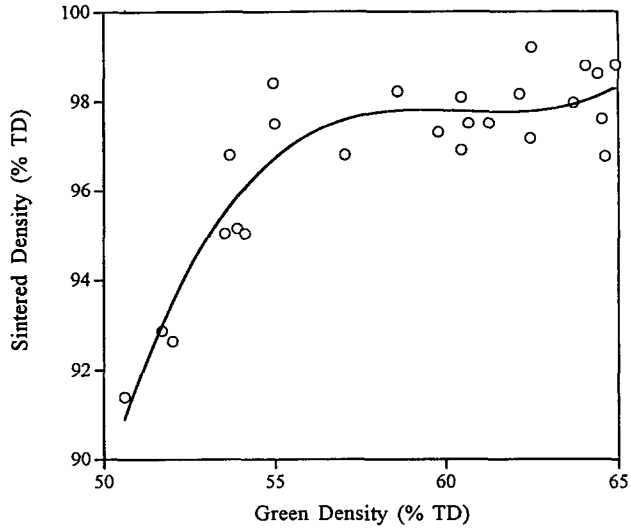

Fig. 2. Densification of the α-SiC powder compacts of varying green densities.

Figure 2 shows the dependence of sintered density upon the corresponding green density (determined by Hg-porosimetry) of the SiC bodies. A sintered density greater than 95% of theoretical density (TD = ~3.25 g/cm^3) was easily obtained when the green density of the powder compacts was greater than approximately 55% of theoretical. However, a poor particle packing (e.g. those below ~55%) caused the densification to be incomplete even with the aid of liquid-phase sintering mechanism, resulting in a less dense sintered body. The sintered density generally increased with increasing green density; however, no complete densification (i.e. porosity = 0) was achieved. This is commonly observed in other reports, resulting in the presence of residual porosity, which is expected to have some influence on mechanical properties.[14,16] As obtained, the sintered SiC bodies exhibited an equiaxed-grain microstructure with grain size ranging from 1 to 3 μm and no exaggerated grain growth to plate-like grains, which may act as stress raisers, was seen.

Specimens with sintered density greater than 95% TD were selected for strength measurement. The porosity–mechanical strength relationship is depicted in Fig. 3. The circle symbols stand for the

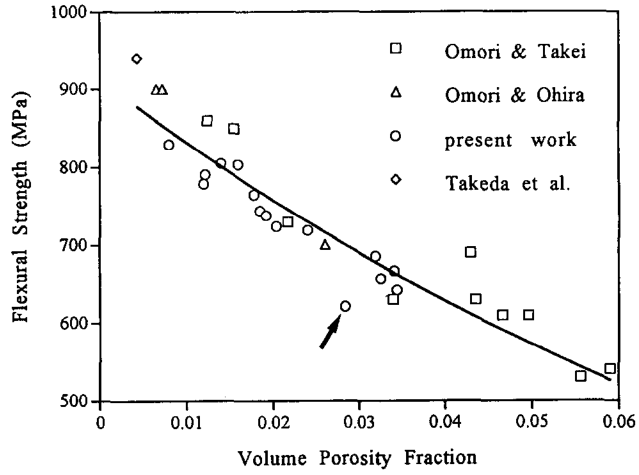

Fig. 3. Flexural strength–porosity behaviour determined experimentally in α-SiC ceramics showed a better correlation with the minimum solid area approach.

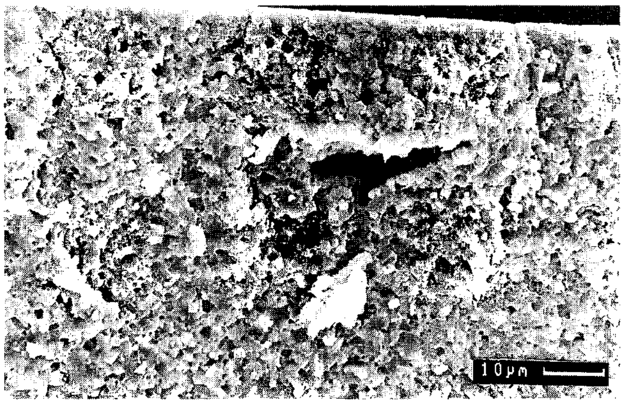

Fig. 4. Scanning electron micrograph of a fractured surface showing the presence of a large oblated-like pore ~ 20 µm in axial length immediately beneath the tensile surface, together with a few small spheroid-like pores.

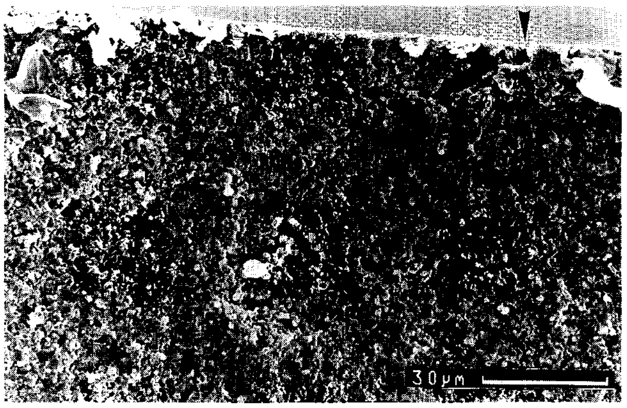

Fig. 5. The presence of surface pore acting as fracture flaw seriously deteriorated the flexural strength of the α-SiC ceramics.

present work, also included are some published data; the choice of data was based on their similarity in testing method (three-point bending), in fabrication method (dry pressing followed by pressureless-sintering) and in sintering aids used (oxides), except for the case of Takeda *et al.*[6] who obtained the SiC ceramic via a hot-pressing technique with metal Al as the sintering additive. No exaggerated grain growth was reported in these literature, instead, equiaxed grains with size ranging from 1 to 3 µm constructed those microstructures.

Upon determining the mechanical strength (σ)–porosity (p) relation, a recent publication by Rice[15] has compared the mechanical property–porosity dependence, especially for elastic modulus, in terms of two basic approaches, namely Rossi's stress concentration approach[17] and minimum solid area approach, and Rice concluded that the latter approach is more fundamentally correlated with mechanical property (particularly for elastic modulus)–porosity behaviour. Since the strength is directly related to the elastic modulus by $\sim(E\gamma)^{1/2}$ and the fracture energy (γ) of polycrystalline solids typically follows a similar trend with porosity as Young's modulus (E) does,[18] the porosity-dependent strength may therefore behave in a similar way to that of the porosity-dependent modulus. The resulting porosity-dependent strength based on the minimum solid area approach can be closely approximated by an exponential function:

$$\sigma = \sigma_0 \exp(-bp) \qquad (1)$$

where σ_0 is the zero-porosity tensile (flexural) strength, σ is the strength at volume fraction porosity p, and the constant b is directly related to the type of porosity and property. For a low concentration of spherical porosity, the constant b is approximately equal to the Rossi's constant (B) in Rossi's equation [$\sigma = \sigma_0(1-Bp)$]. The strength–porosity behaviour shown in Fig. 3 was determined using eqn (1) and resulted in a solid curve (Fig. 3) which correlated well with the literature data and the present work by a correlation coefficient as good as $r^2 \simeq 0.90$:

$$\sigma = 915\exp(-9.0p)\ \text{MPa} \qquad (2)$$

The porosity dependence, i.e. $b = 9.0$, agrees reasonably with the value of the theoretical prediction based on the minimum solid area approach for an extreme of idealized oblate spheroids, i.e. cylindrical pores whose axis is normal to the stress, whereas the constant b may fall into the range of approximately 6–10.[15] However, plugging the data of Fig. 3 into Rossi's equation yielded $B = 7.5$ and a correlation coefficient of $r^2 \simeq 0.85$. The minimum solid area approach is evidently offering a better correlation of σ–p behaviour than that from Rossi's approach.

For a better understanding of the influence of porosity, the fractured surfaces of the tested specimen were examined with SEM. Figure 4 shows a large pore with an oblate-like geometry of approximately 20 µm in axial length, which is located ~ 25 µm beneath the tensile surface, together with a few small spheroids with size comparable to the grain size. However, the real pore structure can't be actually described by means of the two-dimensional fractographic technique. The SEM examination allowed to find a few of such large oblate-like pores but with varying orientations with respect to the stress direction, and distributed sporadically within the sintered bodies. This feature is substantially more complex than the idealized picture, and this may be the main reason for the major deviation in the resultant derivations as detailed by Rice.[15]

Also, a fractographic examination of one specimen, which has a porosity of ~2.8% but with a flexural strength of ~610 MPa (indicated in Fig. 3 with an arrow, and excluded in the derivation of eqn (2)) which is considerably lower than that predicted according to eqn (2), i.e. ~710 MPa, is illustrated in Fig. 5. The fracture origin was evidently due to surface pore (arrow indication), which acts as a failure-causing flaw.[13] This finding, together with the above discussion appears to verify the argument addressed by Rice,[16] according to which the pore shape effect on the strength is limited unless the pore is large enough to act as a fracture flaw, causing a large deviation in prediction of the strength in terms of σ–p dependence by eqn (1) or eqn (2) determined experimentally.

4 CONCLUSIONS

The effect of residual porosity on the flexural strength of sintered α-SiC ceramic was investigated. The flexural strength (σ)–porosity (p) behaviour of the α-SiC ceramics studied in the present work and of similar materials investigated by other authors can be well described by an exponential function: $\sigma = 915 \exp(-9.0p)$ MPa. Pore structure showed little or no influence on the σ–p behaviour. This is consistent with the concept proposed by Rice, unless the pores act as surface flaws leading to a considerable deterioration of the strength.

To minimize the risk (or possibility) of the presence of surface pores as a source of failure, a near-full density of SiC ceramics with controllable flaw size is essentially needed for both reliability improvement and predictable mechanical properties.

ACKNOWLEDGEMENT

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