

# Deformation and Fracture Mechanics of Superior Nanocomposites

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**Abstract.** Lightweight ultra-fine grained (<1  $\mu\text{m}$  size) SiC-ZrO<sub>2</sub>(3Y<sub>2</sub>O<sub>3</sub>) composites, with a combination of high hardness, high bending strength and high fracture toughness, were successfully prepared by high energy mechanical milling followed by heat treatment. The SiC-ZrO<sub>2</sub>(3Y<sub>2</sub>O<sub>3</sub>) composites exhibited high hardness (1707 MPa), high bending strength (as high as 1689 MPa) and high fracture toughness (up to approximately 12.6 MPa.m<sup>1/2</sup>). Such a combination of mechanical properties was attributed to the fine microstructure with a distinct feature consisting of almost continuous network of ZrO<sub>2</sub>(3Y<sub>2</sub>O<sub>3</sub>) phase around SiC grains, or we call harmonic microstructure. It has been demonstrated that a combination of these unique microstructural characteristics was very effective in suppressing the initiation of cracks and governing the path of their subsequent growth during fracture, leading to excellent combination of mechanical properties.

## Introduction

In recent years, silicon carbide (SiC)-based ceramics are very attractive and promising high-temperature structural materials for a variety of applications owing to their excellent combination of optical, thermal, chemical, electrical and mechanical properties [1–5]. In addition, Yttria-Stabilized-Zirconia (ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>)) has major applications as thermal barrier coatings (TBCs) in gas-turbine engines and electrolytes for solid oxide fuel cell (SOFC) as an alternative green energy source due to the excellent combination of high temperature mechanical properties, high oxidation and corrosion resistance, and low thermal conductivity [6,7]. Although an excellent combination of properties makes YSZ the most popular ceramic over other structural ceramics, a very high density (~6.02 g/cm<sup>3</sup>) and, monolithic SiC is a highly covalently bonded silicon and carbon compound that is difficult to densify [8], its low resistance to fracture has impeded its widespread applications.

The development of ceramic-ceramic composites has been an attractive approach to achieve a desired set of mechanical properties which is superior to that of the individual ceramics used in the composites. Therefore, one major approach is to strengthen and toughen silicon carbide and other ceramics, for example with the use of composite technology by incorporating particulate, whiskers, platelets or fiber [9]. Ceramic-based nanocomposite is one of the particulate-reinforced composites in which the nano-sized particulate is dispersed within the matrix grains and/or at the grain boundaries [10]. ZrO<sub>2</sub> has been successfully used as a toughening agent in Al<sub>2</sub>O<sub>3</sub>, SiAlON and Si<sub>3</sub>N<sub>4</sub> matrix composites to improve their mechanical properties [11]. Clausen and Jahn reported that the addition of 20vol% unstabilized ZrO<sub>2</sub> to an Si<sub>3</sub>N<sub>4</sub> matrix improved the fracture toughness thanks to a microcracking toughening mechanism [11].

In the author previous study, it has been successful to create a ceramics composite with the harmonic microstructure controlled formation and its effect on the mechanical properties [12, 13]. However, the higher and higher mechanical properties, especially on the fracture toughness of ceramics composite is still expected. Thus, the objective of the present study is to evaluate the fracture behaviour of SiC-ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) ceramics composite with harmonic microstructure controlled by mechanical milling and heat-treatment parameters.

## Experimental Methods

First, alpha-SiC powders of 2 ~ 3  $\mu\text{m}$  were milled with 50mass% of 30nm  $\text{Y}_2\text{O}_3$ -stabilized- $\text{ZrO}_2$  powders. This mixture was used as a starting material and was mechanically milled with a WC-Co ball and pot with a diameter of 5 and 60 mm, respectively. The MM process on the SiC- $\text{ZrO}_2(\text{Y}_2\text{O}_3)$  powders was performed with a high-energy vibration ball mill speed of 120 Hz. In addition, the milling was performed in dry conditions, and no agent was used. The values chosen for the ball-to-powder weight ratio and the process time these two parameters were 5:1 and from 0 to 144 ks, respectively. Subsequently, the MM powders were heat-treated in a sintering furnace.

Sintering was carried out in a vacuum using a furnace. The sintering process was performed with a pressing die made of graphite under 50 MPa uniaxial pressure. The controlled sintering temperature and heating rates were 1773 K and 373 K per 60 s, respectively. After the mixtures had been soaked at a desired milling time for 0.6 ks, the applied current was cut off, the pressure was released, and the specimen was cooled down to room temperature. Samples sintered measured approximately 15 mm in diameter and 5 mm in thickness.

The sintered samples were cut and carefully polished into rectangular bar specimens (2 mm  $\times$  4 mm  $\times$  15 mm). The bending strength was evaluated by means of the three-point bending method on the rectangular specimens with dimension 2 mm  $\times$  4 mm  $\times$  13 mm specimens using a Shimadzu AG-I-50 kN instrument, at a crosshead speed of 0.5 mm/min. The hardness and the fracture toughness were measured with an HMV-Shimadzu based on Vickers indentations obtained by applying a 98.1 N load for 10 s. The fracture toughness ( $K_{\text{IC}}$ ) of the composite was measured based on the crack length generated by Vickers indentation. The fracture toughness was calculated using the Anstis *et al.* equation, formula given below [14, 15],

$$K_{\text{IC}} = 0.12(c/d)^{-6/5} \cdot H \cdot c^{1/2} \quad (1)$$

where,  $c$  is the average crack length ( $\mu\text{m}$ );  $d$  is the half diagonal of the Vickers indent ( $\mu\text{m}$ ); and  $H$  is the Vickers Hardness (MPa).

## Results and Discussion

From the results of SEM micrographs below before mechanical milling (manual mixing) 0 s, the initial SiC powder shows an irregular shape and an agglomeration in the initial  $\text{ZrO}_2(\text{Y}_2\text{O}_3)$  powder. After mechanical milling for 72 and 144 ks, the SiC powder surface became increasingly covered with  $\text{ZrO}_2(\text{Y}_2\text{O}_3)$  powder, as shown in Fig. 1.

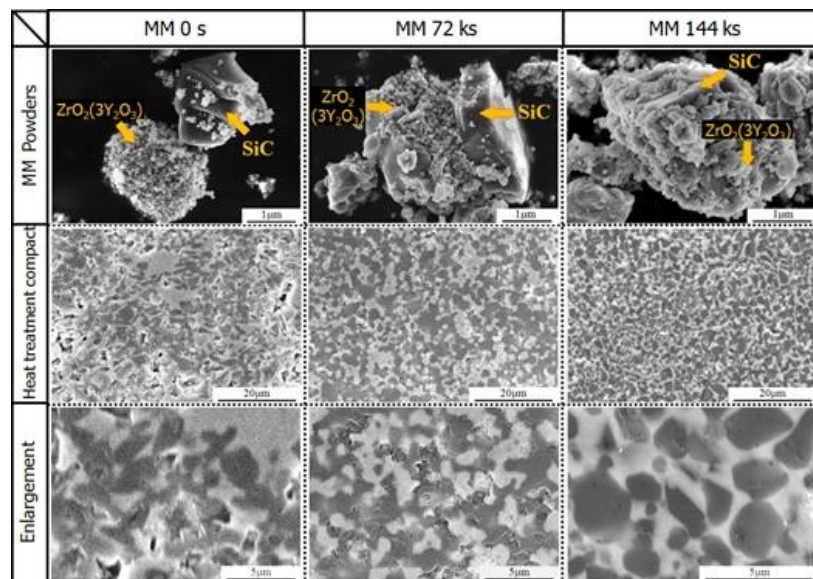


Figure 1. Microstructure of SiC- $\text{ZrO}_2(\text{Y}_2\text{O}_3)$  powders and compacts with milling time from 0s to 144ks

Table 1. Bending strength and fracture toughness of SiC- ZrO<sub>2</sub> and SiC-ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) with MM for 0 s, 72 ks and 144 ks

	MM Time [ks]	Bending Strength [MPa]	Fracture Toughness [MPa.m <sup>1/2</sup> ]
SiC-ZrO <sub>2</sub>	0	270	5.8
	72		8.0
	144	1070	10.2
SiC-ZrO <sub>2</sub> (3Y <sub>2</sub> O <sub>3</sub> )	0	450	6.2
	72		10.5
	144	1689	12.6

The effects of milling time on the bending strength and fracture toughness of the SiC-ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) composite are shown in Table 1. When 50 mass% ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) were added to the initial SiC powders, the densification temperature was decreased to ~1973 K. As the grain size of ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) is smaller than that of SiC the composite becomes easier to sinter. When the milling time was increased to 144ks, the densification temperature of the composite further decreased because of the finer dispersion of ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) produced after the high-energy mechanical milling process. Then, the SiC ceramic with homogeneous fine grains of ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) dispersed on its surface was obtained, and the mechanical properties were improved. It is noted that the sintering temperature depends strongly on agglomerate size [16–18].

However, the longest milling time of 360 ks resulted in lower mechanical properties because of the heterogeneous ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) dispersed half on the SiC surface with the other half of the ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) forming a separate agglomeration, which was not helpful in improving the strength and toughness of the SiC. The mechanical properties test results indicated that the SiC-ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) mechanically milled for 144 ks and heat-treated at 1773 K, the hardness is 1707 MPa, the bending strength is 1689 MPa, and the fracture toughness is 12.6 MPa.m<sup>1/2</sup>.

With milling time for 144 ks, the density of the SiC-ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) could be increased more than 100% of the theoretical. The microstructure of crack propagation shows that the length of crack is about ~5µm. For the MM 144 ks specimens with a harmonic microstructure, the crack is intergranular which is only in the ultra-fine ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>), and there is no crack in the fine SiC grain. The role of fine SiC grains in the crack deflection is as a suppressing in the crack propagation along ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) grains. The combination of two grain sizes with homogeneous dispersion and forming harmonic microstructure is effective to suppressing the crack propagation as shown in Fig. 2 and Fig. 3.

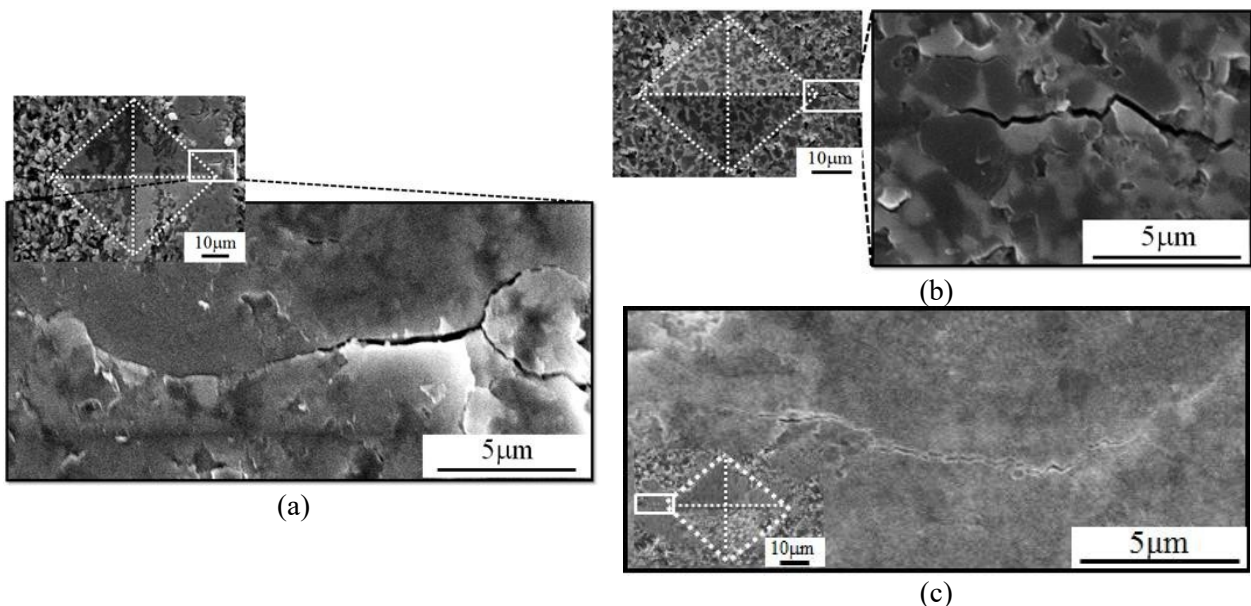


Figure 2. Crack propagation in SiC-ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) compact (a) without MM (0 s), (b) with MM 144 ks and (c) with MM 360 ks obtained by Vickers indenter

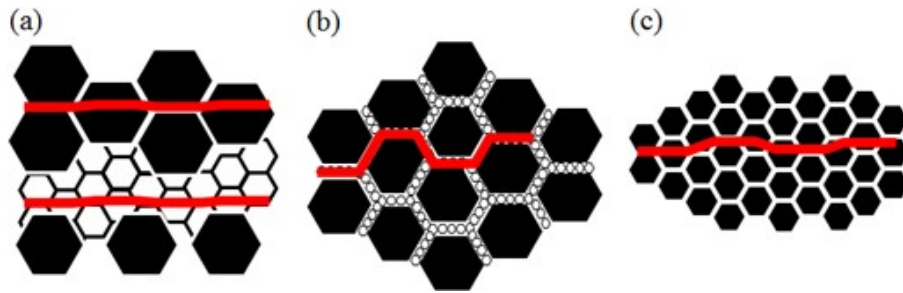


Figure 3. Illustration of the crack propagation of SiC-ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) with milling time (a) 0 s, (b) 144 ks and (c) 360 ks. Black hexagonal indicates SiC fine grain and white hexagonal indicates ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) ultra-fine grain. The red line is illustrating the crack propagation

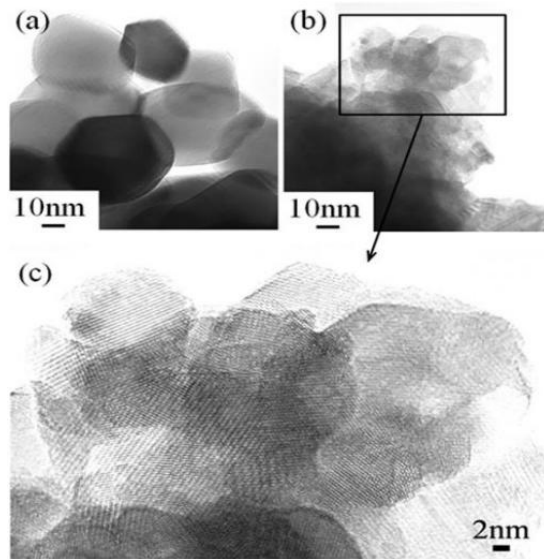


Figure 4. TEM micrographs of ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) particles with milling time for (a) 0 s, (b) 144 ks and (c) enlargement of high resolution TEM with the lattice images

The Energy-dispersive X-ray spectroscopy (EDS) image and peak of SiC-ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) are shown in Fig. 5 and Fig. 6, respectively. The measurement of each element is using K series with average apparent concentration is 2.25, k ratio is 0.018 and total weight sigma is 100%. New phase transformation of Fe formation for SiC-ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) was found in the milled compact, such that SiC and ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) not maintain their cubic and monoclinic structures, respectively.

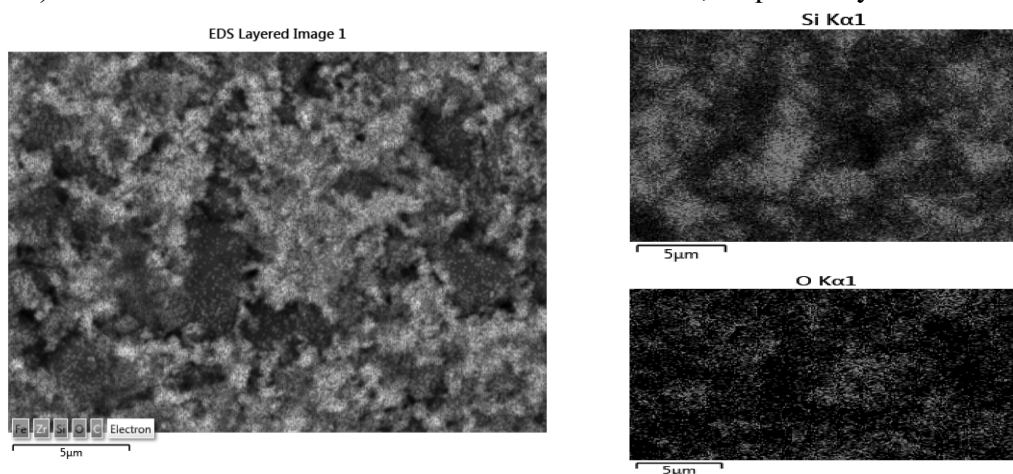


Figure 5. Energy-Dispersive X-ray Spectroscopy image of SiC-ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) compact

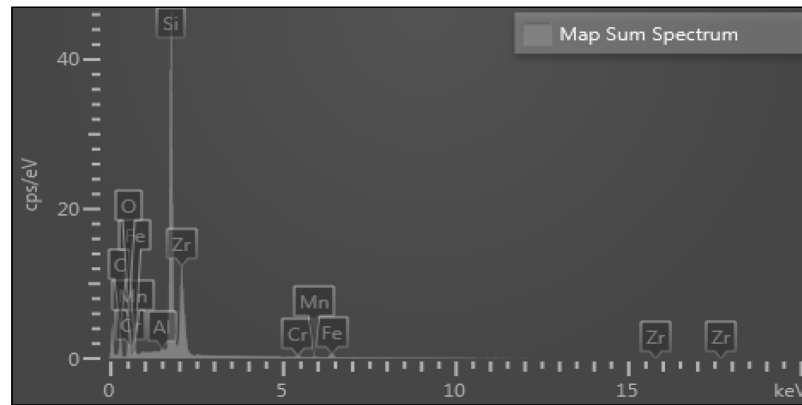


Figure 6. Energy-Dispersive X-ray Spectroscopy peak of SiC-ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) compact

## Summary

Mechanical milling powder, consisting of SiC powder and ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) particles, was sintered, and the deformation and the fracture mechanics of SiC-ZrO<sub>2</sub>(Y<sub>2</sub>O<sub>3</sub>) were investigated. The conclusions obtained are as follows:

1) The bending strength and fracture toughness were simultaneously improved by controlling the microstructure obtained in the high-density materials.

2) By combining of two different grain sizes, the crack propagation could be minimized, when the fine grain dispersed homogeneously and forming harmonic microstructure, the length of the crack further decrease. So, the higher mechanical properties could be achieved.

Those composites with high density were obtained by controlling the intensity of MM. Therefore, the main factors explaining the improvement in the mechanical properties are considered to be the density increase caused by MM and the harmonization of the dispersion.

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