

## Thermal Conductivity of Carbon Reinforced ZrO<sub>2</sub> for Inert Matrix Fuel

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### 1. Introduction

Zirconium dioxide ZrO<sub>2</sub> (Zirconia) is chemically inert, has a low neutron absorption cross section, resistant to the oxidization and reduction [1]; Because of that, it has been used different nuclear applications such as Inert Matrix Fuel (IMF) in the form of partially stabilized zirconia or pure zirconia [2,3]. However, a low thermal conductivity limits the effectiveness of zirconium dioxide and has a negative impact on its general performance. Therefore, improving the thermal conductivity of ZrO<sub>2</sub> can improve its performance and reduces its limitations. By improving the thermal conductivity of ZrO<sub>2</sub>, the temperature gradient, centerline temperature, and the thermal stresses in the IMF can be reduced. Carbon based materials are an attractive choice to enhance the properties of ZrO<sub>2</sub>. They are thermally stable and they have low thermal expansion coefficient, low atomic number, high thermal shock resistance, high melting point and thermal conductivity that spans over a wide range up to ~2000 Wm<sup>-1</sup>K<sup>-1</sup> in diamond or graphene at room temperature [4-5]. In our previous study, it has been shown that the thermal conductivity of ZrO<sub>2</sub> can be enhanced when 3D glassy carbon foam was added to ZrO<sub>2</sub> [6].

In this study, ZrO<sub>2</sub> based composites were fabricated by using 3D glassy carbon foam and equal volumes of carbon fiber through the Spark Plasma Sintering (SPS) method. The samples were characterized by using Scanning Electron Microscope (SEM) and X-ray diffraction to examine the cross sectional microstructure of the samples and identify the phases of the sintered samples, respectively. The effects of adding 3D glassy carbon foam and carbon fiber on the thermal conductivity and flexural strength of ZrO<sub>2</sub> were measured and compared. Finally, an optimized reinforcement material was chosen based on the analysis of the different types of carbon based reinforcements.

### 2. Experimental Procedure

ZrO<sub>2</sub> powder (Sigma-Aldrich) with an average size of 5 μm, vitreous carbon foam (Good Fellow, UK) with an open porosity of 96.5% and Carbon fibers (T700, provided by Toray) with total volume fraction of approximately 6.5% for each reinforcement were used as starting materials in this study. For carbon foam reinforced ZrO<sub>2</sub> composites, the mold was shaken by hands and tapped to assure the ZrO<sub>2</sub> powder is effectively filling the open pores of carbon foam. For carbon fibers reinforced composites, the carbon fibers

and the powder were mixed using ball mill (with 1:10 ratio of the powder to the ball) for 12 hours. Those materials were added to graphite mold (13 mm in diameter) sprayed with BN to prevent the powder from reacting with mold. The samples were sintered using SPS at a heating rate of 100°C/min at temperature of 1700°C for 10 min. Cylindrical shaped specimens for each reinforcement were prepared according to method described elsewhere [6] to measure the thermal conductivity via the laser flash method. The hardness for each specimen were measured using the Vickers hardness tester.

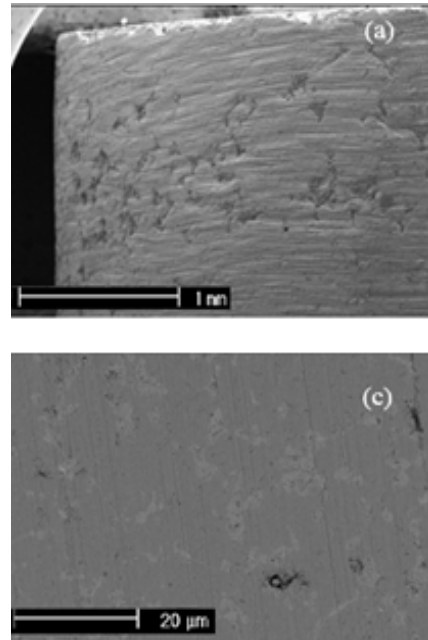


Fig. 1. SEM images of the cross sectional area of the sintered composites reinforced with (a) carbon foam and (b) carbon fiber.

### 3. Results

SEM images of the cross section of carbon foam and carbon fiber reinforced ZrO<sub>2</sub> sintered composite samples are shown in Fig. 1. For carbon foam reinforcement, the interface between the carbon foam and the ZrO<sub>2</sub> was identified and the microstructure of the carbon foam was retained during sintering (Fig.1 a). This might be due to the effective filling of ZrO<sub>2</sub> in the open pores of the carbon foam skeleton. Moreover, the foam layers were homogeneously distributed in the sintered sample. The carbon fibers (Fig.1 c) were not seen as straight long strands in the sintered

microstructure. However, they were seen as agglomerated grey phases entangled in shape. This may be due to the effect of impact of the balls during the mixing process or phase transformation which is accompanied by a change in volume.

Fig.2 shows the XRD analysis of carbon foam, and carbon fiber reinforced composites. As can be seen from Fig.2, no new phase was detected in the carbon foam reinforced composite, while ZrC was detected in carbon fiber reinforced  $ZrO_2$ . Several studies have published about the carbothermal reduction with carbon materials and the main factors that affect the carbothermal reduction process [7-9]. They found that the structure of carbon had an important effect on the carbothermal reaction. Specifically, the larger the surface area and the lower the degree of graphitization, the higher the reactivity of the carbon material. The low surface area of the network of carbon foam makes the carbothermal reaction less likely.

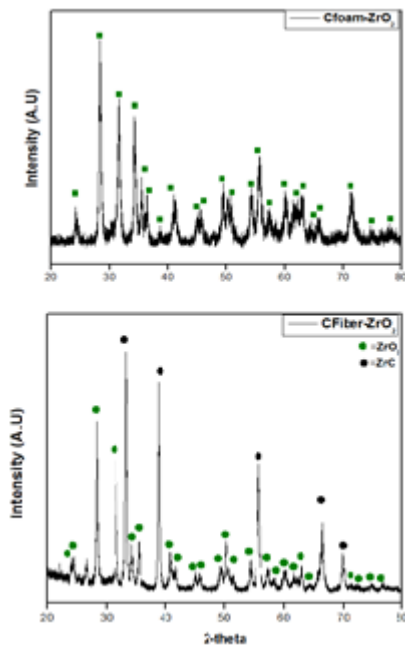


Fig. 2. XRD analysis for three layer carbon foam and carbon fiber reinforced  $ZrO_2$  composites

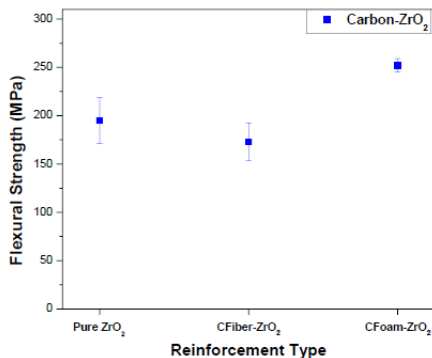


Fig. 3. Flexural strength of the composites

The flexural strength values for composites reinforced with carbon foam, and carbon fiber are shown in Fig.3. The flexural strength of  $ZrO_2$  changed slightly with the addition of reinforcements. Composite reinforced with carbon fibers showed lower values than its counterparts due to the agglomeration of ZrC, a hard and brittle phase which acts as crack initiation sites and serves for premature fracture.

The thermal conductivity of reinforced  $ZrO_2$  composites measured are shown in Fig. 4. Overall, the values of thermal conductivity were enhanced by the addition of carbon reinforcements. The carbon foam reinforced composites showed the highest thermal conductivity of  $3.388 \text{ Wm}^{-1}\text{K}^{-1}$  at  $1100^\circ\text{C}$ . The carbon fiber reinforced composites, due to the formation of ZrC, showed relatively lower values of thermal conductivity than carbon foam reinforced.

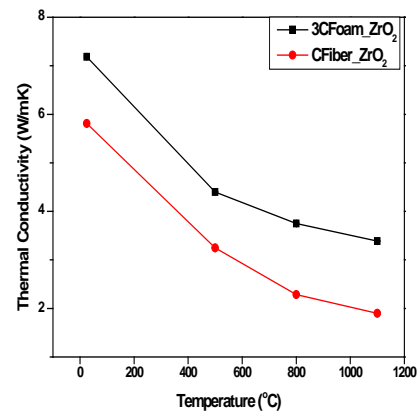


Fig. 4: Thermal conductivity of carbon foam or carbon fiber reinforced  $ZrO_2$  composites.

#### 4. Conclusions

Carbon foam and carbon fiber reinforced  $ZrO_2$ -based composites were fabricated using spark plasma sintering at  $1700^\circ\text{C}$ . The results indicated a general improvement in the thermal conductivities of  $ZrO_2$  based composites and a little increase in the mechanical properties.

The SEM images of the reinforced composites showed that the 3D network structure of the carbon foam was not damaged during sintering and the  $ZrO_2$  powder effectively filled the open pores of the carbon foam. In addition to that no cracks were seen in the composites and the interfaces between the carbon foam and  $ZrO_2$  matrix were intact.

The carbothermal reduction of  $ZrO_2$  into ZrC has an important effect on the thermal and mechanical properties of  $ZrO_2$  based composites. It caused the degradation of the material properties. In this study the carbon foam did not react with  $ZrO_2$  whereas carbon fiber reacted with  $ZrO_2$ . Therefore, carbon foam reinforced  $ZrO_2$  composites showed a better thermal and mechanical properties than carbon fiber reinforced composites.

The 3D network structure of the carbon foam had a positive effect on the improvement of the thermal properties of the reinforced ZrO<sub>2</sub> composites. It provides effective pathways for the heat transfer and homogenous distribution for the carbon foam reinforced ZrO<sub>2</sub> composites.

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