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An investigation into the microstructural and mechanical properties of the ZrB$_2$/SiC composites prepared by silicon infiltration

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Abstract

In this study, the synthesis of ZrB$_2$/SiC composite was carried out via infiltration of silicon melt into a ZrSiO$_4$/B$_4$C preform, and the effect of different ratios of ZrSiO$_4$/B$_4$C and C/ZrSiO$_4$ was investigated on phase and microstructural properties. For this purpose, stoichiometric ratios of raw materials and 10 wt% phenolic resin were used to induce the porosities in the preform. Then the powder mixture was milled and pressed. To perform pyrolysis reaction, it was placed in a vacuum-controlled atmosphere furnace with argon at 650 °C and then the Si infiltration process was performed at 1650 °C for 1 h. The hardness, density, and elastic modulus of the samples were measured. The optimum results were obtained for the composite sample with a ZrSiO$_4$/B$_4$C ratio of 4, which had a density of 5.28 g cm$^{-3}$, elastic modulus of 423 GPa, and hardness of 33.28 GPa. Moreover, scanning electron microscopy and x-ray diffraction analyses confirmed a uniform distribution of ZrB$_2$ and SiC phases and the absence of undesirable phases in the sample, respectively.

1. Introduction

Recently, materials for high-temperature applications have gained a lot of attention especially for power stations, aerospace, and military [1]. ZrB$_2$/SiC composites are considered as candidates for high-temperature servicing conditions due to their unique abrasion resistance and relatively low thermal expansion, which can be used for thermal barrier coatings of gas-turbine blades [2]. Nevertheless, oxidation of refractory diborides in the air at high temperatures has limited their applications. SiC is one of the most popular additives employed to improve the oxidation resistance of ZrB$_2$ in the range of 10–30 vol%, which creates coherent surficial layers of SiO$_2$-based glasses on the ceramics [3, 4]. This continuous scale provides passive oxidation protection at temperatures around 1000 °C–1600 °C [5, 6]. Furthermore, the addition of SiC particles to ZrB$_2$ ceramic matrixes improves the mechanical properties of ZrB$_2$ composite materials [7, 8].

Nayebi et al [9] investigated the effect of temperature on microstructural changes during hot pressing of ZrB$_2$–30 vol%SiC composite. The results showed that the compaction of samples occurs at 2000 °C via the atomic diffusion mechanism. Zamharir et al [10] investigated the mechanical properties of ZrB$_2$–25 vol%SiC composites. The results showed that the fracture toughness of the sample exposed to 16 MPa for 90 min at 1850 °C was 5.9 MPa.m$^{1/2}$. Mashhadi et al [11] studied the effect of SiC addition on the preparation of ZrB$_2$-SiC/Si by pressureless sintering technique under argon atmosphere at 2000 °C–2200 °C for 1 h. The highest relative densities and hardness of 98.12% and 15.02 GPa were reported for sample ZrB$_2$/10 wt%SiC$_n$ sintered at 2200 °C. By the addition of SiC up to 15 wt%, the maximum relative density and hardness of 96% and 14.08 GPa were obtained, respectively. Asl et al [12] studied the hardness and toughness of ZrB$_2$/SiC composites subjected to hot pressing under 30 MPa pressure min at 1700, 1850, and 2000 °C for 30 with different SiC contents (15–30 vol%). The highest values of hardness and fracture toughness of 21.3 GPa and 4.7 MPa.m$^{1/2}$ were observed at 2000 °C.
for samples containing 30 vol% SiC, respectively. An earlier investigation into the crack propagation in ZrB2-SiC composites in the presence and absence of additives showed that the failure mechanism of ZrB2, which has low fracture toughness is joining of intra-granular cracks to form larger transverse cracks along the grain boundary without deviation. By the addition of a small amount of SiC a deviation occurs in the propagation path of the cracks, resulting in a significant increase in the fracture toughness of ZrB2/SiC composites [13].

Due to the complication and cost of using high-temperature furnaces with controlled atmosphere, strong covalent bonding of ZrB2 and SiC and their low self-diffusion coefficients, hard ZrB2 ceramic matrixes containing SiC particles are typically compressed by hot-press and spark plasma sintering techniques [14]. The simple shapes, which are prepared by hot-pressing are required to be machined to the final desirable geometries by high-cost diamond machining processes. Recent investigations demonstrated that ZrB2/SiC composites could reach near theoretical density using pressureless sintering that obviates one of the limitations of these ceramic materials with great strength and high hardness [15].

In this paper, the advantageous infiltration technique was used for the preparation of ZrB2/SiC composites. For this purpose, preforms with different ZrSiO4/B4C and C/ZrSiO4 ratios containing phenolic resin were fabricated and infiltrated by silicon melt. The procedure was maintained as facile as possible to maximize cost efficiency. Finally, the microstructure and mechanical properties of the samples were characterized to determine the optimum preparation conditions.

2. Materials and methods

2.1. Materials
For the preparation of ZrB2/SiC composite specimens, ZrSiO4 and B4C powders with 98% purity and average particle sizes of 146 μm, Novolac phenolic resin as the binder and production of porous structure, and activated carbon with a purity of 99% and 22 μm particle size were used.

2.2. Preform preparation
The required amount of each raw material was determined according to equation (1), which is shown in table 1.

\[2ZrSiO_4 + B_4C + Si + 10C \rightarrow 2ZrB_2 + 3SiC + 8CO\] (1)

The powders of raw materials were milled and mixed with 10 wt% phenolic resin by a ball mill. For the milling process, ZrO2 balls with a ball-to-powder weight ratio of 20:1 were employed at the speed of 150 rpm for 90 min. To remove the moisture, the powder mixture was dried at 80 °C for 4 h. To fabricate the preforms, the samples were pressed under the load of 80 MPa in a steel mold for 10 s.

2.3. Pyrolysis
Pyrolysis was performed in a furnace under a vacuum pressure of 600 mmHg and by importing argon gas at a heating rate of 10 °C min⁻¹. The specimens were then kept at 600 °C for 1 h. Finally, the samples were cooled in the furnace at a rate of 5 °C min⁻¹ to prevent cracking.

2.4. Infiltration process
Samples were placed in graphite containers and insulated with graphite sheets. To perform the infiltration process, the silicon melt was kept at 1650 °C and for 1 h under 600-mmHg vacuum pressure. Argon gas was used to infiltrate the melt into the porous preform. The heating and cooling rates were 10 °C min⁻¹. A schematic of the infiltration process is shown in figure 1.

2.5. Elastic modulus
The pulse-echo method was used to measure Young’s modulus of the composites according to ASTM E797–95 standard using a TG600 ultrasonic thickness gauge at a frequency of 5 MHz (equation (2)).

\[E = \rho V^2\] (2)

where E is the modulus of elasticity (GPa), ρ is the density of the sample (g cm⁻³), and V is the speed of sound in the sample (m s⁻¹).

| Table 1. The chemical ratios of ZrSiO₄/B₄C and C/ZrSiO₄ of the samples. |
|-------------------------|----------------|----------------|----------------|----------------|----------------|----------------|----------------|
| Sample | ZB1 | ZB2 | ZB3 | ZB4 | CZ1 | CZ2 | CZ3 | CZ4 |
| ZrSiO₄/B₄C | 1.8 | 2 | 2.2 | 2.4 | — | — | — | — |
| C/ZrSiO₄ | — | — | — | — | 5 | 6 | 7 | 8 |
2.6. Hardness

The hardness measurement was performed by Vickers microhardness tester (Husser model) according to ASTM C1327 standard under a load of 300 gF. To increase the precision of the data, the test was replicated five times for each sample and the average value was reported.

2.7. Characterizations

The x-ray diffraction (XRD) characterization was performed by an XRD machine (Philips model PW1800, Netherlands) using the Cu-Kα beam (λ = 1.54 Å) and working voltage of 40 kV. The microstructure of the samples was studied by scanning electron microscope (SEM) (Tescan Vega-3 LMU) equipped with energy-dispersive x-ray spectroscopy (EDS).

3. Results and discussion

3.1. Density

Figure 2 shows the bulk density of the samples. According to figure 1 (a), with increasing the ratio of ZrSiO₄/B₄C from 1.8 to 2.4 in samples 1–4, the density was increased from 5.14 to 5.28 g cm⁻³. This increase in density was related to the formation of the ZrB₂ with a higher density of 6.11 g cm⁻³ compared with SiC density (3.2 g cm⁻³). The formation of a liquidus phase and activation of its related mechanisms was also contributed to the increase in density of samples. The increase in sinterability of the samples may also occur due to the formation of the non-stoichiometric ZrB₂ phase, which in turn increases the density. Silicon diffusion coefficient in B₄C and ZrSiO₄ structures is very low due to covalent bonds [14]. Deviation from the stoichiometric state increases the mass transfer due to structural changes or structural defects [16, 17]. With increasing the carbon content, the amount of SiC formed in the system increased, which was another factor in increasing the sample density.

In figure 2(b), by increasing the ratio of C/ZrSiO₄ from 5 to 8 in samples 5–8, the density remained approximately constant. As can be seen, the density of the ZB samples decreased compared to that of the ZB samples. The decrease in density can also be attributed to the increase in the amount of SiC reinforcement, improper distribution of the particles, and/or particle agglomeration [18–23]. In general, it can be concluded that increasing the amount of carbon through opposing factors affects the density, the enhancing factors include

![Figure 1. SEM micrographs of the raw materials: (a) B₄C, (b) ZrSiO₄, and (c) silicon.](image-url)
SiC-producing reactions and formation of the melt phase, while the reducing factors include reactions with gaseous products, formation of phases with densities lower than SiC, especially at high carbon contents. The interaction of these factors determines the density of the samples. In the cases of high carbon contents, reducing factors are dominant and decline the density [24].

3.2. Hardness

Figure 3 shows the hardness of the samples in terms of chemical composition variation. According to figure 3(a), the highest hardness (32.28 GPa) was obtained for sample 4, which in other ZB samples the low hardness is related to their lower density. By increasing the carbon content, silicon melt containing activated carbon, and the carbon networks in the preform, which remained from the process reacts to form hard $\beta$-SiC phase, which itself increases the hardness. The silicon melt rapidly reacts with the free carbon distributed in the structure, while in the absence of free carbon, the formation of SiC is dependent on carbon originating from the B$_4$C [25]. Therefore, as long as free carbon is present in the structure and the necessary thermodynamic and kinetic conditions are provided for the reaction of silicon melt with carbon, no significant change is seen in the composition of B$_4$C and the hardness will remain almost constant. Thus, due to the reduction of residual silicon and the formation of the $\beta$-SiC phase, the hardness increased with the increasing amount of activated carbon. Figure 3(b) shows the maximum hardness (24.48 GPa) for the CZ samples, which is related to sample 5. With increasing the ratio from 5 to 8, increasing the amount of carbon, and improper distribution of particles, the obtained data showed a decrease in the hardness, which was directly attributed to the decrease in the density of the samples. The reduction in hardness may also be due to the formation of microcracks in the intergranular areas between the ZrB$_2$, SiC, and carbon phases [1, 3]. In addition, in samples 5–8, as the amount of carbon increased, an origin for further plastic deformation during the process was provided, which reduced the hardness [26–28]. At temperatures above 1600 °C, as the secondary SiC phase gradually increased and the particles adhered to one another, the cavities were infiltrated by the diffusion mechanism, larger cavities were
formed, and some of these cavities were filled with silicon melt. And eventually, caused a decrease in the hardness of the reaction-bonded ceramics. Thus, with increasing the temperature, an increase in density can be seen, while at temperatures above 1600 °C the hardness decreases due to the formation of cavities, which is consistent with the results attained by Zhang et al [29]. Also, it was observed that the maximum hardness occurred in the samples with the highest density because porosities assist the formation and propagation of cracks. The results obtained from the hardness test were highly consistent with the density measurements.

3.3. Thermodynamic conditions
Phase transformation, reaction spontaneity, and the change in the Gibbs free energy for ZrB2/SiC system were investigated by HSC Chemistry Software (Version 5.0) [30–34]. As shown in figure 4(a), in the equilibrium state, when the temperature reached 1450 °C, the raw materials (ZrSiO4, B4C, and C) reacted and at higher temperatures, ZrB2 and SiC phases were produced. Figure 4(b) shows the Gibbs free energy changes of reactions in terms of temperature. As can be seen, as the sintering temperature increased, the free energy decreased and after a critical temperature, ΔG was shifted to negative values and the reaction occurred.

3.4. Phase analysis
XRD pattern of sample 1 with ZrSiO4/B4C ratio of 1.8 is shown in figure 5(a). As can be seen, after the sintering of the composite, ZrB2 and SiC phases are visible and the diffraction peaks of raw material phases are no longer observed. Additionally, it can be deduced that no secondary phase was formed during the sintering. Figure 5(b) shows the XRD pattern of sample 4 with the ZrSiO4/B4C ratio of 2.4. As in the case of sample 1, in the XRD pattern of sample 4, no evidence of raw materials phases was observed. As can be seen, ΔG of reaction is negative for the reaction of raw materials and formation of ZrB2 and SiC phases, which indicates the completion of the reaction. By comparing the XRD patterns of both ZrSiO4/B4C ratios, it can be seen that although the amount of
silicon remained constant and the amount of carbon increased, the intensity of the peaks were increased, which was attributed to a higher degree of crystallization. In addition, due to the increase in carbon content in sample 4, the amount of SiC phase increased compared to the ZrB₂ phase because of the increased affinity of silicon to bond with carbon. The results of phase percentages of the samples are brought in Table 2, which shows that in addition to the complete reaction of raw materials and the formation of the desirable phases with the optimum contents, the amount of SiC phase is slightly higher due to the presence of excess carbon in the system and its reaction with molten silicon infiltrated into the preform.

Figure 6(a) shows the XRD pattern of sample 5 with a C/ZrSiO₄ ratio of 5. As can be seen, the peaks related to the formation of the main phases of ZrB₂ and SiC can be detected. Also, since the formation of ZrB₂ and SiC is more thermodynamically favored than that of ZrC, and these compounds have higher stability than ZrB₂, the products form ZrB₂ and SiC during the silicon infiltration and was not detected by XRD analysis to indicate the presence of ZrC. However, there are not many additional peaks that can indicate the formation of phases with small quantities during the sintering process. Figure 6(b) shows the XRD pattern of sample 8 with a C/ZrSiO₄ ratio of 8 is given in figure 6(b). By comparing the diffraction pattern of sample 8 with that of sample 5, as it was expected, due to the balanced chemical equation in sample 8 and since the ration of ZrSiO₄/B₄C was constant with increasing the amount of Si and C, the SiC phase fraction was higher than that of ZrB₂. XRD results for CZ samples and determination of phase percentage of raw materials and products are listed in Table 2, which shows that by increasing the amount of carbon, the amount of SiC phase was improved 150% and 450% in sample 5 and 8 compared to that of ZrB₂, respectively.
Figure 5. XRD patterns of: (a) sample 1 with ZrSiO$_4$/B$_4$C ratio of 1.8 and (b) sample 4 with ZrSiO$_4$/B$_4$C ratio of 2.4.

Table 2. Phase quantification of the samples based on the XRD analysis.

| Sample     | Ratio | ZrB$_2$ (wt%) | SiC (wt%) |
|------------|-------|---------------|-----------|
| ZrSiO$_4$/B$_4$C | 1.8   | 48            | 52        |
| C/ZrSiO$_4$ | 5     | 42            | 58        |
| 8          | 40    | 24            | 76        |

3.5. Microstructure observations

Figure 7(a) shows the SEM micrograph of the composite sample with a ZrSiO$_4$/B$_4$C ratio of 1.8. As can be seen, the growth of ZrB$_2$ and SiC grains is not evident at the sintering temperature. However, it can be seen that the ZB samples were completely sintered. A uniform distribution of ZrB$_2$ and SiC micro-sized particles was observed. According to EDS results, the bright and dark areas represent the ZrB$_2$ and SiC phases, respectively. The average particle size of 2–3 μm and the planar morphology is evident. In figure 7(a) a uniform distribution of SiC particles in the ZrB$_2$ matrix is seen, which also implies that there was no detectable sign of agglomeration in the sample. Besides, formation of a continuous network of B$_4$C and ZrB$_2$ is evident in the microstructure.

Figure 7(b) shows the SEM micrograph of sample 4 with a ZrSiO$_4$/B$_4$C ratio of 2.4. Among all the samples, sample 4 had the optimal properties, phase structure uniformity, and more desirable phase content. SEM observations demonstrated the composite grains with dimensions of 4–5 μm. As shown in the figure, the growth of ZrB$_2$ grains was not severe, which could be due to the formation of SiC between the ZrB$_2$ grains, i.e. the presence of SiC particles prevents the excessive growth of the grains. In addition, since there is a considerable
difference between the melting points of ZrB2 (3245 °C) and SiC (2730 °C), and the sintering temperature (1650 °C), the probability of particle growth at the sintering temperature during the very short process time with the presence of micro-sized particles in the sample is low. Furthermore, with increasing ZrSiO4/B4C ratio, the fraction of the ZrB2 phase decreased, while the fraction of the SiC phase increased. According to figure 7(b) it can be seen a uniform distribution of SiC particles in the ZrB2 matrix was obtained. So that there was no observable sign of agglomeration in the micrographs. Moreover, a uniform grain size distribution is evident in the figure and a morphology of symmetrical geometry is attained. The non-uniformity of symmetrical geometry morphology of the particles avoids the formation of areas with stress concentration, which are susceptible to residual stress and crack nucleation [1, 35]. Indeed, the distribution of particles improves the fracture toughness and increases the strength based on the particle morphology [24]. Another clear point in the figure is absence of porosities in the structure. In high-temperature ceramic materials prepared by conventional methods, a wide range of porosities with different sizes are produced due to the exhaustion of volatile materials such as fluorine [1, 35]. Whereas in the following technique the liquid phase fills the porosities and plays an effective role in density of the composite samples. The increase in density is one of the contribution factors to the strength. According to the EDS results, the undesirable phases were either non-existent or present in small and negligible quantities.

Figure 8(a) shows the SEM micrograph of sample 5 with C/ZrSiO4 ratio of 5. Considering the C/ZrSiO4 ratio and the dark areas in the figure, higher Si and C contents caused the formation of the SiC component. Large areas were assigned to Zr and B elements, which showed the formation of ZrB2 phase with a brighter color and planar morphology. Furthermore, the increase in the grain size by increasing the amount of additives was caused by the presence of a high fraction of liquid phase, which led to severe grain growth and this could be one of the
disadvantages of sintering in presence of a liquid phase. Uniform distribution of silicon was observed in dark areas, which improved the mechanical properties of the samples. In sample 5, the buildup of dark areas indicated the agglomeration of SiC particles. Moreover, the bright areas in figure could be due to the presence of free carbon in the composite microstructure. However, it can be seen that the presence of these agglomerates deteriorates the mechanical properties. As can be seen in figure 8(b), with increasing the C/ZrSiO$_4$ ratio and considering the XRD results regarding the high amounts of Si and carbon, the SiC phase is predominant. Also, due to the small size of the formed areas and analyzing the depth of $\sim 1.5 \mu m$ of the EDS, the presence of carbon in the analysis can be related to SiC. This can be observed in the light-gray areas in the matrix. It is apparent that the weight percentage of carbon in these areas is higher than that of the stoichiometric range of SiC, hence the amount of carbon in these areas is higher than the bright areas of the matrix. Likewise, the bright areas that contain zirconium and boron in the elemental analysis, the formation of ZrB$_2$ is confirmed. The particle size was in the micrometer scale and the morphology was planar.

3.6. Elastic modulus

Figure 9 show the elastic moduli of the composite samples. The highest modulus was 423 GPa, which was related to sample 4 with a ZrSiO$_4$/B$_4$C ratio of 2.4, which was confirmed by the density measurements. On the other hand, in samples containing ZB samples by increasing the amount of SiC compared to ZrB$_2$, the elastic modulus reduced from 384 to 365 GPa, which was attributed to the increase in agglomeration and decrease in density. Elastic moduli of ZrB$_2$ is higher than that of SiC. Consequently, for the ZrB$_2$–SiC composites the elastic moduli should be decreased by elevating the SiC content according to the rule of the mixtures. The growth of ZrB$_2$ grains is inhibited and suppressed by the dispersion of SiC particles. In fact, high-stress levels are required for the deformation of the grain-refined ceramic materials. According to the Griffith criterion, the flexural strength of the ceramics can be predicated using the square root of the size of the critical flaw. In the absence of larger-sized
flaws, the strength is inversely related to the square root of the average grain size, i.e. finer grain sizes result in higher flexural strength values.

As mentioned above, it seems that the SiC phase forms a network, which percolates the ZrB₂ structure in the presence of grain-refined particles. For this reason, the elastic modulus of bulk ZrB₂ is higher than that of the ZrB₂ coatings [35–37]. Low elastic moduli are frequently reported in investigations on nano-crystalline materials and thin films [38–40]. This is due to the fact that intrinsically a material stiffness is not dependent on length-scale; the decrease in the elastic modulus is usually affected by the crystallographic textures or structural defects. The reason can be attributed to the subsequent possibilities: porosities, microcracking, presence of the amorphous/nanocrystalline structure, and crystallographic texture [40, 41].

4. Conclusions

ZrB₂/SiC composites were fabricated by inducing porosities using phenolic resin and silicon infiltration at 1650 °C. According to XRD analysis, the desirable phases of ZrB₂ and SiC were formed during the sintering. According to SEM and XRD characterizations, when ZrSiO₄/B₄C ratio was selected as 4, the amount of ZrB₂ and SiC phases was optimum and uniform distribution of both phases was attained in the absence of any the undesirable phases. Microstructure analysis approved the SiC network structure in the ZrB₂ matrix as well as the diffusion of ZrB₂ phase around the SiC grains. As the amount of carbon increased, the predominant phase was SiC, which represented the existence of excess carbon in the matrix and increased the particle agglomeration. It was also observed that the grain size of SiC affected the grain growth and could refine the grain size.

For all samples, the density, modulus of elasticity, and hardness were increased by increasing the ZrSiO₄/B₄C ratio. Furthermore, with increasing the C/ZrSiO₄ ratio the physical properties (density) and mechanical properties (hardness and elastic modulus) decreased due to the enhancing the carbon content in the system, improper distribution of particles, and agglomeration phenomenon. For CZ samples, with increasing the amount of carbon, the fraction of the SiC phase was increased. Physical and mechanical tests confirmed that
the optimum properties (density of 5.28 g cm\(^{-3}\), elastic modulus of 423 GPa, and hardness of 33.28 GPa) were obtained for the composite with ZrSiO\(_4\)/B\(_4\)C = 4.

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