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Densification and Microhardness of Spark Plasma Sintered ZrB_2+SiC Nano-Composites*

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Ultra-High-Temperature Ceramics (UHTCs) such as ZrB_2 and HfB_2 with incorporation of SiC nano-filler are useful as structural materials for applications in propulsion and thermal protection systems such as turbine-engine hot section components, leading edge of hypersonic vehicles, where extremely high heat fluxes generate very high temperatures and steep temperature gradients [1]. Spark plasma sintering (SPS) technique is used for densifying the UHTCs under the influence of uniaxial pressure and pulsed direct current [2]. Here, we study the densification, grain growth, and microhardness of ZrB_2 nano-composites with 15% and 20% SiC consolidated using SPS.

Nano-powders of ZrB_2 (43 nm hexagonal 99% pure) and SiC (40 nm cubic 99% pure) were obtained from US Research Nanomaterials, Inc, Houston, Tx. The nano-powders of ZrB_2 with 15 or 20 vol% SiC were mixed in isopropanol using Thinky planetary mixer and ultra-sonication followed by drying in vacuum at 50°C. The Spark Plasma Sintering (SPS) is performed at 2,100 °C to 1,800 °C at 32-40 MPa at Wright-Patterson Air Force Base, Dayton, OH or at SPS NanoCeramics, LLC, Morton Grove, IL. We used SEM/EDXS for granular and elemental analysis, Archimedes method for density measurements, and microhardness tester for Vickers hardness.

Figure 1 shows the SEM view graph of SPS consolidated ZrB_2+15Vol%SiC nano-composite. The EDXS analysis revealed that though the SiC is distributed throughout the sample, the darker grains have higher SiC composition compared to the rest of the sample. The EDXS analysis also revealed that there is some oxygen content found on Zr rich whiter grains. The x-ray diffraction analysis (reported elsewhere) showed ZrO_2 phase due to oxidation of some Zr during SPS consolidation, which is minimized by Argon-gas purged prior to SPS consolidation. Table 1 shows the densification, microhardness, grain size, and EDX elemental distribution of SPS consolidated ZrB_2+15Vol%SiC and ZrB_2+20Vol%SiC nano-composites at different temperatures and pressures. The density of the composites seems to be influenced by the content of low dense SiC and ZrO_2 in the composites. Figure 2 shows the densification (compared to the theoretical value) dependence on the wt% elemental composition O/Zr ratio obtained from EDXS analysis. It indicates that the densification decreases with increasing ZrO_2 contents. Figure 3 shows that the microhardness is inversely proportional to the pressure and directly proportional to the temperature applied during SPS consolidation. Fig. 4 shows that the average grain size obtained from the SEM viewgraphs of the SPS consolidated ZrB_2+SiC nano-composites is proportional to both the pressure and the temperature used during the SPS consolidation.

The results suggests that there is a need to optimize the SPS parameters to reduce the SiC segregation in order to obtain uniform distribution of SiC throughout the ZrB_2+SiC composite, and eliminate Zr oxidation observed during SPS treatment.

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Table 1: Density, microhardness, grain size, and EDX elemental distribution in SPS consolidated ZrB_2+15Vol%SiC and ZrB_2+20Vol%SiC nano-composites

SiC (Vol%)	Pressure (MPa)	Temp. (°C)	Densification (%)	Hardness (VHN)	Grain Size (µm)	EDX wt% O/Zr
20	40	1950	88.2	1242	2.1	0.033
20	35	1850	86.2	1455	1.6	0.079
20	32	2100	87.2	2304	1.75	0.152
20	32	1800	75.9	1815	0.6	0.330
15	35	1850	89.7	1819	1.78	0.331
15	35	1850	92.2	1845	1.61	0.032
15	32	1800	75	2094	0.544	0.022

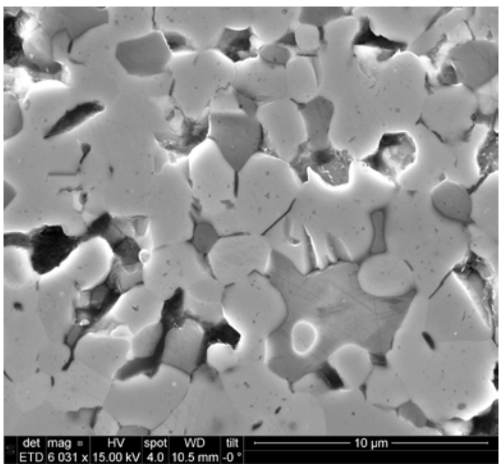

Figure 1: SEM of SPS consolidated ZrB_2+15Vol%SiC nano-composite.

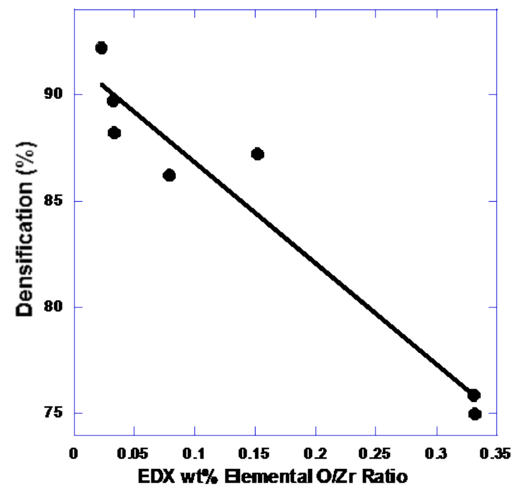

Figure 2: Densification vs O/Zr EDX wt% ratio

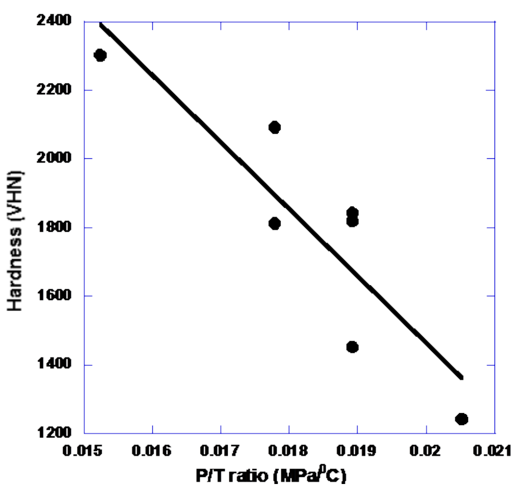

Figure 3: Hardness vs Pressure/Temperature.

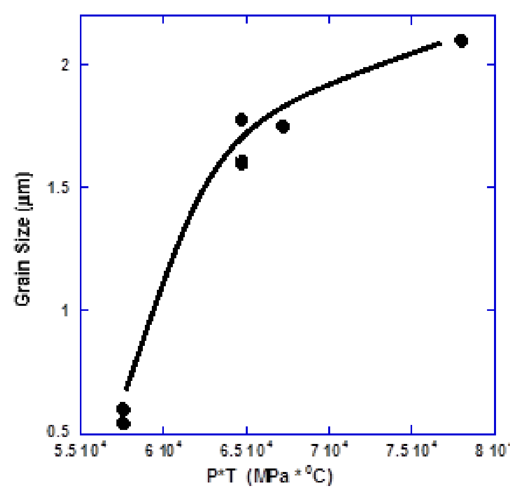

Figure 4: Average grain size vs P*T product.

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