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Densification of B_4C composite through reactive spark plasma sintering using mechanically activated Ti-B reactive mixture as a sintering aid

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High temperature ceramics have wide range of structural, aerospace and energy applications due to their thermal stability, corrosion resistance and superior mechanical properties. B_4C is one of the high temperature ceramics having superior hardness (>35 GPa) along with low density (2.52 $g.cm^{-3}$), high melting point (2763 °C), good wear resistance, corrosion resistance, ballistic efficiency and neutron absorption capacity. Due to its superior properties, it is widely used in wear resistant, neutron absorption and body armor applications in nuclear and defense sectors. However poor sinterability and low fracture toughness (≈ 3 MPa $m^{1/2}$) are the major limitations of B_4C applications. Due to the strong B-C covalent bonding, it is extremely difficult to sinter it below 2000 °C using conventional sintering techniques. Spark Plasma Sintering (SPS) is one of the most promising techniques to sinter the high temperature ceramics at relatively lower temperatures with the advantages of shorter sintering time and limited grain growth.

In the present study, dense Boron carbide was prepared using mechanically activated Ti-B reactive mixture as sintering aid. Ti and B elemental powders were ball milled for 8 h. This Ti-B powder was mixed with Boron carbide in the proportions of 5, 10, 20 wt.% and milled for another 4 h for good dispersion. Spark plasma sintering was carried out at 1400°C, at 50 MPa pressure with 100 °C/min heating rate. Archimedes water immersion method confirmed the samples were dense. X-Ray diffraction studies showed that some amount of WC was picked up during milling. (Ti,W)C, (Ti,W)B_2 phases were observed in XRD. Scanning electron microscopy revealed the distribution and morphology of these phases. EDS studies confirmed the formed phases. Mercury intrusion porosimetry revealed the nano pores with mean pore diameter of 15.4 nm. The B_4C-5 wt.% TiB_2 composition showed highest hardness of 32.2 ± 2.2 GPa.