Microstructural characterization of mechanically alloyed and spark plasma sintered Si-C-B ceramics

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SiC and B_4C ceramics are characterised by their extreme hardness and difficult to sinter to high relative densities without the use of sintering aids. Ball milling can produce metastable structures, and the storage energy during milling can contribute to reduce the sintering temperature. This work reports on the microstructural characterization of Si-C-B ceramics produced by high-energy ball milling and subsequent spark plasma sintering. The elemental powder mixtures of 8SiC:1B_4C and 2SiC:1B_4C (mol-%) were initially milled for 90 h in a planetary Fritsch P-5 ball mill under argon atmosphere using hardened steel vials (225 mL) and balls (12mm diameter), rotary speed of 200 rpm, and a ball-to-powder weight ratio of 10:1. Following, the powders milled for 90 h were sintered at 1300^{o}C for 10 minutes using 20 MPa in a DR. SINTER LAB TM type SPS 211LX (Fuji Eletronic Industrial Co LTD). The starting powders and sintered samples were evaluated by means of X-ray diffraction, electron scanning microscopy, energy dispersive spectrometry, thermal analyses (DSC/TG), and Vickers microhardness. Absence of intense Si, C and B peaks was noted in XRD patterns of starting powders indicating that they are composed of metastable structures such as nanocrystalline and/or amorphous. Dense samples containing a small amount of pores were found after SPS procedure. SEM images also indicated that the 8SiC:1B_4C and 2SiC:1B_4C (mol-%) ceramics presented homogeneous microstructures formed by the SiC and B_4C phases. Coherently, the amount of B_4C precipitates increased in microstructure of the richer-B ceramic.