

STEM-EDX and TEM-PED Characterizations and Directional Properties of Hot Pressed B₄C-TiB₂ Based Composites Doped With WC

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Boron carbide (B₄C) is one of the important non-oxide ceramic material due to its superior physical and chemical properties such as extreme hardness and low thermal conductivity. In order to improve the sinterability of B₄C, sintering additives such as carbon, aluminum, silicon carbide, titanium diboride (TiB₂) phases are added as sintering aid and these additives also improves the mechanical properties.

Aim of this study is the chemical and structural characterization of the solid solution formed during sintering of B₄C-TiB₂ composite and to understand its' formation mechanism as well as effect of uniaxial pressure during sintering over microstructure and mechanical properties. In this study, three different B₄C-TiB₂ (50:50 vol%) composite mixtures were used; (i) ball milled and (ii) high energy milled and (iii) ball milled B₄C-TiB₂ doped with 5 volume % of tungsten carbide (WC). All powder mixtures were sintered with hot pressing (HP) method and characterized by conventional SEM, TEM and STEM imaging techniques and additionally Precession Electron Diffraction (PED) technique is applied to characterize present phases as well as orientation relationship between the different phases. For this purpose, TEM samples were prepared by conventional preparation techniques that is mechanical polishing followed by Ar-ion beam milling as well as focused ion beam milling. Electron transparent sample is examined by using 200 keV field emission TEM (JEOL-JEM2100F) equipped with STEM high angle annular dark field (HAADF) detector (Fischione-Model 3000) and energy dispersive X-ray (EDX) spectrometer (JEOL-JED2300T). The TEM phase and orientation mapping results were obtained with nano sized probe (at NBD alpha 5 and 0.5 nm spot size) that was scanned in a user specified area over the sample up to 0.7° of precession angle and obtained diffraction patterns were analyzed with ASTAR™ V2 via template matching algorithm.

SEM analyses (as given in Figure 1) showed that, microstructure and present phases varies depending on the processing conditions. Significant effect of high energy milling on the particle sizes was observed. All samples mainly consist of TiB₂ and B₄C phases however, depending on milling conditions and WC addition Ti-W-B containing phases with different amounts of W were observed around TiB₂ particles as core-rim structure. Core-rim structures around TiB₂ particles were characterized in detail with STEM-EDX analyses as given in Figure 2. Chemical compositions of rim structures were obtained with EDX analyses as well as PED method. Phase mapping results of these structures support the EDX results. Orientation mapping in TEM also showed that there is a special orientation relation present between core and rim structures.

Furthermore, production methods that include uniaxial pressing may end up with anisotropic properties of the produced materials, therefore microstructure and mechanical properties depending on the sample orientation relative to the pressing direction were investigated. According to the SEM images it was found that morphology and microstructure varies depending on the pressing direction and this difference also present in XRD analyses. Effects of texture on the mechanical properties were measured by hardness and indentation toughness depending on pressing direction and will be presented.

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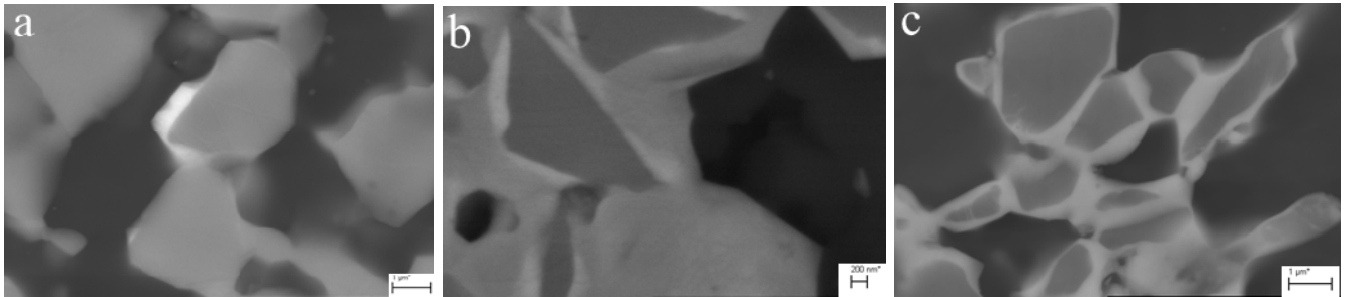


Figure 1. SEM backscattered electron images of (a) ball milled, (b) high energy milled and (c) WC added samples.

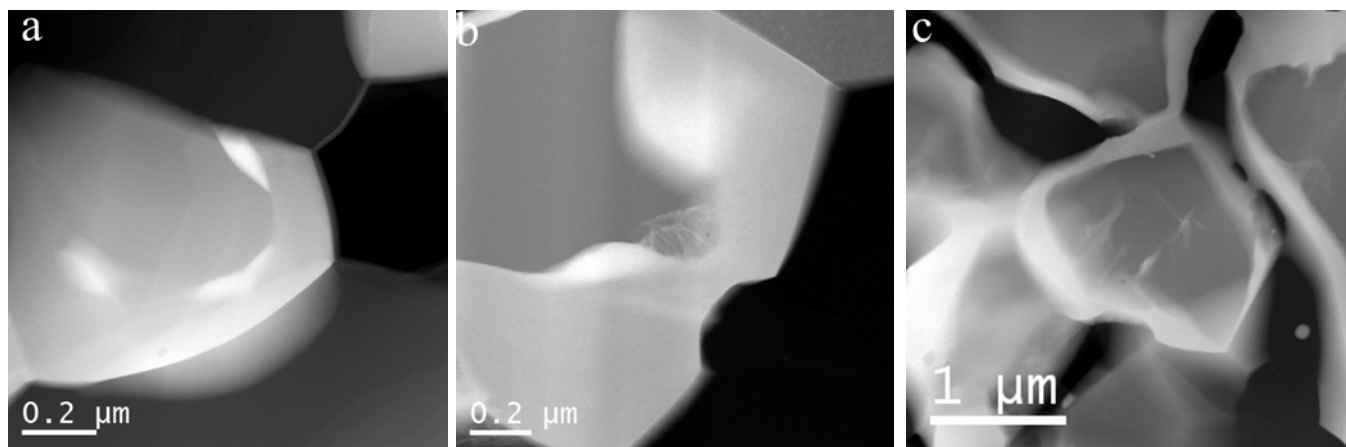


Figure 2. STEM-HAADF images of (a) ball milled, (b) high energy milled and (c) WC added samples.