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Toughness control of boron carbide obtained by spark plasma sintering in nitrogen atmosphere

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Abstract

Boron carbide ceramic was prepared by reactive Spark Plasma Sintering under N_2-atmosphere and for different heating times and maximum pressure regimes. Split-Hopkinson Pressure Bar (SHPB), indentation, XRD and microscopy measurements were performed for samples characterization. It is shown that SHPB toughness control depending on SPS regime is possible and the main reason is introduction of nitrogen into B_4C ceramic. Complex relationships between processing conditions, sintering mechanism, material's specifics, static and dynamic mechanical properties are discussed. Improvement of dynamic toughness is through mechanisms resembling those working for static load conditions such as cracks deflection and pull out, but there are also significant differences.

Keywords: C. Hardness; Boron carbide; SPS; Toughness; SHPB

1. Introduction

Boron carbide (B_4C) is a hard, light-weight, refractory and stable compound useful for different applications [1–3]. Further investigation and development of B_4C-based materials is of much interest. The biggest challenge is to improve toughness of B_4C-based ceramic well known to be a brittle material. Many applications require good mechanical properties under static, dynamic or impact strain-rate mechanical load regimes [4]. A simple and popular strategy for ceramics in general is to generate composites [5,6]. Improvement of toughness usually refers to fracture toughness, i.e. K_{1c}, determined in the static stress–strain load regime and very few data are available for B_4C-based composites in the dynamic or impact regions.

In this work, we present Split-Hopkinson Pressure Bar (SHPB) compressive test and Vickers indentation results on B_4C-based composite ceramic produced by reactive Spark Plasma Sintering (SPS) in N_2 atmosphere and under different regimes (Table 1). Selection of SPS was made considering literature data showing that pulsed current application during SPS processing may generate useful unconventional effects [7] leading to different results [8,9] when compared with resembling hot-pressing that applies conventional heating inside an electrical furnace. In our recent article [10] we have shown that reactive SPS of B_4C in N_2-atmosphere produces a composite in which at grain boundaries a fine mesh-like network of BN (with hexagonal crystal structure according to XRD) wrapped and intercalated by B_xO_y forms. Modified boundaries are expected to provide ductility to B_4C-based composite. Our work demonstrates that through different SPS regimes, the composite can be controlled from the viewpoint of dynamic (SHPB) maximum stress (σ_{max}), maximum strain (ε_{max}) and toughness (S) (Table 1).

2. Experimental

Powder of B_4C (Sinopharm Chemical Reagent Co. Ltd., Singapore, BET specific surface area and average particle size

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Table 1
Samples, SPS regime, static and dynamic mechanical properties parameters, optical microscopy results on samples after SHPB test and crystallite size from XRD.

S	Pressure, *P* [MPa], (atmosphere) and SPS time [min]	*HV* [GPa]	K_{1c} [MPa.m$^{1/2}$]	SHPB max stress, σ_{mas} [MPa]	SHPB max. Strain ε_{max}	SHPB toughness from area *S* [MJ/m^2]	*n*-value	Observations from the optical microscopy after SHPB test (see Fig. 2 a–f)	Crystallite size from XRD [nm]
U	60 (Ar)/ 6 min	22.51	2.21	740	0.0117	6.50	–	Relatively uniform fraction of middle size fractured pieces	101
V	60 (N2)/ 6 min	34.9	4.56	1223	0.0209	18.33	–	Relatively uniform fraction of small size fractured pieces	111
Z	60 (N2/20 min to 1100C and 5 min to 1800C in Ar)	24.4	6.13	522	0.0278	10.44	–	Relatively uniform fraction of small size fractured pieces	99.8
A1	30/(N2) 25 min	27.4	3.17	713	0.0134	6.52	1.50	Relatively uniform fraction of middle size fractured pieces	163
A2	30/(N2) 25 min	–	–	723	0.0124	5.77	–		
B1	60/(N2) 25 min	35.6	4.18	925	0.0209	12.95	2.39	Two fractions very different: 1. Few very large pieces	146
B2	60/(N2) 25 min	–	–	1185	0.0199	14.22	–	2. Fraction of small pieces	
C1	100/(N2) 25 min	34	5.31	1014	0.0598	29.83	2.37	Two fractions very different: 1. Needle-like pieces	131
C2	100/(N2) 25 min	–	–	1270	0.0593	30.93	–	2. Signicant fraction of very small pieces.	
A*	30/(N2) 40 min	24.8	7.55	701	0.0338	15.04	2.32	Two fractions: 1. Relatively uniform fraction of middle size fractured pieces as for A1, 2 2. Fraction of very small particles as for C1, 2	99
B*	60/(N2) 40 min	35.2	6.18	1153	0.0319	20.70	2.37	Two fractions: 1. Relatively uniform fraction of middle size fractured pieces as for A1, 2 2. Fraction of very small particles as for C1, 2	108