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**Mechanical properties of hot-pressed high-entropy diboride-based ceramics**

Ji-Xuan LIU  
*State Key Laboratory for Modification of Chemical Fibers and Polymer Materials, College of Materials Science and Engineering, College of Sciences, Institute of Functional Materials, Donghua University, Shanghai 201620, China*

Xiao-Qin SHEN  
*State Key Laboratory for Modification of Chemical Fibers and Polymer Materials, College of Materials Science and Engineering, College of Sciences, Institute of Functional Materials, Donghua University, Shanghai 201620, China*

Yue WU  
*State Key Laboratory for Modification of Chemical Fibers and Polymer Materials, College of Materials Science and Engineering, College of Sciences, Institute of Functional Materials, Donghua University, Shanghai 201620, China*

Fei LI  
*State Key Laboratory for Modification of Chemical Fibers and Polymer Materials, College of Materials Science and Engineering, College of Sciences, Institute of Functional Materials, Donghua University, Shanghai 201620, China*

Yongcheng LIANG  
*State Key Laboratory for Modification of Chemical Fibers and Polymer Materials, College of Materials Science and Engineering, College of Sciences, Institute of Functional Materials, Donghua University, Shanghai 201620, China*

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Authors
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Ji-Xuan LIU, Xiao-Qin SHEN, Yue WU, Fei LI, Yongcheng LIANG, Guo-Jun ZHANG*

State Key Laboratory for Modification of Chemical Fibers and Polymer Materials, College of Materials Science and Engineering, College of Sciences, Institute of Functional Materials, Donghua University, Shanghai 201620, China

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Abstract: High-entropy ceramics attract more and more attention in recent years. However, mechanical properties especially strength and fracture toughness for high-entropy ceramics and their composites have not been comprehensively reported. In this work, high-entropy (Ti0.2Zr0.2Hf0.2Nb0.2Ta0.2)B2 (HEB) monolithic and its composite containing 20 vol% SiC (HEB–20SiC) are prepared by hot pressing. The addition of SiC not only accelerates the densification process but also refines the microstructure of HEB, resulting in improved mechanical properties. The obtained dense HEB and HEB–20SiC ceramics hot pressed at 1800 °C exhibit four-point flexural strength of 339±17 MPa and 447±45 MPa, and fracture toughness of 3.81±0.40 MPa·m^{1/2} and 4.85±0.33 MPa·m^{1/2} measured by single-edge notched beam (SENB) technique. Crack deflection and branching by SiC particles is considered to be the main toughening mechanisms for the HEB–20SiC composite. The hardness \(H_v\) of the sintered HEB and HEB–20SiC ceramics is 23.7±0.7 GPa and 24.8±1.2 GPa, respectively. With the increase of indentation load, the hardness of the sintered ceramics decreases rapidly until the load reaches about 49 N, due to the indentation size effect. Based on the current experimental investigation it can be seen that the room temperature bending strength and fracture toughness of the high-entropy diboride ceramics are within ranges commonly observed in structure ceramics.

Keywords: high-entropy ceramics; high-entropy diboride; flexural strength; fracture toughness; indentation size effect

1 Introduction

Entropy is an important thermodynamic parameter of a matter. Usually, high-entropy causes low Gibbs free energy and consequently results in high thermal stability of the materials thermodynamically. Based on this principle, a new group of materials—high-entropy materials with equimolar or near-equimolar proportions of five or more elements have been developed in recent years [1,2]. A number of high-entropy ceramic materials have been reported, including high-entropy borides [3–8], high-entropy carbides [9–12], high-entropy oxides [13–16], high-entropy silicides [17,18], high-entropy fluoride [19], as well as high-entropy metal oxycarbonate...
[20]. After the first report by Gild et al. [3] in 2016 on the high-entropy diboride ceramics, much attentions have been attracted on the preparation and characterization of this material, including synthesis [3–8], densification by spark plasma sintering (SPS) [3–6], and microstructure tailoring [5,8].

Mechanical properties are important references for the engineering design and application of a material. Recently we reported the mechanical properties including flexural strength and fracture toughness of (Ti0.2Zr0.2Hf0.2Nb0.2Ta0.2)C monolithic ceramics and its composite with the addition of 20 vol% SiC secondary phase [12]. The results demonstrate that the values of bending strength and fracture toughness at room temperature are typical of structure ceramics. However, the mechanical properties of high-entropy diboride ceramics have not yet been extensively evaluated except for the Vickers hardness as well as fracture toughness by indentation technique [3–7]. It is reported that high-entropy diboride ceramics even with ~8 vol% porosity exhibited higher hardness over the average of the corresponding five individual diboride ceramics [3]. For fracture toughness (KIC), only a few open publications reported values measured by indentation technique and the values are very scattering [4–6]. As the fracture toughness is very sensitive to the measurement techniques, comparable study via different techniques for the evaluation of fracture toughness is meaningful. On the other hand, up to now there is no open report concerning the flexural strength of high-entropy diboride ceramics.

Very recently, we investigated the effects of sintering temperature and the addition of SiC secondary phase on the microstructure of high-entropy (Ti0.2Zr0.2Hf0.2−Nboo.2Ta0.2)B2 ceramics. The results indicate that the addition of SiC secondary particles can obviously inhibit the grain growth of (Ti0.2Zr0.2Hf0.2−Nboo.2Ta0.2)B2 phase and refine the microstructure. However, limited by the sample size (Ø10 mm × 5 mm) prepared by our SPS equipment, only their hardness and fracture toughness were investigated by indentation method in our previous work [5]. In this study, firstly dense high-entropy (Ti0.2Zr0.2Hf0.2−Nboo.2Ta0.2)B2 (HEB) monolithic ceramics and its composite containing 20 vol% SiC (HEB–20SiC) with dimensions of 37 mm × 30 mm × 5 mm are prepared by hot pressing. Then the mechanical properties including Young’s modulus, bulk modulus, shear modulus, Poisson’s ratio, Vickers hardness, four-point flexural strength, and fracture toughness by single-edge notched beam (SENB) technique are systematically investigated. The effect of SiC secondary phase on the microstructure evolution and mechanical properties of hot-pressed HEB ceramics is also discussed.

2 Experimental procedure

2.1 Sample preparation

The following process was used to prepare the diboride-based ceramics. The laboratory-synthesized high-entropy diboride powder with an average particle size of 1.4 μm and residual oxygen content of 1.56 wt% was planetary ball milled without or with the addition of 20 vol% α-SiC powder (> 98.5%, ~0.45 μm) using WC balls as milling media (the ball milling process caused about 0.3 wt% loss of WC balls). Details of the synthesis process for the high-entropy diboride powder and the preparation of powder mixture for the HEB–20 vol% SiC (HEB–20SiC) composite were reported in our previous publication [5]. The obtained powders were put in BN coated graphite mold and then hot pressed at 1800 °C for 1 h in an argon atmosphere (ZT-40-21Y, Shanghai Chen Hua Science and Technology Co. Ltd., China). The as-sintered samples were sectioned into bars followed by grinding and polishing to 0.5 μm diamond paste. The four edges were beveled using 1500 grit diamond disk.

2.2 Sample characterization

The densities of the sintered samples were measured by Archimedes method. To evaluate the relative density (R.D.) of the sintered samples, the theoretical density of HEB was calculated according to its lattice constants. The phase composition of the sintered samples as well as the crystalline cell parameters were studied by X-ray diffraction (XRD, D/max-2550VB+/PC, Rigaku Corp., Tokyo, Japan) with Cu Kα radiation using Si as an internal standard. Field emission scanning electron microscope (FE-SEM, S-4800, Hitachi High-technologies Corp., Tokyo, Japan) equipped with energy dispersive spectroscopy (EDS) was used for analyzing the microstructures of the sintered samples.

According to ASTM standard C1161-18, four-point flexural strength (σ) of the sintered ceramics was measured by Shimadzu universal testing machine (AGS-X, Shimadzu Corp., Tokyo, Japan) using type A bars with dimensions of 1.5 mm × 2 mm × 25 mm. The
inner and outer spans used for the testing were 10 and 20 mm, respectively. The Young’s modulus \((E)\), bulk modulus \((B)\), shear modulus \((G)\), and Poisson’s ratio \((\nu)\) of the sintered samples were determined by Advanced Ultrasonic Material Characterization System (UMS-100, TECLAB Limited Corp., Changsha, China). Vickers hardness \((Hv)\) of the sintered samples was evaluated by Vickers Hardness Tester (HXD-1000TMC&HVS-30ZG, Tai-Ming Optical Instrument Corp., Shanghai, China) using different test loads from 0.49 N (0.05 kgf) to 294 N (30 kgf) and a dwell time of 15 s. For comparing the hardness of HEB and HEB–20SiC ceramics with those of conventional diboride ceramics, \(Hv\) of \(\text{ZrB}_2\) single-phase ceramics and \(\text{ZrB}_2–20\ \text{vol\% SiC (ZrB}_2–20\text{SiC)}\) ceramics were also measured. The R.D. of the \(\text{ZrB}_2\) and \(\text{ZrB}_2–20\text{SiC}\) ceramics is 99.2% and 99%, respectively. The average grain size of the \(\text{ZrB}_2\) grains in \(\text{ZrB}_2\) ceramics is 21.4 μm. The average grain sizes of the \(\text{ZrB}_2\) grains and SiC grains in \(\text{ZrB}_2–20\text{SiC}\) ceramics are 2.7 μm and 1.65 μm, respectively. Details of the preparation and the properties of \(\text{ZrB}_2\) and \(\text{ZrB}_2–20\text{SiC}\) ceramics were reported in our previous work [21,22]. Fracture toughness \((K_{IC})\) was evaluated by SENB method via three-point bending using a crosshead speed of 0.5 mm/min and a span of 16 mm [23]. The dimensions of the bars are 2 mm × 4 mm × 25 mm. The notch was prepared by diamond wire cutting with a wire diameter of 80 μm. The notched width was less than 93 μm and the depth was about 1.8–2 mm. The values of the flexural strength and fracture toughness are the average of five measurements, and those of the other mechanical properties are the average of 10 measurements.

3 Results and discussion

3.1 Phase composition and microstructure

Based on the shrinkage curves of HEB and HEB–20SiC compacts during hot pressing (not shown here), the shrinkage of HEB–20SiC compact finished at about 1750 °C. In contrast, HEB compact stopped shrinking until the sintering temperature reached 1800 °C. The result indicated that the addition of SiC accelerated the densification process of HEB ceramics which was similar to the case in the SPS process of the same system [5]. After hot pressing, both the received HEB and HEB–20SiC ceramics showed R.D. higher than 99%, indicating full densified ceramics have been prepared by hot pressing in this study. Figure 1 shows the XRD patterns and the SEM morphologies of the hot-pressed ceramics. The main phase in the prepared HEB and HEB–20SiC ceramics is high-entropy diboride of \((\text{Ti}_{0.2}\text{Zr}_{0.2}\text{Hf}_{0.2}\text{Nb}_{0.2}\text{Ta}_{0.2})\text{B}_2\). Based on the XRD data, the lattice constants of high-entropy phase were calculated to be 3.100(6) Å and 3.360(4) Å for \(a\) and \(c\), respectively. The values are much close to the data \((a = 3.101\ \text{Å}, \ c = 3.361\ \text{Å})\) reported by Gild et al. [3].

According to the measured lattice constants, the theoretical densities of HEB and HEB–20SiC ceramics were calculated and listed in Table 1. The peak intensity for the \(\alpha\)-SiC phase was relatively very low in the HEB–20SiC specimen owing to the weak diffraction compared to that of the diboride phase. In addition, weak diffraction peaks of \((\text{Zr,Hf})\text{O}_2\) phase were detected attributed to the existence of impurity oxygen in the synthesized \((\text{Ti}_{0.2}\text{Zr}_{0.2}\text{Hf}_{0.2}\text{Nb}_{0.2}\text{Ta}_{0.2})\text{B}_2\) powder and

![Fig. 1 XRD patterns and SEM morphology of the hot-pressed ceramics.](http://www.springer.com/journal/40145)
Table 1 Properties of the hot-pressed HEB and HEB–20SiC ceramics

| Property                  | HEB          | HEB–20SiC   |
|---------------------------|--------------|-------------|
| Theoretical density (g/cm³) | 8.30         | 7.29        |
| Density (%)               | 8.29         | 7.21        |
| Relative density (%)      | 99.8         | 99.0        |
| Average grain size (μm)   | 4.06         | HEB: 2.70, SiC: 1.51 |
| Young’s modulus, E (GPa)  | 448          | 419         |
| Bulk modulus, B (GPa)     | 233          | 221         |
| Shear modulus, G (GPa)    | 190          | 177         |
| Poisson’s ratio, ν         | 0.178        | 0.185       |
| Hardness, Hv (GPa)         |              |             |
| Hv0.2                     | 23.7±0.7     | 24.8±1.2    |
| Hv1.0                     | 22.0±0.9     | 22.4±0.7    |
| Hv5.0                     | 20.2±0.4     | 21.4±0.7    |
| Four-point bending strength, σ (MPa) | 339±17    | 447±45     |
| Fracture toughness, KIC (MPa m¹/²) | 3.81±0.40  | 4.85±0.33  |

Table 1 lists the mechanical properties of the hot-pressed ceramics. As the Young’s modulus of SiC phase (410 GPa [25]) is lower than that of the high-entropy diboride phase, the HEB–20SiC composite shows a decreased Young’s modulus. The four-point flexural strength of the composite improved about 32% when compared to the monolithic. The main contribution of the strength improvement is considered to be the finer grain size of the composite related to the Hall–Petch relationship \( \sigma = \sigma_0 + kD^{-1/2} \), where \( D \) is the grain size of the material.

It can be seen from Fig. 1 and Table 1 that the average grain size of the high-entropy diboride phase is obviously decreased from 4.06 μm in the monolithic to 2.70 μm in the composite. The fracture toughness of the HEB–20SiC composite increased about 27% when compared to the monolithic. According to the model proposed by Taya et al. [25], the tensile residual stress in the high-entropy diboride matrix of the composite (405 MPa in high-entropy diboride phase and −1619 MPa in SiC phase) will decrease the toughness by 0.91 MPa m¹/². During the calculation, the following parameters are used: Poisson’s ratio for HEB 0.178 (see Table 1) and for SiC 0.19 [25], coefficients of thermal expansion (CTE) for high-entropy diboride phase and SiC phase are respectively 7.815×10⁻⁶ K⁻¹ (average CTE of five individual diboride along a and c axis from R.T. to 1500 °C) [26] and 4.02×10⁻⁶ K⁻¹ [25], the measured average interparticulate distance of SiC particles 3.48 μm, the thermal stress relieving temperature 1500 °C. Consequently, other toughening mechanism should be
considered. The crack propagation paths in HEB monolithic and HEB–20SiC composite during Vickers indentation are shown in Figs. 1(b) and 1(c). It is obvious that the crack propagation in HEB monolithic is flat, but it is tortuous in HEB–20SiC composite due to the crack deflection and branching effect by the SiC particles. Accordingly, the crack deflection and branching by the SiC particles is considered to be the main toughening mechanism for HEB–20SiC composite.

As shown in Table 1, the hardness $H_{V0.2}$ of the hot-pressed HEB ceramics is as high as 23.7±0.7 GPa, which is comparable to the previous reported values in the literature [4]. For comparison, the related $H_{V1.0}$ and $H_{V5.0}$ which are often used for evaluating the hardness of ceramics are also listed in the table. Figure 3(a) shows the Vickers hardness of HEB, HEB–20SiC, ZrB$_2$, and ZrB$_2$–20SiC ceramics under different indentation loads. Compared to the monolithic ZrB$_2$ ceramics with coarse microstructure, the higher hardness in the HEB ceramics should be attributed to both the smaller average grain size and the lattice distortion of high-entropy phase. On the other hand, compared to ZrB$_2$–20SiC, HEB–20SiC has similar microstructure but shows higher hardness. The high-entropy ($\text{Ti}_{0.2}\text{Zr}_{0.2}\text{Hf}_{0.2}\text{Nb}_{0.2}\text{Ta}_{0.2}$)B$_2$ phase is actually a kind of solid solutions with multi-principal elements. Because both ZrB$_2$ and high-entropy ($\text{Ti}_{0.2}\text{Zr}_{0.2}\text{Hf}_{0.2}\text{Nb}_{0.2}\text{Ta}_{0.2}$)B$_2$ have hexagonal crystal structure, the high-entropy ($\text{Ti}_{0.2}\text{Zr}_{0.2}\text{Hf}_{0.2}\text{Nb}_{0.2}\text{Ta}_{0.2}$)B$_2$ can be considered as partially substitution of Zr atoms in the crystal lattice of ZrB$_2$ by Ti, Hf, Nb, and Ta with different atom radius. This leads to lattice distortion and consequently increases the energy barrier for the motion of dislocations, which can result in the improvement of hardness. Accordingly, the higher hardness of HEB–20SiC over that of the ZrB$_2$–20SiC should be attributed to the lattice distortion in the HEB phase.

It can be obviously seen from Fig. 3(a) that the hardness of the samples changes under different indentation loads. This is caused by the indentation size effect (ISE) which is resulted from the proportional...
specimen resistance (PSR) [27–30]. With the increasing of indentation loads, the measured hardness decreased fast in the low load range, but gradually reached a stable value at above 49 N. Thus, the indentation load of about 49 N can be considered as the critical indentation load level ($P_c$) and the ISE-boundary for the high-entropy diboride ceramics. Below the ISE-boundary, the ISE contributes obviously to the measured $H_v$. Above this boundary, the ISE is significantly reduced and the measured $H_v$ will become a constant which can be called as load-independent hardness or “true” hardness ($H_{v\text{true}}$) [27]. Based on the PSR model developed by Li and Bradt [27] and the modified-PSR model proposed by Gong et al. [28], the relationship between the indentation load ($P$) and the indentation size ($d$) can be described by the following equation:

$$P = a_0 + a_1 \cdot d + \frac{P_c}{d_c^2} \cdot d^2$$  \hspace{1cm} (1)

Here, $a_0$ relates to the residual surface stresses in the test specimen, $a_1$ is the coefficient relating to the PSR, and $d_c$ is the characteristic indentation size. Besides, the relationship among $H_{v\text{true}}$, $P_c$, and $d_c$ obeys Eq. (2) which is the original formula for the calculation of Vickers hardness of material.

$$H_{v\text{true}} = 1.8544 \cdot \frac{P_c}{d_c^2}$$  \hspace{1cm} (2)

Hence, the value of $P_c/d_c^2$ and $H_{v\text{true}}$ can be derived from the curve fitting of $P$ and the measured $d$ data. Figure 3(b) shows the fit polynomial $P$–$d$ curves calculated by using the $d$ data measured in this experiment. The results show that the measured $P$–$d$ relationship can be perfectly described by the second-order curves which are completely in accordance with Eq. (1). According to the fit polynomial $P$–$d$ curves, the value of $H_{v\text{true}}$ is 19.7 GPa for HEB. The calculated values of $H_{v\text{true}}$ are close to the measured $H_v$ (20.2±0.4 GPa for HEB), which also indicates that 49 N is suitable to be chosen as the ISE-boundary for the high-entropy diboride ceramics.

### 4 Conclusions

In summary, dense (Ti$_{0.2}$Zr$_{0.2}$Hf$_{0.2}$Nb$_{0.2}$Ta$_{0.2}$)B$_2$ (HEB) and its composite containing 20 vol% SiC (HEB–20SiC) are prepared by hot pressing. The effects of SiC secondary particles on the densification, microstructure, and mechanical properties of HEB ceramics are investigated. The addition of SiC not only accelerates the densification process but also refines the microstructure of HEB, resulting in improved mechanical properties. Dense HEB and HEB–20SiC ceramics hot-pressed at 1800 °C exhibit four-point flexural strength of 339±17 MPa and 447±45 MPa, and fracture toughness of 3.81±0.40 MPa·m$^{1/2}$ and 4.85±0.33 MPa·m$^{1/2}$, respectively. Such values of room temperature bending strength and fracture toughness are typical of structure ceramics. Crack deflection and branching by SiC particles is considered to be the main toughening mechanisms for the HEB–20SiC composite. The hardness $H_{v0.2}$ of the sintered HEB and HEB–20SiC ceramics is 23.7±0.7 GPa and 24.8±1.2 GPa, respectively. With the increase of indentation load, the hardness of the sintered ceramics decreases rapidly until the load reaches about 49 N, due to the indentation size effect.
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