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INFLUENCE OF HEATING TO HIGH TEMPERATURES ON MECHANICAL PROPERTIES OF BORIDE-BASED REFRACTORY MATERIALS

The object of research is HfB_2, ZrB_2 and ceramics composition HfB_2-30 % SiC and ZrB_2-20 % SiC, ZrB_2-20 % SiC-4 % Si_3N_4 obtained under high pressure, their mechanical characteristics before and after heating to high temperatures and temperatures of beginning of melting. The research was conducted in order to create new effective refractory materials for use in the aerospace industry. Therefore, the melting temperatures of sintered materials and the effect of heating on their mechanical properties were also studied. Additives (ZrB_2-20 % SiC and HfB_2-30 % SiC) although led to a decrease in specific gravity. But increased hardness (by 17 % and 46 % in the case of ZrB_2 and HfB_2, respectively) and fracture toughness (by 40 % and 21 % in the case of ZrB_2 and HfB_2, respectively). However, significantly reduced the onset of melting temperature in vacuum to 2150–2160 °C.

Materials sintered from ZrB_2 and HfB_2 was not melted after heating to 2970 °C. After heating to a melting point of 2150–2160 °C (in the case of materials with additives) and to temperatures of 2970 °C (in the case of materials sintered with ZrB_2 or HfB_2), the hardness and fracture toughness decreased. Thus, the hardness of the material prepared from ZrB_2 decreased by 19 % and its fracture toughness – by 18 %, and of that prepared from ZrB_2-20 % SiC – by 46 % and 32 %, respectively. The hardness of the material prepared from HfB_2 decreased by 46 %, its fracture toughness – by 55 %, and of that prepared from HfB_2-30 % SiC, after heating decreased by 40 %, but its fracture toughness increased by 15 %. The sintered HfB_2 (with a density of 10.4 g/cm^3) before heating showed a hardness of $H_V(9.8\ N)=21.27\pm0.84$ GPa, $H_V(49\ N)=19.29\pm1.34$ and $H_V(98\ N)=19.17\pm0.5$, and fracture toughness $K_{1C}(9.8\ N)=0.47$ MH·m$^{0.5}$, and ZrB_2 with a density of 6.2 g/cm^3 was characterized by $H_V(9.8\ N)=17.66\pm0.60$ GPa, $H_V(49\ N)=15.25\pm1.22$ GPa and $H_V(98\ N)=15.32\pm0.36$ GPa, $K_{1C}(9.8\ N)=4.3$ MH·m$^{0.5}$. Material sintered with HfB_2-30 % SiC (density 6.21 g/cm^3) had $Hv(9.8\ N)=38.1\pm1.4$ GPa, $H_V(49\ N)=27.7\pm2.8$ GPa, and $K_{1C}(9.8\ N)=8.1$ MH·m$^{0.5}$, $K_{1C}(49\ H)=6.8$ MH·m$^{0.5}$. The sintered with ZrB_2-20 % SiC material had density of 5.04 g/cm^3, $H_V(9.8\ N)=24.2\pm1.9$ GPa, $H_V(49\ N)=16.7\pm2.8$ GPa, $K_{1C}(49\ H)=7.1$ MH·m$^{0.5}$. The SiC addition to the initial mixture significantly reduces the elasticity of the materials.

Keywords: *zirconium diboride, hafnium diboride, silicon carbide, silicon nitride, ultrahigh-temperature ceramics, refractory borides.*

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1. Introduction

The IV–V groups of diborides and carbides of transition metals have a very high melting point (>2500 °C), high mechanical properties, hardness, fracture toughness and high thermal properties, and therefore are recommended as ultra-high temperature ceramics (UHTC) [1–3]. As a result, this material has been proposed for a variety of constructural applications at room and elevated temperature including aerospace applications.

SiC reinforcement of ZrB_2 is known [4] to increase flexural strength, fracture toughness and oxidation resistance and so was chosen as additive in this study. Hafnium diboride has several important advantages over ZrB_2. The fundamental difference in nuclear properties is thermal the neutron capture cross section in hafnium compounds is 3 orders

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of magnitude higher than that of zirconium compounds. The relatively high density of HfB_2 (11.20 vs. 6.12 g/cm^3 in ZrB_2), according to the authors [5], is not a disadvantage and, conversely, can be useful for moving the center of gravity of the High-Lift System Aerodynamics forward in some aerodynamic schemes.

The object of research is HfB_2, ZrB_2 and ceramics composition HfB_2-30 % SiC and ZrB_2-20 % SiC, ZrB_2-20 % SiC-4 % Si_3N_4 obtained under high pressure, their mechanical characteristics before and after heating to high temperatures and temperatures of beginning of melting. *The aim of research* is study the melting temperatures of sintered materials and the effect of heating on their mechanical properties.

2. Methods of research

Under high 4.1 GPa pressure at 1800 °C temperature were prepared samples of ZrB_2, HfB_2, ZrB_2-20 % SiC, ZrB_2-20 % SiC-4 % Si_3N_4, HfB_2-30 % SiC which were approximately 15 mm in diameter and 7 mm in height. During sintering, there were 10 samples. The sintered material was in contact with graphite and hexagonal boron nitride.

Table 1 shows the results of X-ray study of phase composition of initial ZrB_2 (Technical specification 6-09-03-46-75), HfB_2 (Ukraine, Technical specification 6-09-03-418-75) powders (with grain sizes <10 μm which contained 0.1 mass % and 0.2 mass % of admixture carbon, respectively). Additions of SiC and Si_3N_4 were mixed with diborides using a «drunken barrel» mixer for 24 hours.

The samples structure was studied by X-ray diffraction using a DRON-UM1 diffractometer (USSR). To define the phase composition, the monochromatic CuKα radiation (λ=1.541841 Å) in the range of angles 2Θ=8°–88° with a scan step of 0.05° and a time of the exposition at a point of 2 s. As a monochromatic a graphite single crystal installed on a diffracted beam was used. The analysis of the experimental data was made using the PowderCell 2.4 programs, in which the full profile refinement by the Rietveld method was realized [6].

Table 1

Unit cell parameters *a, b, c* and phase composition of the initial powders

Initial powder	Phase composition, mass. %	*a, b, c*, nm
ZrB_2	ZrB_2 – 97 %	*a* – 0.3168, *c* – 0.3530
	ZrO_2t – 1 %	*a* – 0.3604, *c* – 0.5208
	ZrO_2m – 2 %	*a* – 0.5153, *b* – 0.5210, *c* – 0.5310
HfB_2	HfB_2 – 100 %	*a* – 0.3143, *c* – 0.3476
Si_3N_4	A – Si_3N_4 – 4 %	*a* – 0.7747, *c* – 0.5620
	B – Si_3N_4 – 96 %	*a* – 0.7599, *c* – 0.2907
SiC	3C – SiC – 100 %	–

Vickers microhardness and fracture toughness were determined using a FALCON 500 hardness tester (Netherlands) (equipped with an optical microscope, a digital 5-megapixel camera, and a computer). At least 5 indents were made at a load of 9.8 N, 49 N, 98 N.

The modulus of elasticity was calculated according to the method described in [7, 8], using a solution for the lower mode of oscillations of the disks, as it has the greatest practical interest due to the high reliability of the method of recording resonance.

The temperatures of incipient melting were determined with the Pirani-Alterthum technique [9] employing an EOP-68 pyrometer (made by Kharkiv Experimental Plant «Pribor», Ukraine). The instrumental errors of the device are ±4 °C in the range 1400–2000 °C and ±12 °C at 2000–3000 °C.

The Pirani-Alterthum technique and evaluation of measurement errors are described in detail elsewhere [10, 11].

3. Research results and discussion

Table 2 shows the compositions of the initial mixtures, parameters of sintering, the phase composition of the consolidated materials, and the unit cell parameters of the present in the materials phases, determined by X-ray diffraction analysis. Table 3 shows the results of tests of hardness and fracture toughness of materials before and after heating to the temperatures of beginning of melting or mechanical destruction. The results of pyrometric measurement of the melting points in vacuum described in Table 2 (the numbering in Tables 2, 3 is the same).

Table 2

Compositions of the initial mixtures, parameters of sintering (temperature, *T*, pressure, *p*, holding time, τ), the phase composition, density, porosity of the consolidated materials and the unit cell parameters (*a, b, c*) of the phases present in ZrB_2- and HfB_2-based materials

No.	Initial composition	*P*, *T*, t	Phase composition, wt. %, density, g/cm^3, porosity, *P*, %	Unit cell parameters, nm, *a, b, c*
1	ZrB_2	*P*=4.1 GPa *T*=1800 °C τ=0.13 h	ZrB_2=100 %, γ=6.2 g/cm^3, *P*=0 %	*a* – 0.3168 *c* – 0.3528
2	ZrB_2-20 % SiC	*P*=4.1 GPa *T*=1800 °C τ=0.13 h	ZrB_2=79.53 %, β-SiC=20.47 %, γ=5.04 g/cm^3, *P*=1.96 %	*a* – 0.3169 *c* – 0.3508 *a* – 0.4359
3	ZrB_2-20 % SiC-4 % Si_3N_4	*P*=4.1 GPa *T*=1800 °C τ=0.13 h	ZrB_2=77.58 %, β-SiC=19.92 %, β-Si_3N_4=2.50 %, γ=4.98 g/cm^3, *P*=1.77 %	*a* – 0.3168 *c* – 0.3529 *a* – 0.4357 *a* – 0.7609 *c* – 0.2906
4	HfB_2	*P*=4.1 GPa *T*=1800 °C τ=0.13 h	HfB_2=100 %, γ=10.42 g/cm^3, P=1 %	*a* – 0.3141 *c* – 0.3473
5	HfB_2-30 % SiC	*P*=4.1 GPa *T*=1800 °C τ=0.13 h	HfB_2=72.40 %, β-SiC=27.60 %, γ=6.21 g/cm^3, *P*=3.86 %	*a* – 0.3143 *c* – 0.3475 *a* – 0.4358

Table 4 shows the results of estimation of Young's modulus, Poisson's ratio of the samples sintered under high pressure.

The materials were practically non-porous, but the materials with addition of silicon carbide (samples 2, 3, 4 see Table 2) showed somewhat higher porosity. But their porosity was calculated using the results of X-ray phase analysis (obtained by Reitveld refinement) which are not so exact. Additives (ZrB_2-20 % SiC (sample 2) and HfB_2-30 % SiC (sample 5)) led to a decrease in specific gravity and increase in hardness (for 17 % and 46 % as compare with that of ZrB_2 (sample 1) and HfB_2 (sample 4), respectively, Table 3) and fracture toughness (for 40 % and 21 % as compare with that of ZrB_2 and HfB_2, respectively, Table 3), but significantly reduced the onset temperature

of melting in vacuum down to 2150–2160 °C (Table 3). Materials sintered from ZrB_2 and HfB_2 could not be melted when heated to 2970 °C . At this temperature, the samples were cracked and further heating was not possible. If to prepare samples with thicker walls, the power of the available laser occurred to be not enough to heat them at all. (with density 6.21 g/cm^3) had Hv(9.8 N)=38.1±1.4 GPa, H_V(49 N)=27.7±2.8 GPa, and K_{1C}(9.8 N)=8.1 MH·m$^{0.5}$, K_{1C}(49 H)=6.8 MH·m$^{0.5}$. The sintered from ZrB_2-20 % SiC material had density of 5.04 g/cm^3, H_V(9.8 N)=24.2±1.9 GPa, H_V(49 N)=16.7±2.8 GPa, K_{1C}(49 H)=7.1 MH·m$^{0.5}$. The additions lead to decrease of Young modulus and increase of Poisson ratio (Table 4).

Table 3

Vickers hardness and fracture toughness of the ZrB_2 and HfB_2-based materials before and after heating. Numbering is the same as in the Table 2

Sample No.	Initial composition	Heating temperature	Vickers hardness, GPa, under the load			Fracture toughness, K_{1C}, MPa·m$^{0.5}$, under the load		
			9.8 N	49 N	98 N	9.8 N	49 N	98 N
1	ZrB_2	–	17.7	15.4	15.3	4.3	4.2	4.0
1*	ZrB_2	Crack under 2970 °C	14.42	7.85	8.35	3.1	3.46	3.83
2	ZrB_2-20 % SiC	–	24.2	16.7	17.6	–	7.1	6.2
2*	ZrB_2-20 % SiC	2160 °C	12.95	11.29	10.61	2.77	4.84	3.95
3	ZrB_2-20 % SiC-4 % Si_3N_4	–	20.5	18.3	15.8	–	–	9.2
3*	ZrB_2-20 % SiC-4 % Si_3N_4	2160 °C	16.87	11.76	11.32	4.65	5.53	5.07
4	HfB_2	–	21.3	19.3	19.2	–	7.2	5.7
4*	HfB_2	Crack under 2970 °C	11.95	5.05	6.57	1.68	2.14	2.57
5	HfB_2-30 % SiC	–	38.0	27.7	26.3	8.2	6.8	6.4
5*	HfB_2-30 % SiC	2150 °C	22.59	21.52	21.28	–	7.56	7.46

Note: * – sample after heating and pyrometric measurement of melting point

Table 4

Mechanical characteristics of ceramics. Numbering is the same as in the Table 2

Sample No.	Initial composition	Speed of sound, s, m/s	Young's modulus, E, GPa	Log. decrement of oscillations, %	Poisson ratio, μ
1	ZrB_2	8291	418	0.971	0.093
2	ZrB_2-20 % SiC	8599	358	1.576	0.146
3	ZrB_2-20 % SiC-4 % Si_3N_4	8566	358	2.258	0.146
4	HfB_2	8941	847	0.973	0.093

Composite material prepared from ZrB_2-20 % SiC-4 % Si_3N_4 (sample 3) demonstrated somewhat lower hardness than that prepared from ZrB_2-20 % SiC (sample 2), but higher fracture toughness (Table 3).

After heating to a melting point of 2150–2160 °C (in the case of materials with additives) and to temperatures of 2970 °C (in the case of materials sintered with ZrB_2 or HfB_2), the hardness and crack resistance decreased (Table 3). Thus, the hardness of the material prepared from ZrB_2 decreased by 19 % and its crack resistance – by 18 %, and of that prepared from ZrB_2-20 % SiC – by 46 % and 32 %, respectively. The hardness of the material prepared from HfB_2 decreased by 46 %, its crack resistance – by 55 %, and of that prepared from HfB_2-30 % SiC, after heating decreased by 40 %, but its fracture toughness increased by 15 %. The sintered HfB_2 (with a density of 10.4 g/cm^3) before heating showed hardness of H_V(9.8 N)=21.27±0.84 GPa, H_V(49 N)=19.29±1.34 and H_V(98 N)=19.17±0.5, and fracture toughness K_{1C}(9.8 N)= =6.47 MH·m$^{0.5}$, and ZrB_2 with density of 6.2 g/cm^3 was characterized by H_V(9.8 N)=17.66±0.60 GPa, H_V(49 N)= =15.25±1.22 GPa and H_V(98 N)=15.32±0.36 GPa, K_{1C}(9.8 N)= =4.3 MH·m$^{0.5}$. The material sintered from HfB_2-30 % SiC

The X-ray study (Table 2) showed that after sintering materials with additions have similar phase composition as the initial mixture, but SEM EDX study showed that their structures are somewhat more complicated. The structure of the material prepared from ZrB_2-20 % SiC is shown in Table 5, Fig. 1. Thus, sample 2 sintered at high pressures consisted of three main phases, whose stoichiometry is close to the $Z_{0.9}B_2C_{0.12-0.19}$, $Si_{0.92-0.94}C$, $Si_{0.94-0.99}CO_{0.05-0.06}$.

Comparing the results of hardness tests, with the literature data, one can see a pronounced effect of the sintering pressure. The Vickers hardness under indentation load 9.8 N of sample 1 from ZrB_2 is 17.7 GPa, of sample 3 from ZrB_2-20 % SiC is 24.2 GPa, of sample 4 from HfB_2 is 21.3 GPa, while according [12, 13] the materials prepared from the same initial powders and mixtures but under lower pressures (50 MPa) and higher temperatures (1950–2100 °C) demonstrated somewhat lower hardnesses under the same load: 16.5, 21.1and 19.8 GPa, respectively.

Table 5

The results of a microprobe analysis in % of the material prepared from ZrB_2-20 % SiC

Spectrum	B	C	O	Si	Zr	Total	Approximate composition
S1	65.43	5.93	–	–	28.64	100	$Z_{0.9}B_2C_{0.18}$
S2	65.64	3.97	–	–	30.39	100	$Z_{0.9}B_2C_{0.12}$
S3	65.18	4.86	–	–	29.96	100	$Z_{0.91}B_2C_{0.14}$
S4	65.08	6.16	–	–	28.75	100	$Z_{0.88}B_2C_{0.19}$
S5	65.15	5.52	–	–	29.32	100	$Z_{0.9}B_2C_{0.17}$
S6	–	48.09	–	51.91	–	100	$Si_{0.93}C$
S7	–	48.8	2.89	48.31	–	100	$Si_{0.99}CO_{0.06}$
S8	–	49.14	–	50.86	–	100	$Si_{0.97}C$
S9	–	48.41	–	51.59	–	100	$Si_{0.94}C$
S10	–	50.41	2.28	47.31	–	100	$Si_{0.94}CO_{0.05}$
S11	–	47.94	–	52.06	–	100	$Si_{0.92}C$
S12	65.34	4	–	–	30.66	100	$Z_{0.94}B_2C_{0.12}$

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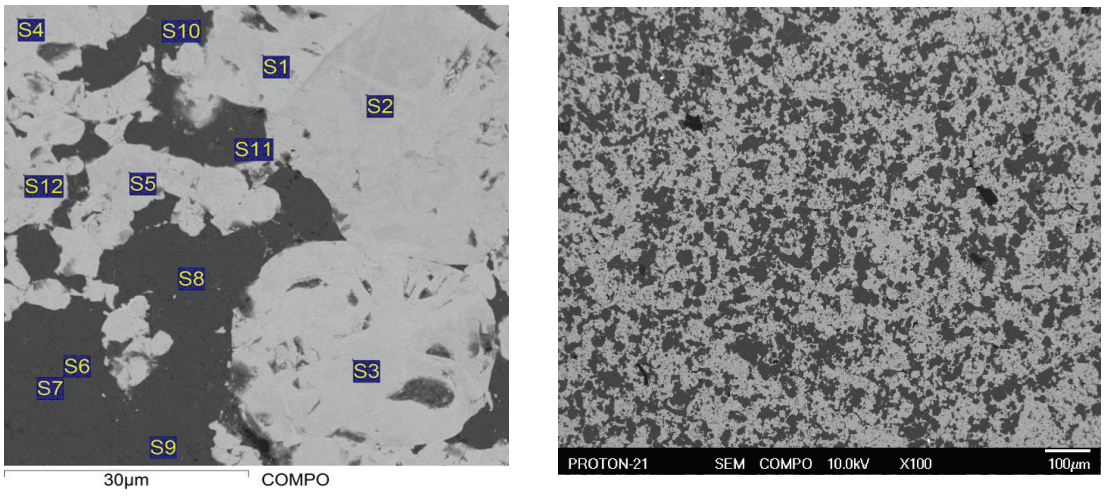

a *b*

Fig. 1. Structure of sample 2 (see Table 2) obtained sintering from ZrB_2-20 % SiC (see Table 2) using the SEM in a regime: *a* – COMPO 30 μm; *b* – COMPO 100 μm

The temperature of the onset of melting of No. 1* (ZrB_2) and No. 5* (HfB_2) could not be measured. The samples were heated to 2970 °C, after which they cracked in half. ZrB_2 is usually melts at 3250 °C according to the phase diagrams [14, 15]. However, other researchers report different melting points, including 3040 °C [16] and 3517 °C [17]. The reason for these discrepancies lies not only in the difficulty of accurately measuring melting points at high temperatures, but also in the fact that these compounds melt incongruently, i. e. they can decompose or dissociate before melting.

The temperature of the beginning of melting of specimens samples 2* (ZrB_2-20SiC), 3* (ZrB_2-20SiC-4Si_3N_4) and 5* (HfB_2-30SiC) were estimated as 2160 °C. The character of melting was violent. As it was mentioned above the additions of SiC to ZrB_2 and HfB_2 lowers the melting points of the materials.

In the works of other researchers, it is reported that ZrB_2 had a Young modulus of 222.1 GPa [18] and that hot-pressed polycrystalline ZrB_2 bars showed Young modulus of 503.3 GPa [19]. The material hot pressed under uniaxial pressure of 32 MPa from ZrB_2-20SiC powder mixtures at a temperature of 1900 °C demonstrated Young's modulus showed 479 GPa [20]. The composite sintered from ZrB_2-20SiC-4Si_3N_4 powders under uniaxial pressure of 30 MPa at 2000 °C in an argon atmosphere showed Young modulus 467 GPa [21]. In our case, ZrB_2 sintered at 4.1 GPa and 1800 °C demonstrated Young modulus 418 GPa (Table 4). The SiC addition to the initial mixture significantly reduces the elasticity of the materials (Table 4).

4. Conclusions

The investigation of structure and materials mechanical properties sintered at high (4.1 GPa) pressure and 1800 °C from HfB_2, ZrB_2 without and with additives of SiC and Si_3N_4 allowed conclude the following.

The additives led to a decrease in specific gravity and increase in hardness and fracture toughness, decrease Young modulus, increase Poisson ratio and significantly reduce the onset of melting temperature in vacuum (down to 2150–2160 °C).

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