Optimization of Effective Parameters on ZrB_2 Ceramic Oxidation

Zohre Balak[1] Mahdi Azizieh[2] Hosin Kafashan[3]

1. Introduction

ZrB_2 is used in different industries due to individual of mechanical and physical properties such as high melting point, high thermal conductivity, flexural strength and hardness. When ZrB_2 is exposed to air at high temperatures, it reacts with O_2 to form ZrO_2 and B_2O_3:

$$ZrB_2 + 5/2O_2 \rightarrow ZrO_2 + B_2O_3(l) \quad (1)$$

Due to high vapor pressure, the B_2O_3 evaporates above 1100 ◦C:

$$B_2O_3(l) \rightarrow B_2O_3(g) \quad (2)$$

The removal of B_2O_3 by vaporization leaves behind a porous ZrO_2 scale, which results in the rapid linear oxidation kinetics above 1400 °C. Thus, the high-temperature applications of monolithic ZrB_2 will be limited by its poor oxidation resistance.

The addition of SiC has been reported to improve the oxidation resistance of ZrB_2. Above 1100 °C, SiC reacts with O_2 according to the following reaction:

$$SiC + 32O_2 \rightarrow SiO_2(l) + CO(g) \quad (3)$$

The SiO_2 and B_2O_3 form a borosilicate liquid, which covers the exposed surfaces. As temperature increases, B_2O_3 is continuously removed from the borosilicate liquid, leading to the formation of a SiO_2-rich glassy layer. Because SiO_2 is significantly less volatile and more viscous than B_2O_3, the SiO_2- rich layer provides effective oxidation protection for ZrB_2–SiC above 1100 °C.

2. Experimental

With respect to high number of effective parameters (Table 1), Taguchi design was used. Table 2 shows the conditions of samples preparation and Table 3 shows the specification of powders. The powders corresponding to 32 different compositions, according to Table 2, were mixed by wet ball-milling at 200 rpm for 3 hours in a zirconia's bottle, using zirconia's balls and ethanol as media.

The mixtures were then dried. The powder mixture was put into graphite die lines with graphite foil which has an inner diameter of 50 mm and sintered using SPS apparatus (SPS-20T-10, China). The sintering was performed at different temperatures (between 1600 °C and 1900 °C), different pressures (between 10 MPa and 40 MPa) and different holding times (between 4 minutes and 16 minutes) based on Taguchi design (Table 1) in vacuum. Oxidation test was conducted in box furnace at 1600 °C for one hour.

Table 1. Effective parameters on oxidation and their levels

TEST	FAC.1	FAC.2	FAC.3	FAC.4	FAC.5	FAC.6	FAC.8	FAC.7	FAC.9
	SiC Vol%	C_f Vol%	M.t hr	$MoSi_2$ Vol%	HfB_2 Vol%	ZrC Vol%	P MPa	T C	t min
L1	5	0	0	0	0	0	10	1600	4
L2	10	2.5	2.5	2	5	5	20	1700	8
L3	15	5	5	4	10	10	30	1800	12
L4	20	7.5	7.5	6	15	15	40	1900	16

Table 2. Conditions of samples preparation

TEST	FAC.1	FAC.2	FAC.3	FAC.4	FAC.5	FAC.6	FAC.7	FAC.8	FAC.9
	SiC Vol%	C_f Vol%	M. t h	$MoSi_2$ Vol%	HfB_2 Vol%	ZrC Vol%	Temp. C	Press. MPa	Time min
1	5	0	0	0	0	0	1600	10	4
2	5	2.5	2.5	2	5	5	1700	20	8
3	5	5	5	4	10	10	1800	30	12
4	5	7.5	7.5	6	15	15	1900	40	16
5	10	0	0	2	5	10	1800	40	16
6	10	2.5	2.5	0	0	15	1900	30	12
7	10	5	5	6	15	0	1600	20	8
8	10	7.5	7.5	4	10	5	1700	10	4
9	15	0	2.5	4	15	0	1700	30	16
10	15	2.5	0	6	10	5	1600	40	12
11	15	5	7.5	0	5	10	1900	10	8
12	15	7.5	5	2	0	15	1800	20	4
13	20	0	2.5	6	10	10	1900	20	4
14	20	2.5	0	4	15	15	1800	10	8
15	20	5	7.5	2	0	0	1700	40	12
16	20	7.5	5	0	5	5	1600	30	16
17	5	0	7.5	0	15	5	1800	20	12
18	5	2.5	5	2	10	0	1900	10	16
19	5	5	2.5	4	5	15	1600	40	4
20	5	7.5	0	6	0	10	1700	30	8
21	10	0	7.5	2	10	15	1600	30	8
22	10	2.5	5	0	15	10	1700	40	4
23	10	5	2.5	6	0	5	1800	10	16
24	10	7.5	0	4	5	0	1900	20	12
25	15	0	5	4	0	5	1900	40	8
26	15	2.5	7.5	6	5	0	1800	30	4
27	15	5	0	0	10	15	1700	20	16
28	15	7.5	2.5	2	15	10	1600	10	12
29	20	0	5	6	5	15	1700	10	12
30	20	2.5	7.5	4	0	10	1600	20	16
31	20	5	0	2	15	5	1900	30	4
32	20	7.5	2.5	0	10	0	1800	40	8

Table 3. Specification of powders

Powder	Grain sizeμ
ZrB_2	20
SiC	25
C_f	T800, 5
$MoSi_2$	25
HfB_2	30
ZrC	20

[1] Corresponding Author: Assistant Professor, Department of materials science and engineering, Ahvaz Branch, Islamic Azad University, Ahvaz, Iran.
Email: zbalak1983@gmail.com
[2] Assistant Professor, Department of materials science and engineering, Ahvaz Branch, Islamic Azad University, Ahvaz, Iran.
[3] Assistant Professor, Department of materials science and engineering, Ahvaz Branch, Islamic Azad University, Ahvaz, Iran.

3. Results and Discussion

Figure 1 shows samples before and after oxidation at 1600 °C for 1 hour. The oxidation of samples was determined visually and the data are given in Table 4. Then the data are entered to the software and the effect of each parameter on oxidation are obtained.

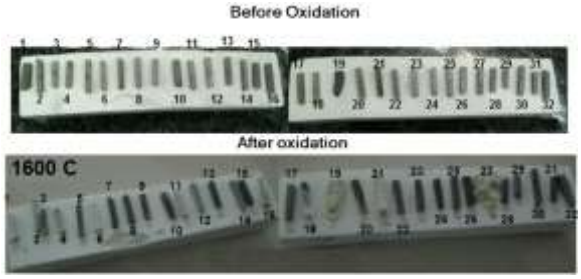

Fig. 1. Samples before and after oxidation at 1600 °C for 1 hour

Table 4. Oxidation of samples

Comp.	Oxidation	Comp.	Oxidation
1	3	17	1
2	2.25	18	3.25
3	1.25	19	4.75
4	3	20	1.25
5	1.5	21	4
6	3.5	22	0.5
7	0.25	23	0.25
8	0.25	24	0
9	0.5	25	0
10	0.75	26	0
11	3	27	5
12	2.25	28	0.25
13	0.5	29	1.75
14	1.75	30	4
15	0.5	31	0.1
16	0	32	0

4. Conclusion

- HfB_2 has positive effect on oxidation resistance of ZrB_2 ceramics.
- By increasing ZrC and C_f, oxidation increases.
- Among the SPS parameters, temperature has highest effect.
- SiC and $MoSi_2$ improve the oxidation resistance.
- Optimum levels for manufacturing composite with the most oxidation resistance are as below:

SiC: 20vol%, C_f: 0vol%, M.t: 5 hr, $MoSi_2$: 6vol%, HfB_2: 15 vol%, ZrC: 10vol%, Temparure: 1800C, Pressure: 30MPa, time: 8min

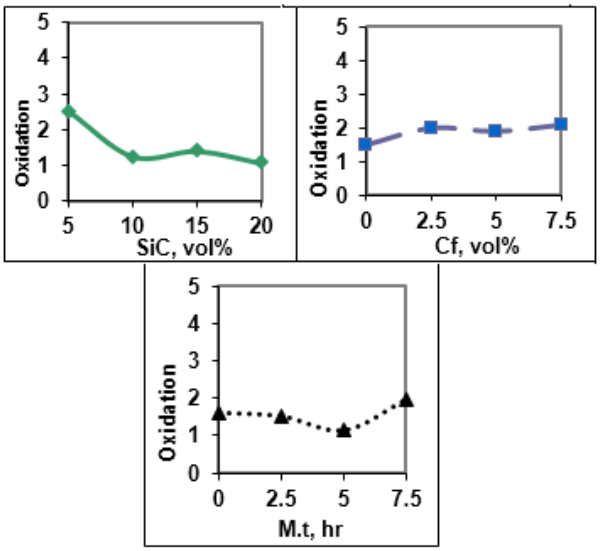

Fig. 2. The effect of SiC, C_f and M.t on oxidation

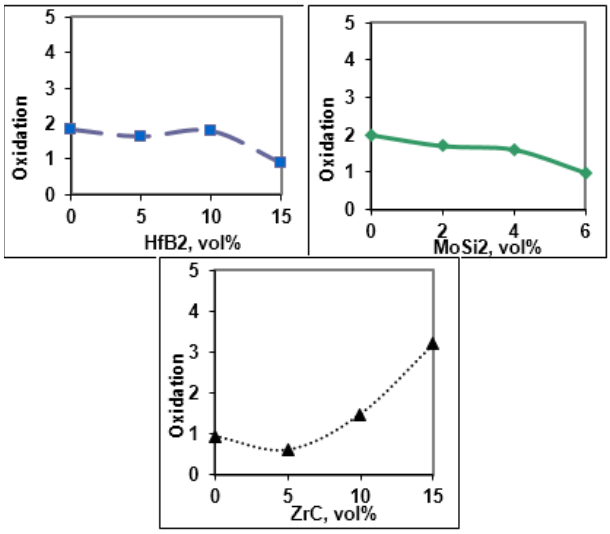

Fig. 3. The effect of $MoSi_2$, HfB_2 and ZrC on oxidation

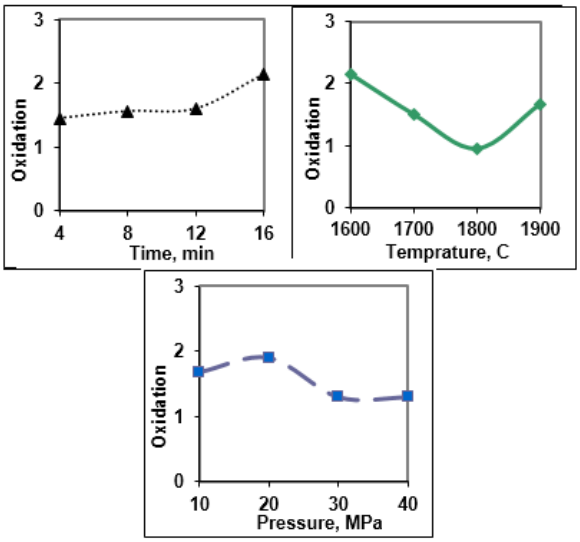

Fig. 4. The effect of temperature, time and pressure on oxidation