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Investigation of Effective Parameters on Densification of ZrB_2-SiC Based Composites Using Taguchi Method

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ABSTRACT

The main goal of this study is optimization of densification of ZrB_2-SiC composites reinforced with chopped C_f prepared by SPS. Taguchi method is employed as statistical design of experiment (DOE) to optimize densification parameters including SiC, C_f, $MoSi_2$, HfB_2 and ZrC content, milling time of C_f and SPS parameters such as temperature, time and pressure. Each of these factors was examined on four levels in order to obtain the optimum conditions. A total of 32 samples were prepared in accordance to the L_{32} array proposed by the Taguchi method.

By using statistical analysis of variance (ANOVA), it has been concluded that the most significant effect on the densification is related to temperature, $MoSi_2$ and time by 50.2%, 20.7% and 9.8% portion, respectively. Also, the results showed that pressure with 0.8%, ZrC with 1.9% and HfB2 with 1.9% portion have the least effect on open porosity. The other parameters including SiC, milling time (M.t) and C_f have 2.9%, 3.6% and 3.8% portion on open porosity respectively.

1. INTRODUCTION

Zirconium diboride (ZrB_2) is a Group IV metal diboride which belongs to the family of ultra-high-temperature ceramics (UHTCs) known for their combination of thermo physical properties, such as high melting point (3245 °C), high hardness (22 GPa), high thermal conductivity (60–140 $W \cdot m^{-1} \cdot K^{-1}$), high electrical conductivity (9.2×10−6Ω·cm), and good corrosion resistance. As a result, ZrB_2-based composite has attracted much attention for various applications, such as thermal protection materials and sharp-leading-edge components in future high Mach (7–20M) hypersonic flight, atmospheric re-entry vehicles as well as rocket propulsion hardware [1-3].

However, it is acknowledged that ZrB_2 has poor sinterability due to its strong covalent bonding and low volume and grain boundary diffusion rate. In studies, nominally stoichiometric ZrB_2 without additives has only been densified by hot-pressing at 2000 °C or higher with pressures of 20–30 MPa, or at reduced temperatures (1790–1840 °C) with much higher pressures (800–1500 MPa)[4].

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Sintering aids are needed to obtain a fully dense bulk material. To improve sinterability of ZrB_2, various metals, nitrides, silicides and carbides, such as Cu, Ni, Fe, Mo, Nb [4] AlN, HfN, ZrN [5,6] Si_3N_4, $MoSi_2$, $ZrSi_2$,$TaSi_2$ [5,6,9] B_4C, WC, VC, Y_2O_3 [5-9] and SiC have been used as sintering aids[5, 10].

Even though such sintering aids have successfully lowered the sintering temperatures by few hundred degrees, more often they have resulted in the formation of deleterious grain boundary phases due to sintering reactions [11]. As an example, the strength of ZrB_2 containing ~3 volume percent Ni has been shown to drop by about 60% between 800 and 1000°C [12].

Recent efforts have directed towards sintering of ZrB_2-based ceramics using spark plasma sintering (SPS), which offers much higher heating rates, lower sintering temperatures and shorter holding times in the presence of pulsed DC electric field and external pressure. SPS technique has yielded favourable results, with respect to lowering of sintering temperature and concomitantly controlling grain growth and formation of secondary phases [11].

In addition, some studies were done to adjust the sintering parameters such as temperature, time and pressure to achieve nearly full densification. So, it is clear that many factors (different additives, sintering

method and parameters of sintering method) can influence on densification.
Investigating all of these parameters in a single work may not be possible. In order to investigate the effect of each factor and attain the maximum density, an optimization strategy is required to find the best experimental conditions. Taguchi method is a very useful tool to solve the complex and confusing problems with fewest variables and fewer tests in many areas. It includes the design of an experiment process using orthogonal arrays that allows independent evaluation of factors through a small number of runs [13].
In this perspective, we focus on the effect of different additives, temperatures, holding times and pressures on densification of ZrB_2-based ceramics. With applying the Taguchi method, the effect of nine parameters such as SiC, C_f, $MoSi_2$, HfB_2 and ZrC content, milling time of C_f and SPS parameters such as temperature, time and pressure at four levels on densification were evaluated and discussed.

2. EXPERIMENTAL PROCEDURES

2.1. Materials

Six basic raw materials including ZrB_2 (20 µm, Northwest Institute for Non-Ferrous Metal Research, China, 99% purity), SiC (25µm), ZrC (20 µm, Alfa-Aesar, 99.5% purity), $MoSi_2$ (25 µm, 99% purity), HfB_2 (30 µm, 99% purity), and T800H fiber (5 µm in diameter) were used to produce the composites. XRD analyses of raw powders are shown in Figure 1.

2.2. Experimental Design

In this study, the Taguchi method was used to design the experiments and to obtain the optimum experimental design by considering the effects of factors on their densification. Nine primary factors were examined followed by a statistical study of the SiC vol% (designated as "A"); the carbon fiber (C_f) vol% as ''B''; the milling time as ''C'' the $MoSi_2$ vol% as ''D''; HfB_2 vol% as ''E''; temperature as ''F''; pressure as ''G'' and time as ''H''. Each factor was examined at four levels. The used factors and levels are presented in TABLE 1. The level of each factor and the values of the tested factors were chosen based on previous researches. Normally, the full factorial design would require $4^9 = 262144$ experimental runs. Apparently, doing these experimental runs is impossible and accompanies with very high cost, while with using Taguchi method only 32 experiments are needed based on the orthogonal array L32. Preparing conditions of each sample are shown in TABLE 2.

2.3. Manufacturing and Characterization

The powders corresponding to the 32 compositions, according to TABLE 2. were mixed by wet planetary ball-milling at 200 rpm for 3 h in a zirconia bottle, using zirconia balls and ethanol as media. The mixtures were then dried.

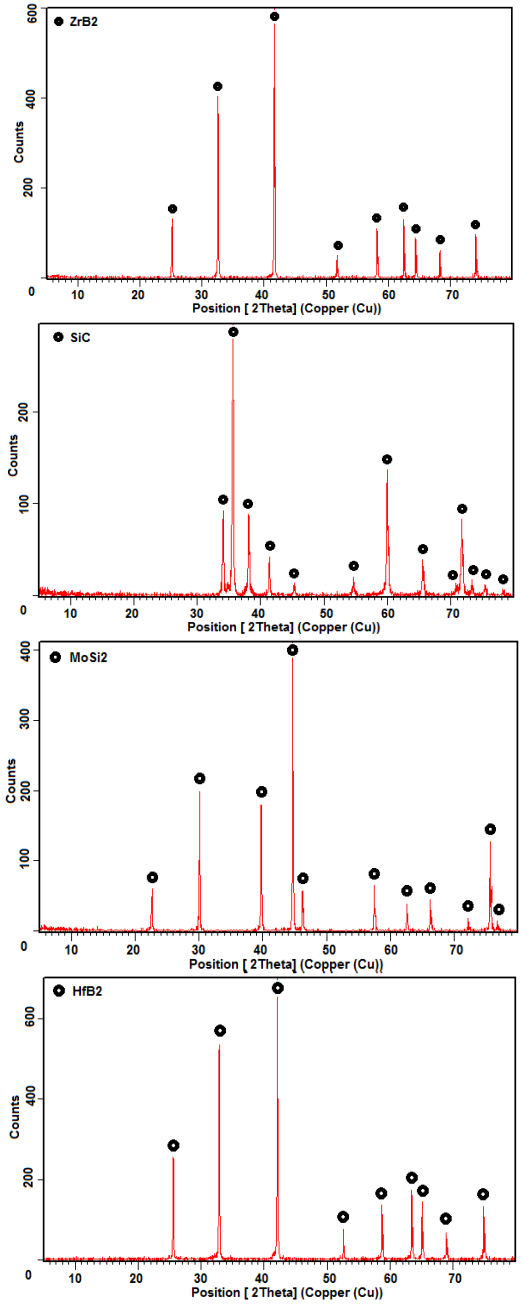

Figure 1. XRD analysis of raw powders.

TABLE 1. Process factors and their levels used in the experiments.

Factors	unit	Sym.	L.1	L.2	L.3	L.4
A: SiC	vol%	SiC	5	10	15	20
B: C_f	vol%	C_f	0	2.5	5	7.5
C: M.t	hr	M.t	0	2.5	5	7.5
D: $MoSi_2$	vol%	$MoSi_2$	0	2	4	6
E: HfB_2	vol%	HfB_2	0	5	10	15
F: ZrC	vol%	ZrC	0	5	10	15
G:Temperature	°C	T	1600	1700	1800	1900
H: Pressure	MPa	P	10	20	30	40
I: Time	min	t	4	8	12	16

TABLE 2. Preparing conditions for each sample

TEST	SiC	C_f	M.t	$MoSi_2$	HfB_2	ZrC	T	P	t
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

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 min and 16 min) based on Taguchi design (TABLE 2.) in vacuum. Final sintered specimen size was 50 mm in diameter and different thicknesses of 2.8 mm and 6.5 mm related to their compositions and SPS conditions. After grinding the surface layer of the obtained disk, the bulk density was measured according to ASTM C 373-88. Then the phase composition was determined by X- ray diffraction (XRD, Siemens, D500) using Cu-Ka radiation on polished composite. Scanning electron microscopy (Sigma / VP, Zeiss) was performed to observe the microstructures of the composites equipped with backscattered electron imaging (BSE).

3. RESULT AND DISCUSSION

In this study, some of ZrB_2–based composites, such as 11, 12, 24 samples, sintered by SPS had a bulk density of 6.22 g/cm^3, 5.67 g/cm^3 and 5.98 g/cm^3 respectively. By conducting the rule of mixture calculation while assuming the true densities were 6.09 g/cm^3 for ZrB_2, 3.21 g/cm^3 for SiC, 6.65 g/cm^3 for ZrC, 11.212 g/cm^3 for HfB_2, 6.24 g/cm^3 for $MoSi_2$ and 1.8 g/cm^3 for C_f, the theoretical densities of composites, 11. 12 and 24 were calculated to be 5.74 g/cm^3, 5.40g/cm^3 and 5.73 g/cm^3 respectively. Based on these true density values, the relative densities of composites 11, 12 and 24 sintered via SPS exceeded 100% because of the phase transformations. So calculating the relative density is impossible and the open porosity percent was chosen instead to evaluate the densification behaviour of the composites in this study.

TABLE 3. shows the results of the open porosity percent for the 32 mixtures prepared. It was repeated four times for each mixture. The results of each times are shown as Trial 1 to 4 in TABLE 4. It is apparent that the average open porosity percent in samples 1, 7, 8, 16, 17, 19, 21, 22, 27 and 28 is more than 10% while it is less than 1% in samples 2, 6, 9, 14, 18, 23, 24, 25 and 29. In other words, these samples have the best densification. SEM images of some of these samples are shown in Figure 2. These images are in accordance with the open porosity results (TABLE3).

TABLE 3. Results of open porosity percent of all samples

Sam.	Average Open porosity %	Sam.	Average Open porosity %
1	29.84	17	14.15
2	0	18	0
3	1.32	19	16.98
4	0.68	20	1.40
5	1.96	21	15.07
6	0	22	10.74
7	12.8	23	0.55
8	10.94	24	0.18
9	1.1	25	0.48
10	9.1	26	7.6
11	3.94	27	15.6
12	1.63	28	12.52
13	1.15	29	0.081
14	0.03	30	6.8
15	3.31	31	3.13
16	13.52	32	6.55

The data were entered to Qualitek-4 software (version 14.5). The results of ANOVA analysis before and after pooling are given in Tables 4 and 5. respectively. It is

clear that temperature with 50.2%, $MoSi_2$ with 20.7% and time with 9.8% have the most portions on the open porosity, respectively. These results indicated that the SPS conditions (temperature and time) and $MoSi_2$ as sintering aid are very important parameters on densification.

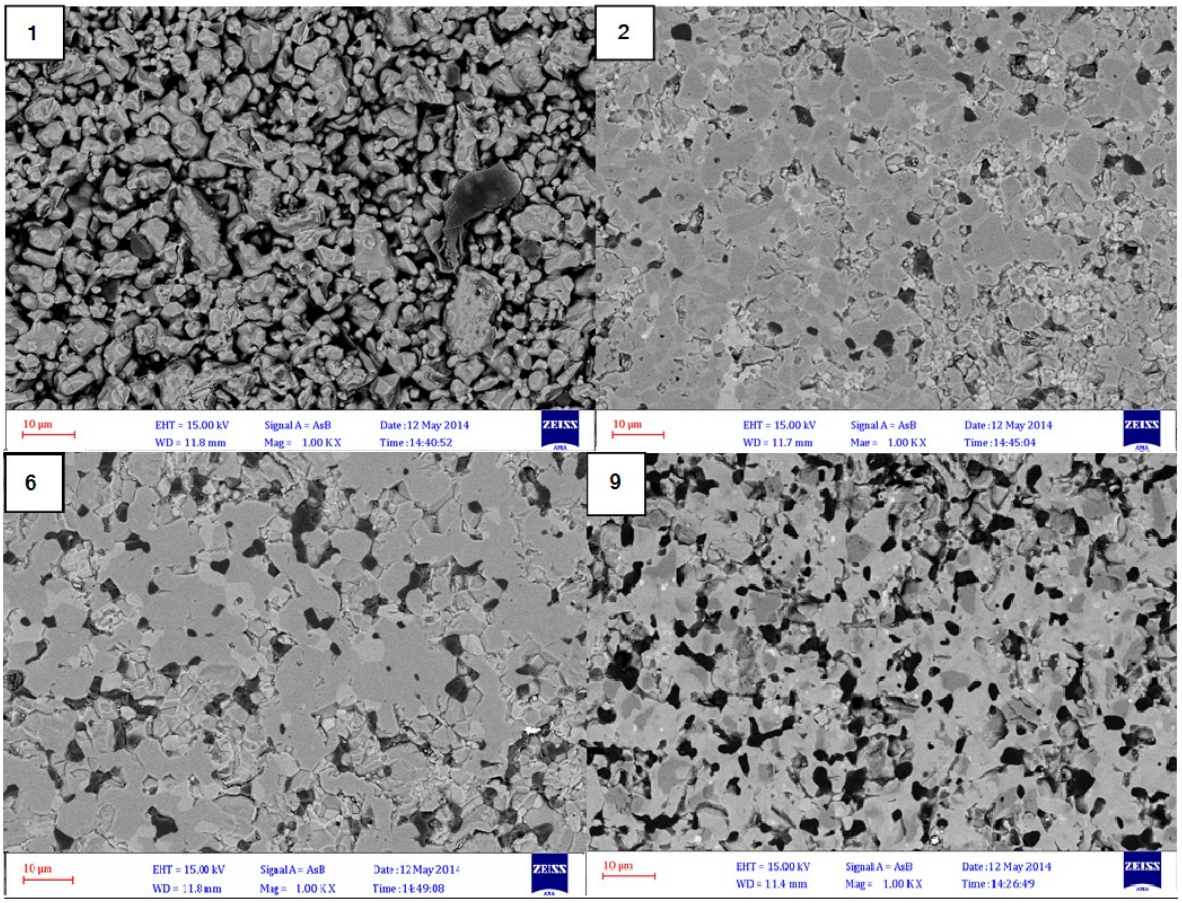

Figure 2. SEM images of samples 1, 2, 6 and 9.

Pressure with 0.8%, ZrC with 1.9%, and HfB_2 with 1.9% have the least portion on open porosity. The other parameters including SiC, M.t, and C_f have 2.9%, 3.6% and 3.8% on open porosity, respectively. Generally, it can be seen that there is no significant difference between data before and after pooling. The only difference is the total error which is higher after pooling due to addition of minor factors portion to it.Standard analysis method was used to investigate the effect of each parameter on densification behaviour.

TABLE 4. ANOVA analysis results for all parameters before pooling.

Fac.	f	S	V	F	s'	P
SiC	3	186.5	62.2	29.3	180.2	2.9
C_f	3	249.1	83	39.2	242.8	3.85
M.t	3	232.9	77.6	36.6	226.53	3.6
$MoSi_2$	3	1313	437.7	206.6	1306.7	20.7
HfB_2	3	124.9	41.6	19.7	118.6	1.9
ZrC	3	129.1	43	20.3	122.8	1.95
Temp.	3	3168	1056	498.6	3161.6	50.2
Press.	3	57.6	19.2	9	51.3	0.8
Time	3	626.1	208.7	98.5	620	9.8
Error		211.8	2.1			4.2
Total						100

TABLE 5. ANOVA analysis results for all parameters after pooling.

Fac.	f	S	V	F	s'	P
SiC	3	186.5	62.2	12.9	172.1	2.8
C_f	3	249.1	83	17.3	234.7	3.7
M.t	3	232.9	77.6	16.2	218.5	3.4
$MoSi_2$	3	1313	437.7	91.1	1298.6	20.7
HfB_2	3	124.9		Pooled		
ZrC	3	129.1		Pooled		
Temp.	3	3168	1056	219.9	3153.6	50
Press.	3	57.6		Pooled		
Time	3	626.1	208.7	43.4	611	9.7
Error		211.8	2.1			9.6
Total						100

3.1. Effect of SiC, C_f and M.t on Open Porosity Percent

Figure 3. shows the effect of SiC, C_f content and M.t on the open porosity percent. Ascan be seen from the figure, open porosity percent decreased by increasing the SiC content. It means the higher SiC content, promotes densification.

It is well-known that the densification of deboride powders like TiB_2, ZrB_2 and HfB_2 are difficult and generally requires high temperatures and external pressures due to their strong covalent bonding and low self-diffusion. In addition, oxygen impurities (B_2O_3 and ZrO_2) present on the surface of starting powder have been shown to inhibit densification because of evaporation of B_2O_3 in the non-oxide ceramic systems[4,8].

Monteverde [4] showed that ZrB_2 with 10 vol% ultra-fine SiC ($d90$ = 0.8µm) achieved full density by HP at 1900 °C and 40 MPa for 20 min in vacuo. Hwang et al. [14] investigated the effect of SiC content on densification behaviour of ZrB_2-based ceramics prepared by HP and reported the same results. The improvement of densification upon addition of SiC was attributed to the formation of intergranular liquid phases during hot-pressing, assisting in densification at lower temperatures. M. Ikegami et al. reported the similar results in ZrB_2-SiC composite prepared by SPS [8].

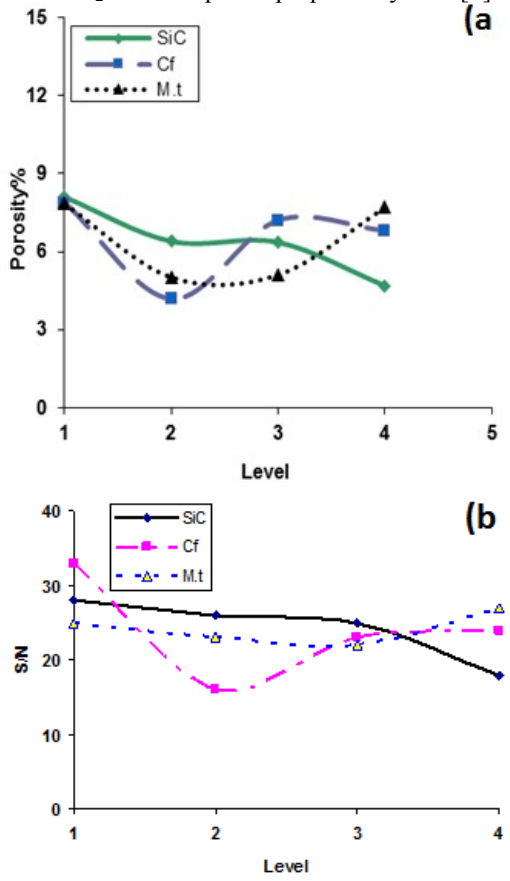

Figure 3. Effect of SiC, C_f and M.t on open porosity percent by a) standard analysis, b) S/N.

Apparently, the presence of SiO_2 prevents evaporation of B_2O_3 during hot pressing and results in formation of a stable liquid phase among the grains, hence improving the sinterability. It is common for B_2O_3 and SiO_2 films to be present on the surfaces of the starting ZrB_2 and SiC powder particles, respectively. According to the phase diagram for B_2O_3–SiO_2, a stable Si–B–O liquid phase is formed among the grains when B_2O_3 and SiO_2 coexist [8].

In our work, we can conclude that the densification improvement is due to the formation of similar intergranular liquid phase.

In fact, the presence of an intergranular liquid phase favours the process of grain rearrangement and improves the packing density of particles, hence improving the densification. In addition, the amount of the intergranular liquid phase increases with increasing SiC content[28].

Figure 3. shows the effect of chopped C_f on open porosity percent. It appears that by increasing C_f content, open porosity percent initially decreased up to 2.5 vol% and then increased with further increasing. It means that low C_f content promotes densification while higher amounts inhibit densification. S. Guo et al. [15] investigated the effect of Short Pitch-based carbon reinforced fibre HfB_2 matrix composites containing 20vol%SiC, with fibre volume fractions in the range of 20–50%, manufactured by hot-press process at 2100 °C and 20 MPa for 60 min. They concluded that the shrinkage behaviour of the composites was linked to fibre volume fraction. The onset temperature of densification was determined to be ~ 1490 °C for HSGF20, ~1510 °C for HSGF30, ~1550 °C for HSGF40, and ~1820 °C for HSGF50. It is evident that the onset temperature of densification was raised with increasing of fibre volume fraction. This indicated that the densifying muechanism of the HfB_2–SiC matrix powder, such as grainboundary diffusion and grainboundary migration, was inhibited due to the presence of the fibre.

In our research, C_f content does not exceed 7.5 vol%. So the effect of C_f on densification is negligible due to its low content (TABLE 5. 3.8%). Also, we concluded that C_f up to 2.5 vol% is not enough to inhibit grainboundary diffusion and grain-boundary migration. So densification does not reduce.

Milling time of C_f up to 5 hr decreases open porosity percent and then increases it by more increasing, as shown in Figure 3.

SEM images of ball milled C_f for different milling times (0, 2.5, 5, 7.5 hr) are shown in Figure 4. It is clear that by increasing the milling time, surface of C_f changes and small particles of C_f are observed. This trend can be seen in all C_f which ball milled for different times.

However in 7.5 hr milling time in addition of small particles of C_f, C_f with different lengths can be seen. It is indicated that 7.5 hr ball milling causes heterogenic C_f lengths to form. So, we can conclude, the open porosity percent decreases until 5 hr milling time may be by filling the pores due to small particles of C_f formed on its surface. Increasing the open porosity percent in 7.5 hr milling time is related to the formation of heterogenic C_f lengths.

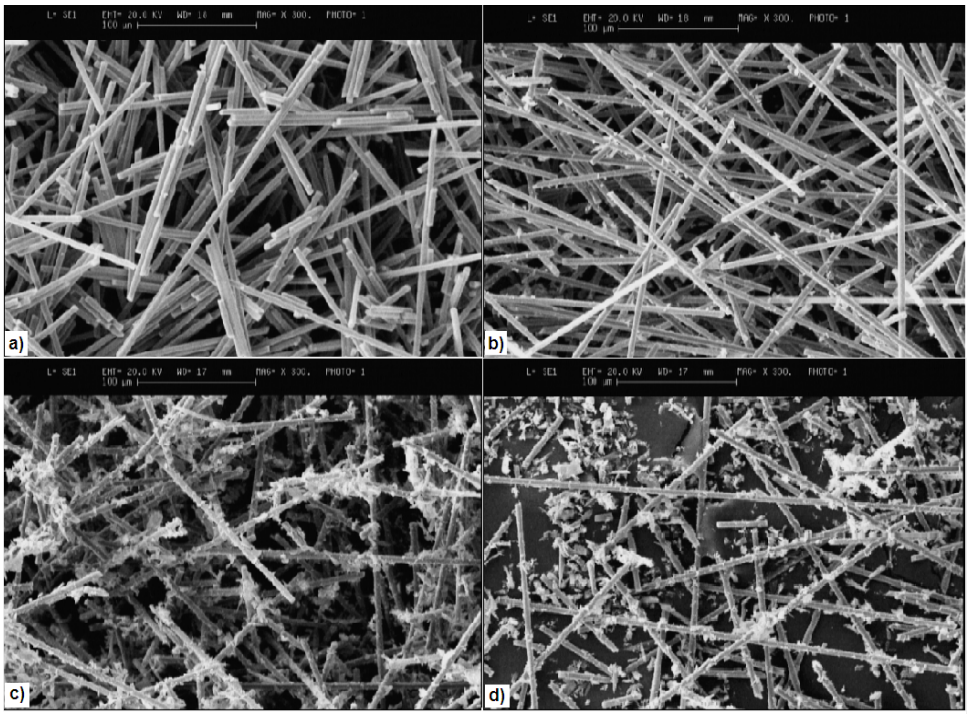

Figure 4. SEM images of C_f ball milled for a) 0, b) 2.5 hr, c) 5 hr d) 7.5 hr.

3.2. Effect of $MoSi_2$, HfB_2 and ZrC on Open Porosity Percent Effect of $MoSi_2$, HfB_2 and ZrC on open porosity is shown in Figure 5. Open porosity percent decreased by increasing $MoSi_2$ content, as shown in Figure 5. $MoSi_2$ is the best sintering aid among other additives used in this paper. According to TABLE 5. its portion on densification is 20.7%. This result is in good acordence with findings of other researches. In the early 1970s, Kinoshita et al. [4] systematically investigated densification behaviour of ZrB_2-based composites with $MoSi_2$. They found that $MoSi_2$ significantly improved sinterability of ZrB_2 powder and relative density exceeding 95% was obtained for ≥20 vol% $MoSi_2$-containing ZrB_2 powder at 2000 °C and 20MPa for 20 min.

The role of $MoSi_2$ was to activate densification by means of chemical reactions involving surface oxides. The reactions lead to the removal of oxide species from ZrB_2 particle surfaces by the formation of a (transient) liquid phase[16-18]. In our study, it is expected that similar intergranular liquid phase forms and improves densification.

F. Monteverde [16] also studied the effect of $MoSi_2$ on densification of ZrB_2 prepared by hot press (HP). He concluded that the rapid increase in the shrinkage rate for the $MoSi_2$-doped compositions above 1650 °C was attributed to the formation of a liquid phase which favours the process of grain rearrangement and also improves the packing density of particles, resulting in improved densification. According to TABLE 4. it can be seen that composites which prepared by sintering at 1600 °C (i.e. composites 1, 7, 10, 16, 19, 21, 28, 30), the open porosity percents are more than 6%.

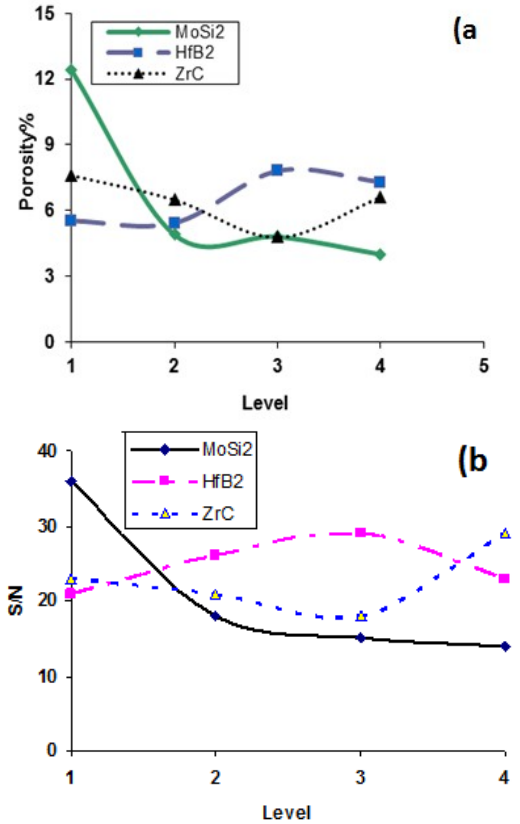

Figure 5. Effect of $MoSi_2$, HfB_2 and ZrC on open porosity percent by a) standard analysis, b) S/N.

It means, it is impossible to reach nearly full densification in 1600 °C even with 6 vol% $MoSi_2$ sintering aid (i.e. composites 7 and 10) while the open porosity percent in composite 2, containing only 2 vol% $MoSi_2$ sintered at 1700 °C, is zero. So, we can conclude that there is a critical temperature for $MoSi_2$ to act as a sintering aid (T≥1700 °C). It is due to the melting point of SiO_2, (~1725 °C)[17]. SiO_2 and Mo borides are produced as a result of reactions between $MoSi_2$ and oxide species (impurity on the surfaces of starting powders). Therefore, sintering temperatures more than 1700 °C is necessary for liquid phase formation.

S. Q. Gue [19] studied densification of ZrB_2-$MoSi_2$-SiC composite and reported similar results.

The ability of $MoSi_2$ as a very good sintering aid, can be clearly seen by comparing the open porosity percent in composites 2 and 6 (TABLE 4). The open porosity percent of both composites is zero. With respect to TABLE 3. the composite 6 was sintered at 1900 °C, 30 MPa for 12 min without $MoSi_2$ while composite 2 was sintered at 1700 °C, 20 MPa for 8 min with only 2 vol% $MoSi_2$. It is concluded that using only 2 vol% $MoSi_2$ can reduces SPS temperature for several hundreds centigrade. In addition, with respect to Tables 3 and 4. it is clear that 2 vol% $MoSi_2$ is enough to reach nearly full densification for composites which were sintered at 1700 °C and 1800 °C by SPS method. Higher $MoSi_2$ does not have more effect. Also, for composites with 1900 °C sintering temperature, no $MoSi_2$ is required to reach nearly full densification (e.g. composite 6). It seems that it is possible to reach full densification by SPS method without using any sintering aid while it is impossible in HP and PLS methods at temperatures lower than 2100°C. This improvement should be reasonably attributed to the higher heating rate and dielectric breakdown mechanisms occurring during the spark plasma sintering process than HP and PLS.

The effect of HfB_2 on the open porosity percent is shown in Figure 5. It is observed that by increasing HfB_2, open porosity percent increases a little at first, then keep nearly constant at higher HfB_2 content. With respect to Zr-Hf-B ternary diagram phase [20], addition of HfB_2 to ZrB_2 causes to inform (Zr, Hf)B_2 solid solution due to their nearly equal lattice parameters. According to E. Rudy [20], by increasing HfB_2 amount, melting point of (Zr, Hf)B_2 solid solution does not change significantly. It originates from nearly equal melting points of ZrB_2 (3245 °C) and HfB_2 (3380 °C). According to TABLE 5. HfB_2 portion on open porosity is very little, 1.9%, which is in accordance with Rudy' s data. We can conclude that HfB_2 does not have any significant effect on open porosity percent and does not act as sintering aid.

Figure 5. shows the effect of ZrC on the open porosity percent. It can be seen that ZrC first decreases open porosity percent (up to 10 vol%), so improves densification. The phase diagram for the Zr–B–C system shows that, the solubility limit for ZrC in ZrB_2 is 4.5 wt%. Also binary diagram phase of ZrB_2-ZrC shows, addition of ZrC decreases liquidus temperature[20].

V. M. Gropyanov and et. al [21] investigated the effect of ZrC and ZrB_2 addition to each other. They found that the sinterability of ZrB_2 is greatly inferior to that of ZrC and that the addition of as little as 10 wt% ZrC to ZrB_2 or 10 wt% ZrB_2 to ZrC substantially improves the sinterability of these materials. A. Snyder et al [22] reported the same results for the effect of ZrC in ZrB_2–SiC–ZrC ultra high temperature composites with four different compositions prepared via SPS at a maximum temperature of 1800 °C.

According to TABLE 5. its portion on open porosity percent is only 1.9%. So, it can be concluded that ZrC does not act as a very good sintering aid like $MoSi_2$. This can be attributed to its strong covalent Zr-C bond which gives this material a very high melting point (~3530 °C). However ZrC is very good grain growth inhibitor which will be discussed in our future work.

3.3. Effect of SPS Conditions on Open Porosity Percent Figure 6. shows the effect of SPS conditions on open porosity percent. It is observed that the open porosity percent decreased significantly by increasing temperature, as shown in Figure 6. A. Teber et al. [23] reached same results by sintering TiC in different temperatures by SPS method. By increasing the temperature from 1350 °C to 1800 °C, the density increases from 93.1 % to about 99.4 %, for 5 min holding time under 80 MPa. They considered that diffusion bounding at the particle boundaries and other high diffusivity paths are enhanced at higher temperatures. So, reorientation of TiC particles occurs and pores are filled as a result of boundary sliding and grain rotation, resulting in higher density materials. In our work, we concluded that the similar mechanism involves in improving densification by increasing temperature.

Figure 6. shows the effect of pressure on the porosity percent. Open porosity percent decreases slightly by increasing pressure up to level 3(30 MPa) and then iexperiences a little increase.

With respect to TABLE 5. the results indicate that, compared with pressure condition (0.8 % portion on densification), temperature and holding time play a more significant role in densification in SPS method by 50.2% and 20.7% portion on densification, respectively. These results are completely consistent with literatures [23, 24].

Shen et al. [25] considered that pressure condition-related enhancement in densification when a higher pressure was applied could be mainly attributed to a higher packing density of the particles. Particularly when the initial powder contains agglomerates, the probability of breakdown of soft/hard agglomeration

increases with rising applied pressure, and such a process also accelerates densification.

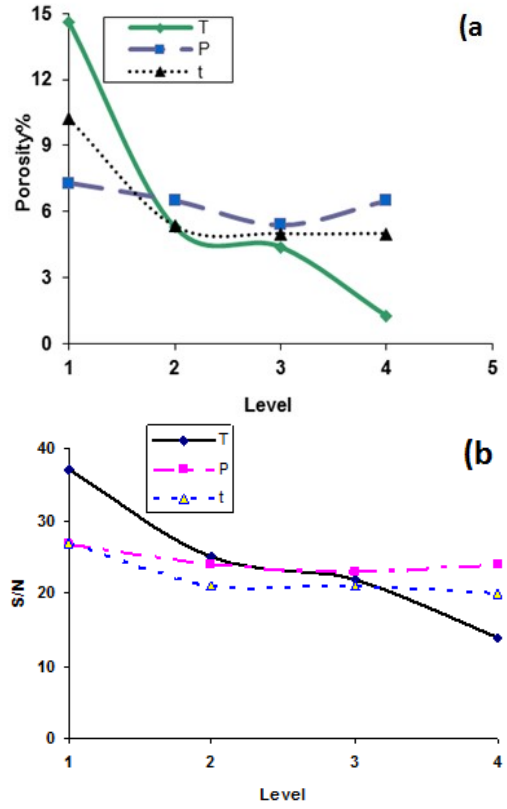

Figure 6. Effect of SPS conditions Temperature, Pressure and Time on open porosity percent by a) standard analysis, b) S/N.

Hu et al. [26] concluded that pressure contributes to the densification by compacting the grains and causing the plastic deformation.

The effect of time on open porosity percent is shown in Figure 6. It is clearly observed that by increasing the holding time open porosity percent decreases.

4. CONCLUSION

1. It has been concluded that the most significant effect on the densification is related to the sintering temperature, $MoSi_2$ content and time by 48.4%, 20.7% and 9.8% portion, respectively.

2. Pressure by 0.9%, ZrC and HfB_2 by 1.9% have the least effect on the densification.

3. Other parameters including SiC, M.t, and C_f have 2.9%, 3.6% and 3.8% effect on open porosity percent, respectively.

4. By increasing the temperature, time, $MoSi_2$ and SiC from level 1 to 4, densification improved continuously while by increasing HfB_2 from level 1 to 4, densification decreased consistently.

5. The optimum condition, to reach the most desiccation is T4, t4, P3, SiC4, C_f2, M.t2 or 3, $MoSi_24$, HfB_21 and ZrC3. The numbers indicate the level of each parameter.

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