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Taguchi analysis on the effect of hot pressing parameters on density and hardness of zirconium diboride

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ABSTRACT

The influence of process parameters on density and hardness of monolithic zirconium diboride ceramic, during hot pressing, has been investigated. This paper publicizes a design of experiment approach, Taguchi methodology, used to improve the processing of ZrB_2 concentrated upon three sintering parameters: temperature, dwell time and pressure. An L9 orthogonal array procedure, involves nine experiments for three factors with three levels, which comprises the signal to noise ratio and analysis of variance was employed to optimize the hot pressing parameters. The "higher is better" condition was selected as the quality characteristic. The statistical analysis recognized consolidating temperature as the most consequential factor affecting the relative density and hardness of ZrB_2. A relative density of ~93% was obtained with a temperature of 1850 °C, dwell time of 90 min and pressure of 16 MPa. The Vickers hardness of ZrB_2 reached ~13.5 GPa at optimal hot pressing conditions. The confirmation tests, carried out under optimum conditions, revealed that the experimental results were in harmony with the expected values from the Taguchi model.

1. Introduction

Zirconium diboride (ZrB_2), prevalently known as a member of ultrahigh-temperature ceramics (UHTCs), has superior combined characteristics of high melting point, high electrical and thermal conductivities, and also fantastic chemical inertness. However, low mechanical properties and poor oxidation resistance are still the impediments for ZrB_2-based materials to be exploited widely. Due to the strong covalent bonding and low self-diffusion coefficient of ZrB_2, it is difficult to consolidate a fully dense ceramic in the absence of secondary phases or any sintering aids [1–5].

Pure ZrB_2 ceramic requires a high hot pressing temperature (>2100 °C), with pressures of 20–30 MPa, or a moderate temperature (e. g. 1800 °C), with much higher pressures (800–1500 MPa) to be densified completely. Hot pressing of coarse ZrB_2 powders (20 μm), even at 2000 °C, leads to a porous sample with a relative density of 73%. Achieving a fully dense ZrB_2 ceramic, with a powder size of 5–10 μm, requires sintering above 2000 °C [6]. Using finer starting materials decreases the hot pressing temperature to 1900 °C, needed to obtain full density [7].

However, it is essential to mention that when consolidating at relatively high temperatures, the densification process is usually accompanied with an undesirable intensive grain growth. For example, grain size of a ZrB_2 ceramic, consolidated at 1850 °C, is larger than that of hot pressed at 1700 °C, by a factor of 1.55. In addition, grain size of another sample hot pressed at 2000 °C is two times larger than that of sintered at 1700 °C [8]. Study of the densification behavior of a bimodal micron/nano-sized ZrB_2 ceramic revealed that a relative density of 99.2% as well as improved mechanical properties can be achievable using a two-step hot pressing (step 1: 1300 °C/30 min/30 MPa; step 2: 1900 °C/30 min/30 MPa) [2].

Results of hot pressing investigations on monolithic ZrB_2 ceramics are listed in Table 1, which contains size of starting powders, hot pressing conditions, and corresponding relative density and Vickers hardness of the sintered samples. There is no systematic investigation performed to infer the effect of process parameters on densification and mechanical properties of monolithic ZrB_2 ceramic (or other UHTCs) during hot pressing. However, a statistical analysis, utilizing the Taguchi method, has recognized consolidating temperature as the most important factor affecting the densification of spark plasma sintered HfB_2–20 vol.% SiC composites [9]. Hence, the target of the current study is to deduce and recognize the crucial hot pressing parameters (temperature, dwell time and pressure) affecting the density and hardness of zirconium diboride. The signal to noise ratio and the analysis of variance approach

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Table 1
Process conditions and corresponding relative density and Vickers hardness of hot pressed ZrB_2 ceramics.

Particle size (μm)	Hot pressing conditions			Relative density (%)	Vickers hardness (GPa)	References
	Temperature (°C)	Dwell time (min)	Pressure (MPa)			
20	2000	20	20	73	–	[6]
2.1	2000	60	30	90.4	–	[10,11]
5–10	1800	60	20	78	–	[6]
6	1900	30	30	86.5	8.7 ± 0.4	[12]
2.5	1900	30	30	80.4	–	[2]
2	2000	30	10	96.8	13.4 ± 0.5	[8]
2	1900	45	32	99.8	23 ± 0.9	[7]
2	1900	60	30	99.8	–	[13]
2	1850	30	10	89	10 ± 0.4	[8]
2	1700	30	10	80	6.5 ± 0.3	[8]
2	1650	120	60	71.6	–	[14]
0.06–0.08 + 2.5	1900	30	30	95.1	–	[2]
0.06–0.08 + 2.5	1300/1900	30/30	30/30	99.2	–	[2]
0.06–0.08	1900	30	30	91.3	–	[2]

are used to estimate the significances and contributions of hot pressing parameters on the relative density and hardness of zirconium diboride.

2. Design of experiment

Experiments were designed established upon the Taguchi method to deduce the effect of three process parameters on density and hardness of ZrB_2. The Taguchi methodology is economical as fewer experiments than the classical techniques are required. Hence, this technique is more versatile than the other procedures [15]. The effect of three hot pressing parameters: sintering temperature, dwell time and applied pressure was investigated in this research. If only three process factors are studied in an analysis, an orthogonal array with nine incorporations of input parameters can be employed. The interactions between the process factors are insignificant with the L9 orthogonal array. The L9 orthogonal array and the levels of the parameters, each parameter was designed with three levels, scrutinized in this study are outlined in Table 2, and the nine combinations of the factors are listed in Table 3.

The output characteristics (e. g. relative density and Vickers hardness) can be analyzed into three classifications; "lower is better", "higher is better" or "nominal is better" condition [16]. In this research, a high relative density and a high hardness are desirable; therefore the statistical analysis is performed with the "higher is better" choice. The Taguchi method employs the signal to noise (S/N) ratio to quantify the quality characteristic divergence from the desired values. The S/N ratio demonstrates the relation between useful outcome (signal) and deviation of measured values (noise). Moreover, this ratio simplifies the optimization process, due to the fact that the larger the S/N ratio, the more optimal outcomes were obtained [15,16]. The S/N ratio for the "higher is better" choice is calculated as per Eq. (1):

$$\frac{\mathrm{S}}{\mathrm{N}} = -10\log\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right) \tag{1}$$

Table 2
Processing parameters and selected levels.

Hot pressing parameters	Range	Level 1	Level 2	Level 3
Temperature (°C)	1700–1850	1700	1775	1850
Dwell time (min)	30–90	30	60	90
Pressure (MPa)	8–16	8	12	16

Table 3
Description of experimental design matrix (L9 orthogonal array).

Trial no.	Notation	Hot pressing conditions		
		Temperature (°C)	Dwell time (min)	Pressure (MPa)
R-1	1700-30-08	1700	30	8
R-2	1700-60-12	1700	60	12
R-3	1700-90-16	1700	90	16
R-4	1775-30-12	1775	30	12
R-5	1775-60-16	1775	60	16
R-6	1775-90-08	1775	90	8
R-7	1850-30-16	1850	30	16
R-8	1850-60-08	1850	60	8
R-9	1850-90-12	1850	90	12

where y_i ($i = 1, 2 \ldots n$) is the response values and n is the number of reiterations. Analysis of variance (ANOVA) was fulfilled to evaluate the importance and contribution of each factor to the overall relative density and Vickers hardness. The data analysis of the results was performed using Qualitek-4 software (Automatic design and analysis of Taguchi experiments, Nutek Inc., Michigan, USA).

3. Experimental procedure

3.1. Material and process

A commercially available ZrB_2 powder (Leung Hi-tech Co., China) with a particle size of ~2 μm and a purity of 99% was used as the starting material. Oxygen content was 0.55 wt.% and other impurities were 0.2 wt.% C, 0.1 wt.% N, 0.05 wt.% Fe and 0.1 wt.% Hf. Five billets, with a diameter of 25 mm and thickness of 5 mm, were prepared for each experiment. In this way, the powders were loaded into a graphite die (Fig. 1) coated with boron nitride and lined with a thin graphite foil. Hot pressing was completed in a graphite resistance-heated vacuum hot press furnace (made by Shenyang Weitai Science & Technology

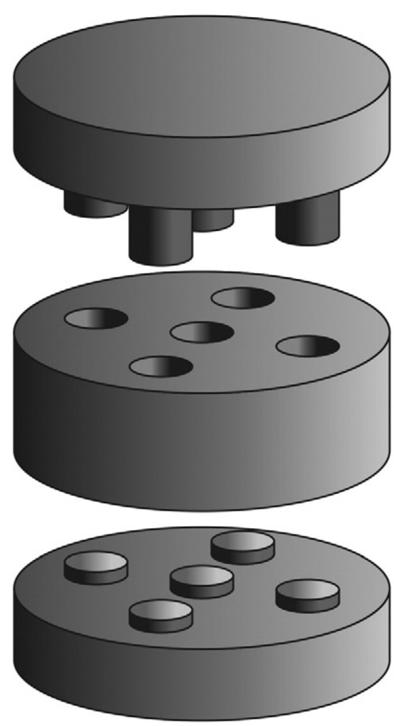

Fig. 1. Schematic of three-piece graphite die was employed for hot pressing of the samples (outer diameter: 150 mm).