Retraction

Retracted: Optimization of the Spark Plasma Sintering Process for High-Volume Fraction Tungsten Carbide/Al 2025 Composites

Advances in Materials Science and Engineering

This article has been retracted by Hindawi following an investigation undertaken by the publisher [1]. This investigation has uncovered evidence of one or more of the following indicators of systematic manipulation of the publication process:

(1) Discrepancies in scope

(2) Discrepancies in the description of the research reported

(3) Discrepancies between the availability of data and the research described

(4) Inappropriate citations

(5) Incoherent, meaningless and/or irrelevant content included in the article

(6) Peer-review manipulation

The presence of these indicators undermines our confidence in the integrity of the article's content and we cannot, therefore, vouch for its reliability. Please note that this notice is intended solely to alert readers that the content of this article is unreliable. We have not investigated whether authors were aware of or involved in the systematic manipulation of the publication process.

Wiley and Hindawi regrets that the usual quality checks did not identify these issues before publication and have since put additional measures in place to safeguard research integrity.

We wish to credit our own Research Integrity and Research Publishing teams and anonymous and named external researchers and research integrity experts for contributing to this investigation.

The corresponding author, as the representative of all authors, has been given the opportunity to register their agreement or disagreement to this retraction. We have kept a record of any response received.

References

[1] C. R. Mahesha, C. Polayya, M. L. Rinawa et al., "Optimization of the Spark Plasma Sintering Process for High-Volume Fraction Tungsten Carbide/Al 2025 Composites," *Advances in Materials Science and Engineering*, vol. 2022, Article ID 6433716, 7 pages, 2022.

Research Article

Optimization of the Spark Plasma Sintering Process for High-Volume Fraction Tungsten Carbide/Al 2025 Composites

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With an orthogonal experimental design, Spark plasma sintering method was used to create tungsten carbide with Al 2025 composites. Pressure, temperature, and hold time all had an influence on the mechanical characteristics of the composites. Pressure had the greatest effect on density and bending strength, followed by temperature and holding duration. After five minutes of sintering at 556°C under 40 MPa, a 50% tungsten carbide/Al 2025 composite material was produced, resulting in a density of 997.7 percent and a spectacular bending strength of 766.65 MPa. This study revealed the ability of high-volume fraction mixes to support huge loads.

1. Introduction

In addition to Si_3N_4, SiO_2, Al_2O_3, B_4C/Al, TiC, and WC, PAMCs have been researched, for their outstanding mechanical, chemical, and electrical qualities [1, 2]. As a result of their extraordinary dimensional stability, high specific modulus mixtures have developed more significantly in the aviation and microelectronics productions [3–5]. Squeeze or stir casting, pouring, power metallurgy, hot pressing, and pressure less infiltration have all been employed to create silicon carbide particle/2xxx aluminium composites [6, 7]. With these processes, it is difficult to limit pores, control bonding and reactivity between interfaces, and produce composite materials with huge volumes [8]. Experiment

failed to eradicate the holes that affect mechanical behavior to decline meaningfully due to high temperatures and lengthy vacuum hot pressing holding times. The tensile properties of hot-pressed tungsten carbide particles/2009 aluminiummixtures were examined by the authors [9] in this paper.

Spark plasma sintering seems to be a pressure-assisted pulsed-current method in which powders are put into an electrically conducting die and fused under pressure gradient. The Spark plasma sintering technique may be employed to produce composite materials with low porosity and good mechanical characteristics [10, 11]. This process has a greater heating rate than standard preparation methods, a lower preparation temperature, and quick

densification of the composite materials [12]. Improved interfacial bonding, cleaner surfaces, and smaller particle sizes are all advantages of SPS. To make 50 percent WC/6061Al composites, the authors in [13, 14] utilised Spark plasma sintering technology. Because of its 99.15 percent density and excellent interfacial attachment among the Al matrix and the tungsten carbide, it possessed a bending strength of 694 MPa. These composites were created by [15] using SPS to manufacture high-density 40 to 55 vol% tungsten carbide particles/pure aluminium composites with densities more than 99 percent [16, 17].

In Taguchi's multifactor orthogonal experimental design, the orthogonal array serves as the foundation [18–20]. It is easy to pick sample points that are equally dispersed across the whole factor test for optimum results in multifactor research. To further understand Spark plasma sintering preparation processing factors for TiB_2-silicon carbide ceramic materials, a total of four treatment variables at three orthogonal experimental design degrees were examined [21, 22]. Vickers hardness was determined to be 28.5 GPa at the ideal processing conditions for this composite. In addition to reducing the number of experiments, this method produces data that can be understood by everyone [23]. The impact of processing parameters on HfB_2, Al_2O_3, ZrB_2-based ceramics, and phenolic fibre has been widely explored in the literature using orthogonal experimental design [24, 25]. The manufacturing of 50 vol percent tungsten carbide/2025Al composites has not yet been reported by Spark plasma sintering through orthogonal experimental design. tungsten carbide/2025Al/SiC composites with large volume proportions of SiC were the focus of this research, and mechanical study was employed to explore the mechanical characteristics of the composite material [26–28]. It was feasible to compare the effects of various factors on the best sintering conditions for 50 vol percent tungsten carbide/2025Al composites using orthogonal experimental design (such as temperature, pressure, and holding time) [29]. In addition to high-volume fraction composite materials, this method's intelligence may be used to other high-volume proportion materials [30].

2. Materials and Methods

2.1. Materials. Tungsten carbide and 2025Al powders with particle sizes of 10 micron were employed as starting materials. Its chemical makeup is composed of Al and 4.7 percent Cu with the remainder being composed of 0.9 percent Mg, 0.6% iron, 0.6% silicon, and just 0.1% magnesium (mass fraction). Utilizing a 3:1 ball-to-powder weightage ratio in a zirconia tank, planetary ball mixing machine ran at 280 rpm for 2 hours to mix the tungsten carbide and 2025 Al particles. Particle fracture or Al flaking was not evident in either powder when comparing tungsten carbide with 2025Al powders. The combined powder was sintering in a graphite mould that had been precompressed at 10 MPa for 5 minutes with a Spark plasma sintering device. To assess the relative importance of various variables during SPS, an orthogonal table is created (Table 1). Although the infrared thermometer's lowest measurement

Table 1: The orthogonal experimental design's factors and levels.

Level	A	B	C
1	500	3	25
2	525	4	35
3	550	5	45

temperature was 100°C, the instrument reported 100°C when the mold's temperature fell below the required minimum.

2.2. Characterization and Evaluation. With the use of a universal testing apparatus, samples measuring $3 \times 4 \times 36$ mm were tested for an average bending rate of 0.25 mm/min. An electronic analytical balance was employed to calculate the density of the composite. For the compactness and bending tests, a minimum of five samples were employed [31].

3. Results and Discussion

3.1. Analyzing Orthogonal Experiment. The L9 table was designated because of its orthogonal design and its three sintering temperatures, holding time, and pressure levels. Table 2 reveals the analysed results of the L9 orthogonal array. With K_{ij} ($i = 1, 2, 3$) and R as key factors, the orthogonal experiment is evaluated using the evaluation indices listed in Table 3. In order to compute R, which is the difference between highest and lowest values in relevant aspect column, K_{max} and K_{min} values are utilised. K_{ij} is the average of the test scores at the jth grade level ($j = 1, 2, 3$). The best factor level will be determined by associating K values. The R value can indicate how much the K value varies.

WC/Al will function better if it has a larger density and flexural strength; consequently, the greatest K value should be used. It was determined that $A2$ (525°C), $B1$ (3 minutes), and $C2$ (35 MPa) were the best parameters for producing a dense composite (35 MPa). These are the best parameters for enhancing the bending strength. Various influences on the WC/2025Al composite are represented by the R value. Bending strength and density were most affected by the sintering pressure, surveyed by the holding duration and sintering pressure, in this study, $B < A < C$.

Orthogonal test analysis cannot predict inaccuracy in testing since it does not know how much each element has changed or whether there has been a random fluctuation in findings, in spite of its simplicity. This is a concern that is often overlooked in orthogonal studies. The test findings were subjected to an analysis of variance in order to address this issue. SPSS Statistics was used to conduct the experimental variance analyses.

The significance of each component was evaluated at the $P = 0.05$ level of consequence for each individual. To be deemed significant, the test index must have an effect on the P value of less than 0.05. A, B, and C can explain 85.4% of the density variation in table, which demonstrates that $R_2 = 0.854$. Temperature, time, and pressure may account for 88% of the density change during sintering according to

Table 2: Analyzing the results of the L9 orthogonal array.

S. No.	A	B	C	Density	Relative density	Bending strength
	C	min	MPa	(g/cm^3)	(%)	(MPa)
1	1	1	1	2.85	96.8	692.42 ± 25.52
2	1	2	2	2.90	98.4	738.12 ± 46.27
3	1	3	3	2.90	98.4	712.87 ± 82.52
4	2	1	3	2.89	98.8	759.25 ± 12.12
5	2	2	1	2.79	96.7	632.17 ± 20.04
6	2	3	2	2.91	99.1	767.75 ± 24.82
7	3	1	2	2.92	97.2	692.24 ± 56.72
8	3	2	3	2.99	99.4	710.26 ± 71.65
9	3	3	1	2.91	94.6	586.42 ± 9.72

Table 3: Calculation indices of the L9 orthogonal array.

Orthogonal array	A	B	C
Density			
K_{11}	2.94	2.90	2.85
K_{12}	2.91	2.90	2.92
K_{13}	2.88	2.91	2.93
R	0.03	0.02	0.06
Level of optimum	A_2	B_3	C_3
Bending strength			
K_{11}	709.1	709.26	632.42
K_{12}	716.12	692.42	732.87
K_{13}	658.18	691.27	726.34
R	51.83	22.94	93.62
Level of optimum	A_2	B_1	C_2

$R_2 = 0.984\%$. $F = 1.891$; $P = 0.346$; $P = 0.159$; and $F = 5.307$; $P = 0.159$; bending strength was not changed by the sintering temperature. Sintering time $F = 0.388$; $P = 0.721$. An error of this magnitude ($df = 2$) and a low degree of freedom ($df = 2$) meant that the test was ineffective in distinguishing between variables of interest. ANOVA a higher sum of squares (SS) shows a bigger effect. $C > A > B$: density and bending strength were most strongly influenced by sintering pressure, whereas the holding time had the least impact.

Before a single index analysis approach is utilised to discover and optimise the complete scheme, the average weighting methodology is often employed to address multi-index orthogonal test concerns. The findings of the study are wrong since this method fails to take into account the differences and relative weights of the various indicators. As a result, the matrix approach was developed to quantify the degree to which each test aspect influences each directory and to swiftly establish the major and minor order of the components based on the weights. With this method, the issue of determining the best design for multi-index orthogonal tests is eliminated. The data are first organized into a three-tiered structural model. Experimentation index layer, component layer, and the flat layer make up the three layers (S). The formulas are given in equations (1)–(3), correspondingly, where $T_i = 1/\sum_{j=1}^{3} K_{ij}$ and $S_i = R_j/\sum_{i=1}^{3} R_{ij}$.

$$M = \begin{vmatrix} K_{11} & 0 & 0 \\ K_{12} & 0 & 0 \\ K_{13} & 0 & 0 \\ 0 & K_{21} & 0 \\ 0 & K_{22} & 0 \\ 0 & K_{23} & 0 \\ 0 & 0 & K_{31} \\ 0 & 0 & K_{32} \\ 0 & 0 & K_{33} \end{vmatrix}, \tag{1}$$

$$T = \begin{matrix} T_1 & 0 & 0 \\ 0 & T_2 & 0 \\ 0 & 0 & T_3 \end{matrix}, \tag{2}$$

$$S^T = \begin{bmatrix} S_1 & S_2 & S_3 \end{bmatrix}. \tag{3}$$

According to equation, the weight matrix (k) which influences the assessment index is listed in the following equation:

$$\begin{aligned} k &= M \times T \times S, \\ k^T &= \begin{bmatrix} k_1 & k_2 & k_3 \end{bmatrix}. \end{aligned} \tag{4}$$

Density was the first sign of success in this endeavor. Equations (5)–(8) demonstrate that to compute the weight matrix (8),

$$M = \begin{vmatrix} 2.93 & 0 & 0 \\ 2.94 & 0 & 0 \\ 2.90 & 0 & 0 \\ 0 & 2,92 & 0 \\ 0 & 2.92 & 0 \\ 0 & 2.93 & 0 \\ 0 & 0 & 2.87 \\ 0 & 0 & 2.94 \\ 0 & 0 & 2.95 \end{vmatrix}, \tag{5}$$

$$T = \begin{matrix} \dfrac{1}{8.72} & 0 & 0 \\ 0 & \dfrac{1}{8.80} & 0 \\ 0 & 0 & \dfrac{1}{8.78} \end{matrix}, \tag{6}$$

$$S^T = \begin{bmatrix} \dfrac{0.04}{0.13} & \dfrac{0.01}{0.13} & \dfrac{0.08}{0.13} \end{bmatrix}, \tag{7}$$

$$k_D = MxTxS = \begin{bmatrix} 0.1034 \\ 0.1037 \\ 0.1023 \\ 0.0255 \\ 0.0255 \\ 0.0256 \\ 0.1006 \\ 0.2061 \\ 0.2068 \end{bmatrix}, \quad (8)$$

$$k_{BS} = \begin{bmatrix} 0.1069 \\ 0.1075 \\ 0.0990 \\ 0.0454 \\ 0.0439 \\ 0.0437 \\ 0.0949 \\ 0.1956 \\ 0.1940 \end{bmatrix}. \quad (9)$$

The weightage matrix of the five pointers that passed the OED test is the sum of $K-D$ and $K-BS$, assuming that the outcomes of these five indicators are of equal importance. The results of the computations may be seen in equation (1).

$$k = \frac{k_d + k_{bs}}{2} = \begin{bmatrix} 0.1052 \\ 0.1056 \\ 0.1007 \\ 0.0354 \\ 0.0347 \\ 0.0347 \\ 0.0977 \\ 0.2008 \\ 0.2004 \end{bmatrix} \quad (10)$$

$$= \begin{bmatrix} A_1 \\ A_2 \\ A_3 \\ B_1 \\ B_2 \\ B_3 \\ C_1 \\ C_2 \\ C_3 \end{bmatrix}.$$

Factor $A1$ is 0.1052, Factor $A2$ is 0.1056, and Factor $A3$ is 0.1007; $A2$ is the most significant factor in the test results. Using the same kind of analysis, $B1$ and $C2$ have the greatest weights. As a result, the ideal orthogonal test scheme is $A_2B_1C_2$, which means that the optimal WC/2025Al sintering process parameters are 550 C, 3 min of holding time, and 45 MPa of pressure. According to [32], TiN materials were synthesized at different temperatures in the radial direction of the specimen. Due to the high temperature at the material's centre, SPS can make Al matrix composites with electrically isolating reinforcing elements. Al will be expelled from the mould with a colossal number of holes after it is taken from the mould. Low speed (500 rpm) and zirconia as the grinding medium and container guaranteed that the particles remained in their original form and did not introduce any impurities into the environment.

High mechanical characteristics may be achieved by combining reinforcement with Al matrix in PAMCs at the right interfacial interface. High temperatures and pressures are commonly used in traditional techniques (such as extrusion and stirring casting) to make WC/Al composites; however, this is because the two materials have limited wettability. Brittle reactants like Al_4C_3 and $MgAl_2O_4$ are common because of this. Remarkably, the material properties, thermal conductivity, and resistance to abrasive corrosion are all affected by the hydrolysis of the flake-shaped Al4C3. No other current methods for preventing the production of Al_4C_3 have a negative impact on densities (such as reducing processing temperatures and holding times) or performance (such as adding Mg or Si or doing a pre-oxidation treatment with WC). Low temperature, short-term preparation methods such as SPS have been described in the literature as being capable of producing more easily interfacial products-free composites, according to the literature.

$$4AL(1) + 3SiC(s) \longrightarrow Al_4C_3(s) + 3Si(1),$$
$$4SiO_2(s) + 4Al(1) + 2Mg(1),$$
$$Al_4C_3s + 12H_2O_g \longrightarrow 4Al(OH_3)(s) + 3CH_4(g)$$
$$\longrightarrow MgAl_2O_4(s) + 5Si(1),$$
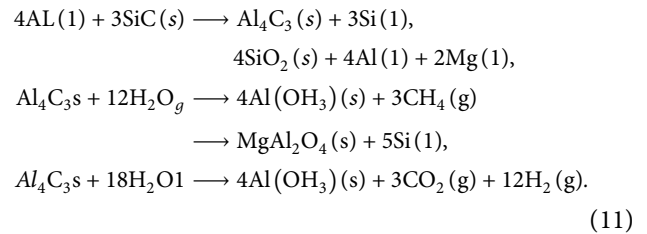$$Al_4C_3s + 18H_2O1 \longrightarrow 4Al(OH_3)(s) + 3CO_2(g) + 12H_2(g).$$
$$(11)$$

Figure 1 depicts WC/2025Al composites with variant SPS preparation circumstances. A summary of its entire behavior is seen in Table 2.

When heated to 525°C for five minutes under 35 MPa, the material had a bending strength of 766.65 Mega Pascal ($A_2B_3C_2$). Sintering at 550°C for 5 minutes under 30 MPa ($A_3B_3C_1$) reduced the mixture's bending strength (584.83 mpa) and elasticity ($A_3B_3C_1$) due to the material's large holes. WC/Al 2025 amalgamated density and contact configurations are affected by the sintering temperature, according to Yu and colleagues. Because of the alloy leakage, the density of the material reduced to around 96%, and the strength decreased from 367 to 211 MPa at 530°C. Figures 2(a) and 2(b) display density and bending strength values in the form of graph. After 2 minutes of holding time, the density of WC/Al 2025 went from
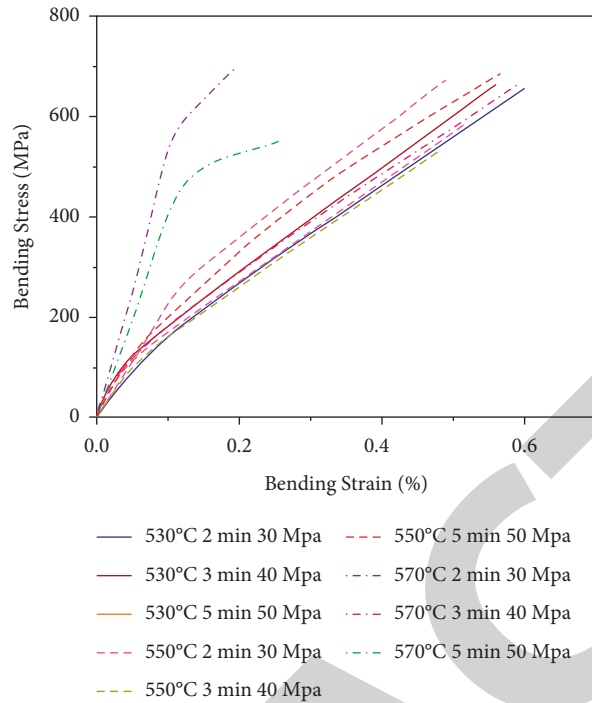
Figure 1: Bending curves of the 50 percent WC/Al 2025 composites made by Spark plasma sintering through variant conditions.
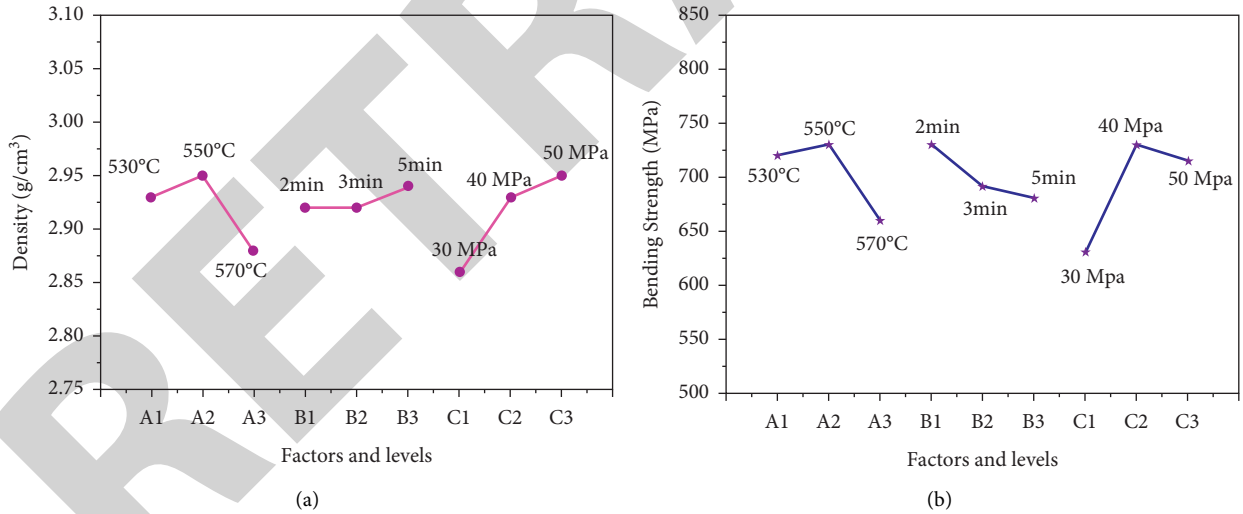

(a)

(b)

Figure 2: Impact factors of (a) density and (b) ending strength.

$2.93 \, g/cm^3$ to $2.94 \, g/cm^3$, which may be ascribed to the molten Al filling the space between the WC and filling the larger holes, but not filling smaller ones. An increase in density of $2.90 \, g/cm^3$ occurred as the sintering temperature reached $570°C$. Bending strength declined from $713.93 \, MPa$ to $690.78 \, MPa$, a 3.2 percent drop, and the composites' density was $2.92 \, g/cm3$ after two and three minutes of holding. However, holding for 5 minutes,

bending strength dropped to 692.45 Mega Pascal, while density raised to $2.95 \, g/cm^3$. The test's sensitivity might have been low due to the experiment's low degree of freedom and large error. A $40 \, MPa$ boost to the sintering pressure improves density by 0.34 percent but reduces bending strength by 0.80 percent. Figure 3 shows the comparison between WC/Al composite bending strength and temperature preparation.
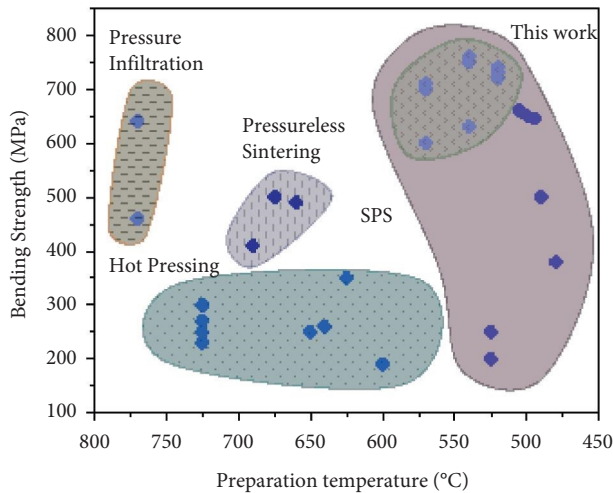
FIGURE 3: A comparison between WC/Al composite bending strength and temperature preparation.

4. Conclusions

A 50 vol% tungsten carbide/Al 2025 composite was made using Spark plasma sintering in this investigation. When performing visual, ANOVA, and matrix analyses, orthogonal experimental design was employed to find the best possible combination of each index. The following findings can be drawn from this investigation:

(1) The sintering pressure, temperature, and holding time have most impact on SPS density and bending strength. Density and strength increase first when pressure and temperature rise.

(2) On five minutes run at 525°C and 35 MPa produced a tungsten carbide/Al 2025 combination with a 99.7% density and 766.65 MPa bend strength. The developed composite material had no brittle interfacial products, the Al matrix was equally dispersed, and the interface was strongly bound. The tungsten carbide particles in the material shattered, allowing the particles to be drawn out, causing the material to fracture.

Data Availability

The data used to support the findings of this study are included within the article. Further data or information are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

Acknowledgments

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