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Optimizing the Thermal Spray Parameters for Producing High-Performance Mo/ZrB_2 Metal Matrix Composites Using the Taguchi Method

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Abstract: The process of Thermal spray is effective for creating a metal matrix composite (MMC) by embedding zirconium diboride reinforcement into a molybdenum matrix. Both materials Mo and ZrB_2 have different characteristics, but they have good thermal shock resistance, maintain strength at elevated temperatures, and stability in extreme environments. The study focused on creating $MoZrB_2$ composites using a thermal spray coating technique, varying the parameters of spraying distance, number of passes, and gas pressure, and testing the resulting castings to evaluate their hardness and Young's Modulus. The primary objective of the research was to use the Taguchi technique for identifying the optimal parameters for generating the highest Young's Modulus and hardness for the castings. The Taguchi method integrates experimental and analytical principles to identify the most significant parameter affecting the response, which can significantly enhance overall performance. The study found that the optimal parameters were a spraying distance of 20 cm, gas pressure of 6 bar, and the number of passes of 18. The Taguchi method accurately predicted the parameters that produced the highest properties for the composite coatings, which demonstrated good surface formation without hot cracks and fewer pores, with well-formed metallurgical bonding between the coating and the substrate.

Keywords: flame spray; coating parameters; spraying distance; molybdenum; zirconium diboride

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Citation: Mihoob, M.M.; Albarody, T.M.B.; Ahmad, F.; Alnarabiji, M.S. Optimizing the Thermal Spray Parameters for Producing High-Performance Mo/ZrB_2 Metal Matrix Composites Using the Taguchi Method. *Coatings* **2023**, *13*, 1620. https://doi.org/10.3390/coatings13091620

Academic Editor: Shibo Li

Received: 6 May 2023
Revised: 13 July 2023
Accepted: 17 July 2023
Published: 15 September 2023

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1. Introduction

Metal matrix composite materials are considered as sophisticated materials that integrate a resilient metallic matrix with either a rigid ceramic or a soft reinforcement [1]. These materials possess exceptional properties in comparison to monolithic materials and can be customized to meet specific application requirements. Metal matrix composites show advantages in a great number of specific applications [2,3]. Aerospace, power generation, heavy machinery, and other industries all use thermal spray (TS) techniques extensively for coating applications. In these procedures, feedstock—materials that have been heated or melted—are sprayed onto a surface to create a coating. However, different process variables have a direct impact on the characteristics of the coated materials [4–7]. The quality of any composite material is influenced by varying processing parameters. Among these variables, there must be a pair of variables that exert the most significant influence.

It has been recognized that the complete economic and technical capability of any manufacturing procedure can only be attained when the procedure is executed with the most favorable variables. The Taguchi method stands out as a highly significant optimization approach. Thicker coating forms on the substrate as the number of passes during the TS process is increased, and a denser microstructure is produced when the coating's porosity decreases with increased particle temperature [8]. Additionally, the flow of oxygen

and fuel reduces the coating's porosity. Greater oxygen and acetylene fuel rates create a rise in particle velocity; this lead to an increase in both the pressure inside the combustion chamber and the degree of contact [9]. To prevent coating problems, process variables must be carefully managed.

An inferior base material can be swapped out with a coating that delivers superior surface characteristics and performance using coating modification technologies. This method is very helpful in challenging circumstances, such as corrosive environments, conditions requiring wear protection, thermal insulation, or high levels of stress [10,11].

Experiments have been conducted to optimize the process parameters of flame-spraying to produce high-quality coatings. The effectiveness of the coating is determined by the cohesiveness and adhesive strengths of the coating, which are regulated by the spraying process parameters used during the coating deposition process [12,13]. In industrial settings, flame-sprayed molybdenum coatings are commonly used to improve the performance of engineering components such as shafts, pistons, and piston rings. By adjusting the spraying settings, the coating efficacy of metallic glass based on Fe was improved, revealing that the amorphous phase initially increased but decreased as power was increased [14]. It was believed that the oxidation and melting phases of the powders, as well as the heat radiation from the flame, contributed to this phenomenon. The correlation between the micro-hardness and spraying power was positive.

Zirconium diboride is a top-tier material in the ultra-high-temperature ceramics (UHTCs) group, valued for its outstanding combination of chemical stability, high thermal and electrical conductivity, and resistance to erosion and corrosion. It is suitable as a thermal protection system. Enhancing the coating's strength, hardness, and wear resistance can be achieved by optimizinzsg the spray parameters. Spraying settings are vital for enhancing the properties of coatings [15].

Several processing parameters can impact the quality of composite materials, with one or two being particularly significant. To fully maximize the economic and technical potential of any manufacturing process, it is crucial to operate it with the most optimal parameters. The Taguchi method is an important optimization process that can help achieve this [16,17]. The Taguchi method of parameter design offers a reliable technique for developing high-quality systems through a systematic, efficient, and simple approach that aims to improve design performance, quality, and cost-effectiveness. This approach is especially advantageous in cases where the design parameters are subjective and limited in scope. By adjusting the design parameters, the Taguchi parameter design boosts the performance characteristics and minimizes the impact of fluctuations in the system's performance [18]. The Taguchi method facilitates a thorough comprehension of the separate and combined impacts from a minimal amount of simulation attempts. This method consists of several steps that adhere to a specific sequence of experiments, which eventually lead to an enhanced understanding of the performance of a product or process.

The increased need for materials that can endure challenging industrial environments and the limitations of conventional materials has spurred the creation of new alloys for thermal coating. Flame-sprayed molybdenum coatings are a prominent illustration of such coatings that are extensively used in multiple industrial settings to enhance the functionality of engineering components, such as shafts, pistons, and piston rings. These coatings exhibit superior thermal stability, corrosion resistance, and wear resistance. The coating's effectiveness is dependent on its cohesive and adhesive strengths, which are influenced by the coating deposition process's spraying parameters. This study focuses on optimizing the flame-spraying process parameters to create top-notch coatings, with previous research successfully using Taguchi methods for optimization [19,20].

The process of thermal spraying involves a variety of parameters that can affect the properties of the coatings, such as spraying distance, gas pressure, temperature, number of passes, and flow rate. According to research, longer spraying distances can lead to smoother and more uniform coatings, while shorter distances can result in coatings with higher hardness values. It is important to choose the right spray process and parameters,

such as fuel gas type, particle velocity, and spray distance [21]. By using thermal spray (TS) technology, the thickness of the coating on the treated substrate can be increased by increasing the number of passes during the spraying process [22], and porosity decreases with higher particle temperature [23].

The microstructure of the coating becomes denser when exposed to higher rates of oxygen and acetylene fuel, which leads to a decrease in the coating's porosity. Additionally, when the particle velocity increases due to elevated oxygen and acetylene fuel rates, the pressure within the combustion chamber rises, and particle spread improves, enhancing contact and extending the particle's kinetic energy [24]. Hence, controlling process parameters is crucial to avoid coating defects.

Zirconium diboride is a high-quality ultra-high-temperature ceramic material that has outstanding physical, structural, transport, and thermodynamic properties. As a result, it can be used in a variety of applications, including refractory linings [25,26], electrodes [27], microelectronics [28], and cutting tools [29]. ZrB_2 is an excellent material for thermal protection systems due to its high melting temperature, chemical stability, and resistance to erosion/corrosion, as well as its exceptional electrical and thermal conductivity. Additionally, adjusting the spraying settings is crucial for enhancing the coating's characteristics [15,30]. The proposal was made that the phenomenon was influenced by the oxidation and melting levels of the powders, as well as the heat radiation from the flame. An association was observed between spraying power and microhardness. In general, improving the spray parameters can increase the coating's strength, hardness, and resistance to wear.

The quantity of times the process is carried out: The size and arrangement of particles present in the input material can affect the coherence and permeability of the coating. Particles ranging from 1 to 50 µm undergo partial melting and are swiftly propelled by either an electric arc or flame before being scattered onto a surface to produce a coating [31].

Spray distance: The thickness, density, and mechanical properties of a coating can be influenced by the distance and angle at which it is sprayed. Generally, the spraying distance falls in the range of 100–300 mm, and the spray angle is between 45 and 90 degrees [32].

Gas pressure: The melting and speed of the feed-stock material can be affected by both the spray temperature and pressure, ultimately affecting the microstructure and mechanical characteristics of the coating. The temperature at which the spray is released can vary from 3000 to 11,000 °C, while the pressure can range from 1 to 10 MPa [33].

Measuring the density of cut stainless steel samples accurately is challenging, and optimizing thermal spray parameters to achieve dense coating surfaces is crucial. To address these issues, the primary objective of this study was to create a $MoZrB_2$ composite coating using the thermal spray (TS) process. The Taguchi approach was employed to optimize torch parameters.

To achieve a $MoZrB_2$ composite coating with high wear resistance, an L9 orthogonal array was utilized in the Taguchi method to determine the main effect of various parameters. The coating's composition was discovered to be primarily influenced by the operational parameters, including spraying distance, gas pressure, and number of passes, among others. Nevertheless, the chemical composition of the coating was indirectly taken into account as it is modified by varying the operational parameters, rather than being treated as an independent parameter. The use of L9 helped to remove the interaction between the parameters. Statistical analysis software MINITAB 17 (Version: Minitab 17 Creator: Minitab, LLC) was employed to design and analyze the experiments in this study.

2. Materials and Methods

2.1. Material

In this experiment, Stainless steel type 316 was utilized as the substrate. To prepare samples, the substrate was cut using an abrasive cutter, resulting in dimensions of 3 × 2.5 × 0.3 cm. Next, the samples were polished using P 120, 400, 800, 1000 and 1200 grit to remove dust and carbon from the surface, and the composition of the substrate contains

some coating materials such as molybdenum. For the ceramic top-coat material, molybdenum and zirconium diboride powders with the same weight ratios were employed. The wt. % of both powders was maintained at 50 wt. %. To obtain the desired particle size, the molybdenum and zirconium diboride powders were separately sieved using a sieve shaker for 1 h each. The powders were then ball milled at 121 rpm for 4 h using a roller ball mill for homogeneous mixing.

Finally, the dried powder was ground and sieved using a 140 mesh, and the molybdenum and zirconium diboride powder was classified into two sizes, fine and coarse.

2.2. Process Parameters

Various parameters had a significant impact on the spray process, as shown in Table 1, which presents the parameters and their respective levels used in this study. The primary process variables that influenced the spraying process were the spraying distance, gas pressure, and number of passes. In addition, the porosity and corrosion resistance of the coatings were significantly impacted by the spray distance [34].

Table 1. Thermal spray process parameters and their levels.

Parameter	Units	Levels
Spraying Distance	cm	20, 25, 30
Number of Passes	-	12, 18, 24
Gas Pressure	bar	2, 4, 6

2.3. Signal-to-Noise Ratio (SN Ratio)

To evaluate the performance of the process response, Taguchi proposed the signal-to-noise (SN) ratio, which is the mean-to-standard deviation ratio and effectively accounts for the variations encountered in a series of trials. Depending on the response's characteristics, the SN ratio can be classified into three categories: smaller is better (SB), higher is better (HB), and nominal is better (NB). Regardless of the chosen criteria for the SN ratio, the level with the maximum value is considered the optimal level for each parameter. In the present study, the goal is to minimize weight loss during the wear test and maximize the Mo/ZrB_2 composite coating's hardness. Hence, these two outputs are considered as response variables, and the SB criterion is appropriate for the SN ratio for the first output, whereas the HB criterion is appropriate for the SN ratio for the second output based on different requirements, Taguchi et al. suggested an equation for the SN ratio as follows (Equation (1)):

Higher is better:

$$SN_{HB} = -10 \times \sum_{i=1}^{1} \frac{1}{\frac{2}{i}Y} \quad (1)$$

2.4. Design of Experiment (DOE)

The Design of Experiments (DOE) is a crucial statistical technique that employs Taguchi orthogonal arrays (OAs) to systematically investigate controllable factors and their associated responses at multiple levels. This approach reduces the number of tests required, saving both time and resources. According to the Taguchi method, the L9 orthogonal array (OA) with four parameters (factors) and three levels was the most suitable for the experimental activities as a DOE tool parameter. The coating data were analyzed using Minitab 17.0 software, and randomized OAs were used to perform nine sets of thermal spray (TS) tests, starting from trial 1 and ending with trial 9. The experimental results for the coating process experiments are shown in Table 2.

Table 2. L9 orthogonal array and parameters.

Trials	Spray Distance (cm)	Number of Passes	Gas Pressure (bar)
1	20	12	2
2	20	18	4
3	20	24	6
4	25	12	4
5	25	18	6
6	25	24	2
7	30	12	6
8	30	18	2
9	30	24	4
Output		Signal-to-noise ratio	
Hardness (HV)		Higher is better	
Young's Modulus (N/m^2)		Higher is better	

To focus on the primary effects, Taguchi suggests using standard orthogonal arrays for experiment design. For this study, the L9 orthogonal array was selected, consisting of nine experiments. This design allows for the analysis of up to eight parameters, with one parameter set at two levels and the other at three levels. The parameters and their corresponding levels are listed in Table 3, where each row represents a single trial, with the first row serving as column labels for each variable. Each row represents one trial. The first row is for column labels and each column represents one variable.

Table 3. L9 orthogonal array and parameters.

Trials	Spray Distance (cm)	Number of Passes	Gas Pressure (bar)	Young's Modulus (N/m^2)	Hardness (HV)
1	20	12	2	24.2	225
2	20	18	4	18.259	220
3	20	24	6	10.904	260
4	25	12	4	18.008	233
5	25	18	6	26.152	200
6	25	24	2	23.27	243
7	30	12	6	21.2295	212
8	30	18	2	14.456	223
9	30	24	4	24.711	243

2.5. Experiment Procedures

2.5.1. Hardness Testing

For each of the processing states, the hardness properties of the alloys were evaluated and measured. In order to ensure that the surfaces of the cube samples used for measuring hardness were perfectly parallel to one another, the samples needed to be machined into cubes first. Following the heat treatments, the oxide layer that had formed on the surface was polished away using a rubbing compound. In accordance with ASTM E18-19 [34], the instrument that was utilized for the hardness measurement was a Mitutoyo HR-400 Rockwell Hardness Tester (Mitutoyo Corporation, Takatsu-ku, Kawasaki, Kanagawa, Japan) as shown in Figure 1, which featured a conical diamond indenter and a weight of 150 kg. Before beginning the tests, a Mitutoyo 50 HRC standard hardness block with the part number 19BAA039 was applied to the machine in order to check that it was correctly calibrated. On each plane (XY and XZ), there were a total of ten measurement points that were utilized for the purpose of computing average values and standard deviations.



Figure 1. Mitutoyo HR-400 Rockwell Hardness Tester.

2.5.2. Nanoindentation Testing

The nanoindentation tests were performed to determine the main material parameters, hardness and Young's Modulus. Both hardness and elastic modulus were calculated using an Anton Paar Bioindenter (Step 300, UNHT3, Graz, Austria as shown in Figure 2, and tests on nanoindentation were conducted. The indenter was a ruby ball with a diameter of 1 mm, and the applied load was 200 µN, the Table 4 shows the indentation parameters.

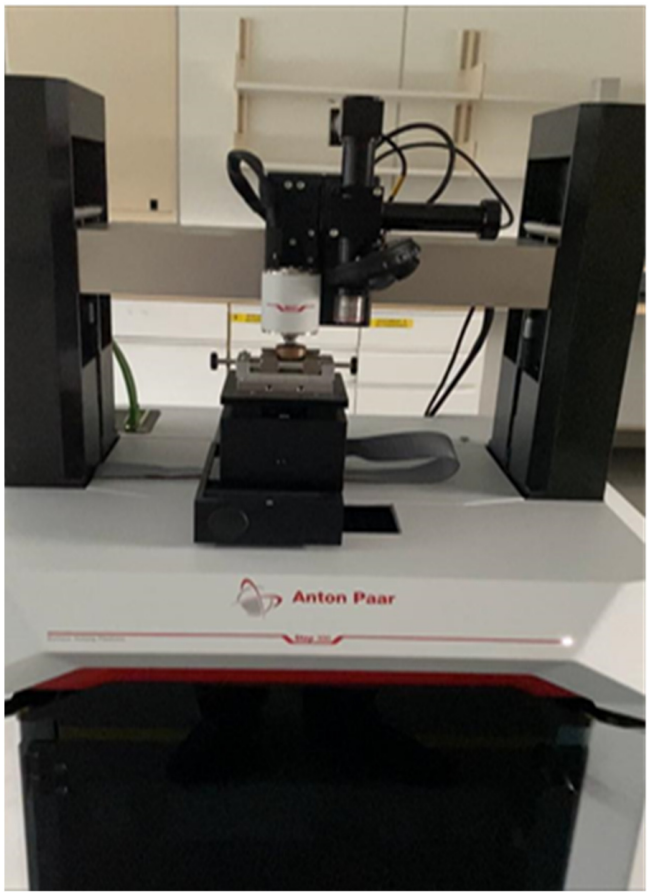


Figure 2. Anton Paar Bioindenter (Step 300, UNHT3) Tester.

Table 4. Indentation parameters.

Acquisition Rate	20 Hz
Max load	10 mN
Loading rate	100 mN/min
Approach distance	2000 nm
Approach speed	4000 nm
Stiffness threshold	500 μN/μm

The spherical nanoindentation P-h responses of Mo/Zrb_2 for the five maximum loads Pmax = 2, 4, 6, 8, 10 mN with the constant loading rate dP/dt = 45 mN/s. For each maximum load, the representative P-h curves are shown in Figure 3.

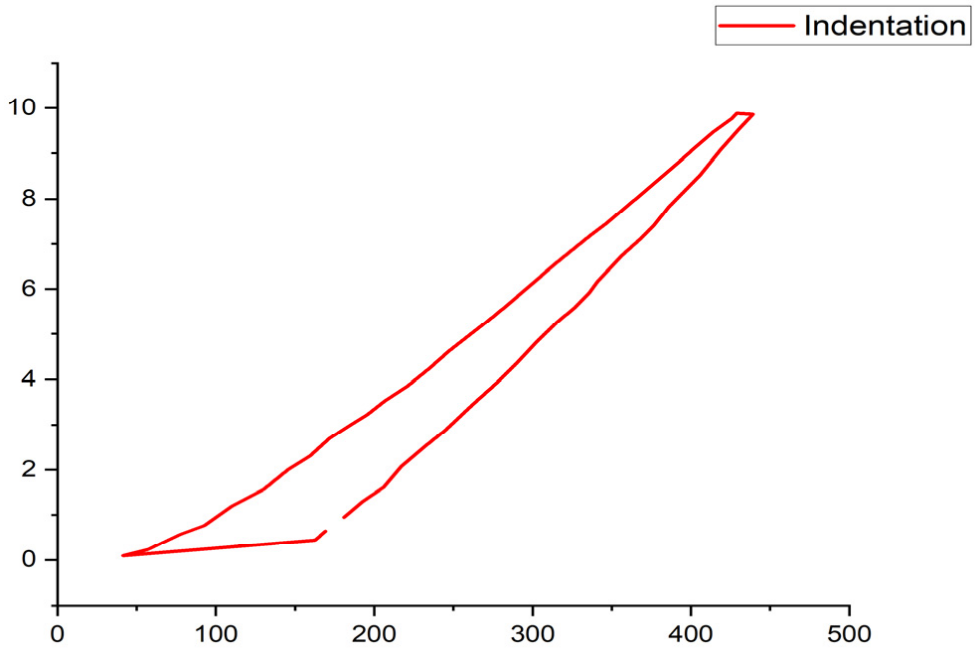


Figure 3. Sample load-displacement curve.

3. Results and Discussion

3.1. Taguchi Method

Taguchi devised a method using statistics to enhance the quality of produced items. Taguchi's loss function transformed the discrepancy between experimental data and targets into an S/N ratio, signifying the mean-to-standard deviation ratio. Taguchi utilizes the signal and noise to represent desired and undesired response values. The S/N ratio is classified into three categories based on the preferred response level: the medium-the-better, the larger-the-better, and the lower-the-better [6]. The larger-the-better level characterized the responses in this research for Cd.

3.2. Optimization of Parameters via Taguchi Method

Choosing the ideal coating parameters, Taguchi's orthogonal array is a great option. With orthogonal arrays, it is possible to determine the impact of parameters on the response variable and specify which parameters produce the best hardness results [35]. Under the experimental conditions specified, the results of the hardness test were analyzed using the SN ratio shown in Tables 5 and 6. The study focused on changes in the parameters mentioned in the manuscript to optimize the coating process and achieve the desired weight loss. Taguchi's "smaller is better" formula was used to calculate the SN ratio. Figure 4 shows the SN ratio effects that were used for optimization, and MINITAB 17 was used to generate plots for the SN ratio. The delta is the difference between the highest average SN

ratio and the lowest average SN ratio for each parameter. Ranks were assigned based on delta values, with Rank 1 given to the highest delta value, Rank 2 to the second-highest delta value, and so on. According to Figure 5, the parameter with the greatest difference in delta value among all design parameters has the most significant effect on the response. The gas pressure had the highest delta value for the maximum-minimum value, and thus it is the most significant parameter affecting the wear rate (Rank 1), while the number of passes has the least effect (Rank 3) as shown in Tables 7 and 8. Additionally, the parameter with the largest slope has the most significant effect, whereas the parameter with the smallest slope has the least significant effect. For the minimum Young's Modulus, the optimal combination of coating parameters can be seen from the main effects plot [36]. Consequently, the best value for each parameter was determined by selecting the level with the highest SN ratio, which was considered the optimal value. Therefore, the optimal deposition parameters for producing a low-weight $MoZrB_2$ composite coating were identified.

Table 5. Response Table for Signal to Noise Ratios.

Level	Spraying Distance (cm)	Gas Pressure (bar)	Number of Passes
1	47.40	46.97	47.24
2	47.03	46.61	47.30
3	47.07	47.91	46.95
Delta	0.37	1.30	0.35
Rank	2	1	3

Table 6. Response Table for Signal-to-Noise Ratios.

Level	Spraying Distance (cm)	Gas Pressure (bar)	Number of Passes
1	24.55	26.44	26.07
2	26.93	25.59	26.07
3	25.87	25.32	25.21
Delta	2.38	1.13	0.86
Rank	1	2	3

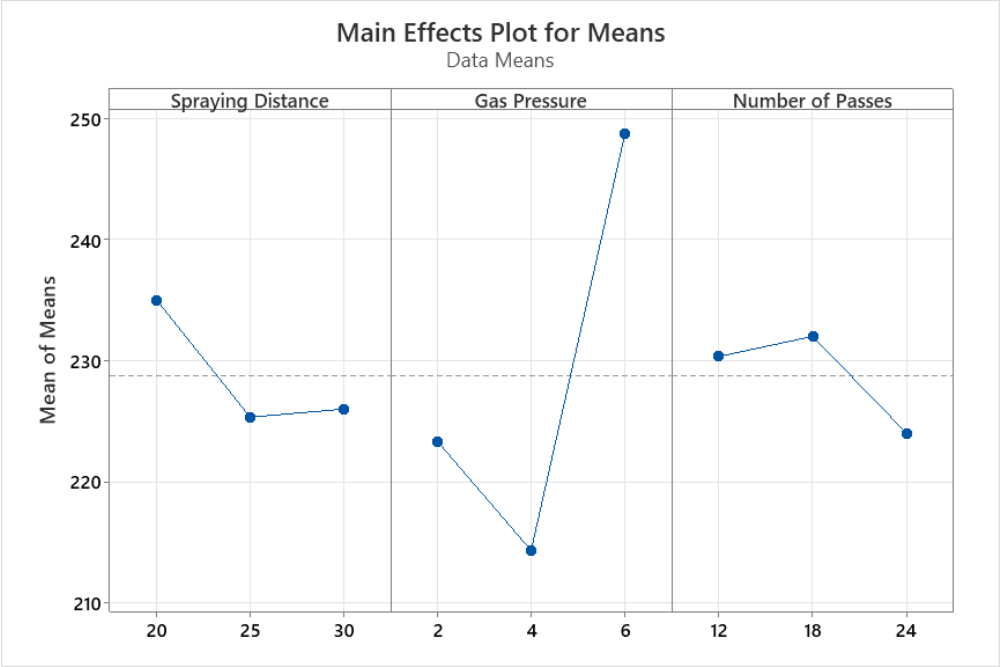


Figure 4. Hardness main effects plot of means.

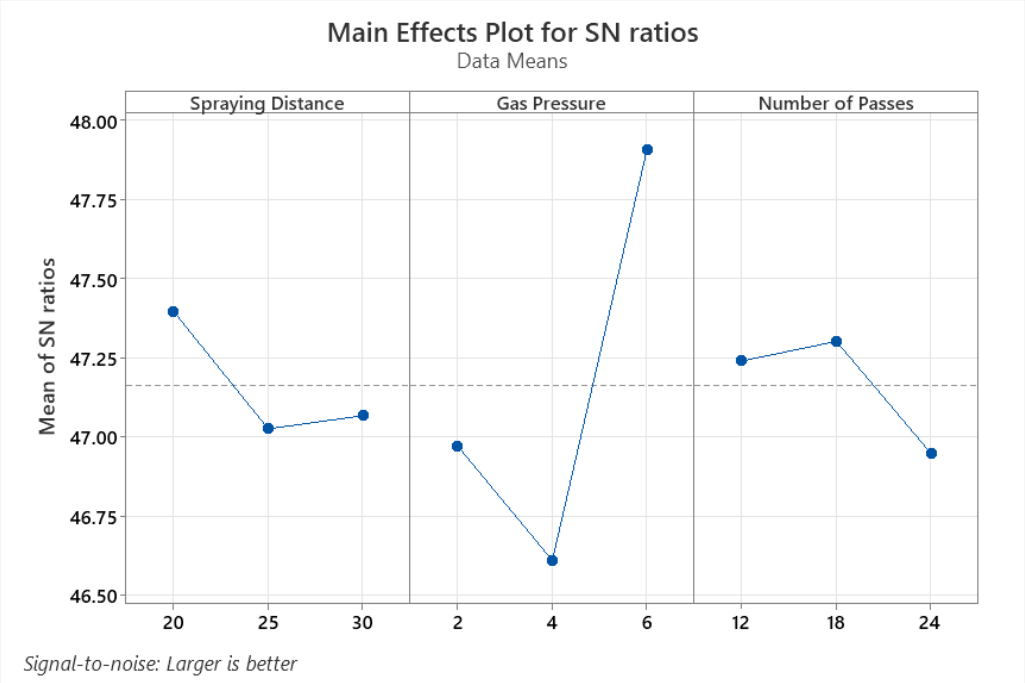


Figure 5. Hardness main effects plot for SN ratio.

Table 7. Response Table for Means.

Level	Spraying Distance (cm)	Gas Pressure (bar)	Number of Passes
1	235.0	223.3	230.3
2	225.3	214.3	232.0
3	226.0	248.7	224.0
Delta	9.7	34.3	8.0
Rank	2	1	3

Table 8. Response Table for Means.

Level	Spraying Distance (cm)	Gas Pressure (bar)	Number of Passes
1	17.79	21.15	20.64
2	22.48	19.62	20.33
3	20.13	19.63	19.43
Delta	4.69	1.52	1.21
Rank	1	2	3

It is evident from Figure 6 that the minimum at the first level of spray distance (A), second level of gas pressure (B) and third level of number of passes (C).

In Figure 7 the the maximum at the second level of spray distance (A), first level of gas pressure (B) and third level of number of passes (C).

This study aimed to optimize the hardness and Young's Modulus of a $MoZrB_2$ composite coating by varying different deposition parameters. The Taguchi method was used to evaluate these parameters, and a confirmation experiment was conducted to validate the results [37]. The surface morphology of the coatings produced was also analyzed to determine their impact on tribological and hardness properties. The study revealed the optimal deposition parameter levels required to produce a $MoZrB_2$ composite coating with high hardness and a high Young's Modulus. The optimal spraying distance, gas pressure, and number of passes for high hardness were 20 cm, 6 bar, and 18, respectively, while for the high Young's Modulus, they were 25 cm, 2 bar, and 12, respectively. The scanning electron microscope (SEM) (TESCAN VEGA's 4th generation, Kohoutovice, Czech Republic)

micrographs in Figure 8 showed that the coatings had laminar structures consisting of stacked particles and pores resulting from the short time of the material in the flame and the high cooling rate of the molten material during the coating's formation. The SEM of the coatings displayed stacked splats as well as defects from the technique, such as pores, cavities, oxides, and splat boundaries.

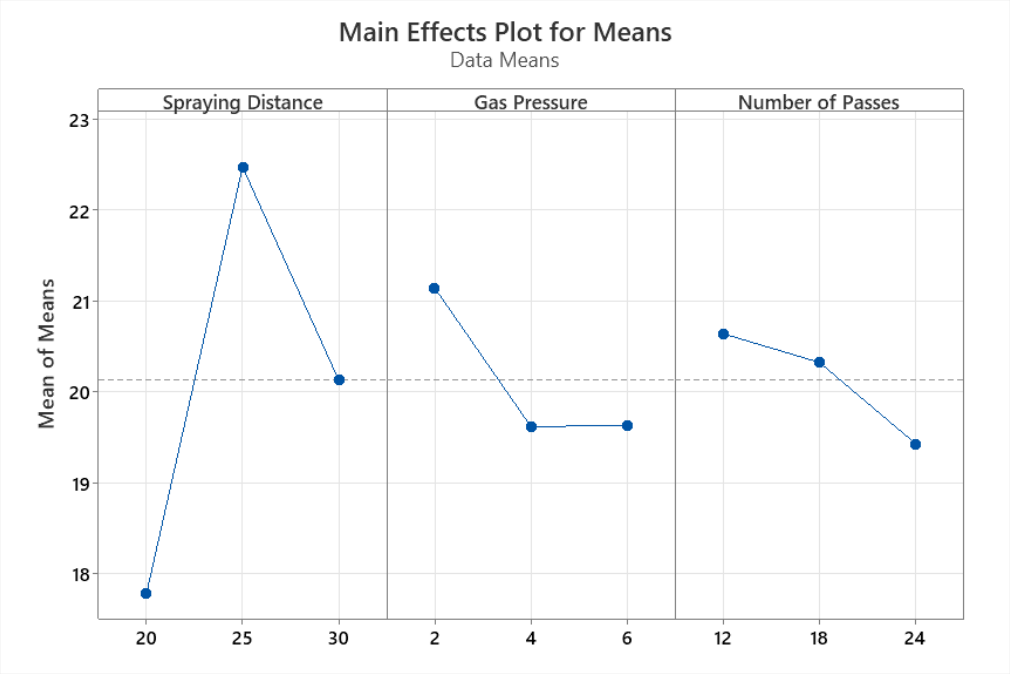


Figure 6. Young's Modulus main effects plot of means.

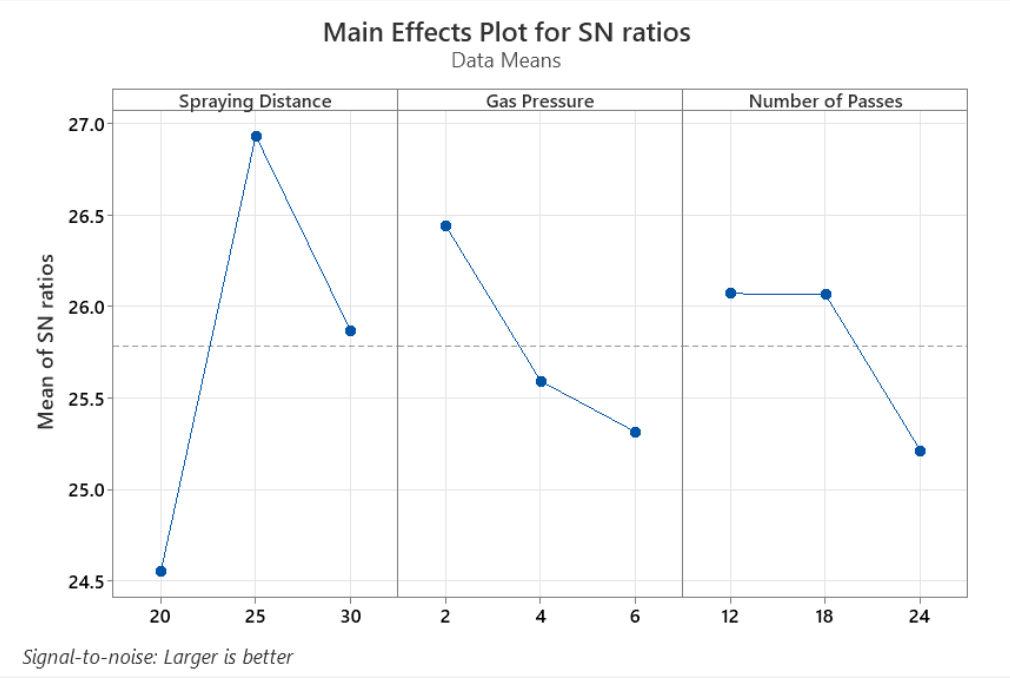


Figure 7. Young's Modulus main effects plot for SN ratio.

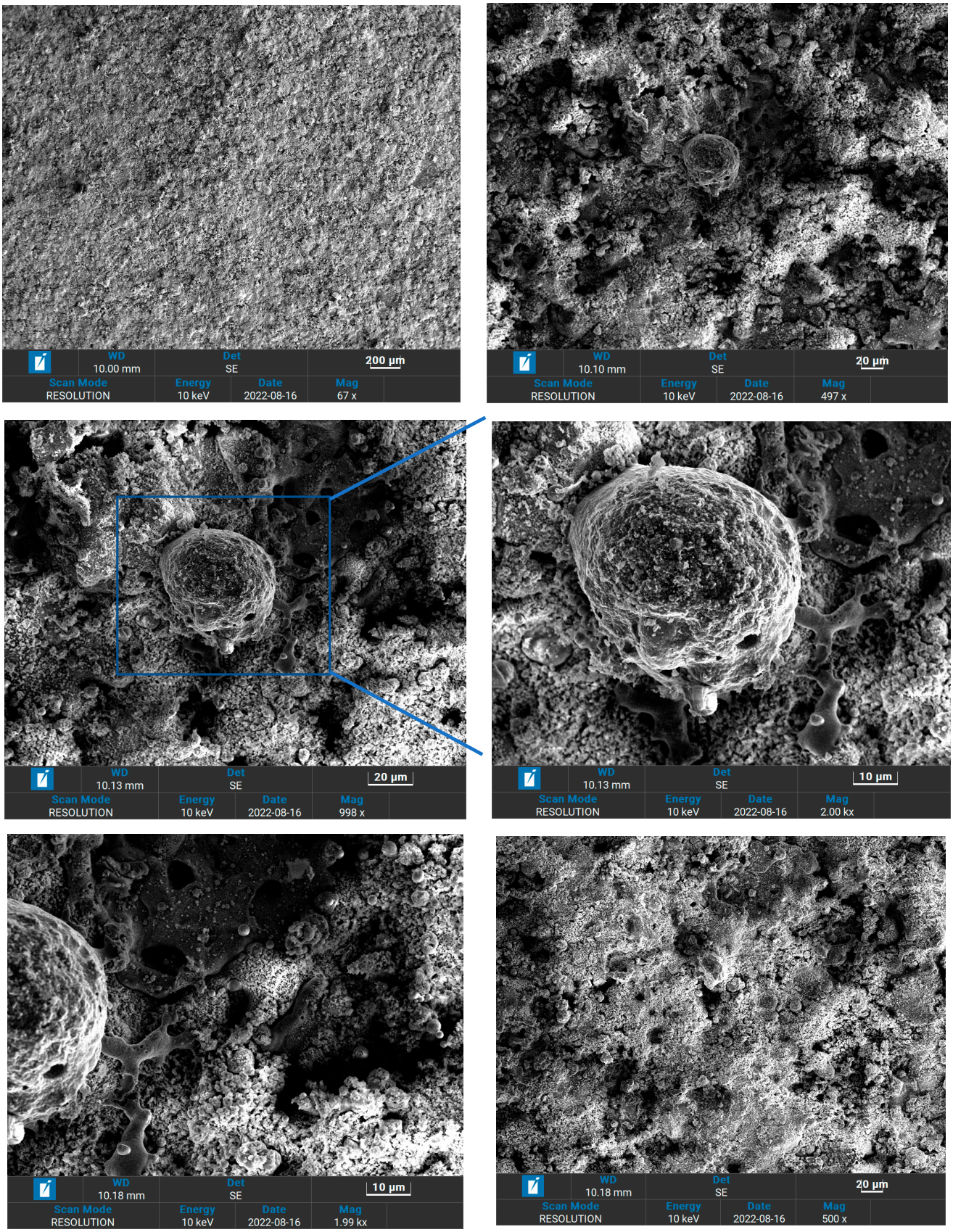


Figure 8. SEM morphology for Mo/ZrB_2 under optimal parameter.

4. Conclusions

In this study, samples with Mo/ZrB_2 coating deposited on stainless steel 316 L substrate were investigated in terms of coating hardness and Young's Modulus properties using two different methods. Nanoindentation is the most popular method currently used to characterize a coating's parameters. In this paper, the Taguchi approach was used to optimize the coating parameters for achieving a high coating hardness, Young's Modulus and hardness developed with calculations by Anton Paar. The following observations resulted from the evaluation. By using the Taguchi method, the optimal coating parameters for achieving high hardness were found to be a spraying distance of 20 cm, gas pressure of 6 bar, and a number of passes of 18. Another possibility for optimal conditions was found to be a spraying distance of 25 cm, gas pressure of 2 bar, and a number of passes of 12, as there was no significant difference between the SN ratios of Mo content at levels 2 and 3, according to Figure 5. The optimal coating parameters suggested that TS could be a promising method for producing a highly dense coating surface. The SEM images revealed that the TS coating had a good morphology due to the accumulation of optimal spraying distance, gas pressure, and number of passes, which resulted in less porous and well-bonded coatings. In future research, further exploration of the relationships between different parameters can be conducted, and comprehensive models with finite equations can be developed. Additionally, the effect of changing the ratio of molybdenum to zirconium to three to one and vice versa on the different mechanical properties of the coating can be examined.

Author Contributions: Data curation M.M.M. and T.M.B.A.; formal analysis, M.M.M.; funding acquisition, T.M.B.A.; investigation, M.M.M., F.A. and M.S.A.; methodology, M.M.M. and T.M.B.A.; project administration, T.M.B.A.; resources, T.M.B.A.; software, M.M.M.; supervision, T.M.B.A., F.A. and M.S.A.; validation, M.M.M. and T.M.B.A.; writing—original draft, M.M.M.; writing—review and editing, M.M.M. All authors have read and agreed to the published version of the manuscript.

Funding: The authors would like to acknowledge Universiti Teknologi PETRONAS for its financial support, under the grants (MRA-015MD0-151) and (015BD1-001), which is gratefully appreciated.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Conflicts of Interest: Authors declare no conflict of interests.

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