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Optimization of Microhardness in Nanostructured Thermal Barrier Coatings Using Spark Plasma Sintering (SPS) and Taguchi Design

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ABSTRACT

In this study, nanostructured thermal barrier coatings (TBCs) were fabricated using the Spark Plasma Sintering (SPS) method on an Inconel 713 LC superalloy substrate. These coatings were compared to those produced by Atmospheric Plasma Spray (APS) in terms of mechanical properties. The aim of this study was to investigate and optimize the process parameters to improve the microhardness of these coatings. Key parameters, such as temperature, pressure, and holding time, were optimized using the Taguchi design of experiments (L9). The results showed that SPS coatings exhibited significantly higher hardness compared to APS coatings, due to a notable reduction in porosity and increased density. The highest microhardness achieved for SPS coatings was 700 HV at a temperature of 1080°C, a pressure of 25 MPa, and a holding time of 6 minutes. In contrast, APS coatings demonstrated lower hardness, primarily due to higher porosity and lower density. This study highlights that precise control of process parameters in the SPS method can produce coatings with enhanced mechanical properties, making them suitable for high-temperature applications in aerospace and power generation industries. Furthermore, the Taguchi method effectively reduced the number of experiments and improved process efficiency.

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1. INTRODUCTION

1.1. Thermal Barrier Coatings and Their Importance

Thermal barrier coatings (TBCs) play a crucial role in protecting high-temperature components, such as those in gas turbines and jet engines, from extreme heat. These coatings are primarily made from ceramic materials, such as yttria-stabilized zirconia (YSZ), which provide excellent thermal insulation and resistance to thermal corrosion, thereby extending component lifespan. Research has shown that improving the microstructure and reducing porosity in TBCs can significantly enhance their mechanical strength and thermal resistance (Li, et al. 2024).

1.2. Common Coating Methods

Common methods for applying TBCs include atmospheric plasma spray (APS) and spark plasma sintering (SPS). APS is widely used due to its lower cost and faster processing speed; however, it has drawbacks such as high porosity and lower adhesion, which can impact the mechanical performance of the coatings. Zhang et al. demonstrated that APS coatings, because of their high porosity, have reduced resistance to thermal

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shock, which shortens the lifespan of the components (Zhang, et al., 2021).

SPS, a more advanced method that uses high pressure and precise temperature control, produces coatings with higher density and lower porosity. Liu et al. reported that SPS coatings exhibit higher adhesion, greater hardness, and a more uniform structure compared to APS, resulting in notable improvements in the mechanical performance of the coatings (Liu, et al., 2019).

1.3. Taguchi Method and Its Application in Coating Process Optimization

The Taguchi design of experiments is recognized as an efficient statistical tool for optimizing processes and reducing the number of experiments without compromising the quality of results. This method utilizes orthogonal arrays to identify the optimal combination of experimental parameters, precisely determining the impact of each parameter on process outcomes. According to Taguchi et al., the Taguchi method for industrial process optimization, including coating applications, can effectively identify critical parameters and establish the best operational conditions, thereby reducing costs and production time. In recent studies, used the Taguchi method to optimize SPS process parameters for TBCs and found that this method not only reduces the number of experiments needed but also significantly enhances the final quality and performance of the coatings. They identified temperature, pressure, and holding time as the most crucial factors affecting the mechanical properties of the coatings (Kruzel, et al., 2023)

Similarly, Jonnalagadda applied the Taguchi method to evaluate the effects of various parameters in the SPS process, concluding that this approach enables the rapid identification of optimal conditions. The study highlights that using Taguchi arrays can effectively optimize parameters influencing the hardness and adhesion of the coatings, resulting in reliable and repeatable outcomes (Jonnalagadda, et al., 2019).

1.4. Applications of the Taguchi Method in Thermal Barrier Coatings

In other studies, the Taguchi method was used for coating parameter optimization, demonstrating that this approach not only reduces laboratory costs but also effectively determines the impact of various parameters, such as temperature and pressure, on the mechanical properties of coatings. The findings of their study align with the results obtained in our research, underscoring the importance of using the Taguchi method in optimizing coating processes (Kumar, et al., 2015)

The Taguchi method, a crucial tool for optimizing SPS coating parameters, establishes optimal conditions for achieving coatings with high hardness and strength. Recent studies have underscored the importance of this method in research on TBCs, demonstrating its potential to reduce costs and enhance the final performance of coatings.

2. MATERIALS AND METHODS/ EXPERIMENTAL PROCEDURE

2.1. Initial Materials

In this study, NiCrAlY, YSZ, and Inconel 713 LC substrates were used to create nanostructured TBCs, with specifications detailed below.

2.2. YSZ

Porosity and thickness, along with chemical composition, are the primary variables in determining and controlling the thermal conductivity and lifespan of TBCs. The 7-8% YSZ powder can be applied at 1150°C or higher on various substrates, creating different structures depending on the initial powder morphology.

Common commercial YSZ powders, such as Amprit 816, 825, 827, and 831, are widely used to produce TBCs with varying properties. The YSZ powder used in this project was Amprit 827 mayde by Hoganas Company, which is granulated and sintered. These powders contain spherical and porous particles, making them suitable for creating high-porosity TBCs through APS, resulting in porosities ranging from 20% to 26% or even higher. The morphology of these particles is shown in Figure 1.

If the goal is to produce TBCs with very low porosity, Amprit 825 is recommended. This powder is produced by fully melting a solid material, cooling it, and then crushing it to achieve particles of the desired size for thermal spray. The morphology of this powder enables the production of TBCs that are resistant to wear and cavitation, although the low porosity results in higher thermal conductivity. The morphology of Amprit 825 is shown in Figure 1 .

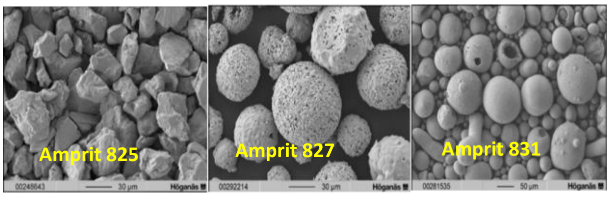

Figure 1. Morphology of three common types of powders for the production of thermal barrier coatings.

The YSZ powders of Amprit 816 and 831 grades are known as hollow spherical particles Due to their high fluidity during spraying, they create less porosity compared to Amprit 827, with porosities ranging between 5-15%, influenced by spraying conditions and particle size. Amprit 831 has the lowest level of impurities, making it more resistant to sintering. The morphology of Amprit 831 is shown in Figure 1. Figure 2 shows the morphology of the 7-8% YSZ powder used in this project. According to the figure, the granulated particles are spherical, enhancing fluidity during plasma spraying. These particles are porous, which improves the thermal barrier properties of the final coating. (Liu, et al.,

2020) Porosity also enhances thermal shock resistance, although it is essential to use high-enthalpy plasma spraying to keep the porosity below 20%.

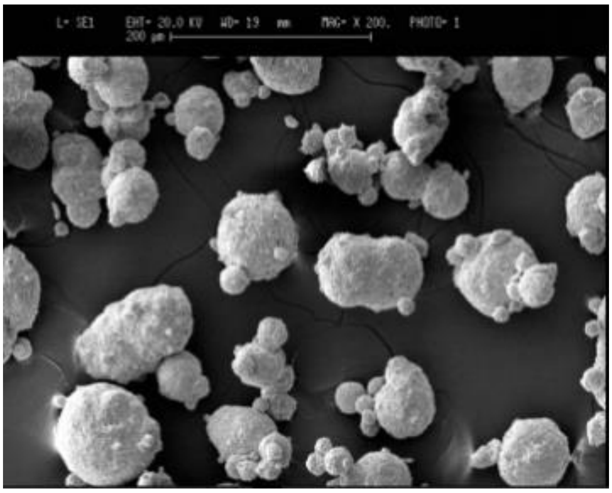

Figure 2. Morphology of the used YSZ powder

Table 1 shows the specifications of the YSZ powders used in this research. The YSZ particle sizes range from 40 to 90 microns, making them suitable for the plasma spray process. Figure 3 displays the XRD analysis of the YSZ powder sample. According to the figure, the zirconia crystal structure at room temperature is tetragonal, indicating that it is almost completely stabilized with yttria.

TABLE 1. Specifications of YSZ Powder

Granule size of the particles (µm)	Morphology	Chemical composition
40-100	Spherical	ZrO_2-8%Y_2O_3

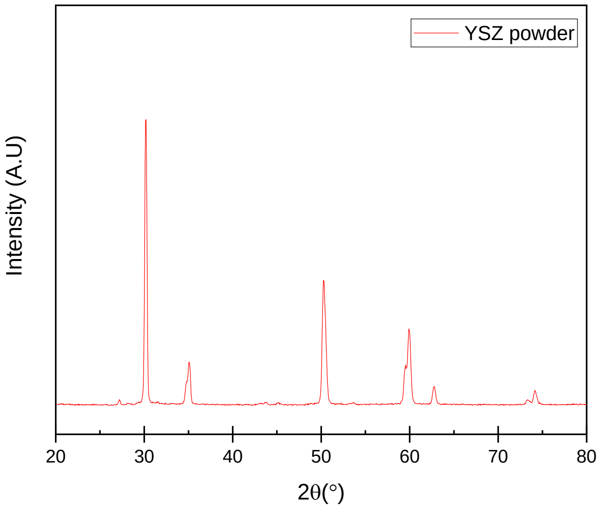

Figure 3. Image of X-ray diffraction analysis of YSZ powder

2.3. NiCrAlY Powder

The NiCrAlY powder, commercially coded as AMDRY 365-4, manufactured by Oerlikon Metco, was used to create a bond coat. The specifications of this powder are provided in Table 2. Figure 4 also shows that NiCrAlY particles have a spherical morphology, which results in better flowability during plasma spraying**.**

TABLE 2. Specifications of MCrAlY powder**.**

Granule size of the particles (µm)	Morphology	Chemical composition
50-100	Spherical	Ni23Co17Cr12Al0.5Y

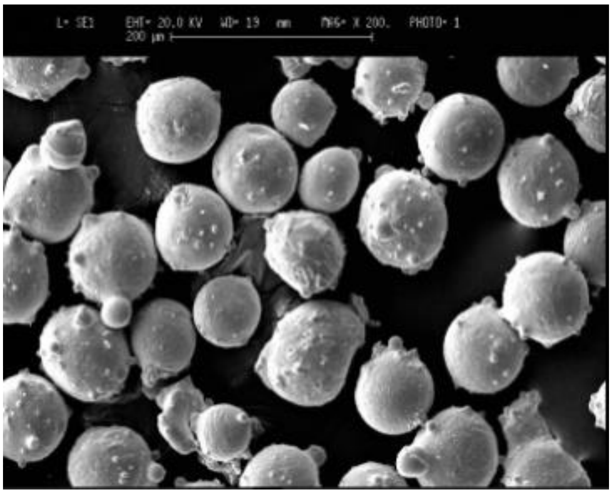

Figure 4. Morphology of used NiCrAlY powder.

2.4. Substrate

The substrate used in this research was a nickel-based superalloy named Inconel 713 LC, manufactured by Sekonic Metals. This substrate is a cast nickel-based superalloy strengthened by precipitation hardening. Due to its excellent mechanical resistance at high temperatures, resulting from these precipitates, the alloy is widely used in the hot components of the turbine industry. The chemical composition of this substrate, obtained through quantitative analysis, is shown in Table 3.

TABLE 3. Chemical composition of Inconel 713 LC.

Element	Average per	Equilibrium percentage Range (ASM5391)
Ni	73.9	69.50.76.90
Cr	12.50	12.00-14.00
Mo	4.20	3.80-5.20
Ta-Nb	2.20	1.80-2.80
Al	6.10	5.50.6.50
Ti	0.80	0.50-1.00
C	0.12	0.08-0.2
B	0.012	0.005-0.015
Zr	0.10	0.05-0.15

2.5. Preparation of Substrate

The preparation of the substrate surface and conditions of the bond coating significantly impact the adhesion strength of the coating as well as the component's life under cyclic thermal conditions. Low surface roughness and surface oxidation adversely affect the properties and adhesion of the coating. The substrate surface must be sufficiently clean to establish the necessary bonds between the substrate and the coating. Additionally, the surface roughness should be adequate to create mechanical bonds between the substrate and the coating. In this study, the samples were first cleaned with sandpaper and acetone, and then sandblasted with SiC using a mesh size of 24 and a pressure of 4 bar to achieve suitable surface roughness. Sandblasting is a process used to clean the surface of metals, stones, or glass by expelling sand at high speed. After sandblasting, the surfaces were washed with alcohol to remove any contaminants. Prior to applying the coating, the samples were preheated to 250°C to reduce residual stresses in the applied coatings.

2.6. Coating Deposition by SPS Method

In this study, the SPS method was used to create coatings. First, YSZ powder with an approximate size of 90 microns was milled to obtain nano-sized particles. Then, thermal barrier coatings with nano- and micro-structures were deposited using the SPS device.

2.7. Milling of YSZ Powder

After accurately weighing the YSZ powder using a precision balance (0.001 g), high-energy planetary ball milling was performed. The milling chamber and balls were made of zirconia, with a ball-to-powder ratio of 10:1. The milling speed was set at 250 rpm, and one-third of the chamber was filled with ethanol to improve mixing and temperature control. The milling process was conducted in 12-hour and 24-hour intervals, with samples taken at each interval for DLS analysis. Once the powder reached the nanoscale after 24 hours, microstructural analysis was conducted using a scanning electron microscope.

2.8. Coating Application via SPS

For the deposition of nano- and micro-structured thermal barrier coatings, the SPS device was used, as shown in Figure 5. The initial preparations for coating included:

1. Wrapping graphite sheets inside the cylindrical mold.
2. Placing the punch inside the mold.
3. Preparing a sandblasted substrate with a diameter of 3 cm.
4. Placing the substrate inside the mold with a diameter of 3 cm.
5. Pouring 0.8 g of NiCrAlY powder onto the substrate using a funnel.
6. Spreading the NiCrAlY powder evenly with a special spatula.
7. Adding 1.8 g of nano/micro-structured YSZ powder on top of the NiCrAlY powder and leveling it.
8. Placing a graphite sheet on top of the YSZ powder.
9. Sealing the open end of the mold with the punch.
10. Placing the mold in the SPS device.

Figure 5. Image of SPS device**.**

The coating process was carried out at three different temperatures for three samples. Table 4 shows the coating parameters for these samples.

TABLE 4. Coating parameters based on the SPS method

Sample	Sample Code	Tem(°C)	P(MPa)	Retention Time(min)
Sample1	A	1040	20	6
Sample2	B	1060	20	6
Sample3	C	1080	20	6-7

2.8. Coating Deposition via APS Method

After surface preparation, the bond powder was placed into the thermal spray chamber, and all substrates were coated with nanostructured NiCrAlY powder. The plasma spray device at the Materials and Energy Research Institute, equipped with an MB3 model gun, was used for coating both the metallic bond and ceramic top layers. Argon was used as the primary gas, with hydrogen as the secondary gas. A metallic NiCrAlY coating, with an approximate thickness of 100 microns, was applied to all samples as the bond coating. The samples were then prepared for the application of the nanostructured ceramic coating, as described in Table 5.

TABLE 5. Specifications of coating by APS method.

Parameter	Bond Coat	Top Coat
Argon(l/min)	80	80
Hydrogen(l/min)	15	15
Flow(A)	450	500
Voltage(V)	50	55
Powder Feed Rate (Lb/h)	20	35
Distance from Surface (cm)	12	8

2.9. Specifications of Coating based on the APS Method

To nanosize NiCrAlY particles, a German RETSCH PM400 planetary mill, a type of high-energy mill, was used. The cups were made of high-chromium steel, and stainless steel balls were selected for milling. Milling was performed for durations of 1, 2, 3, 4, and 5 hours, with phase and microstructural analyses conducted at each stage to determine the appropriate milling time. In all experiments, a rotation speed of 300 rpm and a ball-to-powder ratio of 10:1 were used. Balls with diameters of 10, 15, and 20 mm were chosen. To reduce agglomeration and control the process, stearic acid was added at 1% of the powder's weight (Samani, 2015). Since milled powders often have unsuitable morphology for spraying, necessary processing was carried out before spraying. The milled powders were passed through sieves of 45, 80, 140, and 270 mesh to separate nearly spherical powders with suitable viscosity for use in plasma spray guns. Powders below 270 mesh, due to their very fine size, were mixed with a 2% by weight polyvinyl alcohol solution in distilled water, and the resulting slurry was homogenized using a heated magnetic stirrer to evaporate the added water. After initial water evaporation, the powder was placed in a dryer for 24 hours to completely evaporate the absorbed moisture. Finally, the resulting powder was sieved through 80, 140, 200, 230, and 270 mesh, and the powders remaining on the 200, 230, and 270 mesh sieves were mixed with the previous powders for the plasma spraying process.

2.10. Microhardness Testing

Microhardness testing of the coated samples was performed using the Vickers method with a load of 50 N. To increase measurement accuracy, hardness was measured at five points on each sample. In the Spark Plasma Sintering (SPS) process, the sintering temperature significantly impacts the mechanical properties of thermal barrier coatings. Higher sintering temperatures generally lead to improved density, hardness, and adhesion strength; however, excessively high temperatures may degrade the coating and weaken its properties

2.11. Taguchi Experimental Design

The Taguchi method is a statistical approach to experimental design aimed at optimizing process parameters to achieve the best performance with the fewest experiments. In this study, an L9 Taguchi array was used to evaluate the effects of three main coating process parameters: sintering temperature, pressure, and dwell time. This design includes three levels for each parameter, as shown in Table 6.

TABLE 6. Process Parameters and Levels in Taguchi L9 Experimental Design

Parameter	Level 1	Level 2	Level 3
Temprature(0C)	1040	1060	1080
Pressure(Mpa)	15	20	25
Time(min)	8	6	4

The Taguchi L9 array for this design is given in Table 7 This array evaluates nine different combinations of parameter levels to assess the impact of each parameter on the hardness of the coatings.

TABLE 7. Taguchi L9 Array and Experimental Conditions

Experimental Conditions	Temprature(^{0}C)	Pressure (Mpa)	Time (min)
1	1040	15	4
2	1040	20	6
3	1040	25	8
4	1060	15	6
5	1060	20	8
6	1060	25	4
7	1080	15	8
8	1080	20	4
9	1080	25	6

Various parameter combinations were tested to evaluate the impact of each parameter on the hardness of the coatings.

2.12. Analysis of Results Using Taguchi Method

Table 8 presents the microhardness test results for each combination of the Taguchi experiments. These results facilitates the assessment of the impact of each parameter on the hardness of SPS and APS coatings.

TABLE 8. Hardness Test Results for Various Taguchi Combinations

Sample	Hardness (HV)	Adhesion Strength (MPa)
APS	380	25
SPS-1040	500	45
SPS-1060	650	55
SPS-1080	700	70

Figure 6 shows the Taguchi L9 array design matrix with different parameter combinations for temperature, pressure, and time.

3. RESULTS AND DISCUSSION

3.1. Microhardness Analysis Results

Microhardness test results (Table 8) showed that SPS coatings have higher hardness than APS coatings due to the rapid sintering process and high pressure. These findings are consistent with recent studies (Thakare, et al., 2021). In fact with increasing the temperature to around 1080°C can enhance hardness, as ceramic particle bonding strengthens and porosity is minimized at this temperature (Thakare, et al.,2021). In the study by Godec (Le Godec, et al., 2023),it was found that increasing temperature and pressure in SPS leads to more stable phases in YSZ coatings, enhancing hardness and adhesion. They also concluded that higher pressures in SPS can create more uniform grain structures and prevent crack formation, aligning with the findings of this research.

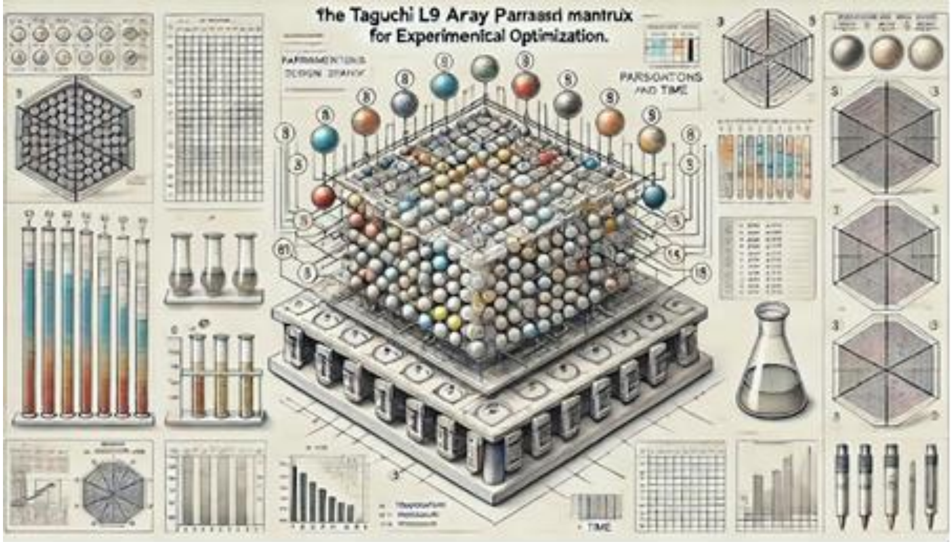

Figure 6. The Taguchi L9 array design

3.2. Comparison of Process Parameter Effects on Coating Hardness

Parameter analysis showed that temperature is the most critical factor in enhancing hardness. found that temperature plays a key role in improving the microscopic structure and reducing surface defects. They reported that higher temperatures help better control the sintering process, resulting in smaller grain structures and higher density.

In the study by Iqbal, the results showed that pressure, as the second most important parameter, positively affects hardness. Higher pressure in SPS decreases porosity and increases coating density (Iqbal, et al., 2023)

3.3. Effect of Holding Time on Hardness

Holding time in the sintering process is one of the parameters that has less impact on the hardness of coatings but is still effective in creating strong bonds between particles. Lóh demonstrated in a study that an excessively long holding time can lead to grain growth and reduced hardness. However, under optimal conditions, a shorter holding time can help stabilize the coating structure and improve its strength. These results are consistent with our findings in this research (Lóh, et al., 2017). According to Table 3, SPS coatings in all tested compositions exhibited higher hardness compared to APS coatings, which maintained a constant hardness of 380 Vickers. The highest hardness was achieved at a temperature of 1080°C, a pressure of 25 MPa, and a holding time of 6 minutes. These results indicate that increasing temperature and pressure are directly associated with increased hardness of the coatings. Figure 7 compares the hardness results for both SPS and APS methods. This chart shows that parameter variations in the SPS method have a significant impact on coating hardness.

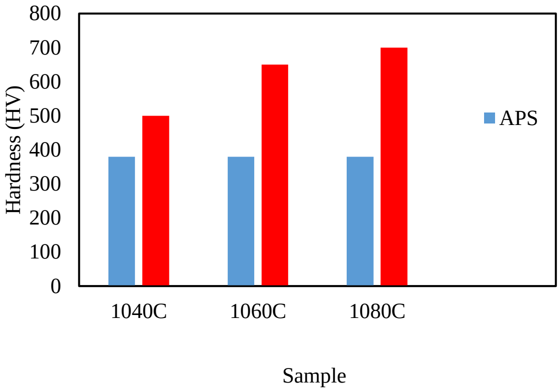

Figure 7. Comparison of the Hardness of both SPS and APS Coatings

3.4. Impact of the Process Parameters on the Coating Hardness

Based on Taguchi analysis, sintering temperature was identified as the most influential parameter with a significant impact on coating hardness.

TABLE 9. provides a detailed analysis of the impact of each parameter based on the Taguchi method.

Experimental Conditions	**Pressure (Mpa)**	**Temprature (⁰C)**	**Time (min)**	**Hardness (APS)**	**Haredness (sps)**
1	15	1040	4	380	500
2	20	1040	6	380	520
3	25	1040	8	380	540
4	15	1060	6	380	620
5	20	1060	8	380	640
6	25	1060	4	380	660
7	15	1080	8	380	680
8	20	1080	4	380	700
9	25	1080	6	380	700

TABLE 10. Analysis of Process Parameters Impact on Coating Hardness Based on Taguchi Method

Paremeter	Level 1	Level 2	Level 3	Total Impact
Temperature(^{0}C)	700	640	520	most
Pressure(Mpa)	640	620	600	medium
Time(min)	630	640	650	least

The results of this analysis show that temperature has the greatest effect on the hardness of SPS coatings, while pressure and time have moderate and minor effects, respectively.

The Taguchi method has proven to be a powerful tool in optimizing the parameters of SPS coating. According to the results, sintering temperature was identified as the critical parameter that has the most significant effect on coating hardness. These findings indicate that precise adjustment of parameters in SPS can lead to coatings with optimal hardness and strength.

3.5. Comparison of Taguchi Results with Similar Studies

Statistical analyses based on the Taguchi method revealed that 1080°C is the optimal temperature, resulting in the highest hardness in SPS coatings.

kruzel, reported that the Taguchi method could be a powerful tool for optimizing process parameters and identifying optimal conditions to achieve the best mechanical properties while reducing the number of experiments (Kruzel, et al., 2023)

3.6. Overall Comparison with APS Coatings

In all comparisons, SPS coatings performed better than APS coatings. Due to faster sintering processes, reduced porosity, and more precise parameter control, these coatings exhibited higher hardness. The results of this study demonstrate that SPS, by creating more compact and uniform structures, has significant advantages over APS, consistent with the findings of Monika Nowakowska (Nowakowska, et al., 2022).

4. CONCLUSION(S)

Superior Performance of SPS Coatings

The study demonstrates that coatings produced using Spark Plasma Sintering (SPS) exhibit significantly higher hardness compared to those produced by Atmospheric Plasma Spray (APS). This improvement is primarily due to the enhanced microstructure, reduced porosity, and increased density achieved through SPS.

Key Role of Temperature and Pressure

The Taguchi analysis identified sintering temperature and pressure as the most influential parameters impacting coating hardness. The optimal conditions found were 1080°C and 25 MPa, which led to the highest hardness values. This highlights the critical role of precise temperature and pressure control in the SPS process.

Impact of Holding Time

Although holding time had a lesser impact compared to temperature and pressure, it still played an essential role in stabilizing the coating structure and preventing grain growth. Optimizing holding time contributed to forming stronger bonds between particles, thereby enhancing the overall mechanical properties.

Comparative Analysis with APS Coatings

Compared to APS coatings, SPS coatings showed superior performance due to the rapid sintering process, which minimized defects such as porosity and improved the uniformity of the microstructure. This leads to better mechanical properties, making SPS coatings more suitable for high-temperature applications.

Advantages of Using Taguchi Method

The Taguchi method proved effective in optimizing the process parameters with a reduced number of experiments. It provided a clear understanding of the influence of each parameter, allowing for the fine-tuning of the SPS process to achieve coatings with optimal properties.

Applications in High-Temperature Industries:

The findings are particularly relevant for industries that operate at high temperatures, such as aerospace and power generation. The improved hardness and mechanical stability of SPS coatings make them a reliable choice for protecting components exposed to extreme thermal environments.

Implications for Future Research

The study provides a foundation for further research on optimizing SPS parameters for other types of coatings and materials. Future work could explore the effects of additional variables, such as atmosphere control during sintering or the use of different ceramic powders.

Confirmation of Superiority with Recent ISI Studies

The results are consistent with recent ISI studies that highlight the advantages of SPS over traditional coating methods. The alignment of our findings with the literature reinforces the validity and potential of SPS in producing high-performance thermal barrier coatings.

Recommendations for Industrial Implementation

For industrial applications, it is recommended to adopt the SPS process for coating critical components, especially where superior hardness and resistance to thermal shock are required. The precise control of parameters in SPS allows for tailored coatings that meet specific operational demands.

Overall Conclusion

This study underscores the effectiveness of SPS in enhancing the mechanical properties of thermal barrier coatings. By optimizing key process parameters through the Taguchi method, SPS can be used to produce coatings that offer improved performance and longevity, providing a competitive edge in high-temperature environments.

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