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Development & characterization of alumina coating by atmospheric plasma spraying

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Abstract. Ceramic coatings are applied on metals to prevent them from oxidation and corrosion at room as well as elevated temperatures. The service environment, mechanisms of protection, chemical and mechanical compatibility, application method, control of coating quality and ability of the coating to be repaired are the factors that need to be considered while selecting the required coating. The coatings based on oxide materials provides high degree of thermal insulation and protection against oxidation at high temperatures for the underlying substrate materials. These coatings are usually applied by the flame or plasma spraying methods. The surface cleanliness needs to be ensured before spraying. Abrasive blasting can be used to provide the required surface roughness for good adhesion between the substrate and the coating. A pre bond coat like Nickel Chromium can be applied on to the substrate material before spraying the oxide coating to avoid chances of poor adhesion between the oxide coating and the metallic substrate. Plasma spraying produces oxide coatings of greater density, higher hardness, and smooth surface finish than that of the flame spraying process Inert gas is often used for generation of plasma gas so as to avoid the oxidation of the substrate material. The work focuses to develop, characterize and optimize the parameters used in Al_2O_3 coating on transition stainless steel substrate material for minimizing the wear rate and maximizing the leak tightness using plasma spray process. The experiment is designed using Taguchi's L9 orthogonal array. The parameters that are to be optimized are plasma voltage, spraying distance and the cooling jet pressure. The characterization techniques includes micro-hardness and porosity tests followed by Grey relational analysis of the results.

1. Introduction
Ceramic coatings are applied on metals to prevent those materials from oxidation and corrosion at room as well as elevated temperatures. These coatings are high temperature coatings based on oxides, silicates, nitrides, carbides, cermet and super porcelains and other inorganic materials. There are a wide range of ceramic coatings that can be applied to metal components in order to enhance their functional properties. The advantages of ceramic coating on materials are high chemical resistance, excellent wear resistance, good reflectivity, fine electrical resistance and prevention of hydrogen diffusion. Ceramic coatings have got a wide variety of applications like Furnace fixtures, Textile, thread and fiber parts, Mechanical seal areas (sleeves, journals, etc.), Impellers, Molds, Pistons, Valves, Wear rings etc.

2. Literature Review
Ceramic coatings on metal surfaces can improve the performance of the metallic substrates [1,2].The special properties of Alumina, a ceramic coating includes high hardness, chemical inertness, wear resistance and a high melting point. These excellent properties enabled alumina ceramics to be widely used in many applications subjected to extreme pressure, force and temperature conditions. [3-6]. The force of adhesion between the ceramic sprayed coating and the substrate metal is usually not very

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strong due to chemical mismatch and other problems. A viable solution to this problem is to insert a metallic bond coat between the substrate and the coating. This bond coat is sprayed onto the substrate before the ceramic coating [7].Various coating methods can be used to produce ceramic coatings on the surface of materials. These methods differ from each other in terms of the coating quality they can achieve, efficiency of deposition, complexity of the process and cost of investment [8]. Thermal spraying is the application of the consumable material in to a substrate by melting the consumable material into fine droplets and impinging those droplets on to a substrate to form a uninterrupted coating [9]. Thermal spray coatings can be used as an effective means of producing such wear resistant surfaces [10]. Plasma spraying is a versatile technology that has been successful as a reliable cost- effective solution for many industrial problems. A variety of coating functions like wear resistance, heat and oxidation resistance, corrosion resistance, electrical or thermal conductivity [11]. While plasma spraying, a number of parameters needs to be given special attention. Studies showed that a number of parameters, relating mainly the current, voltage, stand-off distance, coating powder, base metal, coating method, and environmental effects during coating process, will greatly influence the quality of the resultant coating.

3. Experimental Details

The test substrate of 20mm x 20mm x 6mm was machined from 07X16H6 transition stainless steel. The chemical composition of the substrate material is shown in Table 1. Heat treatment of the material includes austenizing, sub-zero treatment and tempering. The substrate was then grinded to obtain a finish below 20 μm. The component was thoroughly cleaned with Percloro-ethylene and checked with ultra violet lamp for the surface cleanliness. The part was then grit blasted with abrasive grit (fused brown Aluminium Oxide of size ≤ 0.5mm). After grit blasting, the surface roughness of the sand blasted surface was more than 6.3 microns, which was checked with a comparator. The grit blasting equipment and its set up ensured a uniform blasting.

Table 1. Chemical Composition of 07X16H6 Substrate Material

Element	Cr	Ni	Si	Mn	C	P	S	Fe
% Composition	16	6	0.56	0.54	0.07	0.008	0.006	Balance

Atmospheric Plasma spraying was performed on a Metco 9MC plasma-spraying machine with Metco 9MB spraying gun and Metco 9MP feeder. NiCr powder was initially plasma-sprayed on the surface of the specimens to form a thickness of 50- 100 μm as the intermediate bond coat to avoid the mismatch betwwen the metallic substrate and the oxide coating. Pure Al_2O_3 ceramic powder was sprayed over the intermediate bond coat as the top coat for a thickness of 250 – 300 μm. Detailed spraying parameters are listed in Table 2.

Table 2. Plasma spraying parameters employed in the present study

Parameter	Coating	
	Ni-Cr Bond Coat	Al_2O_3 Coating
Primary Gas	Ar, 90 SCFH	Ar, 140 SCFH
Secondary Gas	H_2	H_2, As per Voltage
Powder Carrier Gas	Ar	Ar
Powder Feed Rate	25 g/min	30 g/min
Voltage	65 V	As per Design
Current	500 A	500 A
Spraying Distance	5”	As per Design
Cooling Jet Pressure	0 psi	As per Design

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4. Design of Experiments

Design of Experiments makes controlled changes to input variables in order to gain maximum amounts of information on cause and effect relationships with a minimum sample size. Ignition resistance, leak tightness and wear resistance are the main characteristics that are required for the material. Hardness and porosity are the two main factors that are properties of the above requirements. So the micro hardness and porosity of the coating are treated as the response variables. Previous literatures show that there are a lot of factors affecting the desired characteristics. In this work Plasma Voltage, Spraying Distance and Cooling Jet Pressure are selected as the control factors. The levels of factors are selected as three based on Taguchi design. The L9 Orthogonal Array (OA) is selected according to the number of control factors and their different levels. Table 3 shows the experimental design as per L9 Orthogonal Array

Table 3. L9 Orthogonal Array Experimental Design

Exp. No	Plasma Voltage (volts)	Cooling Jet Pressure (psi)	Spraying Distance (inch)
1	70	0	3.5
2	70	1	3
3	70	2.8	2.5
4	74	0	3
5	74	1	2.5
6	74	2.8	3.5
7	78	0	3
8	78	1	3.5
9	78	2.8	2.5

5. Characterization

The characterization techniques includes micro hardness and porosity tests. The coated samples were mounted using epoxy powder. The mounting was done using an automatic hot compression mounting press. The curing time was 10 minutes and cooling time was 5 minutes. The pressure and temperature were 150 bar and 160^0 C respectively. The polishing steps included rough polishing followed by emery paper polishing having grit number 220, 320, 400, 600, 800 and 1000. Polishing using alumina paste, diamond paste and using aerosol spray on yellow cloth was done as the subsequent steps. Micro hardness was evaluated using Vickers Hardness test using Qness Q10 A+ Vickers hardness test. Axio Lab A1 was the optical microscope used for the microstructural evaluation and porosity test.

6. Results and Discussion

6.1. Micro Hardness

The micro hardness was evaluated using Vickers micro hardness test. The load used for indentation was 300 gf. The indentation was made using a square based pyramidal diamond indenter having an included angle of 136^0. The specimen was prepared according to ASTM 382 standard for the evaluation. The micro hardness test results of the experiments are shown in Table 4.

6.2. Microstructure evaluation

The specimen was prepared according to the ASTM standards. Figure 1 and Figure 2 shows the microstructural images of the specimen in different magnifications. The coated specimens showed a uniform distribution of the porosity over the entire surface area. Cracks or similar defects were not present in the coatings. The bonding between the bond coat and the top alumina coating as well as the bottom metallic substrate was found to be visually stronger. The coating thickness of the bond coat was around 60µm and that of the top coating was slightly above 250µm.

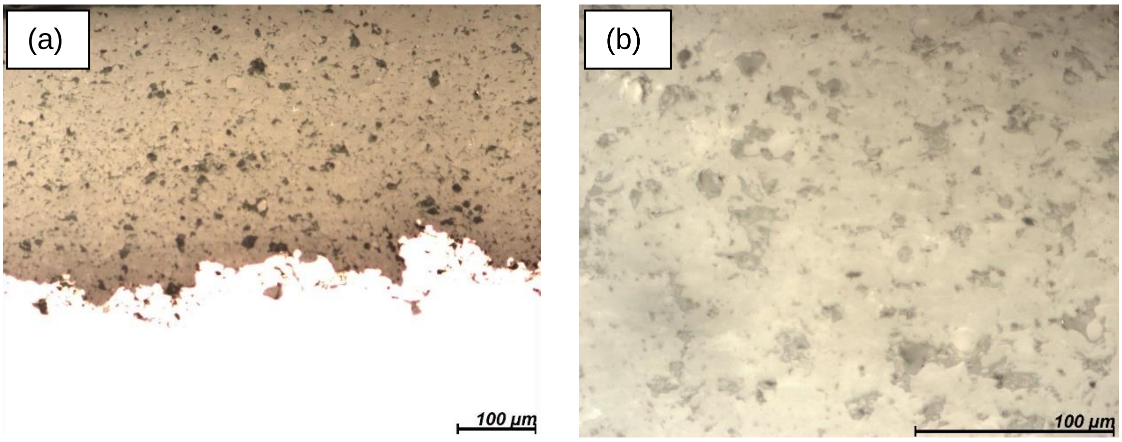

Figure 1: (a) Microstructural image of the specimen under 200x magnification and (b) Microstructural image of the top coating under 500x magnification

6.3. Porosity Evaluation

The sample preparation for porosity evaluation was done according to the standard ASTM E2109 – 01. The image was taken with a microscope and was evaluated using an automatic image analyzer to analyse the porosity. Random samples were taken from the entire area of coating and the porosity values mentioned in Table 4 are the arithmetic average of all the values obtained from a single sample. The porosity size varied and was almost uniformly distributed.

Table 4: Micro Hardness and Porosity of the Experiments

Exp. No	Micro Hardness ($HV_{0.3}$)	Porosity (%)
1	761	3.55
2	602	3.24
3	901	2.35
4	897	2.93
5	738	2.22
6	597	2.20
7	794	2.09
8	634	3.94
9	778	2.09

6.4. Grey Relational Analysis

There are two performance characteristics that need to be optimized (maximization in case of micro hardness and minimization in case of porosity). The problem can be converted into a single objective problem using grey relational grade. The experimental results (micro hardness and porosity) are normalized in the range between 0 and 1 in the initial step which can be termed as grey relational generation. Grey relational coefficient can be calculated to establish the relation between the actual and desired experimental data from the initial step. Grey relational grade is calculated by computing the average of grey relational coefficients.

Micro Hardness is to be normalized according to Larger the better condition and Smaller the better condition is to be used for analysing the porosity. Eq. 1 and Eq. 2 are used for hardness and porosity respectively.

$$x_i^*(k) = \frac{x_i^0(k) - min x_i^0(k)}{\max x_i^0(k) - \min x_i^0(k)} \quad (1)$$

$$x_i^*(k) = \frac{max x_i^0(k) - x_i^0(k)}{\max x_i^0(k) - \min x_i^0(k)} \quad (2)$$

Where, i vary from 1 to 9. The deviation, Δ_i is calculated as shown in Eq. 3 whereas Eq. 4 is used to calculate the grey relational coefficient, $\varphi_1\ (k)$.

$$\Delta_i = |1 - x_i^*(k) \quad (3)$$

$$\varphi_1\ (k) = \frac{\Delta_{min} + \delta\Delta_{max}}{\Delta_i\ (k) + \delta\Delta_{max}} \quad (4)$$

Where k is the current response factor number. The grey relational grade is the average of grey relational coefficients. The grey relational grade, γ_i can be defined mathematically as shown in Eq. 5.

$$\gamma_i = \frac{1}{n}\sum_{k=1}^{n} \varphi_i\ (k) \quad (5)$$

Where, n = Number of response factors. Table 5 shows the Grey relational Analysis results. The experiment number 3 with 70V, 2.8 psi cooling jet pressure and 2.5” spraying distance showed the maximum grey grade, 0.89.

Table 5. Grey Relational Analysis Results

Exp. No	Normalized Values		Deviation		Grey Relational Coefficient		Grey Grade	Rank
	Hardness	Porosity	Hardness	Porosity	Hardness	Porosity		
1	0.539	0.212	0.461	0.788	0.521	0.388	0.454	7
2	0.016	0.378	0.984	0.622	0.337	0.446	0.391	8
3	1	0.859	0	0.141	1	0.781	0.890	1
4	0.987	0.545	0.013	0.455	0.974	0.523	0.749	2
5	0.464	0.928	0.536	0.072	0.483	0.875	0.678	5
6	0	0.943	1	0.057	0.333	0.898	0.616	6
7	0.648	0.999	0.352	0.001	0.587	0.997	0.792	3
8	0.122	0	0.878	1	0.363	0.333	0.348	9
9	0.595	1	0.405	0	0.553	1	0.776	4

Table 6 gives the response table of the grey relational analysis. Level 1 of plasma voltage is used in experiments 1, 2 and 3. Similarly level 2 in 4,5,6 and level 3 in 7,8,9.the GRG of each level is the average of the GRG of the particular set of experiments. The same method is adopted in case of CJP and SD. Maximum values of GRG are obtained at levels 2,3 and 3 for plasma voltage, cooling jet pressure and spraying distance respectively. Based on the difference between the maximum and minimum values of GRG, they are ranked. So the optimum levels of parameters are plasma voltage = 74 V, cooling jet pressure = 2.8psi and spraying distance = 2.5”. That is, PV_2 CJP_3 SD_3.

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Table 6. Response Table for Grey Relational Grade

Process Parameters	Grey Relational Grade				Rank
	Level 1	Level 2	Level 3	Difference	
Plasma Voltage (PV)	0.578	*0.681*	0.639	0.103	3
Cooling Jet Pressure (CJP)	0.665	0.473	*0.761*	0.288	2
Spraying Distance (SD)	0.473	0.639	*0.787*	0.314	1

7. Conclusions

The Grey relational analysis based on Taguchi L9 orthogonal array optimized the process variables in atmospheric plasma spraying of alumina on stainless steel. The analytical results can be summarized as follows:

1. The response table of the average Grey relational grade of each parameters on different levels show that that the largest value of the Grey relational grade was obtained for the plasma voltage 74V, cooling jet pressure 2.8psi and spraying distance 2.5”. These are the recommended levels of the controllable parameters of the plasma spraying process as the maximization of micro hardness and minimization of porosity are considered together.
2. The order of the importance for the different controllable factors is in the order of spraying distance, cooling jet pressure and plasma voltage. That is, among the various listed parameters, spraying distance shows the maximum correlation to the desired outputs.

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