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SENSITIVITY ANALYSIS OF ATMOSPHERIC PLASMA SPRAYING PROCESS PARAMETERS ON POROSITY OF ALUMINA COATING ON AZ31B MAGNESIUM ALLOY

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ABSTRACT

This paper focuses on the development of empirical relationship for the prediction of porosity of plasma sprayed alumina coated AZ31B magnesium alloy. Experimental part of the study is based on five level central composite designs of three (process) parameters. In order to investigate the effects of input parameters on porosity, an empirical relationship is constructed by multiple regression analysis. A sensitivity analysis is carried out and compared the relative impact of input parameters on porosity in order to verify the measurement errors on the values of the uncertainty in estimated parameters. The results obtained show that developed empirical relationship can be applied to estimate the effectiveness of process parameters for a given porosity. The input power is more sensitive than standoff distance and powder feed rate.

Keywords: *Atmospheric Plasma Spraying, Alumina Coating and Porosity*

1. Introduction

Magnesium alloys are an alternative to iron and aluminium as structural materials since their low density and high strength-to weight ratio offer a multitude of possibilities in potential applications in which weight reduction and appropriate mechanical properties are important requirements to be achieved. Automobile and civil aviation have increased their interest by Mg-alloys as a result of actually restrictive environmental legislation on greenhouse gas emissions like CO_2 [1] because weight reduction is a good option for significant reduction of fuel consumption and CO_2 emissions.

Thermal spraying is a highly complex deposition process with a large number of interrelated variables. Due to the high velocity and temperature gradients in the plume, even small changes in the controllable or uncontrollable parameters can result in significant changes in the particle properties and thus in the microstructure of the coatings. Thermally sprayed Al_2O_3 coatings have been widely studied as wear, corrosion resistant as well as thermal or electrical insulative coatings in order to improve the surface characteristics of industrial components. Many techniques have been used to spray such kind of materials. Atmospheric plasma spraying (APS), due to its relatively high deposition efficiency, flexibility, and easy automation, has become a commercial process to deposit Al_2O_3 coatings [2-3].

Porosity is the basic and key quality characteristics to understand the microstructure and properties of thermal spray coatings. During plasma spraying, the pores and micro-cracks can be generated from different sources, such as the entrapped gases, the incomplete filling in the rapidly solidifying splats, and the shrinking of the splats during rapid solidification etc. If no distinction is made of the nature of pores and the micro-cracks, the porosity in plasma-sprayed coatings can verify from less than 2% to more than20%, depending on the type of powders and the deposition parameters used [4]. For instance, when the spherical-shape pores are closed in the coating, the entrapped gases during spraying process are often considered as the main source. The intra-lamella cracks, which can occur within the splats, are generally believed to be formed due to the shrinking of the splats during rapid solidification. Among these features, porosity level is a key parameter describing the anisotropy of sprayed coatings and controlling their properties.

In aggressive environments, one of the major problems in using plasma-sprayed coatings is the presence of the open pores, closed pores and micro-cracks in the coatings [5-6]. Moreover, the presence of even insignificant micro-pores can substantially reduce the coating's mechanical and protective properties, such as elastic modulus, micro-hardness and bonding strength, etc. Therefore, reduction of porosity of the

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sprayed coatings plays a key role in improvement of the corrosion resistance of the coatings.

In this paper, an empirical relationship between APS process parameters and porosity was constructed based upon the experimental data obtained by three parameters-five levels central composite design. The empirical equation, simulating the APS process, was carried out by Multiple Regression Analysis (MRA) and a sensitivity equation was derived from this basic equations. This analysis generally requires a definition of an objective function and design parameters. In this study, the objective function was chosen as porosity, whereas process parameters (input power, standoff distance and powder feedrate) were selected as the design variables. The present study mainly focuses on the determination of sensitivity characteristics of design parameters and the prediction of fine-tuning requirements of these parameters in APS process. The results revealed considerable information about the effect of process parameters and optimum spraying conditions.

2. Experimental Work

2.1 Identifying the important process parameters

From the literature [7-10] and the previous study done in our laboratory, the predominant factors (APS process parameters) that have a greater influence on the coating properties were identified. They are (i) the power (kW), (ii) the standoff distance (cm), (iii) the powder feed rate (gpm).

2.2 Finding the working limits of the parameters

A large number of spray trial runs were carried out on grit blasted extruded AZ31B magnesium alloy (16 mm in diameter and 15 mm in thickness) to determine the feasible working limits of APS parameters. The chemical composition and mechanical properties of the base metal are presented in Tables 1 and 2 respectively.

Table 1: Chemical Composition (wt %) of AZ31B Mg Alloy

Al	Mn	Zn	Mg
3	0.2	1	Balance

Plasma spray deposition was carried out using an APS system 40 kW IGBT-based Plasmatron (Make: Ion Arc Technologies; India. Model: APSS-II).The feed stock was H.C. Stark, AMPERIT 740.1 powder (Al_2O_3) with particle size of -45+20µm. Different combinations of APS process parameters were used to carry out the trial runs. Prior to deposition, specimens were sandblasted, in order to increase their surface roughness and achieve better adherence between the ceramic coating and the metallic substrate. Grit blasting was carried out using corundum grits of size of 500 +320 µm and subsequently cleaned using acetone in an ultrasonic bath and dried. After grit blasting, the average surface roughness was measured using the surface roughness tester. (Make: Mitutoyo, Japan; Model: Surf test 301).The average roughness was found to be 5µm.Plasma primary and auxiliary gases were Ar and H_2, in addition Ar was used as carrier gas. Coating thickness for all the deposits was maintained at 200 ± 15 µm.Photograph of alumina coated specimens shown in Fig.1.

Table 2: Properties of AZ31B Mg Alloy

Yield strength (MPa)	Ultimate tensile strength (MPa)	Elongation (%)	Hardness(Hv) at 0.05 kg load
171	215	14.7	69.3

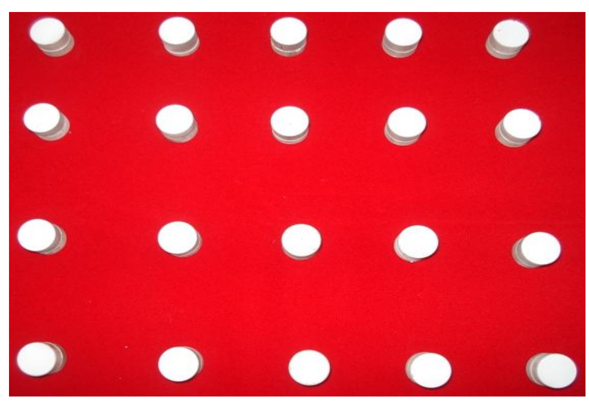

Fig. 1 Alumina Coated Specimen's Photo

2.3 Developing the design matrix

By considering all the above conditions, the feasible limits of the parameters were chosen in such a way that the AZ31B magnesium alloy should be coated using atmospheric plasma spraying process by varying plasma spraying parameters such as power, standoff distance and powder feed rate. So that reasonably good adherent coatings could be obtained. Central composite rotatable design of second order was found to be the most efficient tool in response surface methodology (RSM) to establish the mathematical relation of the response surface using the smallest possible number of experiments without losing its accuracy [11]. As the range of individual factor was wide, a central composite rotatable three factor five level factorial design matrix was chosen to optimize the experimental conditions. With a view to achieving the aforementioned aim, statistically designed experiments based on a factorial technique were used to reduce the cost and time and to obtain the required information pertaining to the main and interaction effects on the response parameters. Table 2 presents the ranges of factors considered, and table 3 shows the 20 sets of coded conditions used to form the design matrix. The design matrix is consisting 20 sets of coded conditions and comprising a full replication three factor factorial design of 8 points, six corner points and six centre points. All of the variables at the intermediate (0) level constitute the centre points while the combinations of each process variable at either the lowest (-1.682) or the highest (+1.682) value with the other four variables of the intermediate levels constitute the star points. Thus, the 20 experimental runs allowed for the estimation of the linear, quadratic, and two-way interactive effects of the variables on the APS-Al_2O_3 coating deposits. The method of designing such a matrix is dealt with elsewhere. For the convenience of recording and processing experimental data, the upper and lower levels of the factors are coded here as +1.682 and −1.682, respectively. The coded values of any intermediate value can be calculated using the following relationship:

$$X_i=1.682[2X-(X_{max}+X_{min})]/(X_{max}-X_{min}) \qquad (1)$$

Where X_i is the required coded value of a variable X and X is any value of the variable from X_{min} to X_{max}; X_{min} is the lower level of the variable; X_{max} is the upper level of the variable.

2.4 Experimental investigation

In this investigation, 20 coating deposits were prepared using different combinations of APS process parameters, as prescribed by the experimental design matrix (Table 3).The experiments were conducted in a random order to prevent systematic errors from infiltrating the system.The porosity was analyzed on the polished cross section of the coating as per ASTM B276 standard [12] using optical microscope (Make: MEIJI, Japan; Model: MIL-7100) equipped with image analyzing system.

Table 3: Important APS Process Parameters and their Levels

Factors	Not.	Units	Levels				
			-1.682	**-1**	**0**	**+1**	**1.682**
Power	A	kW	18	19.4	21.5	23.6	25
Standoff distance	B	cm	10	10.6	11.5	12.4	13
Powder feed rate	C	gpm	15	20	25	30	35

The morphologies of the powder and coating were observed with a scanning electron microscopy (SEM; JSM 6400, JEOL, Tokyo, Japan).The powder is fused and then crushed, which gives its characteristic angular shape with a size distribution ranging between 2-8μm as shown in Fig. 2.The backscattered scanning electron micrographs of the cross-sections of the alumina coating revealed the very rough surface, interconnected pores randomly distributed within the layer and poor bonding at the substrate/coating interface(Fig.3). Spattering pattern appears on the surface, which indicates the occurrence of spraying molten drops during coating process. The alumina coatings consist of countless single-spots from which a few circular pores are present on the coating surface, the non-uniform growing pattern of the coating and trapping of oxygen bubbles in the coating growth process may be responsible for the extensive porosity of the ceramic coating.

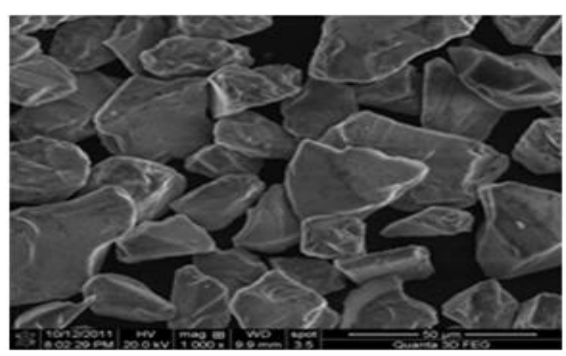

Fig. 2 SEM Micrograph of the Al_2O_3 Powder

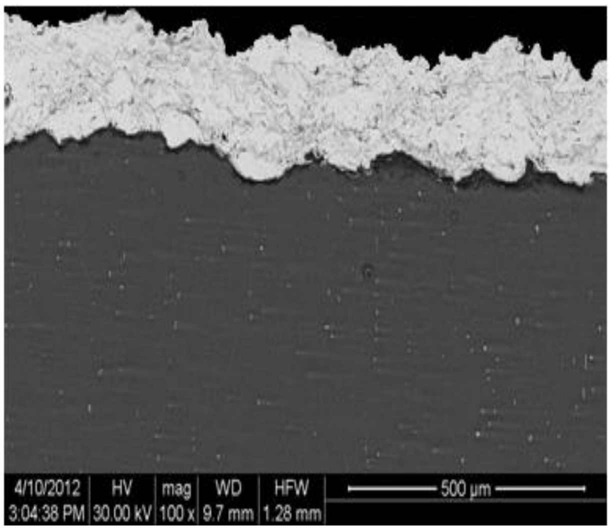

Fig. 3 Back scattered scanning electron micrograph of the cross section of alumina coating

3. Developing an Empirical Relationship

The porosity is a function of power (A), standoff distance (B), powder feed rate (C) and hence it can be expressed as,

$$P = f\{A, B, C\} \qquad \ldots\ldots (2)$$

The second-order polynomial (regression) equation used to represent the response surface Y (P) is given by,

$$Y=b_0+\Sigma b_i x_i+\Sigma b_{ii} x_i^2+\Sigma b_{ij} x_i x_j, \qquad \ldots\ldots (3)$$

And for three factors, the selected polynomial could be expressed as

$$P=b_0+b_1(A)+b_2(B)+b_3(C)+b_{12}(AB)+b_{13}(AC)+b_{23}(BC)+b_{11}(A^2)+b_{22}(B^2)+b_{33}(C^2) \qquad \ldots\ldots (4)$$

Where b_0 is the average of the responses and b_1, b_2, b_3… b_{11}, b_{12}, b_{13}… b_{22}, b_{23}, b_{33}, are regression coefficients that depend on respective linear, interaction, and squared terms of factors. The value of the coefficient was calculated using the following expressions,

$$b_0=0.166338(\Sigma X_0 Y)+0.05679(\Sigma\Sigma X_i Y) \qquad \ldots\ldots (5)$$

$$b_i=0.166338(\Sigma X_i Y) \qquad \ldots\ldots (6)$$

$$b_{ii}=0.0625(\Sigma X_{ii} Y)+0.06889(\Sigma\Sigma X_{ii} Y)-0.056791(\Sigma\Sigma X_0 Y) \qquad \ldots\ldots (7)$$

$$b_{ij}=0.125(\Sigma X_{ij} Y) \qquad \ldots\ldots (8)$$

Where i^2 varies from 1 to n, in which X_i is the corresponding coded value of a factor and Y is the corresponding response output value (porosity) obtained

4. Sensitivity Analysis

Sensitivity analysis, a method to identify critical parameters and rank them by their order of importance, is paramount in model validation where attempts are made to compare the calculated output to the measured data. This type of analysis can be useful to find out, which input parameter must be most accurately measured, thus determining the input parameters exerting the most influence upon model outputs [13]. Mathematically, from the experiment and 'n' is the total number of combinations considered (in this case n=20) porosity

$$P=\{5.32+2.50(A)+1.69(B)+1.30(C)0.87(AB)+0.88(AC)+1.38(BC)+1.54A^2)+2.42(B^2)+1.72(C^2)\}(vol\%) \qquad \ldots\ldots (9)$$

Sensitivity of a design objective function with respect to a design variable is the partial derivative of that function with respect to its variables. To obtain the sensitivity equation for input power, Eq. (4) with non significant terms is differentiated with respect to input power. The sensitivity Eqs. (10), (11) and (12) represent the sensitivity of porosity for input power, standoff distance and powder feed rate, respectively

$$\partial P/\partial A=-2.50-0.87B+0.88C+3.08A \qquad \ldots\ldots (10)$$

$$\partial P/\partial B=1.69-0.87A+1.38C+4.84B \qquad \ldots\ldots (11)$$

$$\partial P/\partial C=1.30+0.88A-1.38B+3.44C \qquad \ldots\ldots (12)$$

In this study, the aim is to predict the tendency of porosity due to a small change in process parameters for plasma spraying process. Sensitivity information should be interpreted using mathematical definition of derivatives. Namely, positive sensitivity values imply an increment in the objective function by a small change in design parameter whereas negative values state the opposite [14-16].

Table 3: Design Matrix and Experimental Results

Spray condition	Coded values			Original value			Porosity, vol.%
	A	B	C	A (kW)	B (cm)	C (gpm)	
1	-1	-1	-1	19.4	10.6	20	12
2	1	-1	-1	23.6	10.6	20	7
3	-1	1	-1	19.4	12.4	20	14
4	1	1	-1	23.6	12.4	20	6
5	-1	-1	1	19.4	10.6	30	10
6	1	-1	1	23.6	10.6	30	9
7	-1	1	1	19.4	12.4	30	18
8	1	1	1	23.6	12.4	30	13
9	-1.682	0	0	18	11.5	25	14
10	1.682	0	0	25	11.5	25	5
11	0	-1.682	0	21.5	10	25	9
12	0	1.682	0	21.5	13	25	15
13	0	0	-1.682	21.5	11.5	15	8
14	0	0	1.682	21.5	11.5	35	12
15	0	0	0	21.5	11.5	25	5
16	0	0	0	21.5	11.5	25	6
17	0	0	0	21.5	11.5	25	5
18	0	0	0	21.5	11.5	25	6
19	0	0	0	21.5	11.5	25	5
20	0	0	0	21.5	11.5	25	5

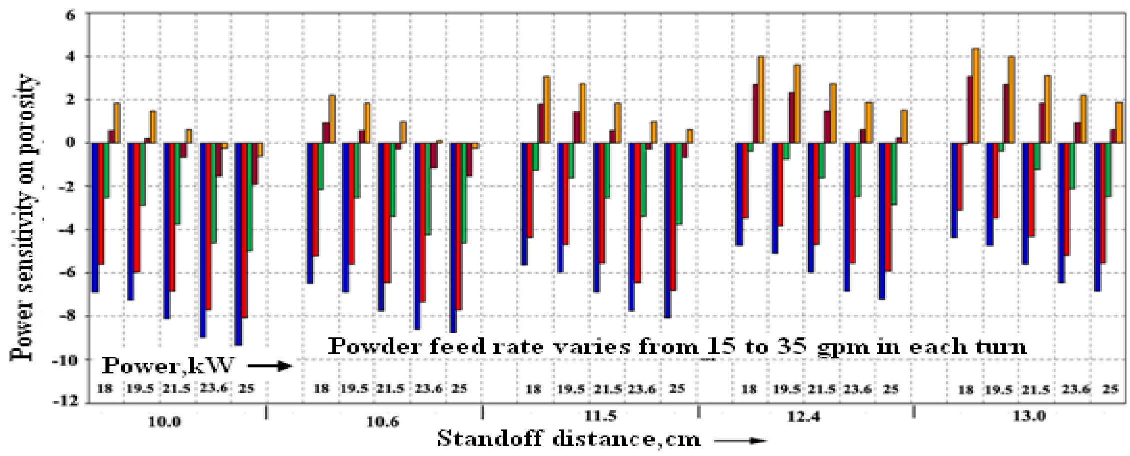

Fig. 4(a) Input Power Sensitivity of Porosity

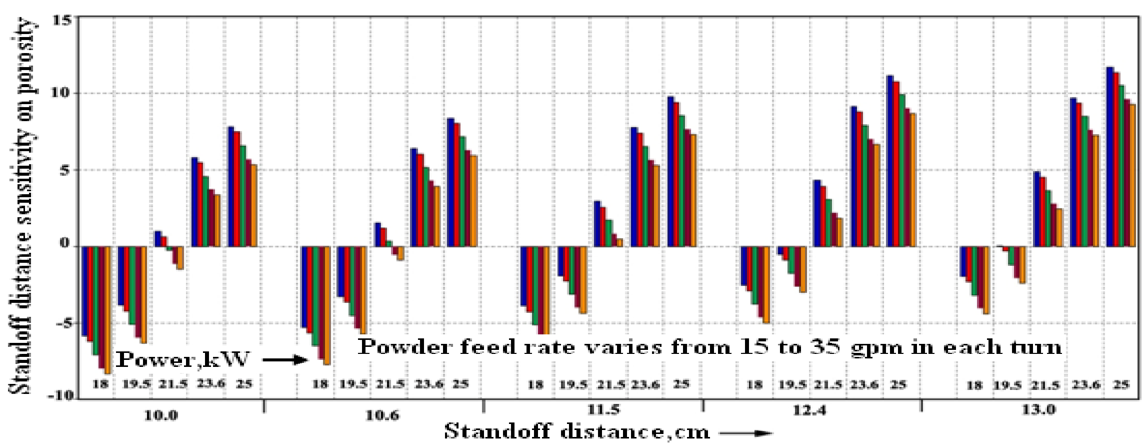

Fig. 4(b) Standoff Distance Sensitivity of Porosity

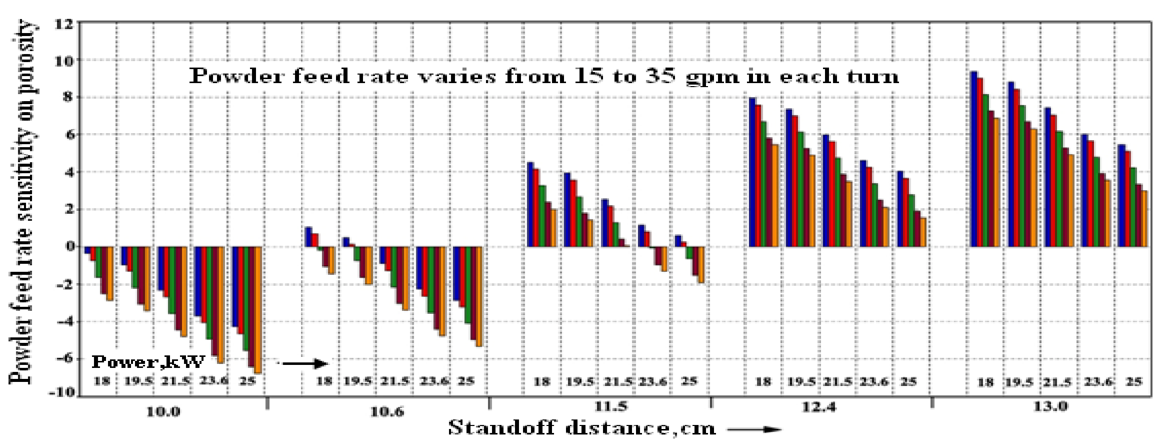

Fig. 4(c) Powder Feed Rate Sensitivity of Porosity

Fig. 4 Sensitivity Analysis Result

Fig. 4 displays the input power, standoff distance and powder feed rate sensitivity maps on porosity respectively. Standoff distance values less than 11.5 cm indicates positive values and greater than 11.5 cm shows negative sensitivity. The small variation of input power causes large changes in porosity. The results reveal that the porosity is more sensitive to input power than standoff distance and powder feed rate.

5. Conclusions

The following important conclusions are obtained from this investigation

i. An empirical relationship was developed to predict the porosity of plasma sprayed alumina coated AZ31B magnesium alloy, incorporating process parameters. The developed relationship can be effectively used to predict the porosity of alumina coated AZ31B magnesium alloy at 95% confidence level.

ii. Input power was more sensitive than the other parameters such as standoff distance and powder feed rate.

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