Optimization Of Plasma Parameters For Al_2O_3-40wt%8YSZ Composite Ceramic Coating Using Response Surface Methodology

G. Perumal and N. Alagumurthi
[a]V.R.S College of Engineering and Technology, Arasur, Villupuram-607 107

[b]Pondicherry Engineering college, Pondicherry

Abstract

Plasma spraying process is a more successful technique to develop ceramic coating for many industrial applications, where high wear and corrosion resistance with heat resistance are critical. But, the presence of porosity is the main issue for thermal spray coatings. The present work deals with the investigation on the significance of response surface methodology for minimizing the porosity in Al_2O_3-40wt%8YSZ composite coating developed on Ti-6Al-4V alloy and the effect of plasma parameters on porosity of coated alloy. Input power, primary gas flow rate and spraying distance are chosen as plasma parameters to assess the porosity of composite coating. The variation of porosity with plasma parameters are mathematically modeled using response surface methodology. Optimum parameters for minimum porosity are also determined. The effect of plasma parameters on porosity is investigated by developing surface response plots for selected plasma parameters within the design range. The results indicate that the input power has a largest effect on porosity of the coatings and followed by spraying distance and primary gas flow rate. Both predicted and measured values of porosity reasonably agree with each other. It indicates that the predicted model is more effective to determine the plasma parameters to develop Al_2O_3-40wt%8YSZ composite coating on Ti-6Al-4V alloy with minimum porosity.

Keywords: Ti-6Al-4V alloy, Plasma spray, Porosity, Response surface methodology

1. Introduction.

Ceramic coating is the best alternative to the metal components to modify their surface properties to prevent them from surface degradation. In ceramic coatings, ceramic provides resistance to wear, corrosion, erosion etc. and substrate provides mechanical properties like strength and stiffness. The combined properties of both ceramics and metallic substrate extend the life period of the components and thereby it reduces the requirement of repetitive maintenance and expensive components replacement. Among the different surface modification technique, the positive features like high temperature (15000 k) ,fast deposition rate and high energy density of plasma spraying process can deposit high melting point materials, such as ceramics make this process precious to develop different kinds of coatings for various applications. Recently, Plasma sprayed alumina/zirconia composite coatings are found to have superior mechanical properties and tribological properties than monolithic alumina or zirconia[1,2,3,4].However, the porosity of the thermally sprayed ceramic coatings is the main issue that determines the durability and functional characteristics of the coatings [5, 6]. Previous studies have also reflected that the porosity, size of pores and distribution of pores in the coatings are strongly influenced by the plasma spraying parameters [6, 7, 8, 9].Utilization and recognition of any coating system depend on the suitable quality of the coatings. In order to get good quality, it is necessary to characterize, and interpret the performance of the coatings before it is employed for a particular application. It need to be employed optimization techniques to find optimal plasma parameters and theoretical models for prediction of coating quality with minimum experimental trials. In case of thermal spraying technique, the optimization of the spraying parameters is not an easy task. Because, number of processing parameters involved are more in the thermal spraying technique. Design of Experiments (DOE) is an effective tool for conducting minimum number of experiments to get optimal plasma parameters for enhanced coating properties. Many research reports reveal that the DOE techniques have been successfully employed to optimize plasma parameters [10-16]. Dyshlovenko et.al, [11] studied the effect of parameters on plasma spraying and laser treatment of hydroxyapatite coatings using two level full factorial designs. Hasan et.al [12] has utilized two level factorial designs to optimize the parameters of the high velocity oxy-fuel coating of hydroxyapatite for orthopedic applications. Azarmi et.al [13] used D-optimal design to optimize the Plasma spray parameters to deposit inconel 625 alloys on mild steel substrate, who reported that the spray distance, particle size, and arc current have a largest effect on the porosity. Taguchi's method as well as response surface methodology is an efficient means to correlate the plasma parameters with coating properties and for determining the effects of plasma parameters on the measured responses. Taguchi's experimental design was used by Saravanan et.al [15] to correlate the Plasma parameters and detonation gun-spraying process parameters with properties of alumina coatings and the results demonstrate that the input power, primary gas flow rate and spray distance are more influencing factors on porosity of plasma sprayed alumina coatings. Bor-Tsuen Lin et.al [16] has used the response surface methodology to optimize the plasma parameters to obtain higher hardness in the plasma sprayed yttrium stabilized zirconia coating. The results show that the most significant variables that affect the hardness are arc current,powder feed rate and primary gas flow rate. To the best of the author's knowledge, there is no such optimization studies have been carried out to develop Al_2O_3-40wt%8YSZ composite coating on Ti-6Al-4V alloy using plasma spraying technique. The present work aims at developing the composite coating on Ti-6Al-4V alloy using Al_2O_3-40wt%8YSZ composite blend by varying the most influencing parameters which are identified from previous reports. Response surface methodology has been used to optimize plasma parameters for minimum level of porosity. Response plots and mathematical model have also been developed to correlate the plasma parameters with porosity.

2. Experimental methods

2.1. Materials and processing

Commercially available Al_2O_3 with particle size 5-45µm and 8mole% of yttrium stabilized ZrO_2 with particle size 15-45µm were used to obtain the composite feed stock powders. The as-received Al_2O_3 powders are angular and irregular morphology, whereas the yttrium stabilized ZrO_2 powders are of spherical morphology. Composite feed stock powder was obtained by blending the 60wt% of Al_2O_3 and 40wt% of yttrium stabilized ZrO_2 powders using planetary ball mill at a speed of 200rpm for the duration of 3hours without addition of alumina ball in order to prevent the breaking of particles. Fig.1. shows the SEM morphology of composite Al_2O_3- 40wt%ZrO_2 composite powder after the blending. It reveals that zirconia powders uniformly distributed with alumina powders. All the coatings were deposited on Ti-6Al-4V alloy (grade5) of dimension 30x25x3 mm^3. The chemical composition of the substrate material used for this study is shown in Table.1

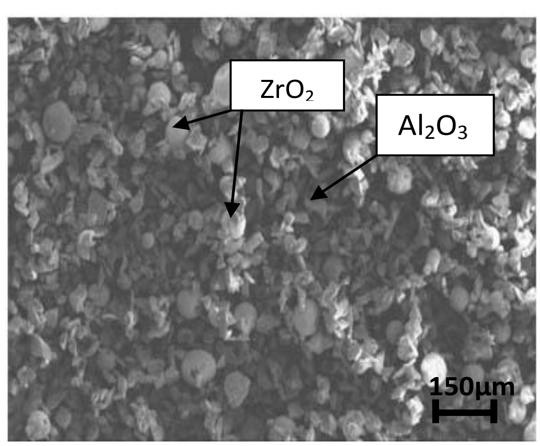

Fig.1 .SEM morphology of as-blended Al_2O_3- 40wt%ZrO_2 composite powders

Table.1Chemical composition of substrate materials

Substrate	Composition (wt %)						
Ti-6Al-4V	C	Fe	V	N	Al	O	Ti
(grade5)	0.08	0.2	4.06	0.009	6.48	0.13	Remaining

2.2. Response surface methodology

The porosity of the plasma sprayed coatings is critical for many engineering applications which have significant effect on wear, corrosion and thermal resistances etc. Porosity is one of the factors to assess the coating quality. Usually, porosity level in the coatings developed using atmospheric plasma spraying process is ranging from 1 to 10%. Minimum porosity can be achieved only by employing appropriate plasma parameters. Hence, it is essential to know in advance the surface quality and properties of the coatings for the implementation to the specific applications. Theoretical response models need to be developed and the developed response model is able to predict porosity at different plasma parameters for a good quality coating. To develop the model for coating porosity needs an appropriate design of experiments and consistent statistical analysis. Response surface methodology (RSM) is a set of mathematical and statistical techniques that is practical for the modeling and analysis of problems in which a response of interest is influenced by several factors and the objective is practically used to optimize this response [17].In case of RSM problems, the form of relationship among the response and the independent variables are unknown. Hence, initially a polynomial type of a model is chosen and it establishes the relationship between the response and independent process parameters employed. Since, the relationship between input and output response of the plasma spray system is nonlinear, first order models may not be sufficient. Therefore, more detailed plasma spraying process representation, it needs a second-order response surface model [16]. In the perspective of polynomial regression, an experiment is performed for plasma spraying process parameters x_1, x_2. . . $x_{p,}$ and the measured response of experiment *y* is represented in the following form

$$y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_i \sum_j \beta_{ij} x_i x_j + \varepsilon \qquad (1)$$

Where β_0, β_i, β_{ii}, β_{ij}, are coefficient for corresponding terms and ε *is* random errors. The set of regression coefficients β's are unknown and are estimated by the method of least squares.

2.3 Coating development and Characterization

Metco 3MB plasma gun with 40KW atmospheric plasma spray system was used to develop the coatings. All the coatings were deposited on Ti-6Al-4V alloy of size 30x25x3 mm^3. Before the deposition, the substrate was sand blasted using corundum of size # 24 mesh at an air pressure of about 50psi to improve the adhesion strength between the coating and substrate. The experiments are conducted using response surface methodology with central composite rotatable design which helps to reduce the number of experiments. The plasma parameters chosen for the present study are input power (P), spray distance(S) and primary (Ar) gas flow rate (A). Since, the variables considered for coatings are multi-level variables and their resulting effects are not linearly related, it was decided to use five level experiments for each variable. Previous studies on the effect of plasma parameters on ceramic coating demonstrate that higher input power, higher primary gas flow rate, lower spraying distance and lower powder feed rate were found to cause high hardness and lower porosity of the coatings [15, 16]. Hence, large numbers of experiment trials were carried out to deposit composite powder on Ti-6Al-4V alloy to achieve reasonably closer limits of plasma parameters for quality coatings. The main plasma parameter which was varied and their levels are shown in Table.2. Some parameters were kept constant throughout the experiments as shown in Table.3. The coded values and actual setting values of plasma parameters are presented in Table.4.

Surface morphology of the feed stock powders and micro structure on the cross section of the as-sprayed coatings was investigated using scanning electron microscope (Hitachi, S-3400N) and optical microscope**.** Porosity measurements were done using the optical microscope **(**Carl Zeiss, Canada**)** attached with clemex image analyser. Porosity was measured on cross section of the coatings at seven areas. Prior to the microstructural investigation and porosity measurement on cross section of the coatings, samples were mounted using baklite powder and then polished using SiC papers of grit sizes ranging from 120μm to 1600μm followed by mirror polishing with diamond paste of size 1μm.Phase analysis of the feed stock powders and the as-sprayed coatings was also performed using X-ray diffractrometer (Brucker, D8Advance) with Cu Kα radiation. The current and voltage were set at 40kV and 20mA and the all the readings were collected in the 2θ ranges from 10^0 to 90^0 in a step scan mode with a step of 2^0/ min.

Table.2. Main plasma parameters and their levels.

Process parameters	symbol	levels of experiment				
		- 1.682	-1	0	1	1.682
Power (Kw)	P	28	30	33	35	37
Sraying distance(mm)	S	58	75	100	125	142
Primary gas flow(l/min)	A	32	36	42	48	52

Table.3. Constant plasma parameters

Process parameters	Value
Powder feed rate (gm/min)	9
Carrier gas flow rate (l/min)	5
Secondary gas flow(l/min)	6.5
Nozzle diameter (mm)	8

3. Results and discussion.

3.1 Second-order quadratic model for porosity

The second-order response surface model, representing the porosity (Vol%) can be expressed as a function of plasma parameters such as input power (P), spraying distance (S), and primary (Ar) gas flow rate(A) .The relationship between the porosity and the plasma parameters has been developed in the form of non-reduced final equation in terms of coded factors as followed.

$$\text{Porosity (Vol \%)} = 1.89-1.41P+0.36S-0.27A+0.77P^2+0.75S^2+0.33A^2-0.49PS+0.40PA-0.23SA$$

The relationship between the porosity and plasma parameters has been developed in terms of actual factors is as follows.

$$\text{Porosity (Vol\%)} = 182.7607-8.87336P+0.093403S-1.52322A+0.122715P^2+0.001196S^2+0.00927A^2-0.00782PS+0.026583PA-0.00153SA$$

This model can be used to determine the porosity of composite coating at particular design parameters. An analysis of variance on the established quadratic model is shown in Table.5 to evaluate the influencing factors on porosity. It reveals that first order and second order of input power (P) and spraying distance(S) have a significant influence on porosity. In addition to that, the p-value of the model tends to be zero which indicates that the model is significant. Further, adequacy of regression model is verified by means of residual analysis, testing for lack of fit, and calculation of R^2. In residual analysis, normally distributed residual indicates that the model is adequate [10]. This verification is usually carried out by constructing a normal probability plot of the residuals. If the residuals lie approximately on a straight line, the normal distribution of residuals is confirmed. Fig.2 shows the normal plot of residuals for the porosity model. It reveals that the residuals approximately fall on the lines and concentrated on the center parts of the lines which are better indication of model adequacy. Calculation of F-value for the lack fit is used to examine if the model fits the data or not. Larger F-value indicates that the model is inadequate to fit the data. The F-value of lack of fit for the porosity model is 0.68.It implies the lack of fit is not significant relative to the pure error. There is a 65.8% chance that the lack of fit F-value could occur due to noise. The insignificant lack of fit demonstrates that the model is adequate. Calculated R^2 value for the model is 0.986. In design of experiments, adjusted R^2 is employed to overcome the increase in R^2 with the addition of insignificant terms to the model. Adjusted R^2 for the model is 0.972 indicates the model is satisfactory to predict the porosity.

Table.4.Experimental conditions and results

Test	coded value			actual value			Porosity (Vol %)
	P	S	A	P	S	A	
1	-1	-1	-1	30	75	36	4.51
2	1	-1	-1	35	75	36	2.25
3	-1	1	-1	30	125	36	7.23
4	1	1	-1	35	125	36	2.21
5	-1	-1	1	30	75	48	4
6	1	-1	1	35	75	48	2.76
7	-1	1	1	30	125	48	5.13
8	1	1	1	35	125	48	2.38
9	-1.682	0	0	28	100	42	6.47
10	1.682	0	0	37	100	42	1.54
11	0	-1.682	0	33	58	42	3.4
12	0	1.682	0	33	142	42	4.5
13	0	0	-1.682	33	100	32	3.32
14	0	0	1.682	33	100	52	2.21

15	0	0	0	33	100	42	1.7
16	0	0	0	33	100	42	1.82
17	0	0	0	33	100	42	2.10
18	0	0	0	33	100	42	1.7
19	0	0	0	33	100	42	1.7
20	0	0	0	33	100	42	2.41

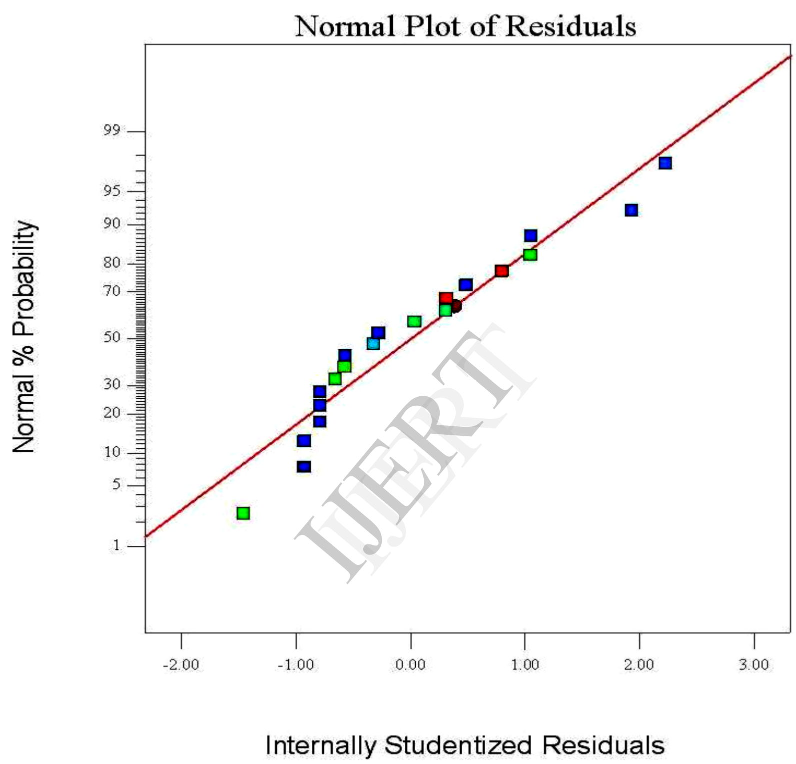

Fig.2. Normal plot of residuals for the model of porosity

Table.5.Analysis of variance for quadratic model

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	48.95	9	5.44	81.80	< 0.0001	significant
P	27.00	1	27.00	406.0	< 0.0001	
S	1.77	1	1.77	26.66	0.0004	
A	1.00	1	1.00	15.05	0.0031	
P^2	8.48	1	8.48	127.49	< 0.0001	
S^2	8.05	1	8.05	121.11	< 0.0001	
A^2	1.56	1	1.56	23.40	0.0007	
PS	1.91	1	1.91	28.74	0.0003	
PA	1.27	1	1.27	19.13	0.0014	

SA	0.42	1	0.42	6.30	0.0310	
Residual	0.66	10	0.07			
Lack of Fit	0.27	5	0.05	0.68	0.6585	not significant
Pure Error	0.40	5	0.08			
Cor Total	49.61	19				
R^2	0.986					
Adj R^2	0.972					
Pred R^2	0.942					
Adeq precision	28.47					

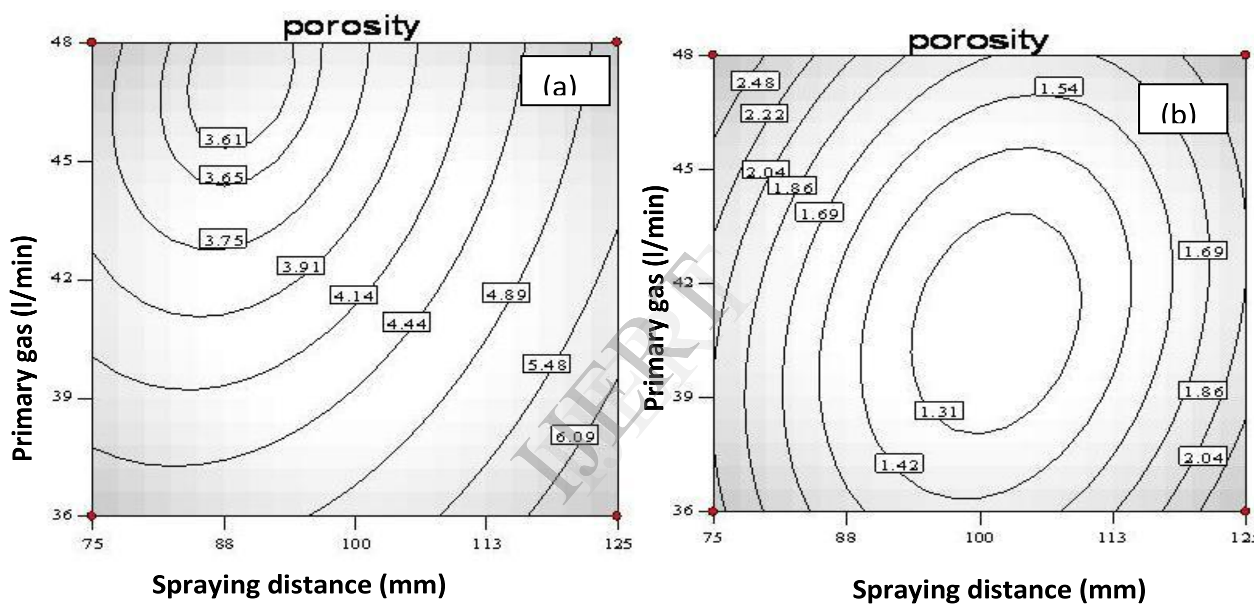

Fig.3 Response plot for porosity at different input power (a) P= 30Kw (b) P= 35Kw

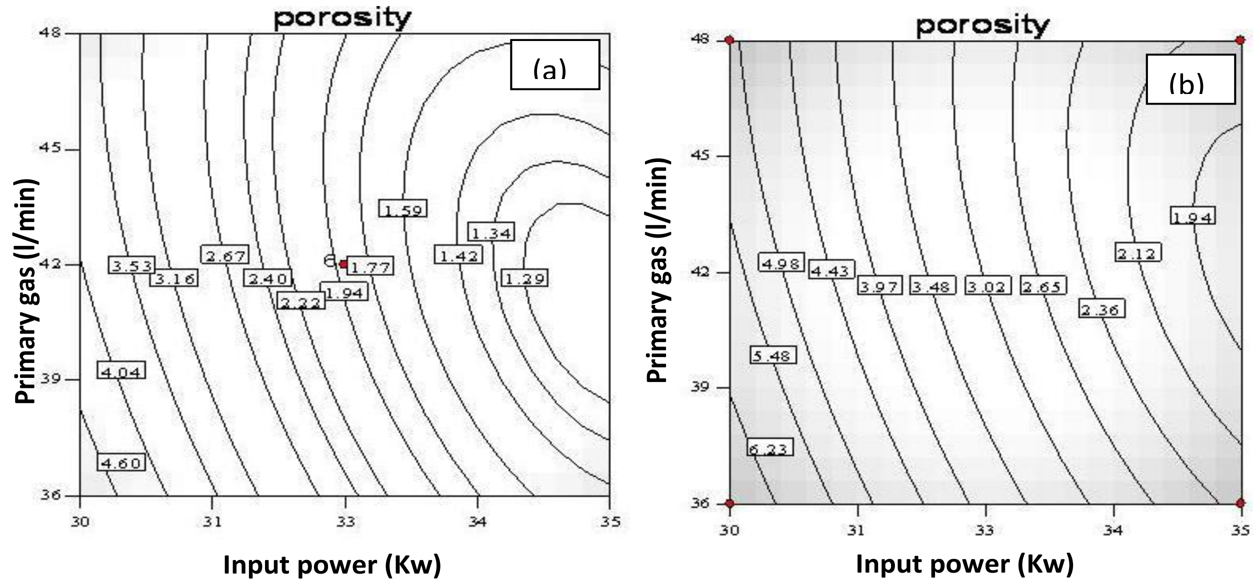

Fig.4 Response plot at different Spray distance (a) S= 100mm (b) S= 125mm

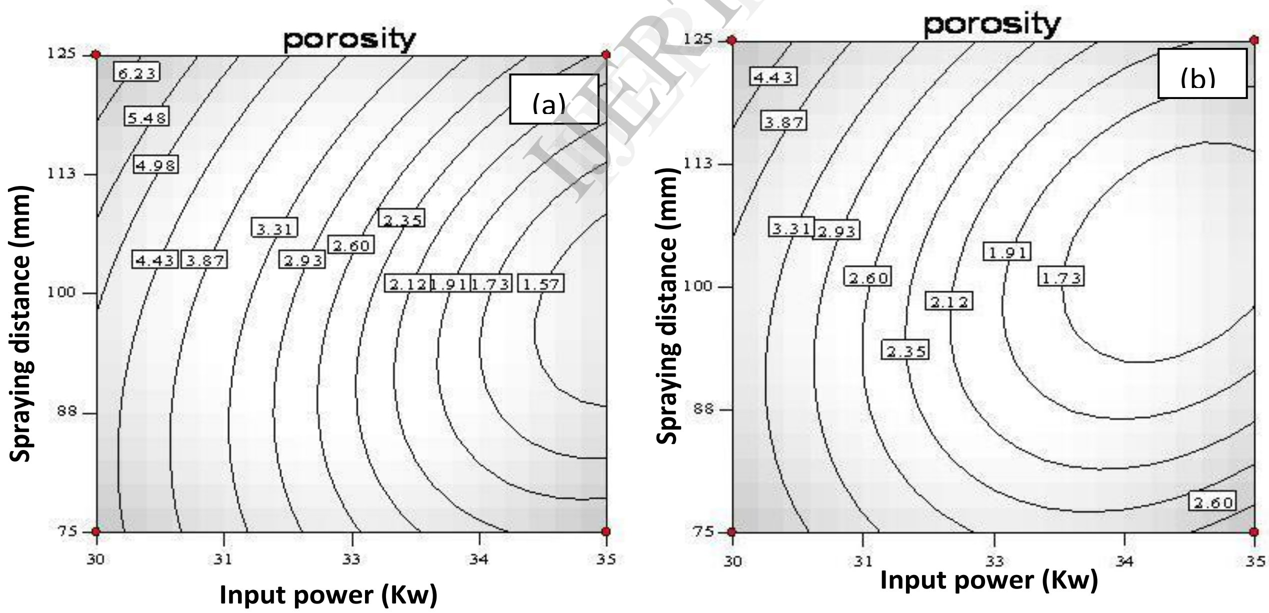

Fig.5 Response plot at different primary gas flow rate (a) A= 36l/min (b)A=48l/min

3.2 Influence of plasma parameters on porosity

The influence of the selected plasma parameters on the coating porosity can be evaluated from the quadratic second-order response function. To predict the optimal spraying parameters, the contour plots were also plotted using the developed responses of spraying processes. The contour surface plots based on the equation were generated with ISO activity line as a function of a pair of significant parameters and keeping the third significant parameter as constant for each response. These response surface contour plots help in the prediction of the coating porosity at any region of the experimental domain. Eq. (2) is plotted as response surface contours (Figs.3–5) at the two levels of input power, spraying distance and primary gas flow rate respectively. The concentric ellipses or saddle responses in the plot reflect a region of lower coating porosity at approximately the stationary points of each plot. It is clear from these figures that the porosity decreased with the increase of input power and primary gas flow rate. However, porosity increases with the increase of spray distance. Obviously, the relationship between the porosity and plasma parameters and the porosity for every stationary point can be easily estimated from these figures. The minimum porosity was measured as 1.29 percentages. 35Kw of input power, 99 mm of spray distance and 43 l/min of primary gas flow rate were identified as the resulting stationary point (optimal parameters) corresponding to the minimum porosity (optimal parameters in the coded form 1,-0.040,0.17).Three confirmation experiments were conducted to compare the experimental results with the predicted results under the optimal parameters. The mean experimental porosity (Vol %) was obtained as 1.31percentage.

3.3 Confirmation of experimental results.

Confirmation tests are used to predict and verify the accuracy of the porosity model developed for plasma sprayed composite ceramic coatings with respect to the selected plasma parameters within the experimental domain. Fig.6 shows the confirmation of experimental results for the porosity of composite coatings. It shows the experimental values of porosity and their corresponding predicted values of porosity by response model. The confirmation experiments were conducted for randomly selected plasma parameters (test numbers 2, 9, 15) and for optimal plasma parameters. It can be clearly observed from the Fig.6 that the predicted values were very close to the experimental value. In case of optimal condition, experimental porosity (Vol %) is 1.31 which is very close to the predicted value of 1.29. The error percentage observed between experimental and predicted value is 1.53 % which indicates that the model is significant to predict the coating porosity. The confirmation test clearly shows that porosity model of the plasma spraying process was significantly improved by the optimal setting of plasma parameters.

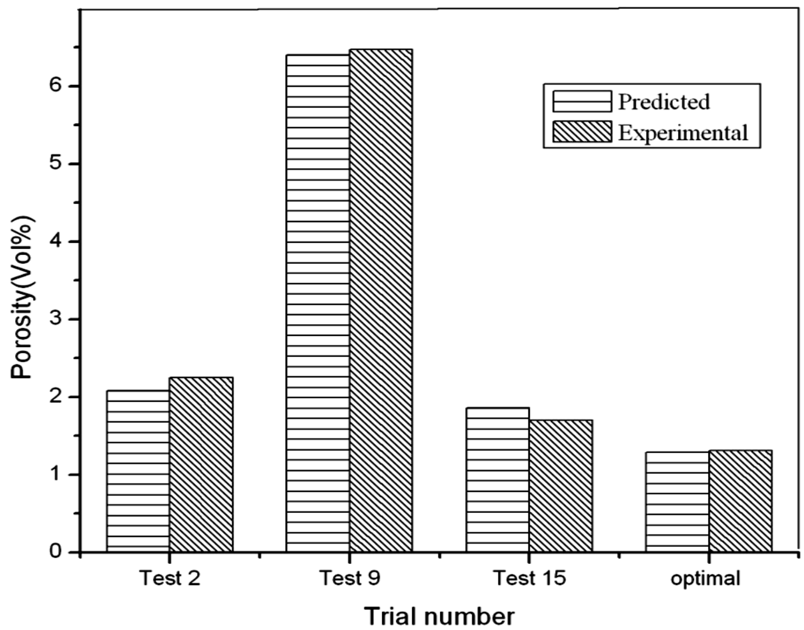

Fig.6 Confirmation experimental results

3.4 Characterization.

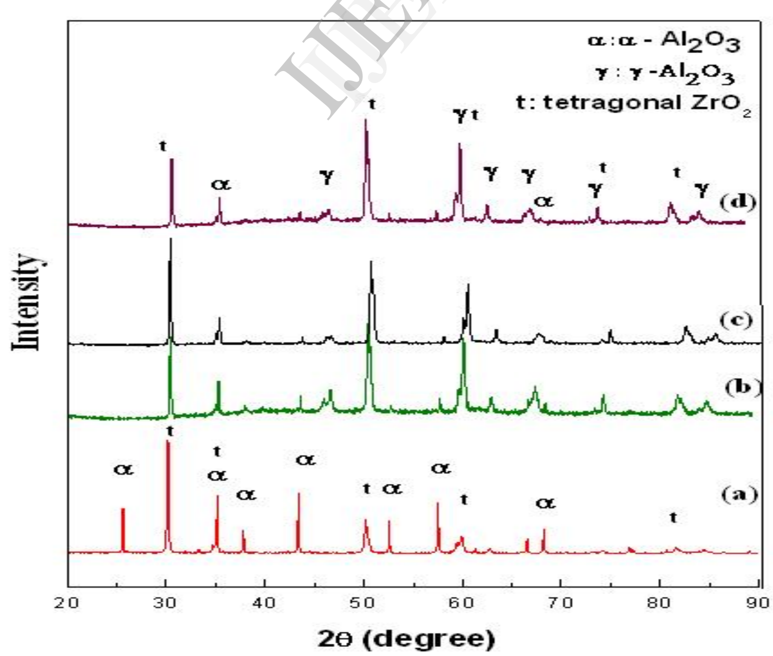

Fig.7 XRD pattern of (a) composite powder and as sprayed coatings of (b) test 9 (c) test 15 and (d) optimal condition

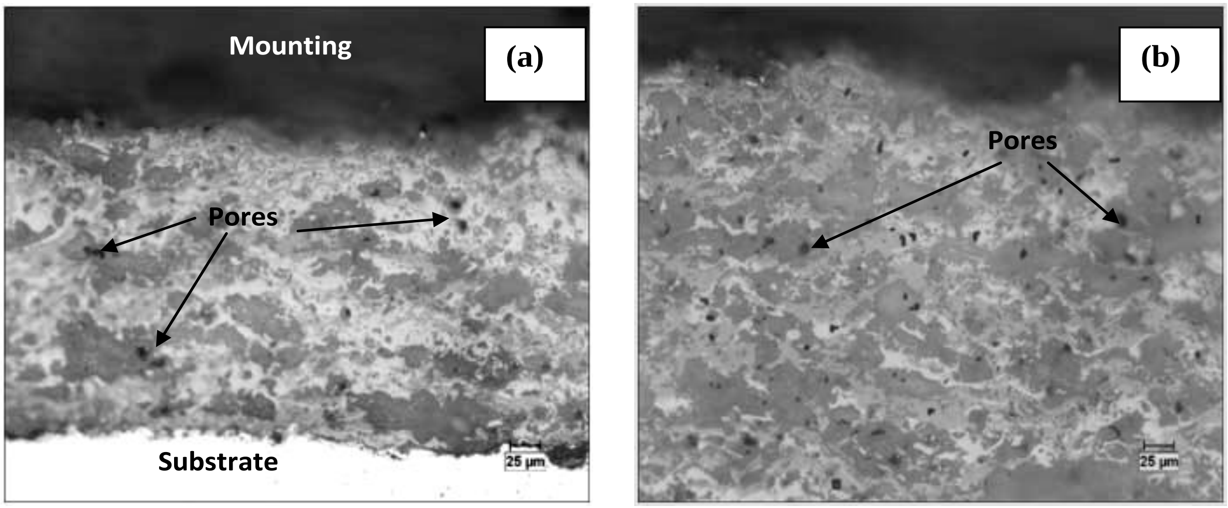

Fig.8.Optical micrograph of cross section of coating (a) Test 15 (b) Test 9

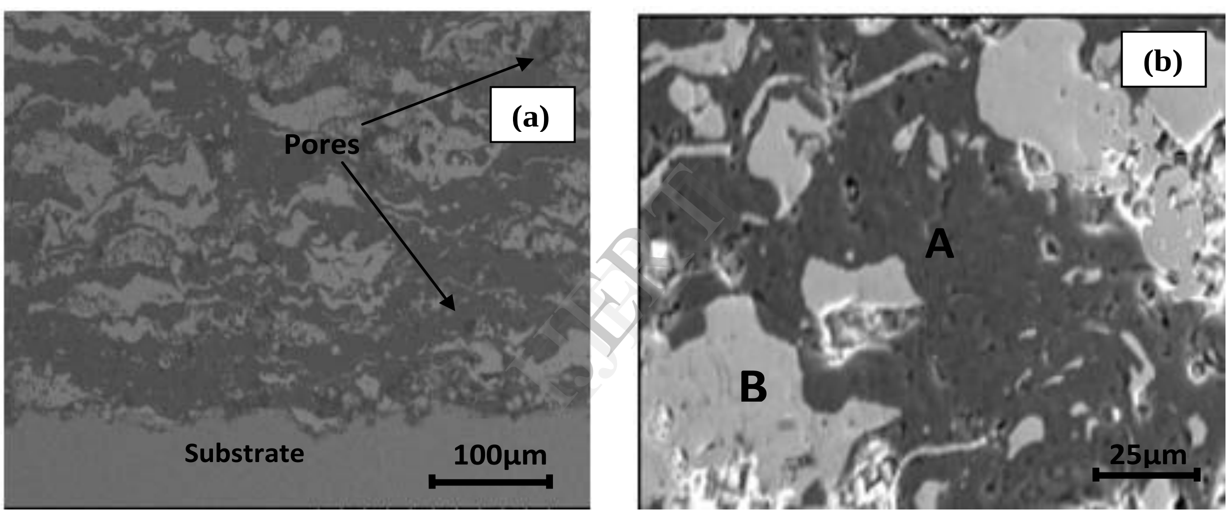

Fig.9.SEM micrograph of cross section of coating at optimal condition (a) lower magnification (b) higher magnification

Table.6 EDS Analysis on alumina/zirconia coatings

Elements	Point "A"		Point "B"	
	Wt%	Atomic%	Wt%	Atomic%
O	40.66	53.96	21.75	60.21
Al	58.16	45.77	1.56	2.56
Zr	1.19	0.28	76.69	37.23

To demonstrate the effect of plasma parameters on coating porosity, XRD results and optical micrographs and SEM micrographs of coating with selected parameters are presented in Fig.7 to Fig.9. The XRD pattern of coating developed by the condition of test 9, test 15 and optimal process conditions are shown in Fig.7. XRD pattern of the entire coated specimen mostly consist of γ-Al_2O_3 with smaller percentage of α-Al_2O_3 and tetragonal-ZrO_2, which is because of the high temperatures and rapid solidification involved in the thermal spray coating process. Al_2O_3 powders in the composite are subjected to high temperature during melting and most of α- Al_2O_3 in the starting powders gets transformed into γ-Al_2O_3 phase which is intermediate phase of alumina. After impact onto the substrate, γ-Al_2O_3 nucleated first over α-Al_2O_3 due to rapid quenching of melts (10^6 k/s) because the critical free energy for nucleation from liquid is less than that for α-Al_2O_3 [4]. Hence, γ-Al_2O_3 phase typically appears more in the plasma sprayed alumina or alumina rich coatings. The presence of α-Al_2O_3 in the as-sprayed coatings is due to the un-melted or partially melted particles. The presence of lower intensity of α-Al_2O_3 in the coating reflects the feed stock powers are subjected to the extensive meltings. Porosity of the coating mainly depends upon the melting state of the feed stock powders. If the feedstock powders attain the extensive melting, this results in lower porosity. The XRD results of coating developed by optimum condition and test 15 shows the less intensity of α-Al_2O_3 and higher intensity of γ-Al_2O_3.phase which demonstrates that the feed stock powders experienced the high temperature and consequently higher melting rate than the coating developed by the condition of test 9.Hence, the coating developed by the condition of test 9 exhibits higher porosity. Lower melting rate of the coating developed under the condition of test 9 is attributed to the low input power than the optimal conditions.

The optical and SEM micrographs of the coating cross section are shown in Fig.8 and Fig.9 It can be seen that coatings are formed by layered structure. Formation of layered structure is typical for plasma sprayed coatings. It can also be observed from the micrographs that the coating composed of alternative layer of bright and dark lamella. Both layers were analysed using EDX at point "A" and "B" (Fig 9.b) and the results are presented in the Table.6, which confirmed the presence of zirconia and alumina in the composite coatings. The bright lamella (point "B") is matching to the ZrO_2 with negligible amount of alumina and dark lamella (point "A") concerning to the mixture of major amount of alumina and small amount of zirconia. Fig.8. shows the optical micrograph of coating developed using conditions of test 15 and test 9. It clearly demonstrates that the coating developed using the condition of test 15 shows less porosity level as compared to the coating developed using test 9 .Fig.9 shows the SEM micrographs of coating developed using optimal conditions presents less porosity than the coating developed using other conditions. Lower porosity of the coating is attributed to the process conditions used for the development of coatings. Coatings developed by optimal conditions and test 15 uses higher power than the test 9.It indicates that most of the feed stock powders get melted at higher power and uniformly flattened over the substrate upon impact, which forms the coating with less porosity that appeared as dark circular shapes. However, all the coating presents homogeneously distributed and irregularly shaped pores of size ranging from 2 to 5μm. The XRD results and micrograph of the coatings clearly demonstrate that the porosity of the coatings depends on the process conditions used to develop the coating. Both XRD and micrographs of the coating also shows that the developed model is significant, as these results are closely related with the predicted results.

4. Conclusions.

The porosity in the plasma spray process has been measured for Al_2O_3-40wt%8YSZ composite coating on Ti-6Al-4V alloy under different process conditions and their optimization and responses have been obtained through response surface model. Based on the experimental and analytical results, the following conclusions were arrived.

- The influence of plasma parameters on the coating porosity has been evaluated with the help of response surface methodology and optimal plasma parameters has been identified for minimum coating porosity
- The input power is the most influencing parameter for coating porosity followed by the spraying distance and primary gas flow rate.
- Higher input power, shorter spraying distance and higher primary gas flow rates are the preferential plasma parameters to achieve dense coating on Ti-6Al-4V alloy.
- A second-order response surface model for coating porosity has been developed from the observed data. The predicted and measured values are comparatively close to each other. This demonstrates that the developed model is very useful to predict the coating porosity at 95% confidence intervals within the experimental domain.
- The developed response surface contour plots for coating porosity are effective in the prediction of relationship among the coating porosity and plasma parameters at any area of the experimental domain.

- The confirmation test results revealed that the determined optimal combination of plasma parameters satisfy the actual needs of plasma spray process for developing dense coating on Ti-6Al-4V alloy.
- XRD results and micrographs of the coatings developed for selected parameters are also consistent with the predicted and experimental results, which indicate the developed model is significant.

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$$\text{Porosity}=182.7607-8.87336P+0.093403S-1.52322A+0.122715P2+0.001196S2+0.00927A2 - 0.00782PS+0.026583PA-0.00153SA$$