Effect of experimental parameters on the micro hardness of plasma sprayed alumina coatings on AZ31B magnesium alloy

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Abstract

Surface treatment of engineering materials has recently become important for serviceable engineering components. Many techniques such as thermal and thermo chemical surface treatments have been used to develop surface characteristics of materials. Hardness is the most important property, which influences considerably service life characteristics of coatings. In this investigation, alumina coatings were deposited by atmospheric plasma spray technique under different levels of power, stand-off distances and powder feed rates. Empirical relationship was developed to predict the micro hardness of alumina coatings by incorporating the plasma spray process parameters. The input power and the stand-off distance appeared to be the most significant two parameters affecting the hardness of the coating among the three investigated process parameters. Further, correlating the spray parameters with coating properties enables the identification of characteristics regime to achieve desired quality of coatings.

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Keywords: Hardness; Plasma spraying; Alumina coating; Mg alloy

1. Introduction

Magnesium alloys are considered as good candidates for many structural components of automobile, aerospace and military industries to satisfy the demand for weight reduction, improving fuel efficiency and reducing greenhouse gas emissions [1]. In addition, magnesium alloys (AZ31) are attractive increasingly for their combination of outstanding properties such as low density, high specific strength and stiffness and high mechanical damping capability [2,3]. Magnesium has good castability, machinability and easy recycling ability. Furthermore, it can also be used in the communication and electronics industry for good electromagnetic shielding characteristics [4]. More recently the usage of magnesium alloy has increased gradually as magnesium alloy has the potential to replace aluminum and some plastics in a variety of applications in the automotive and aerospace industries. However, the application of magnesium alloy has been restricted because of poor surface property. In order to further expand the application of magnesium alloys, surface modification processes such as chemical conversion coatings [5], plasma electrolytic oxidation (PEO) [6,7], physical vapor deposition (PVD) [8] and laser surface treatment [9,10] have been applied to improve surface properties of Mg alloy.

Among these techniques, atmospheric plasma spraying is one of the most commonly used thermal spraying processes because of its flexibility, high deposition rates and multifunction. Plasma spraying is employed to deposit coatings of almost all materials including metal alloys, ceramics and cermet with a congruent melting point onto the substrate. It is known that the properties of plasma spraying coating is related to many parameters of plasma spraying process such as the powder feed rate, the spraying power and the spraying distance. These parameters directly influence the heat and mass transfer between particles and plasma jet, and then affect the degree of melting of particles, the temperature and in-flight velocity of droplets before they impact on the substrate [11].

Ceramic coatings are commonly employed for thermal and environmental protection of metal components operating at severe working conditions [12]. Their application is able to improve the resistance and the durability of the underlying components, thus reducing their placement of worn out parts.
and the relative idle times. Among them Al₂O₃ coatings are good candidates for anti-wear and anti-corrosion applications, due to their high hardness, chemical inertness and high melting point, as well as to their high resistance to abrasion and erosion [13].

The microstructure and the mechanical properties of the coating are influenced by the spraying parameters, such as the spraying power, stand-off distance, powder feed rate, etc. These parameters affect the thermal energy and kinetic energy of particles. If particles are subjected to an excess of thermal energy, they can be vaporized in the plasma jet rather than arriving at the substrate in the fully molten condition [14]. However, if the particles receive too little thermal energy, they arrive at the substrate in an unmelted condition. In the last decades, the importance of the link between the spraying parameters and coating quality has been appreciated. As for all materials, the hardness of a coating is a measure of the resistance to plastic deformation. It is widely recognized that the hardness increases with the increasing coating density, i.e. decreasing number of pores and microcracks [15].

Venkataraman et al. [16] studied the influence of porosity, pore size, spatial and topological distribution of pores on microhardness of plasma sprayed ceramic coatings and reported that among several microstructural features, porosity seems to have a stronger influence on mechanical property such as micro hardness. Prystay et al. [17] studied the correlation between the particle temperature and velocity and the structure of plasma sprayed zirconia coatings, to determine which parameter most strongly influences the coating structure. They inferred that the temperature of the sprayed particles has a larger effect on the coating properties than the velocity. Ruiz-Luna et al. [18] investigated the effect of HVOF processing parameters on the properties of NiCoCrAlY coatings by design of experiments. The results of their investigation showed that the response surface, the empirical relationships among the variables, and the response parameters allowed the selection of optimum deposition parameters and the improvement of coating properties. Saravanan et al. [19] reported that the coating quality is directly related to the corresponding coating microstructure, which is significantly influenced by the spray parameters employed. A study by Yong Yang et al. [20] opined that the coating prepared by applying spraying power of 30 kW had the maximum microhardness, which was attributed to the maximum Al₂O₃ content present in the coating and the most uniform microstructure of the coating. A factorial designed experiment was used by Jandin et al. [21] to analyze the correlation between operating conditions and the microstructure and mechanical properties of twin wire arc sprayed steel coatings. Results show that direct relationships do exist between spray conditions, oxide content in the coating, and microhardness.

Conversely, role of spraying parameters such as power, stand-off distance, and powder feed rate on the micro hardness of plasma sprayed ceramic coatings on magnesium alloy has not yet been reported in the literature. Hence, the present investigation was carried out to develop an empirical relationship to predict the hardness of plasma sprayed alumina coatings. The effect of input power, stand-off distance and powder feed rate on micro hardness of alumina coating is reported in this paper.

2. Methodology

2.1. Identifying the important process parameters

An initial step in the design of experiments is to select independently controllable process parameters. It has been widely recognized in the thermal spray community that there are many hundreds of parameters, which can potentially influence the properties of the coatings. For economic (time requirements) and theoretical reasons (interdependence of parameters), it is not possible to control all possible parameter variations. According to the literatures [16–21] and our laboratory experiences [22], the predominant factors which are having more influence on coating characteristic in plasma spraying process were identified. They are as follows:

(i) input power (kW);
(ii) stand-off distance (mm);
(iii) powder feed rate (mm).

These are the primary operational parameters contributing to the melting and flattening of the powder particles, subsequently, influencing the coating characteristics of plasma sprayed coatings.

2.2. Finding the working limits of the parameters

A large number of spraying trials were conducted on grit-blasted 16-mm-thick AZ31B alloy substrate coupons to determine the feasible working range of the above factors by varying one of the APS spray parameters and keeping the rest of them at constant value. The chemical compositions of the AZ31B alloy used in this study are as follows (in wt.%): Al 3.0, Zn 0.1, Mn 0.2 and Mg balance. Plasma spraying of the alumina powder was carried out using an APS system 40 kW IGBT-based Plasmatron (Make: Ion Arc Technologies, India; Model: APSS-II). Before spraying, the substrate was grit blasted with corundum at a pressure of 4.2 bars and cleaned with ethanol to remove any remaining dust or grease from the surface. Deposition was performed using argon and nitrogen as plasma-forming gases. The necessary number of spraying passes was carried out to obtain a ceramic layer thickness of 240 μm. The working limits of the spraying parameters were discussed elaborately in our previously published paper [22].

2.3. Metallographic preparation

Metallographic cross sections of the coatings were prepared for the porosity and microhardness measurements. The samples
were first carefully cut to the specific dimensions (10 × 10 × 2 mm³). They were then mounted with low viscosity epoxy resin under vacuum environment. The mounted samples were successively ground with 600, 800, 1000 and 1500 grit SiC papers and eventually polished using diamond slurries of 10–8, 8–5, 5–2, 2–0.5, 0.5–0 mm during 5, 5, 7, 10 and 10 min, respectively. Because of pullouts in brittle materials, it is difficult to establish and evaluate true porosity in a metallographically prepared spray coating. As metallographic grinding and polishing, if not carried out correctly, can introduce artifacts which are not part of the coating structure. Ceramic coatings are brittle and particles break out of the surface during grinding. If not polished thoroughly, these break out leave an incorrect impression of a high porosity. Similar procedures were followed by the other investigators [23,24].

2.4. Developing the design matrix

With a view to study the effects of the considered process parameters on the micro hardness, 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 the interaction effects of the parameters on the response. Table 1 presents the process factors with their corresponding levels and Table 2 presents the design matrix according to coded levels.

Table 1
Important APS process parameters and their levels.

<table>
<thead>
<tr>
<th>Factors</th>
<th>Notations</th>
<th>Units</th>
<th>Levels</th>
</tr>
</thead>
<tbody>
<tr>
<td>Power</td>
<td>P</td>
<td>kW</td>
<td>−1.682 −1 0 +1.682</td>
</tr>
<tr>
<td>Stand-off distance</td>
<td>S</td>
<td>cm</td>
<td>10 10.6 11.5 12.4 13</td>
</tr>
<tr>
<td>Powder feed rate</td>
<td>F</td>
<td>gpm</td>
<td>15 20 25 30 35</td>
</tr>
</tbody>
</table>

2.5. Recording responses

In this present investigation, the plasma spraying was carried out according to the design of experiments, at each condition, three specimens were coated as prescribed by the design matrix. The experiments were conducted in a random order to prevent systematic errors infiltrating the system.

2.5.1. Porosity analysis

As per the procedure laid out in ASTM B 276 standard [25], the porosity measurement was carried out on the well-polished cross-sectional area of the coating, using an optical microscope (Make: Meiji, Japan; Model: MIL-7100) equipped with an image analyzing software (Metal Vision Version 6). In this study, the images captured under 1000× magnification by optical microscopy were chosen for porosity analysis as desired features like open pores and network of cracks were properly revealed. Initially, a 400 × 400 μm square area was selected on the polished cross-section of the coating and the image was analyzed. The same procedure was repeated at five random locations to find out the average percentage volume of porosity. It was explained in Fig. 1.

2.5.2. Microhardness measurements

Microhardness measurements were operated by indenting on the metallographic cross sections under 300 g load for 15 s using a Vickers microhardness tester (Make: Shimadzu, Japan; Model: HMV-2T). For each coating sample, the measurement series comprised 20 random indentations. Distance between indentations was kept three times longer than the indentation diagonal to prevent the effects of the stress field of nearby indentations.

The Scanning Electron Microscope (Make: Jeol, Japan; Model: 6410-LV) was used to analyze the size and morphology of the parent materials. The powder is fused and then crushed,
which gives its characteristic angular shape as shown in Fig. 2. SEM micrographs of the plasma-sprayed alumina coating are shown in Fig. 3a, b. A number of microcracks were observed on the surface of the coating. Fig. 4a, b shows the optical microstructure and SEM images of the alumina coating. From these micro-graphs, it can be seen that the coating microstructure consists of completely melted splat structures, unmelted particulate regions, pores between the splats and cracks within the splats. EDS analysis of the interface area (Fig. 5) detected the presence of Al and O in the coating.

3. Predictive statistical model for microhardness

In this study, a response surface model-building technique was utilized to predict the microhardness in terms of the input power, the stand-off distance and the powder feed rate. Details of the model building technique are discussed below.
3.1. Response surface methodology (RSM)

RSM is an experimental strategy that explores the space of the process independent variables, and an empirical statistical modeling, to develop an appropriate relationship between the responses (output) and the process variables or factors (input). In the present investigation, to correlate the process parameters and the micro hardness, an empirical relationship was developed to predict the responses based on experimentally measured values. The response is a function of power ($P$), stand-off distance ($S$), powder feed rate ($F$) and hence it can be expressed as

$$CR = f(P, S, F)$$

The empirical relationship chosen includes the effects of the main and interaction effect of all factors. The construction of empirical relationship and the procedure to calculate the values of the regression coefficients can be referred elsewhere [26]. In this work, the regression coefficients were calculated with the help of Design Expert V 8.1 statistical software. After determining the coefficients (at a 95% confidence level), the final empirical relationship was developed using these coefficients. The final empirical relationship to estimate the response is given below:

$$\text{Microhardness (HV)} = 1075.033821 - 122.0188(P) - 61.4927(S) - 30.8938(F) + 8.334(PS) - 49.347(PF) - 51.869(SF) - 91.00244853(P^2) - 103.1103121(S^2) - 102.743(F^2)$$

3.2. Checking the adequacy of the model

In this investigation, analysis of variance (ANOVA) is used to check the adequacy of the developed empirical relationship. ANOVA test results are presented in Table 3. The adequacy of the model was tested using the ANOVA technique. In this study, the model $F$ value and the associated probability values are checked to confirm the significance of the empirical relationships. Further, using the $F$-values, the predominant factors which have the major and minor effects on the responses could be assessed. From the $F$ value assessment, it was found that the predominant factors which have direct influence on the responses as per hierarchy are power, stand-off distance and powder feed rate. The determination coefficient ($R^2$) indicates the goodness of fit for the model. In all the cases, the value of the determination coefficient ($R^2 > 0.99$) indicates that less than 1% of the total variations are not explained by the empirical relationships. The value of the adjusted determination coefficient is also high, which indicates the high significance of the empirical relationships. The predicted $R^2$ values also show good agreement with the adjusted $R^2$ values. Adequate precision compares the range of the predicted values at the design points with the average prediction error. At the same time, a relatively low value of the coefficient of variation indicates the improved precision and the reliability of the conducted experiments. The value of probability $> F$ in Table 3 for the empirical relationship are less than 0.05, which indicates that the empirical relationships are significant. Lack of fit was not significant for the developed empirical relationship as desired [27]. The normal probability plots for the response are shown in Fig. 6. From the figure, it could be inferred that the residuals fall on the straight line.
line, which shows that the errors were distributed normally. Collectively, these results indicate the excellent capability of the regression model. Further, correlation graphs were drawn relating experimental values and predicted values as shown in Fig. 7 and it is found that the developed empirical relationships can be effectively used for prediction purpose.

4. Results and discussion

4.1. Effect of input power on microhardness

In the plasma spray process, the input power is controlled by variation of arc current. It indicates higher arc current leads to higher input power and conversely. The variation of response with input power levels are displayed in Fig. 8a. Lower power levels offered improper melting quality coatings in terms of higher porosity. With low spraying powers, the powder particles are poorly melted. When they impact on the substrate or the already formed coating, they are not able to spread out completely to form splats and therefore, could not conform to the surface [28]. In such a case, the interlamellar pores and cracks will be formed due to the solidification of the splats. Moreover, when the spraying power is relatively low, numerous unmelted and partially melted particles existed in the coating. As the arc current increases, the total and the net available energies in the plasma increase. This condition leads to a better in-flight particle molten state and higher velocities. It is well known that during plasma spraying, an electric arc is initiated between the two electrodes using a high frequency discharge and then sustained using power. The arc ionizes the gas, creating high-pressure gas plasma containing higher heat content. The resulting increase in gas temperature, which may exceed 30,000 °C, in turn increases the volume of the gas. The coatings

<table>
<thead>
<tr>
<th>Source</th>
<th>Sum of squares</th>
<th>df</th>
<th>Mean square</th>
<th>F value</th>
<th>p-Value</th>
<th>Prob &gt; F</th>
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<td>580.7121</td>
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<tr>
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<td>10</td>
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<tr>
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<td>208.287</td>
<td>3.013734</td>
<td>0.1249</td>
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</tr>
<tr>
<td>Pure error</td>
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<td>5</td>
<td>63.06667</td>
<td></td>
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<tr>
<td>Cor total</td>
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<td>19</td>
<td></td>
<td></td>
<td></td>
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<tr>
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<td>R-squared</td>
<td>0.995562</td>
<td></td>
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<tr>
<td>Mean</td>
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<td>Adj R-squared</td>
<td>0.991567</td>
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<tr>
<td>PRESS</td>
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<td>Adeq precision</td>
<td>46.90273</td>
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</table>

df: degrees of freedom; CV: coefficient of variation; F: Fisher ratio; p: probability.
are formed by spreading of melted droplets. Complete melting of the powders and higher velocity will yield lesser porosity coatings and make the melted droplets spread adequately. The optical micrograph of the coated specimens Fig. 9a reveals the at lower power levels, the alumina coated specimen shows extensive cracking of the coating due to the applied indentation load. Increasing the power level which increases the enthalpy in the plasma flame is likely to melt the particles, which in turn increases particle-melting ratio subsequently enhancing good compaction of the coating obtained during the coating buildup. Further, effective flattening and solidification of the particles over the deposited layers will lead to reduction in porosity [29] and increasing microhardness values. Porosity decreases under high power levels because the particles are more likely to melt at high plasma energy levels, thereby enhancing flow and compaction of the coating during its build up. If the velocity of the particles is increased and/or the viscosity is decreased, then particle spreading tends to increase. The presence of non-molten particles will also increase the roughness of the coating and will lower the values of hardness because of low particle cohesion. This also should increase the porosity of the coatings. An increase in porosity will lower the coating stiffness, producing a decrease in the values of hardness. Under very high power levels, gas entrapment upon impact occurs because of the high pressure in the gas layer just prior to impact. During the rapid spreading and quenching of splats, gas escape can be suppressed resulting in escalating gas pressure in the splat center, which can create the thin cap of a gas bubble, leaving behind a residual hole causing an increase in porosity level and the reduction of hardness values [30]. In the case of the coating produced under high power levels (Fig. 9b), the cracks are very few. During the indentation process, a complex elastic-plastic field is formed beneath the indentation. When porosity or an equivalent defect is present under the indentation, it creates a multiaxial stress state and causes a local strain concentration in its vicinity. Porosity tends to reduce the effective area supporting the load and is detrimental to the strength of the coating. Pores present in the coating accommodate deformation without resistance and thus decrease the hardness of the coating. Splat separation together with multiple cracks were consistently found throughout the coating deposited under the lower power levels (Fig. 9a). This can be seen in the spreading of the indent as the splats separate. Splat separation and multiple cracks also suggest that weak bonds are formed at the splat interfaces. These weak bonds allow the splats to separate easily under the shear stresses imposed by the indenter [31]. The low level of splat interaction is most likely caused by low particle velocities and lower particle temperatures. Splat separation is still visible in the coating sprayed under higher power level (Fig. 9b), but only pore separated splats are affected. Both the experimental and the predicted results agree in describing these effects.

4.2. Effect of stand-off distance on microhardness

The effects of stand-off distance on microhardness of the coatings are displayed in Fig. 8b. According to the figure, it is seen that the hardness has an inversely proportional relationship with the stand-off distance. The stand-off distance mainly controls the cohesion between splats because the temperature and velocity of particles in the plasma flame significantly change with stand-off distance. Therefore, better spreading and cohesion would be achieved with shorter spraying distances. At smaller stand-off distance, possibilities of splashing of molten particles and quench cracks end up with increased level of porosity [32]. Stand-off distance is of substantial importance because adequate distance must be provided for heating and accelerating the powder, but too great a distance will allow the powder to cool and lose velocity, because the gas stream is rapidly expanding, cooling, and slowing will end up with
molten droplets land on substrate without enough kinetic energy to form splats. These droplets can stay on the substrate by themselves [33] and act as a stress concentrator which resulted in crack propagation in multiple directions. Lowering spray distance firstly increases deposition rate but problems appear by strongly increasing heat load. Coatings are dense but large cracks form in the coating due to internal stresses that arise during the deposition process. The optical micrograph of the coating exposure is shown in Fig. 9c, where very few microcracks of the coating can be observed.

On the other hand, by increasing the spray distance coating compact is reduced and hardness significantly reduces due to a reduced particle temperature and a lower particle impact. Also by lowering the average impact temperatures of the droplets with the substrate surface, the lower kinetic energy and particle temperatures reduce the plastic deformation of the particles leading to a substantial decrease microhardness value [34]. As explained, increase in stand-off distance causes prevention of splashing of material with possible fragmentation and quenching cracks. This condition reduces the porosity and has positive effect on hardness. With longer stand-off distance, the enthalpy of the molten ceramic particles is largely lost, and the particles are decelerated in a relatively longer flight path because of the interaction with the surrounding air. Under such conditions, the particles striking on the substrate will not be adequately flattened to overlap the layers, resulting in higher porosity and reduces the hardness values. It is clear from Fig. 9d, cracks were observed on the surface of the material.

4.3. Effect of powder feed rate on microhardness

Fig. 8c indicates the effect of powder feed rate on the response of the coatings. From the figure, it can be inferred that the hardness decreased with the increase in powder feed rate. In plasma spray processing, the powder feed rate refers to a per-
percentage or number of particles which share the kinematic and thermal energies in the flame. The numbers of particles in the flame are influential on particle velocity and their temperature. Too low a powder feed rate will result in vaporization, and over melting of the particles resulting in cracks (decreases hardness) [35], splashing, and high porosity levels, whereas too high a feed rate will end up in poor melting of the powder particles resulting in a decrease of the splot flattening ratio and an increase in the porosity [36]. Inspection of the coating morphology produced under lower powder feed rate reveals the presence of micro cracks (Fig. 9e). This suggests that a good contact between the oxide scales of different splats might have been caused by particle velocities and temperatures.

Increase in powder feed rate will lead to better vaporization and improve the deposition efficiency. It also prevents formation of quench cracks and splashing of molten particles. Hence, this phenomenon increases the hardness values. On the other hand, when the powder feed rate goes beyond 25 gpm, increase in the number of powder particles may lead to deviation of them from injection axis. Hence, it leads to a change in the powder inlet condition and increases particle collisions that change the trajectories of the colliding particles. Large or small number of particles will also float on top of the flame or penetrate it to reach the lower part. These particles, not attaining to sufficient velocity and temperature, however, either reach the coating or stick to it. Under such condition, the porosity of surface increases and leads to low hardness [37]. But by further increase in powder feed rate, the kinetic and thermal energies in the flame increase and lead to increase in temperature and velocity of particles. It also prevents formation of quench cracks and splashing of molten particles [38]. Hence, the porosity decreases at high powder feed rate and hardness increases, subsequently. When the powder feed rate was increased to 35, the alumina coated specimen exhibited a higher amount of cracks observed than those in the lower powder feed rate (Fig. 9f).

4.4. Relationship between porosity and microhardness of alumina coatings

The coating porosity and the microhardness obtained from the experimental results are related as shown in Fig. 10. The experimental data points are fitted by a straight line. The straight line is governed by the following regression equation:

\[
\text{Microhardness (HV)} = 1.305.4 - 47.047 \times \text{(porosity in vol. %)}
\]

(3)

The slope of the estimated regression equation (−47.047) is negative, implying that as porosity decreases, microhardness increases. The coefficient of determination is \( R^2 = 94.6\% \). It can be interpreted as the percentage of the total sum of squares that can be explained by using the estimated regression equation. The coefficient of determination \( R^2 \) is a measure of the goodness of fit of the estimated regression equation [39].

The fitted regression line (Eqn. 3) may be used for two purposes:

(a) To estimate the mean value of microhardness for the given value of coating porosity.

(b) Predicting an individual value of microhardness for a given value of coating porosity level.

The confidence interval and prediction interval show the precision of the regression results. Narrower intervals provide a higher degree of precision (Fig. 10). Confidence interval (CI) is an interval estimate of the mean value of \( y \) for a given value of \( x \). Prediction interval (PI) is an interval estimate of an individual value of \( y \) for a given value of \( x \). The estimated regression equation provides a point estimate of the mean value of microhardness for a given value of porosity. The difference between CI and PI reflects the fact that it is possible to estimate the mean value of microhardness more precisely than an individual value of microhardness. The greater width of the PI is reflecting the added variability introduced by predicting a value of the random variable as opposed to estimating a mean value. From Fig. 10, it is also inferred that the closer the value to “X” (15.21vol %) the narrower will be the interval.

5. Conclusions

The following important conclusions are obtained from this investigation

- Empirical relationship was established using RSM to predict the micro hardness of plasma sprayed alumina coatings, incorporating APS spray operational parameters. The developed relationship can be effectively used to predict the micro hardness of alumina coatings on AZ31B magnesium alloy at 95% confidence level.
- The input power was found to have greater influence on the micro hardness of plasma sprayed alumina coatings followed by process parameters such as stand-off distance and powder feed rate.
- A regression equation has been developed incorporating coating porosity and microhardness of the coating. This equation can be effectively used to predict microhardness of alumina coating, if coating porosity is known.
References