Optimizing Coating Thickness of Electrophoretic Deposition Overlay on Plasma Sprayed YSZ Coating Using Taguchi Method

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Abstract. In this study, nano sized yttria stabilized zirconia (YSZ) suspension in organic solution was deposited by electrophoretic deposition (EPD) method as a protective layer on substrate that was previously plasma sprayed thermal barrier coating (TBCs). In order to improve the performance of TBC from degradation by melt ingression of fuel impurities. Design of experiments (DOE) by Taguchi method was used to optimize the controlled variables of EPD process. A crack free YSZ overlay coating was carried out at different variables; applied voltage (20, 40, 60) V, deposition time (3, 5, 7) min and suspension concentration (5, 10, 15) g/l using DC current. Morphological appearance and cross section of the investigated coating specimen were done using optical and field emission scanning electron microscope. Optimizing process and analysis of variances (ANOVA) were performed by “Minitab 18” software. The results indicate that best condition of coating thickness can be obtained at 40V, 5min and 10g/l when applying signal-to-noise ratio “Larger is better”.

Keywords. Thermal barrier coatings (TBCs), Electrophoretic deposition (EPD) overlay, Design of experiments (DOE), Analysis of variances (ANOVA).

1. Introduction

The continuous progress demand for the thermal barrier coatings has prompted the researchers to investigate new materials technology for gas turbine engine [1]. Plasma spraying method was widely used to produce thermal barrier coatings [2, 3], due to the plasma spray mechanism. It has the ability to obtain a coating with certain specifications that provide desired porosity ratio, which increases the thermal insulation of the hot metal parts of the turbine engine [4, 5]. The main motivation to enhance TBCs is to develop appropriate coating systems to provide surface protection at high temperatures for long-term efficiency and durability [6]. Laser sealing of the porous YSZ topcoat was also studied [7]. However, during laser sealing of YSZ topcoat, a rapid quenching encourages the formation of the metastable tetragonal-prime phase (t'-ZrO2) [8,9]. Phase t' helps to prevent the phase transformation to equilibrium phases of monoclinic (m) during service. It is important to know that the transformation to m phase is accompanied by detrimental volume change (3 to 5%) that could lead to spallation and delamination of the ceramic topcoat [10]. A dense barrier
overlay coating by electrophoretic deposition technique, which has been widely developed as a method for depositing wide range of materials for different applications [11]. EPD is widely employed as a cost-effective and easy technique for a diversity of coating uses. Electrophoretic deposition is a material processing method where charged particles are moved during the solvent in a stable colloidal suspension due to electric field and deposited on an oppositely charged substrate [12]. Depending on surface charge of the suspended particles, two type of EPD can be identified. If the particles have positively charged, they will move towards the electrode with the opposite sign, the cathode, and this process is called cathodic EPD. In the contrary, if the dispersed particles have negatively charged, they will be attracted by the positively charged electrode, the anode, and this process is named anodic EPD [13]. It must follow by sintering process at high temperature to increase the density and adhesion of green structure [14].

Two groups of parameters that effect on the determine and characteristics of EPD process are (i) parameters related to the suspension and (ii) parameters related to the deposition process [15]. If the suspension variables were constant, so, process variable is mostly affected coating quality.

In this study, a dense YSZ overlay is deposited on the YSZ plasma sprayed coating by EPD under different process parameters (voltage, time and concentration suspension). Design of experiments by Taguchi method was used to select the optimum conditions that give best results of the output response (coating thickness) depending on signal-to-noise ratio “larger is better”. Verification of the best conditions were also performed.

2. Experimental work:

In order to optimize the performance enhancing of TBCs by applying overlay EPD coating under various parameters, Air plasma sprayed YSZ and CoNiCrAlY coatings were prepared in this study. TBCs samples are composed of bond layer and ceramic layer in which (Amdry 365-2) powders with average size (–75 +38 μm) were thermally sprayed as bond layer on clean and grit blasted Ni- based Substrate (ϕ= 20mm, h= 4.5 mm). Surface roughness (Ra) after grit blasting by alumina was in the range (5.5-6.5) μm. Using similar APS process, 8wt% yttria stabilized zirconia powder (Sulzer metco 204NS) with average particle size (–125 +11 μm) were sprayed on the bond layer as ceramic layer. Table 1 presented the processing parameters used in this investigation. After that the resultant TBCs samples were electrophoretic deposited using 8 wt. % (5 mole) yttria stabilized zirconia (YSZ) nano powders. The primary size of the fine powder in the range (15-50 nm) and the morphology was shown in the figure 1. A stable colloidal suspension of nano YSZ powder in an organic solvent (ethanol) was prepared with the use of 1.0% of the acetic acid. Then, the purity of the solvent ethanol added with 2.5% of the distilled water. Iodine (I$_2$) at 0.5 g/l a concentration was utilized as an additive. To obtain a stable colloidal suspension of individually dispersed YSZ particles, the suspension was stirred mechanically for (24) hr and after that subjected to ultrasonication for (40) min to break down the particle conglomerate. In order to apply regular deposition, the working electrode must be electrically conducting surface. Therefore, a thin layer of Pt (~ 50 nm) utilized (pt) D.C. sputter coating system was employed on APS YSZ coatings. The YSZ coated with Pt was used as a cathode and (stainless steel 316L) disk of the same surface area and shape was used as anode. The distance between two electrodes was kept parallel at (1 cm) in the cell, which was placed in the YSZ suspension under gentle continuous stirring to ensure the uniformity of the suspension and to reduce the sedimentation of YSZ particles during deposition. Zeta potentials of the YSZ suspensions in the solvent were measured using device (Model: Vasco, Company: Corcoran, Technology Country: France), for the purpose of ensuring suspension stability. Value pH was observed by using a pH meter (pH =4). Positively charged YSZ particles were then deposited
electrophoretically onto the cathode, using design of experiment by taguchi method. In this study, thickness of the deposited particles controlled by three factors: (1) concentration of the suspension (C), (2) applied voltage (V), and (3) the deposition time (t). The three values (level) of each parameters are listed in Table 2. The three parameters and their three corresponding levels were analyzed by using Taguchi design of Experiments of orthogonal arrays $L_9 (3^3)$. Table 3 lists the Taguchi orthogonal array used in this investigation. Green YSZ coating which obtained from each case (experiment) was dried over the night and the field emission scanning electron microscope was used to characterize the surface morphology and cross section of the coating.

![Figure 1](image_url)

**Figure 1.** The morphology of (a) micro YSZ and (b) nano YSZ powders used in plasma spraying and electrophoretic deposition process respectively.

| Coating material type | Plasma gun | Spray distance (mm) | Primary Gas (Ar) Flowrate (SCFH) | Secondary Gas (H2) Flowrate (SCFH) | Spraying Current (A) | Carrier Gas (Ar) Flowrate (SCFH) |
|-----------------------|------------|---------------------|----------------------------------|-----------------------------------|---------------------|----------------------------------|
| YSZ (204NS)           | Sulzer Metco 3 MB | 100 | 85 | 15 | 500 | 50 |
| CONiCrAlY (Amdry 365-2) | Sulzer Metco 3 MB | 120 | 85 | 15 | 450 | 40 |

**Table 1.** List of APS processing parameters utilized to fabricate TBCs.

**Table 2.** Controlled parameters and their levels used for EPD YSZ overlay on plasma sprayed YSZ coating.
Applied voltage (v), V
Deposition time (T), min
Concentration of the suspension (c), g/l

| No. | V (V) | T (min) | C (g/l) |
|-----|-------|---------|---------|
| 1   | 20    | 3       | 5       |
| 2   | 20    | 5       | 10      |
| 3   | 20    | 7       | 15      |
| 4   | 40    | 3       | 10      |
| 5   | 40    | 5       | 15      |
| 6   | 40    | 7       | 5       |
| 7   | 60    | 3       | 15      |
| 8   | 60    | 5       | 5       |
| 9   | 60    | 7       | 10      |

Table 3. Taguchi orthogonal array for EPD overlay YSZ coating on plasma sprayed TBCs L9.

3. Results and Discussion:

As plasma sprayed YSZ topcoat samples were described for initial microstructure and phase constituents. Fig. 2a reveals the first architecture of APS sample which used as a reference sample in this investigation. Its present typical lamellar microstructure resulted from successive impact and frosting (solidification) of the accelerated droplets on the superalloy substrate and also, it illustrates the porous topcoat microstructure. Coatings sprayed sample from a spray distance of 100 mm show the best degree of melting particles compared with other samples at 70 and 130 mm [16]. Investigation of the cross section of APS YSZ coating fracture surface reveals bimodal microstructure composed of columnar grains and nanosized un-melted powders as shown in figure 2b. Higher inter-lamellar pores were relatively generated because of non-complete perpendicular between the solidified splats and substrate called closed porosity. The volume fraction of closed voids and porosity was lower than voids and porosity formed at the topcoat (open porosity). It’s important to note, desirable amount of closed porosity had a positive effect on the performance of TBCs. Because it increases the thermal insulation required during services [17].
Table 4 and Figure 3 show mobility and zeta potential of the preparation of solutions utilized in the precipitation coating layer. It is important to understand electrophoretic mobility values through deposition in order to control the EPD process. All values are positive because the pH value was made and adjusted at 4. High absolute values of zeta potential were observed, which indicates a high stability and a good dispersed suspension and also expected cathodic precipitation due to the addition of iodine, so that all suspensions are suitable for EPD. The movement also increases with an increase in the absolute value of the zeta potential [18].

**Table 4. Value of zeta potential and mobility.**

| Concentration of YSZ | Zeta potential (mv) | Mobility |
|----------------------|---------------------|----------|
| 5 g/L                | 34.38               | 0.71     |
| 10g/L                | 55                  | 1.14     |
| 15g/l                | 48.13               | 1        |
Figure 3. Represent values of zeta potential and mobility for suspensions of concentration YSZ at (a) 5 g/L, (b) 10g/L, (c) 15g/l.

Figure 4 and Table 5 depict detailed micrographs of electrophoretic deposition on the plasma sprayed YSZ coating using design of experiments by taguchi. These experiments explain the behavior of deposition thickness response depending on the variables controlling the electrophoretic deposition process, in order to reach the largest thickness that can be obtained from this method. Especially, in this study, nano ceramic coating materials are deposit on a ceramic coating. It should be noted that a thin platinum layer has proven to be effective in significantly increasing the deposition rate of all the experiments. Due to maximum thickness of EPD coating is required, therefore, signal-to-noise ratio was selected to be “larger is better”. The behavior of SNs for coating thickness at different levels for all variables studied is shown in figure 5. The best level of all variables that governed coating thickness represented in table 6 considered the optimum condition for this response. Taguchi design of experiments was used to optimize the desirable coating thickness using EPD process on plasma sprayed YSZ TBCs.

It was found that all the EPD process parameters such as: applied voltage, deposition time and suspension concentration have obvious effect. The exceedingly important established result is that the suspension concentration is the mostly dominant parameter on the coating thickness obtained from EPD method. This can be observed from the results of the ANOVA that were conducted for the nine experiments shown in the table 7, depending on the signal-to-noise ratio as larger is better for the responding coating thickness. As well as, the analysis of variance gives the percentage contribution of each variable on the coating thickness. Figure 6 clarifies the contour and 3D surface plot of Coating thickness as a function to voltage and concentration (The two most influential variable). It shows non-uniform behavior for the 9 experiments because all the variables affect the coating process change with each other, there is no change for one of the variables at the expense of the constancy of the rest. In general, high value of thickness can be obtained at relatively concentration range 5-15 g/l and at different value of voltage range 20-60 V. It must be noted that
increasing the concentration more than 10 g/l, applied voltage more than 40V and the deposition time more than 8 min, produce a poor quality and irregular coating layer.

**Figure 4.** Optical micrographs of electrophoretic deposition on the plasma sprayed YSZ coating using design of experiments for all 9 experiments.
Table 5. Mean and signal-to-noise ratio of EPD nano YSZ coating thickness on plasma sprayed TBCs

| Variables       | Value | level |
|-----------------|-------|-------|
| Applied voltage (v), V | 40    | 2     |
| Deposition time (T), min | 5     | 2     |
| Concentration of the suspension (c), g/l | 10    | 2     |

Table 6. Best level values for each variable governed Coating thickness

| Variables studied | Value | level |
|-------------------|-------|-------|
| Applied voltage (v), V | 40 | 2 |
| Deposition time (T), min | 5 | 2 |
| Concentration of the suspension (c), g/l | 10 | 2 |

Table 7. ANOVA of coating thickness of nano YSZ EPD on plasma sprayed YSZ coating for each variables

| Variable | DF | Sum of squares (s) | Variance | Contribution% |
|----------|----|--------------------|----------|---------------|
| V (v)    | 2  | 4.8548             | 2.4274   | 19.47         |
| T (min)  | 2  | 3.6553             | 1.8277   | 14.66         |
| C (g/l)  | 2  | 16.2039            | 8.1020   | 64.99         |
| Error    | 2  | 0.2174             | 0.1087   | 0.871         |
| Total    | 8  | 24.9314            |          |               |
Figure 6. Contour and 3D surface plot of Coating thickness vs. applied voltage and concentration.

After obtaining the optimal values for the variables used in table 6, it is important to apply these values to verify the chosen conditions. In order to understand the performance of the coating and compare it with the plasma sprayed coating, it is necessary to make comprehensive description. It’s clear visible from surface morphology in Figure 7 (a) crack-free powder compact of YSZ overlay obtained from EPD after drying. The lack of pores was also observed, and this is important in preventing or protecting the plasma coating from penetration of harmful substances during services. Interface between the EPD YSZ overlay coating and APS YSZ, given in Figure 7(b), obviously offers a good adhesion of the EPD overlay coating. A dense continuous overlay coating can be obtained from sintering process at high temperatures.
Figure 7. FESEM of EPD on plasma sprayed sample (a) upper plan view (b) cross section, for the optimum condition obtained from Taguchi method.

4. Conclusions

Electrophoretic deposition (EPD) technique was successfully used to fabricate a dense-continuous overlay coating of YSZ for APS YSZ TBCs. Design of experiment by Taguchi was used to obtain the optimum conditions of the controlled variables that affect EPD process. Suspension concentration the mostly dominant variable on the coating thickness resulting from EPD method followed by the applied voltage and then the deposition time. Optimum condition result from DOE was 40V, 5min and 10g/l according to the studied variables and their levels.

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