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# The Effect of Surface Roughness of Inconel 738 Superalloy on the Adhesive Properties of 8YSZ Coatings Deposited by Electrophoretic Deposition (EPD)

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**Abstract**

The electrophoretic deposition coating method has recently received attention as a new method for thermal barrier coating due to its advantages such as the simplicity of the method, controllability of the process, and the need for simple equipment. In this study 8 wt % yttria-stabilized zirconia was deposited on Inconel 738 substrate at room temperature by electrophoretic deposition. Before deposition, the substrates were treated by sand blasting treatment (by silica sands with different sizes) and roughness in the range of $R_a$=1 and $R_a$=3 was achieved on the substrate surfaces. The coated samples were sintered at 1100 °C temperature under controlled atmosphere (Ar atmosphere). X-Ray diffraction, scanning electron microscopy and thermal shock resistance technics were used to investigating of structural and phase properties and adhesiveness between coatings and substrates. Results from thermal shock resistance test show that by increasing roughness number, the thermal shock resistance ability first increased and then decreased. In addition, according to SEM images, increasing of roughness number, causes decrease the microcracks that creating in the coating surface. The best result was observed for coatings deposed on substrate with $R_a$=2.

## 1. Introduction

Thermal barrier coatings used for insulation of turbine blades. With these coatings, the substrates of the blades are protected against high temperature and corrosion of hot gases. These coatings usually manufacture by Electron beam physical vapor deposition (EB-PVD) [1,2], and air plasma spray (APS) [3,4]. However, EB-PVD is costly [5,6] and if the suitable and direct accessibility to the place of deposition is not provided, non-uniformity in coatings is created (some areas of sample remain without coatings) [6,7]. APS also needs too many Equipment and creating of stable plasma arc is difficult in this process [6,8]. In addition, it must say that organometallic materials must use in other methods like chemical vapor deposition (CVD)which these materials have high cost [6,9].

All of methods that mentioned above have some important disadvantages. These methods are costly, complicate and need to expensive equipment. Recently, in the laboratory, researches have been focused on manufacturing of these coatings by electrophoretic deposition (EPD) technique [10,11]. In recent years, (EPD) is taken in to consideration in both industrial and academic sectors; not because of the versatility of this method with different materials specially compounds, but also because of its simple and cheap equipment that needed for deposition [12,13]. EPD method has been used successfully for gas diffusion electrodes [14] and sensors [12], silica thin films [15], layered ceramics [16], multi-layer composites [17], hydroxyapatite Coatings [18], layers contained carbon nanotubes [19], functionally graded ceramics [20], and piezoelectric materials [21]. So, the mentioned coating method is not a new method. However, little research has been done regarding its use in order to cover TBC. One of the disadvantages of the EPD coating method can be attributed to the improper adhesion of the coating to the substrate. Based on this, in previous researches, the intermediate layer was used to increase adhesion to the substrate [7,10,11]. So far, no research has been done regarding the effect of surface roughness on the amount and quality of YSZ coating coated by EPD method and its adhesion to the substrate. Therefore, according to the mentioned cases, TBC coating by electrophoretic deposition method and also investigating the substrate effect can be an important and valuable research.

In this paper, 8YSZ was deposited by EPD on Inconel 738 superalloy substrates at room temperature. The substrates before EPD were sand blasted by silica sands with different sizes and different surface roughness was created on the surface of these substrates. The deposited samples by EPD were sintered in optimum temperature. Finally, microstructures, phases and the amount of adhesion between coatings and substrates were investigated while studying the effect of surface roughness on the adhesion of the YSZ coating on the superalloy substrate, the optimal mode is determined.

## 2. Experimental Procedures

### 2.1 Preparation of Suspension

An industrial nickel sheet and Inconel 738 with dimension of 4 x 10 x 25 $mm^3$ were used for the anode and cathode (substrate), respectively. 8 mol% YSZ with average particle size of 70 microns and high purity was used. Ethanol and acetone with laboratory purity as solvent and iodine as dispersant were used, respectively. 20 gr/l of YSZ powder was added to 100 cc of solvent with a ratio of three to one (ethanol to acetone). 0.4 g/l of iodine was added as a dispersant in order to disperse the solid particles in the suspension. According to previous researches, the addition of iodine is an effective factor to prevent the precipitation of particles before the coating process and to improve the coating quality [7,11]. The volumetric flask that contained solvent and raw materials was placed on ultrasonic bath (manufactured by Elmasonic, model: S15h) for 1 hour. The ultrasonic bath cause that the agglomerations of raw materials are dispersed.

**Table 1** *Raw materials and solvents for preparing of suspension*

| Raw Material and Solvent | Concentrates or Volume ratio | Manufacturer |
|---|---|---|
| 8YSZ | 20 g/l | Sigma-Aldrich (USA) |
| Iodine | 0/4 g/l | Merck (Germany) |
| Solvent (acetone to ethanol) | 3 to 1 (Volume ratio) | Dr. Mojallali (Iran) |

### 2.2 Preparation of Cathode

The samples of Inconel 738 superalloy were cut to dimension of 4 x 10 x 25 $mm^3$. Then the totally 6 samples were treated by sand blasting treatment (by silica sands with different sizes) and roughness in the range of $R_a$=1 to $R_a$=3 was achieved on the substrate surfaces. Table 2 shows the average roughness of these samples for three points. Every sample before EPD process, were cleaned on ultrasonic bath for 5 minutes in a mixture of acetone to ethanol (3 to 1 volume ratio). For each test, a control sample also is used. The surface of control samples was polished by sandpapers (until sandpaper with number 2000).

**Table 2** *The results of roughness test on substrates after sand blasting treatment.*

| Samples | A | B | C | D | E | F |
|---|---|---|---|---|---|---|
| Ra | 1.05 | 1.08 | 2.01 | 2.09 | 3.07 | 3.10 |

### 2.3 Preparation of Anode

An industrial nickel was used for anode in this research. This anode, before EPD, was polished by sandpaper (Number 5000) and then was cleaned on ultrasonic bath for 5 minutes in a mixture of acetone to ethanol (3 to 1 volume ratio).

## 2.4 Deposition

After preparing of electrodes (anode and cathodes), suspension and cell (Fig. 1), prepared suspension was added to deposition cell. The EPD process was carried according to Table 3.

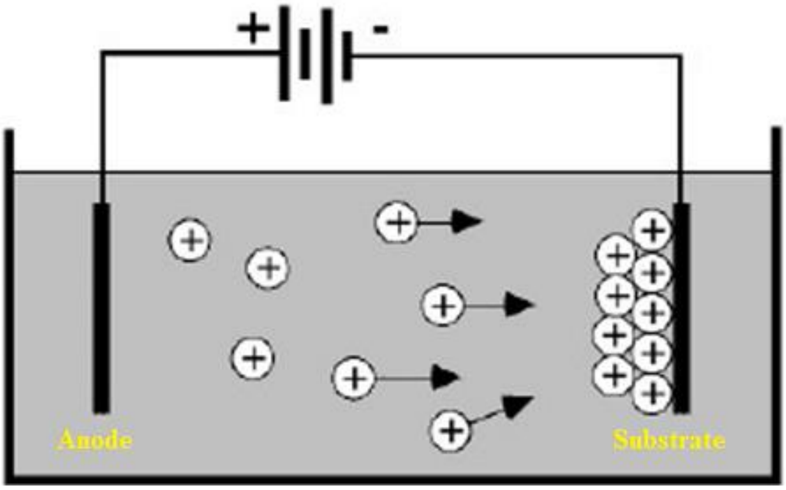


**Fig. 1** *EPD cell image*

**Table 3** *The parameter of EPD process for 8YSZ coatings on Inconel 738 superalloy*

| EPD Parameters | Amount (Unit) |
|---|---|
| Time | 2 min |
| Temperature | 25 °C |
| Voltage | 10 V |
| Electrode distance | 1 cm |

## 2.5 Drying

The coated samples were maintained for 24 hours in room temperature. Then the samples were dried in 80 °C for 4 hours.

## 2.6 Sintering

The samples were semi-sintered in a tube furnace after drying and sintering. The heating rate was 5 °C. After arriving of temperature to 1100 °C, the samples were sintered in this temperature for 4 hours. Then the samples were cooled to room temperature in the tube furnace. To protect of substrates from corrosion, Argon gas (more than 99% purity) was used. According to previous research, the subsequent sintering process plays an important role in increasing the adhesion of YSZ particles to each other [7,8,11].

The phases of YSZ coatings were investigated by X-Ray diffraction (XRD). The microstructure of coating also was investigated by scanning electron microscopy (SEM). The adhesiveness also was investigated by comparing of thermal shock resistance of coated samples with standard samples (the samples with polished substrates), according to ASTM B517-91.

## 3. Results and Discussion

Fig. 2 shows the typical thickness of a coating. The mean thickness of the YSZ coating was about 60 micrometers. The reported thickness was calculated by averaging between all samples. According to this fact, suspensions were binder-free, so this thickness is significant.

Also based on software analysis by Clemex vision on the YSZ coating, the calculated density was 74% of theoretical density. In other words, the coating density of YSZ coating by EPD method was 74% of the raw material powder used in the experiment. Obtaining 26% porosity in the coatings produced by electrophoretic method indicates that this amount of porosity is suitable for the production of thermal barrier coatings.

Fig. 3 shows the scanning electron microscope images form YSZ coatings. These coatings were sintered in 1100 °C for 4 hours. The present of roughness, causes that the amount of surface cracks was decreased. The surface cracks were Minimum in samples with 2-micrometer roughness in substrate. Ceramic materials are brittle so that decreasing of surface cracks causes to improvement of mechanical properties.

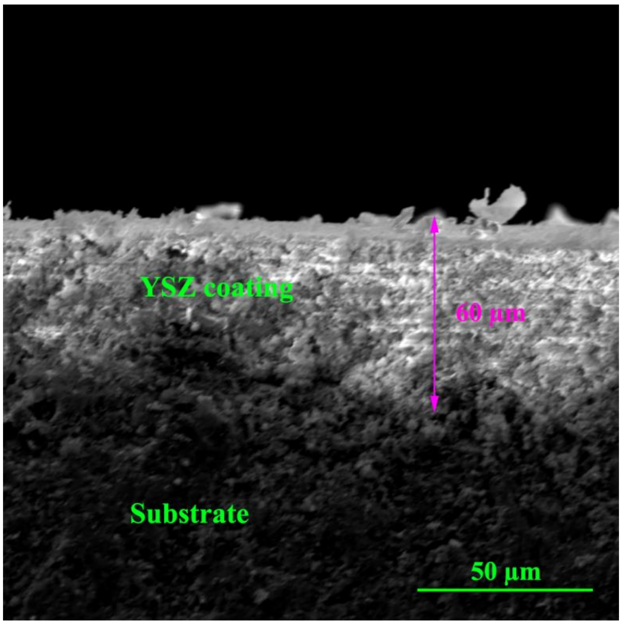


**Fig. 2** *Cross section of YSZ coated samples on Inconel 738 superalloy substrate*

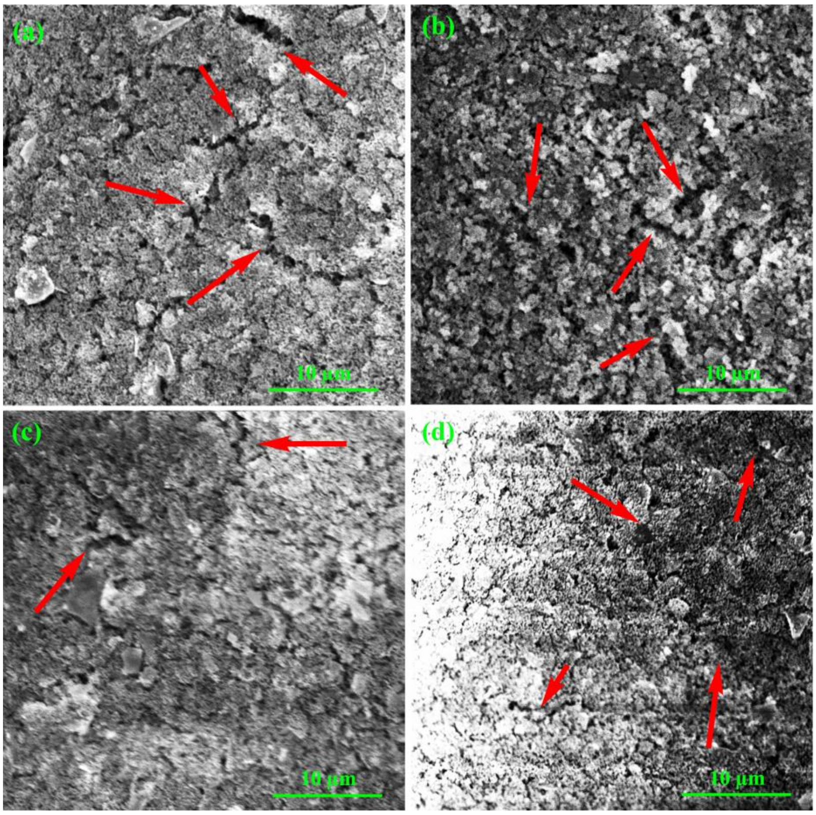


**Fig. 3** *Scanning electron microscope images from YSZ coating surfaces on a) standard substrate (with polished surface), b) substrate with $R_a$=1 micrometer, c) substrate with $R_a$=2 micrometer, d) substrate with $R_a$=3 micrometer*

Fig. 4 shows XRD patterns of sintered coatings in 1100 °C for 4 hours. According to analysis by X'Pert HighScore Plus, there are three phases of Cubic, Tetragonal and Monoclinic of YSZ in all samples. By comparing of peaks intensities of YSZ powder and YSZ coatings in Fig. 4, it's concluded that the amount of monoclinic phase, compared to Cubic and Tetragonal phases, was increased after sintering. The most of Cubic and Tetragonal phases peaks of this material are overlapped, so concluding about the change of the amounts of Cubic and Tetragonal phases is not correct. Totally, it has concluded that the percent of Cubic and Tetragonal phases are increased. In addition, according to XRD pattern of samples with polished substrate, it is understood that roughness has not influence on phase formations [22,23]. It is necessary to explain that due to the slow cooling of the samples in the furnace, the possibility of forming the non-equilibrium phase of zirconia is very low.

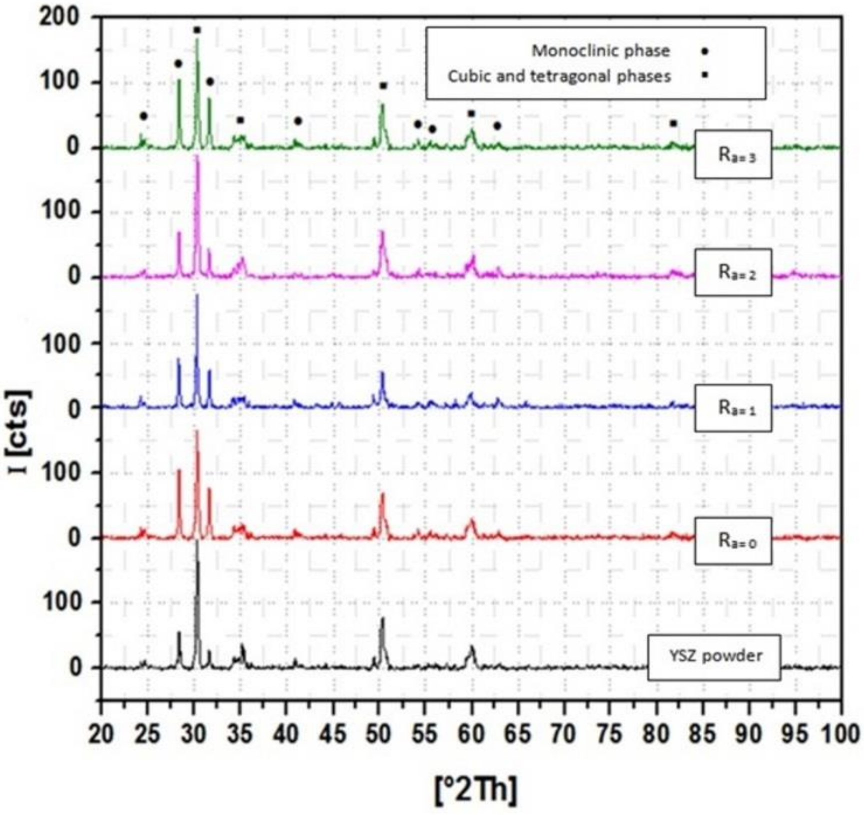


**Fig. 4** *XRD patterns of YSZ coatings on substrates with different roughness*

Fig. 5 shows the number of thermal shock cycles of samples with different roughness. It has showed that the roughness had caused increasing in the number of thermal shock cycles that samples could tolerate.

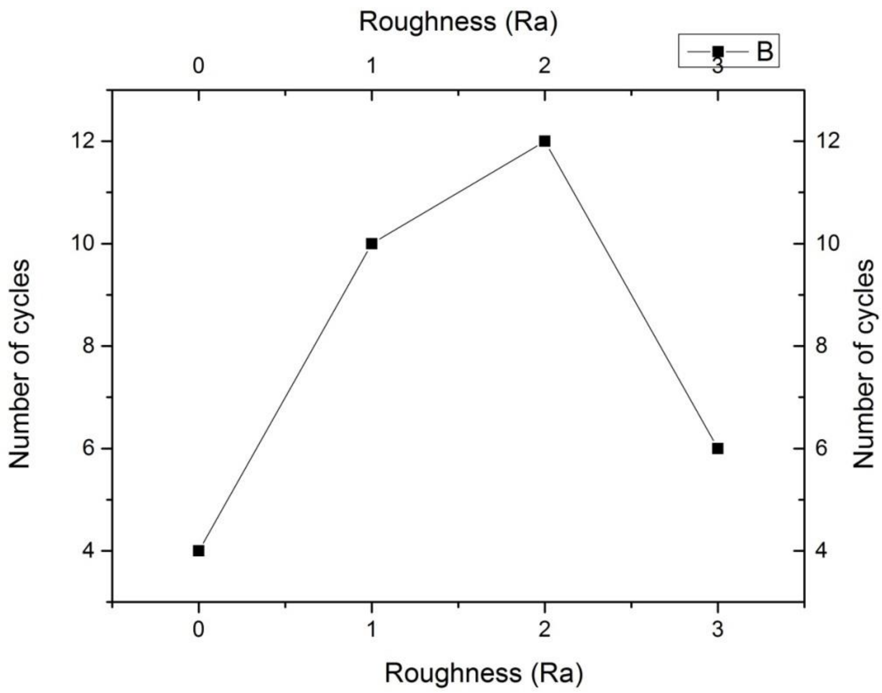


**Fig. 5** *Number of thermal shock cycles of samples with different roughness*

According to Fig. 5, it has concluded that the optimal roughness in the studied roughness range equals 2 micrometer. The sample with $R_a = 2$, could withstand until 12 cycles of thermal shock.

## 4. Conclusion

This research studied the effect of the surface roughness of nickel-based superalloy (Inconel 738) on the properties of YSZ coatings produced by the electrophoretic deposition (EPD) method. By investigating of XRD patterns of YSZ powder with YSZ coatings, it has showed that coatings have three phases of Cubic, Tetragonal and Monoclinic. In addition, the amount of Monoclinic phase increase by sintering. Also sand blasting (roughness) has not influence on phase formations. Results of thermal shock resistance test show that by increasing of roughness, at the first, the ability of thermal shock resistance is increased and then this ability is decreased. The samples with $R_a = 2$, could withstand until 12 cycles of thermal shock and have the best resistance.

## Conflict of Interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

## Author Contribution

*The authors confirm contribution to the paper as follows:* ***study conception and design****: A. Akbari, H. Alikhani;* ***data collection****: H. Alikhani, A. Taherizadeh, A. Khezrloo;* ***analysis and interpretation of results****: A. Akbari, H. Alikhani, A. Taherizadeh, A. Khezrloo;* ***draft manuscript preparation****: H. Alikhani, A. Taherizadeh, A. Khezrloo . All authors reviewed the results and approved the final version of the manuscript.*

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