Journal of Physics: Conference Series

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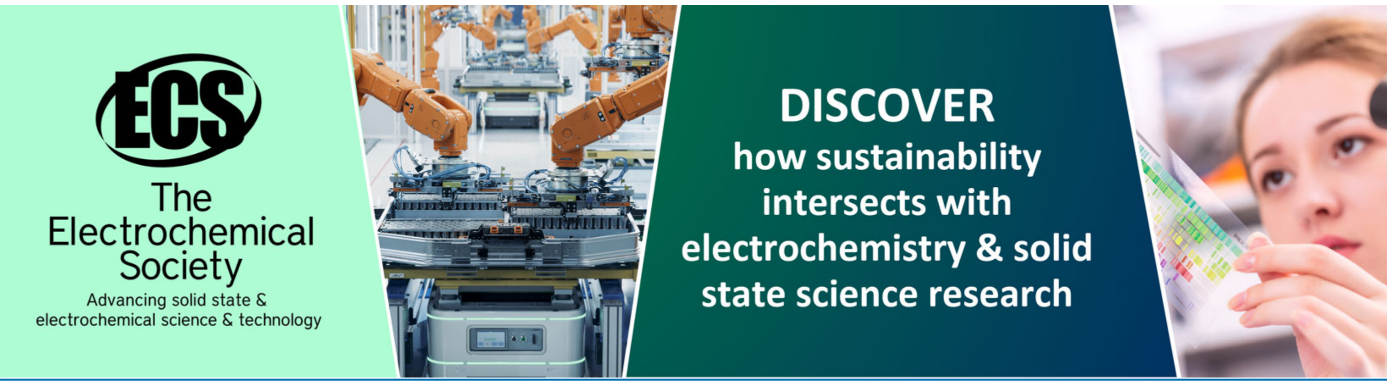
To cite this article: S Liscano and L Gil 2013 *J. Phys.: Conf. Ser.* **466** 012008



Journal of Physics: Conference Series **466** (2013) 012008 doi:10.1088/1742-6596/466/1/012008

Performance of air plasma spraying parameters in the fabrication of ZrO_2–10%Y2O_3–18% TiO_2 thermal barrier coatings

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Abstract. In this research, ZrO2–10%Y2O3–18% TiO2 thermal barrier coatings (TBCs) were sprayed by employing an air plasma thermal spray (APS) technique following a 23 factorial design experiment in order to obtain the best favorable spraying conditions able to enhance their properties. The effect of the spraying parameters on the porosity, microhardness, microstructure and morphology were determined by using different techniques such as optical microscopy (OP), image analysis, Vickers indentation and scanning electron microscopy (SEM) technique, this latter coupled with X-Ray microanalysis (EDS). It was found that both the arc voltage and the powder feed rate, as well as their interaction had a significant effect on the values of the reported hardness and these results were related to the existing level of porosity in the coatings. It was concluded that the best coatings properties, for the level of the variables studied in this work, could be obtained if the arc voltage is maintained at 36 V, the arc current at 900 A and the powder feed rate to 24 g/min.

1. Introduction

Plasma-sprayed yttria-stabilized zirconia (YSZ) continues to play an important role in enhancing performance of components for different industrial applications [1, 2]. Tailoring the microstructure and properties of these thermal barrier coatings towards achieving both prime reliance and manufacturing reproducibility is a complex task due to the multitude of interrelated parameters that influence both the plasma spray process and the deposit formation dynamics [3, 4]. The atmospheric plasma sprayed process involves melting of the feedstock materials in a plasma plume and rapidly transporting these molten particles to the substrate, where rapid solidification of individual particles occurs upon impingement. The successive build-up of these "splats" results in a layered arrangement in the coating, analogous to a brick-wall-like structure where the splats are intertwined in complex arrays [3]. This splat-based layered microstructure leads to an intrinsic anisotropy of the coating in the direction perpendicular to the spray direction. The splats are separated by interlamellar pores resulting from rapid solidification of the lamellae, globular pores formed by incomplete inter-splat contact or around unmelted particles as well as intrasplat cracks, which are due to thermal stresses and tensile quenching stress relaxation. The proper characteristic of the TBC originates from the existing porosity, the microcracks and their inherent microhardness [4, 5]. Although numerous studies involving processing parameters effects on the characteristics of such deposits have been carried out, a rigorous analysis on the effects of the APS parameters on the morphology and the mechanical properties of

YSZ plasma sprayed coatings employing the Experimental Design study has been little attempted; particularly, for the chemical composition of the powders used in the present investigation, which implies the addition of TiO2 to the traditional ZrO2-YO1.5 system, addition which is related to the enhancement of toughness, phase stability and sintering resistance, all of which are desirable for TBC applications [7].

The present study was conducted with the aim of the relationship between different deposition parameters of APS ZrO2–10%Y2O3–18% TiO2 coatings and their microstructural features and microhardness. The empirical relationships amongst the independent variables were achieved by employing both, statistical experimental design methods and the response surface methodology, described in detail elsewhere [7].

2. Experimental details

The TBCs, composed of a bond coat and a top coating, were industrially deposited by air plasma spray (APS) onto AISI 1020 steel coupons of 19.05 mm diameter. The powder used for the bond coating was Ni-5%Mo-5.5%Al (Metco® 447NS), which was deposited by using a Sulzer Metco® model 5P-II torch (manual operation). A Praxair model SG-100 torch coupled to mechanical arm was used to deposited ZrO2–10%Y2O3–18%TiO2 (Metco® 143) with powder size -75 +5 μm, as top coating. The industrial operational parameters are presented in table 1.

In this research, a 2^3 factorial design experiment was employed to establish the effects of the variables on the coating quality. Three operational parameters were varied: the arc current, the arc voltage and the powder feed rate, and setting to two levels and employing a central point (obtained of conditions of plant), which implies that nine experiments, were necessary for to explore the variation of these variables at the chosen levels. Two replicate experiments were carried out in each case. The test matrix is displayed in table 1.

Table 1. Industrial operational parameters and Test matrix for the 23 factorial experiment design.

Run Nº	Arc current (A)	Arc voltage (V)	Powder feed rate (g/min)	Spray Distance (mm)	Ar Flow Primary (SCFH)	H2 Flow Secondary (SCFH)
Conditions of plant (Central point)	850	35,5	20	76	75	15
1	800	35	24			
2	900	35	24			
3	900	36	24			
4	800	36	24			
5	800	35	16			
6	900	35	16			
7	900	36	16			
8	800	36	16			

The experiments were carried out following a random sequence and the data were obtained for the response properties as: porosity and microhardness. These results were subject to an analysis of variance (ANOVA), based on F-test, in order to measure whether a factor contributes significantly to the variance of a response. The degree of the influence was represented as a frequency histogram. Response surfaces methodology (RSM) was used to describe empirical relationships amongst the three independent variables investigated. The statistical analysis of the result was conducted by using a commercial statistical analysis package [7].

In order to have reproducible results for the metallographic apparent porosity studies, samples cross section have been prepared following the procedure recommended in the literature for the thermal spray coatings [9]. The porosity was determined by carrying out the study of twenty fields captured on the cross section of the sample by employing digital image analysis (IA32 LECO Image Analysis System, USA). The microstructure of the coatings was examined by using a scanning electron microscope (Quanta FEG 600 SEM) coupled with a standard energy dispersive X-ray microanalysis attachment (EDAX). The hardness of the coatings was obtained using a Vickers indenter with the load of 300g applied for 15 seconds on the sample cross section. Results of ten measurements were averaged to obtain the reported microhardness values.

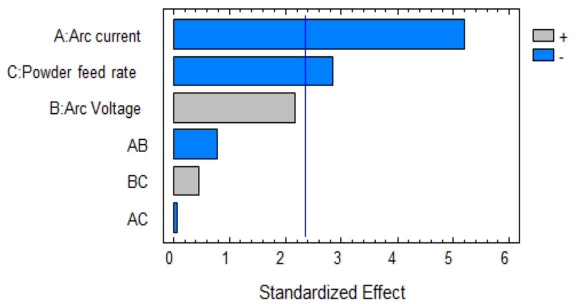
3. Results and discussion

3.1. Porosity

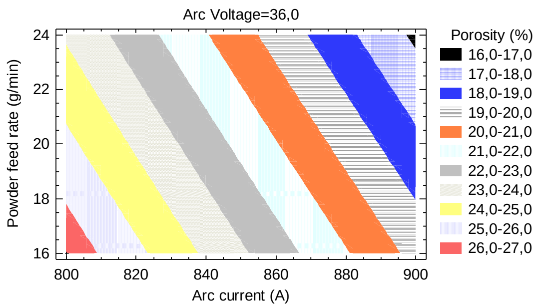
The average porosity and standard deviations values, as revealed by image analysis, are listed in Table II. As it can be observed, the average porosity of these coatings varied ranged between 14.7% to 26.5 %. From the analysis of the variance (ANOVA) it was found that arc current (A) and powder feed rate (C) were the only independent variables that contributes significantly to the variance of the response, at 95 % confidence level, for the interval studied in the present investigation. This could be observed in figure1 (a), where the frequency histogram shows the influence of each factor on the response function, i.e. on porosity.

Table 2. Summary of the characteristic results of the APS coatings for the 23 factorial experiment design.

Run Nº	Microhardness ($HV_{0,3}$)	Porosity (%)
1	330	17,8±2,1
2	486	17,3±3,2
3	798	14,7±2,5
4	734	26,5±3,7
5	895	26,3±0,8
6	788	16,5±0,6
7	775	22,3±3,4
8	828	24,5±2,4
(central point)	591	18,5±3,2



(a)



(b)

Figure 1. (a) Frequency Histogram shows the Influence of each factor on the response function, i.e. on porosity. (b) Response Surface Contours of porosity for a constant value of Arc Voltage= 36,0 V and like a funtion of variation of Powder feed rate (g/min) and Arc current (A).

Since no substantial differences in porosity were found when the arc voltage was varied between 35V to 36 V, figure. 1 (b) represents the typical contours of porosity corresponding to the APS ZrO2–

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10%Y2O3–18% TiO2 coatings as a function of the both arc current and powder feed rate values for a constant value of the arc voltage of 36V. For example, the figure 1 (b) shows that an increase of the arc current from 800 to 900 A, produced a decrease in the average porosity of 30%, when powder feed rate was fixed in 24 g/min. This result is expected since an increase in arc current develops more heat available in the spraying jet. Measurements performed by Vardelle et al. [9] when spraying alumina powder have indicated that a variation of 10% in the value of the arc current leads to a variation of 20% in the value of the process thermal efficiency. On the other hand, in the same figure, when powder feed rate varies from 16 to 24 g/min produces a decrease in the average porosity of 16% when the arc current is fixed in 900 A. These results corroborates those reported by Fleury et al. [10], where the porosity level linearly decreases as the powder feed rate increases. As expected, excessive feed rates may result in inadequate or partial melting of the powder. However, the powder feed rate should be adjusted to avoid the over-oxidation of the powder.

3.2. Microhardness

The hardness values of the coatings obtained during different tests were listed in table 2. As it can be observed, the average hardness values corresponding to the nine processing conditions varied between 330 to 895 HV300. Similar hardness values were reported by Shin [5], for coatings of plasma-sprayed ZrO2 coatings. From the ANOVA test, it was found that hardness depends of the powder feed rate (C), the arc voltage (B) and the interactions between the arc voltage and the powder feed rate (BC) and between arc current and the powder feed rate (AC), since they were determined as being significant from the statistical point of view, as shown in Figure 2a. The results of empirical modeling of the microhardness are also presented in figure 2. This figure presents the contours of micohardness for two conditions each one with constant value of the powder feed rate: 16 g/min (figure 2 (b)) and 24 g/min (figure 2 (c)). The axes, in both cases, correspond to the arc voltage (horizontal axis) and arc current (vertical axis).

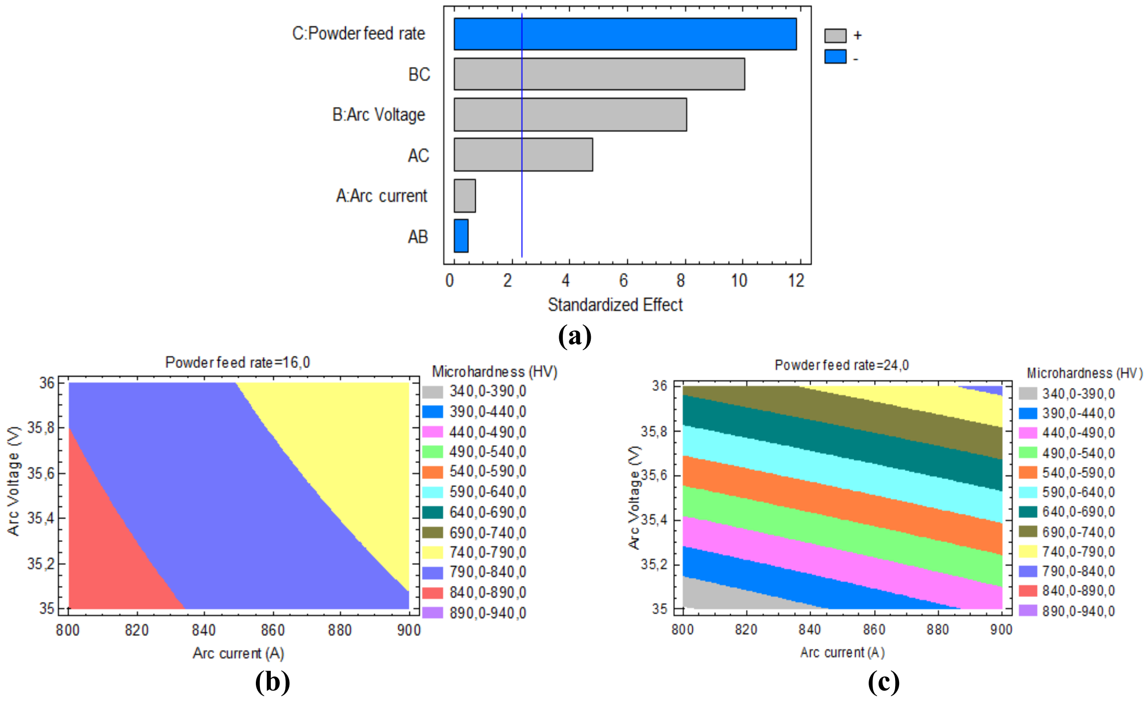

Figure 2. (a) Frequency histogram showing the influence of the variables and their interactions on microhardness. (b) Response Surface Contour of microhardness for the variables, current and voltage, the speed of the powder was maintained between values 16g/min. (c) 24g/min.

From the analysis carried out in figures 2 (b) – (c) it could be observed that power feed rate has a marked influence on the hardness value of the coating. In the figure 2 (b), at value constants of arc voltage and power feed rate in 16g/min, the hardness value starts to decrease when the value of the arc current is increasing. In contrast, when the powder feed rate increases to 24g/min, hardness behavior becomes opposite: increases with the rise of current arc (figure 2 (c)). This behavior in the mechanical properties has been attributed in the literature to interaction of other variables such as those related to the existing phases in the coating microstructure, as well as the adhesion between splats [10].

4. Conclusions

APS process can be investigated efficiently using methods based on factorial experimental designs. Empirical modeling could be successfully used to identify parameters that have a significant influence on coating properties. Analysis of the coatings characteristics has showed that the arc current, the arc voltage and the powder feed rate as well as their interaction had significant effects on the porosity and microhardness. In this study, the lowest porosity levels were obtained for the case of Run N°3 (highest parameters value), while Run N°5 (lowest parameters value) produced the hardest coating.

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