ISSN 2176-5480

22nd International Congress of Mechanical Engineering (COBEM 2013)
November 3-7, 2013, Ribeirão Preto, SP, Brazil


# STUDY OF THE INFLUENCE OF DEPOSITION PARAMETERS IN THE POROSITY AND OXIDATION OF BOND COATINGS DEPOSITED BY HIGH VELOCITY OXI-FUEL PROCESS

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***Abstract.*** *Thermal Barrier Coating (TBC) is commonly used for protecting surfaces against oxidation at high temperatures. The Top Coating provides Thermal isolation to the alloys that work at high temperatures and the Bond Coating protects the substrate from oxidation. Denser coatings and with low inclusion of oxides usually result in Bond Coats which are more resistant to oxidation. In general, Bond Coats are deposited by Thermal Spray processes and, inherently to this process, the formation of oxides and pores in the coating occurs. The correct choice of parameters can lead to a lower level of oxides and pores in the Bond Coat. This paper aims to analyze the influence of the deposition parameters (Feed Rate, Distance of Deposition and Carrier Gas Pressure) in the levels of porosity and oxidation in Ni22Cr10Al1Y coatings deposited as Bond Coat for High Velocity Oxy-Fuel process (HVOF). After deposition the samples were metallographically prepared and examined via Optical Microscopy with the aid of a software program for measuring the porosity and percentage of oxides. The experiments followed a full factorial design of the type L8. Through analysis of variance was found that the parameters who most influence in the oxidation and porosity level is the feed rate and carrier gas pressure, showing that despite expectations, the distance of deposition has no influence on the oxidation and oxide contents of the particles during deposition.*

***Keywords:*** *HVOF; oxides; porosity; Bond coats*

## 1. INTRODUCTION

Gas Turbine is a machine that converts energy generated by combustion fuel in mechanical energy for propulsion, work or generation of electrical energy. Initially designed for aeronautics industry, gas turbines are each time more used in thermoelectric power generation (Eskner, 2004). The efficiency of a turbine depends on the inlet and outlet gas temperatures. In moderns gas turbines the inlet gas temperature may exceed 1673 K. Due to the high inlet temperatures, it is necessary that the vanes used in these turbines have a high resistance to oxidation in these temperatures (Tamarin, 2002).

Therefore it is important the use of coatings applied in these vanes in order to increase the corrosion and oxidation resistance of these materials at high temperatures (Stöver and Funke, 1999). The great advantage in using coatings is that by changing just the surface of the component, one can provide mechanical properties completely different to the substrate (Rodriguez et al, 2007; Trevisani et al, 2011). Some of the benefits reached are: reduction of the maintenance costs, higher operating temperatures, reduced thermal load, increase in the wear, erosion, oxidation and corrosion resistance in ambient and in elevated temperatures (Aguiar, 2012).

Turbine components exposed to high temperatures are normally coated with alloys resistant at elevated temperatures, such as (Ni, Co)-Cr-Al-Y alloys, also known as Bond Coats. They are called “Bond Coats” because they form a bond coat between the substrate and a ceramic coating used as thermal barrier (Guo et al, 2009).The literature reports that Bond Coats with lower levels of oxides and porosity result in coatings more resistant in cyclic oxidation tests at temperatures above 1173 K (Peng et al, 2011).

The techniques normally used to deposit the Bond Coat are Electron Beam Physical Deposition (EBPVD), Plasma Spray (PS) an High Velocity Oxi-Fuel (HVOF) (Peichl et al, 2003).

In this paper the HVOF technique was employed. This process uses an internal combustion jet fuel (propylene, acetylene, propane or hydrogen gas) to produce a gas temperature higher than 3000 K and to generate a supersonic gas

ISSN 2176-5480



velocity (Li et al, 2003). The powder particles are injected into the gas jet, and simultaneously heated, and propelled toward the substrate. HVOF is one of the most used technique in the applications of Bond Coats, producing coatings both resistant to isothermal and cyclical oxidation (Tamarin, 2002). During the HVOF thermal spray process, the particles being sprayed are exposed to a high-temperature, oxidizing atmosphere, due to this, it is inherent to this process the formation of oxides, pores and voids in the cooling of the coating (Davis, 2004). It was deposited a Ni22Cr10Al1Y alloy by HVOF process in order to produce coatings with low level of porosity and oxides and with this purpose it was performed a statistical analysis to determine which parameters are most influential in the deposition of this alloy. The parameters analyzed were distance of deposition, carrier gas pressure and feed rate.

## 2. METHODOLOGY

The sample used were made of austenitic stainless steel plates AISI 304 with dimensions of 75x37x6,35 mm. As Bond Coat it was deposited the NI-343 alloy (Ni22Cr10Al1Y), using the HVOF process by a 6-axis manipulator, brand ABB. The substrates were previously prepared through grit blasting, which provides both the cleaning and the roughness required by the processes of Thermal Spray. It was used alumina in powder grain size 36/ Alundum 38A in a shot-blasting machine of 100 psi in the “Thermal Spray and Special Welding Laboratory” (LABATS) from UFPR. The depositions by HVOF were performed in the company REVESTEEL in the city of Pinhais-PR.

### 2.1 Definition of process parameters

The definition of the deposition parameters were based on studies in literature and technical bulletins the manufacturers of the powders (Aguiar, 2012). Following these studies, preliminary tests were performed seeking to define the choice of parameters. Through these tests, it was verified that the parameters that presented the greatest influence on the final content of pores and oxides in the coating were deposition distance (mm), carrier gas pressure (psi) and Powder Feed Rate (g /min).

### 2.2 Experimental design

Each of the chosen process parameters was analyzed on two levels and for this cause it was decided to choose a full $2^k$ factorial design, with k=3 (each one of the process parameters), which allows to evaluate up to three parameters at two levels each. The experiment was conducted with two replicates and the analysis of variance (ANOVA) was performed.

The parameters used and the levels chosen are shown in Tab. 1. The levels presented were chosen according to preliminary tests (bending test and visual analysis by optical microscopy), in which all specimens were approved.

Table 1. Parameters and levels used in the deposition and ANOVA.

| **Level** | **Factors** | | |
|---|---|---|---|
| | **(A) Deposition Distance (mm)** | **(B) Feed Rate (g/min)** | **(C) Carrier Gas Pressure (psi)** |
| **-1** | 150 | 20 | 110 |
| **1** | 250 | 40 | 150 |

### 2.3 Characterization of oxidation and porosity

As the response, it was analyzed the content of porosity and oxides in the cross section of the deposited coatings. The samples were metallographically prepared through sanding with particle sizes of 320, 400, 600 and 1200 mesh, followed by polishing cloth with diamond suspension of 3 µm, 0.25 µm, 0.1 µm and colloidal silica of 0.04 µm, all these procedures were carried out in a automated equipment, brand Buehler, model Vector 60.

The analysis of porosity and oxide content was performed on an Olympus Optical Microscope, model BX60, with image acquisition through a digital camera Mediacybernetics, model EvolutionLC. The images were converted into 8-bit grayscale format and then calibrated and adjusted with the image analysis software AnalySIS 5.1 in the Materials Laboratory of UFPR.

## 3. RESULTS AND DISCUSSION

The experiment was conducted with two replicas. After analysis of porosity and oxidation, the responses (content of pores and oxides) were measured. Figure 1 shows the cross section of each sample, i.e., for each treatment analyzed.

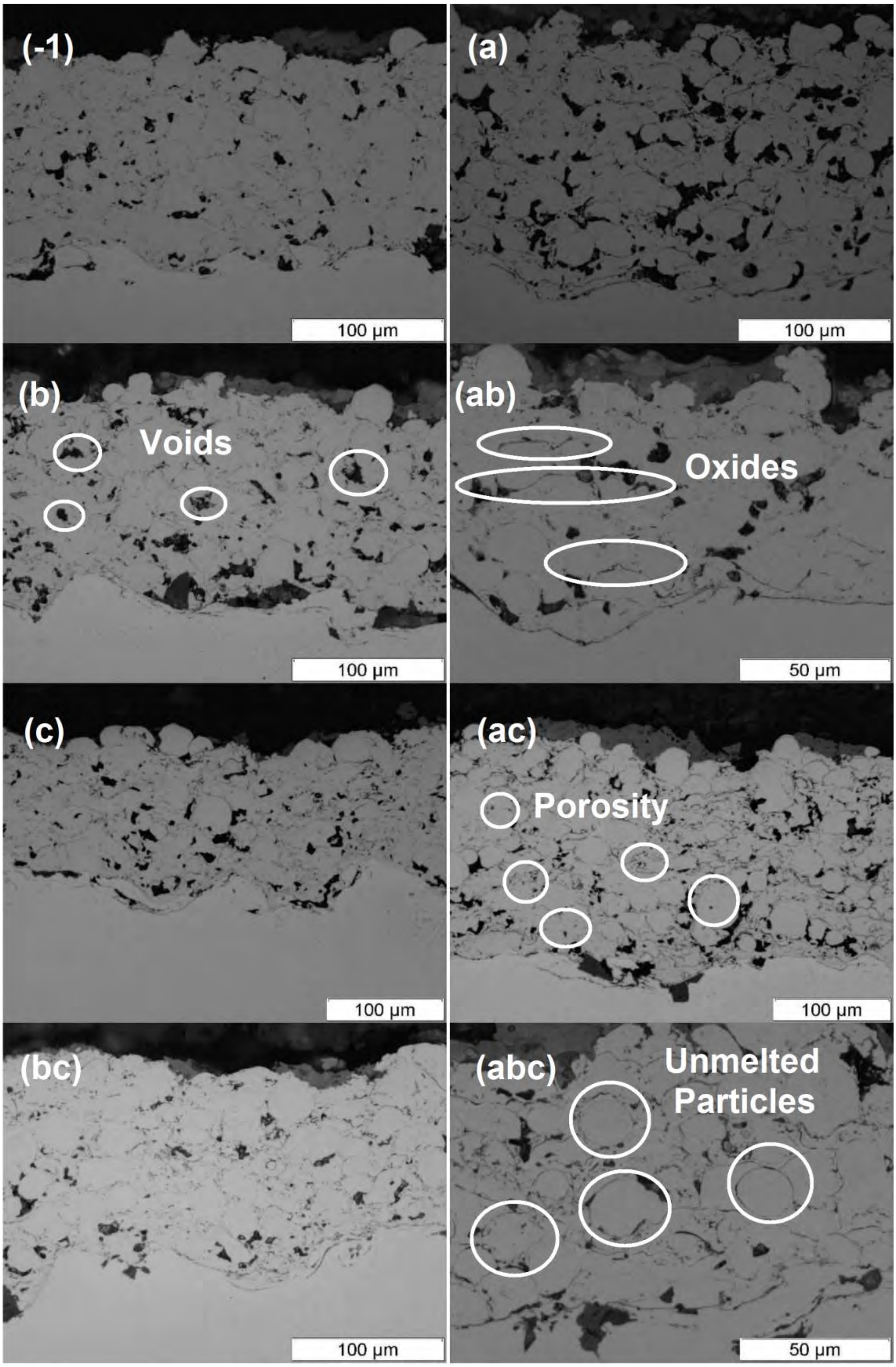


Figure 1. Cross section of the coatings for each treatment analyzed- Replica 1. Featured in the images are the pores, voids, oxides and unmelted particles which occurred during deposition by HVOF.

ISSN 2176-5480



With the aid of an image analysis software, the percentages of oxides and pores were calculated and are shown in Tab.2.

It can be observed in the samples shown in Fig. 1 the presence of oxides, pores and voids, which are inherent to the process. The oxides are formed mainly in the boundary of the splats and the surface of the coating, pores occur normally inside of the splats and voids are formed in the interlamellar regions during the cooling of the deposited coating (Paredes, 2006). The porosity analysis in itself is difficult, the image analysis software tends to analyze the tonalities and both pores and voids commonly have the same tonalities. Therefore, when referring to porosity in this paper it is referred to the sum of pores and voids.

It can also be observed the presence of some unmelted particles in some samples (such as treatment "abc"). These particles typically occur when part of the powder propelled by the jet does not melt, or does not reach the substrate with sufficient thermal energy to deform in the typical lamellar structure of the coatings deposited by Thermal Spraying (Pawlowski, 2008).

The analysis of the experiments was performed using the software Minitab 16, which enables calculating both the effect of each parameter and the analysis of variance (ANOVA). For this analysis, it was raised the hypothesis that the parameters deposition distance, feed rate, carrier gas pressure and the interactions between them were significant. Observing the images and the calculation of pores and oxides, it can be seen that the percentage of oxides for all samples were below 5% (ranging from 1.9 to 4.6%) showing a low content of oxides in the end of the deposition.

Table 2. Levels of porosity and oxides after running the $2^3$ factorial design with two replicas.

| **TREAT-MENTS** | **(A) Deposition Distance (mm)** | **(B) Feed Rate(g/min)** | **(C) Carrier Gas Pressure (psi)** | **Porosity (%)** | | **Oxides (%)** | |
|---|---|---|---|---|---|---|---|
| | | | | **Replica 1** | **Replica 2** | **Replica 1** | **Replica 2** |
| -1 | 150 | 20 | 110 | 3,6 | 4,6 | 3,5 | 4,6 |
| a | 250 | 20 | 110 | 7,9 | 4,5 | 3,7 | 3,5 |
| b | 150 | 40 | 110 | 6,1 | 1,8 | 2,2 | 2,5 |
| ab | 250 | 40 | 110 | 1,5 | 3,1 | 2,3 | 2,6 |
| c | 150 | 20 | 150 | 3,1 | 3,8 | 3,4 | 3,1 |
| ac | 250 | 20 | 150 | 3,2 | 2,2 | 2,3 | 2,2 |
| bc | 150 | 40 | 150 | 1,0 | 0,3 | 1,9 | 2,8 |
| abc | 250 | 40 | 150 | 2,3 | 0,7 | 2,2 | 2,6 |

By analyzing the results, the effects were calculated and were performed the analysis of variance for the results of the porosity measured. Effects and analysis of variance are presented in Tab. 3. The normal plot of the standardized effects for the porosity is presented in Fig. 2. Through the graphic analysis, it can be viewed that the most significant effects were B (feed rate) and C (carrier gas pressure). The negative effects suggest that for higher feed rates and pressures there will be a decrease in the final porosity of the coating. In order to confirm the graphic analysis, the analysis of variance for porosity was calculated.

Table 3. Analysis of variance for porosity.

| **Source of Variation** | **Effects** | **Sums of Square** | **Degrees of Freedom** | **Mean Square** | **F** | **P** |
|---|---|---|---|---|---|---|
| Distance (A) | 0,134 | 0,0716 | 1 | 0,0716 | 0,03 | 0,867 |
| ***Feed Rate (B)*** | ***-2,009*** | ***16,1403*** | ***1*** | ***16,1403*** | ***6,71*** | ***0,032*** |
| ***Carrier Gas Pressure (C )*** | ***-2,056*** | ***16,9127*** | ***1*** | ***16,9127*** | ***7,04*** | ***0,029*** |
| Distance*Feed Rate(AB) | -0,531 | 1,1289 | 1 | 1,1289 | 0,47 | 0,513 |
| Distance*Pressure | -0,054 | 0,0116 | 1 | 0,0116 | 0,00 | 0,946 |
| Feed Rate*Pressure | -0,011 | 0,0005 | 1 | 0,0005 | 0,00 | 0,989 |
| Distance*Feed Rate*Pressure | 1,341 | 7,1958 | 1 | 7,1958 | 2,99 | 0,122 |
| Residual Error | -- | 19,2292 | 8 | 2,4037 | -- | -- |
| **TOTAL** | -- | **60,6905** | **15** | -- | -- | -- |

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For a level of significance of 5 % the F of Snedecor is $F_{0,05,1,8}$=5,32. Using ANOVA it was confirmed that the significant parameters are the feed rate (B) and carrier gas pressure (C), which had an F value greater than 5.32, which were 6.71 and 7.04 respectively. The same was observed in the graph in Fig. 2. It is known in the literature that higher carrier gas pressures result in higher velocity of the particles and therefore a greater kinetic energy at the time of the impact of the particles with the substrate. This increased impact energy generates denser coatings with lower porosity content (Davis, 2004).

However increasing carrier gas pressures above 150 psi can be harmful to adherence of the coating. According (Pawlowski, 2008) the pressure used in the deposition of NiAl based alloys typically range from 100 to 200 psi. Control of Powder Feed Rate is critical. This rate coupled with the Voltage of the equipment, directly influences the rate of deposition per pass and the deposition efficiency (Davis, 2004). If using a very high deposition rate for a given Voltage, part of the powder will not be melted, harming the adhesion of particles and generating more porous coatings. A lower feed rate can result in low deposition efficiency.

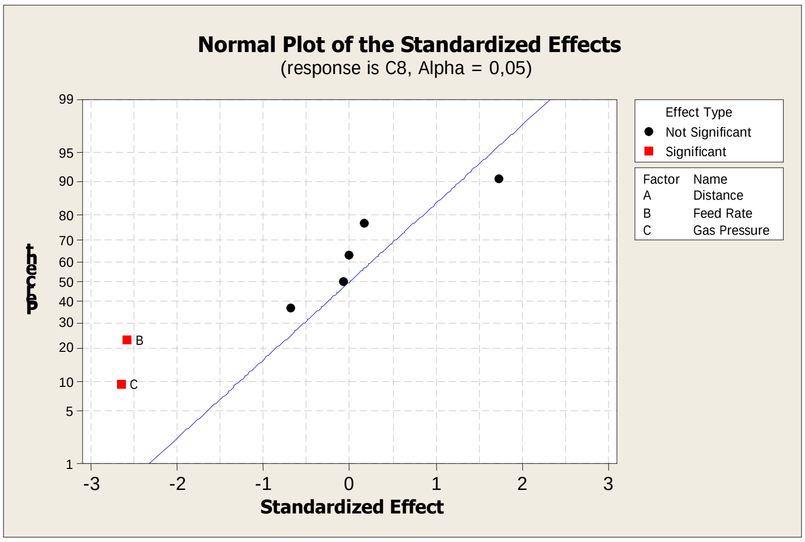


Figure 2. Normal plot of the standardized effects for the porosity.

After the analysis of porosity, it was performed the analysis of results of the oxide contents. The effects were calculated and ANOVA was performed for the oxide content measured in the coatings, as shown in Tab. 4.

Table 4. Analysis of variance for the oxide contents.

| Source of Variation | Effects | Sum of Squares | Degrees of Freedom | Mean Square | F | P |
|---|---|---|---|---|---|---|
| Distance (A) | -0,3187 | 0,40617 | 1 | 0,40617 | 2,74 | 0,137 |
| ***Feed Rate(B)*** | **-0,8953** | **3,20594** | **1** | **3,20594** | **21,6** | **0,002** |
| ***Carrier Gas Pressure (C )*** | **-0,5363** | **1,15055** | **1** | **1,15055** | **7,75** | **0,024** |
| *Distance*Feed Rate* | 0,3979 | 0,63316 | 1 | 0,63316 | 4,27 | 0,073 |
| *Distance*Pressure* | -0,1478 | 0,08739 | 1 | 0,08739 | 0,59 | 0,465 |
| ***Feed Rate*Pressure*** | **0,5314** | **1,12962** | **1** | **1,12962** | **7,61** | **0,025** |
| Distance*Feed Rate*Pressure | 0,1663 | 0,11066 | 1 | 0,11066 | 0,75 | 0,413 |
| Error | -- | 1,18727 | 8 | 0,14841 | -- | -- |
| **TOTAL** | -- | **60,6905** | **15** | -- | -- | -- |

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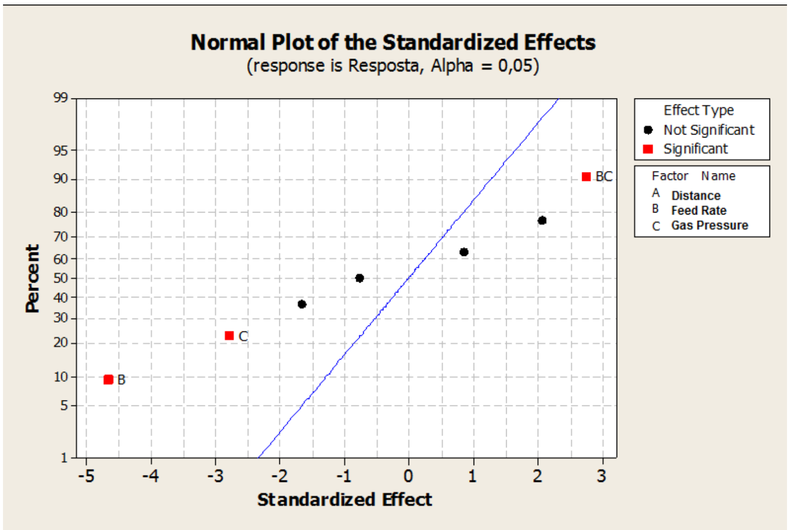


Figure 3. Normal plot of the standardized effects for the oxide contents.

Figure 3 shows the normal plot of the standardized effects for the oxide contents. As can be seen both in Tab. 4, as in the graph of Fig. 3, the parameters that had the greatest influence on the formation of oxides were also the Powder Feed Rate and carrier gas pressure. As aforesaid, higher carrier gas pressures result in higher velocity of the particles. With higher velocities, the propelled particles have a shorter exposure to air and therefore a lower oxidation occurs. The adequate feed rate allied with the proper carrier gas pressure results in better deposition efficiency by reducing the amount of oxides formed.

According to the experimental design and analysis of variance evaluated in this study, it was found that the parameters that most influence the porosity and oxidation of Bond Coats deposited by HVOF are the carrier gas pressure and the feed rate. To analyze in which of the levels studied occurred the lowest levels of pores and oxides, two graphs were plotted with the main effects for the Powder Feed Rate and carrier gas pressure, one for the level of porosity in Fig.4 and another for the content of oxides formed in Fig.5.

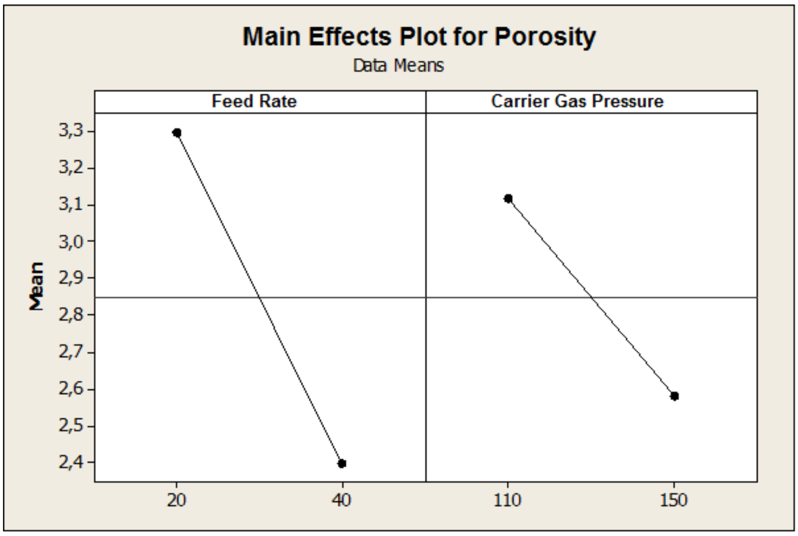


Figure 4. Main effects plots of feed rate and carrier gas pressure for porosity.

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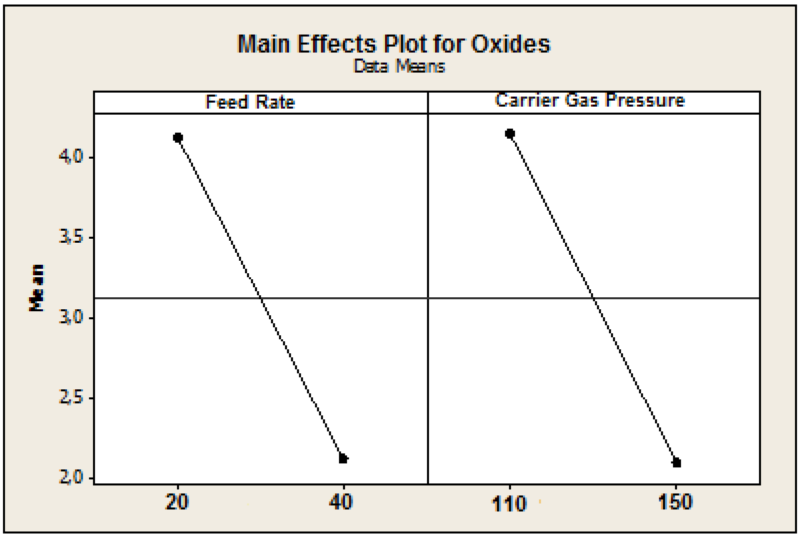


Figure 5. Main effects plots of feed rate and carrier gas pressure for oxide contents.

Analyzing both graphs, it is noticed that the highest levels of the feed rate (40 g/min) and carrier gas pressure (150 psi) result in coatings with lower levels of oxides and pores. This fact is confirmed by Tab. 2, where it is observed that the treatment BC which used these parameters had a mean value for the pore and oxide content of 0.6% and 2.3%, respectively, the smallest values of all the coatings.

An important observation is that contrary to what is stated by the literature (Pawloski, 2008), the parameter distance deposition, measured between the nozzle of the gun and the surface of the substrate, was not significant in the formation of oxides and porosity for the alloy deposited. The literature reports that this parameter has a direct influence on the temperature and velocity of the particle at the time that this collides with the substrate. At smaller distances, the particles reach the substrate with higher thermal energy and kinetic energy, deforming and forming denser coatings with lower porosity (Li et al, 2003).

However, very small distances should also be avoided in order to prevent overheating of the substrate and the shock, fragmentation and repulsion of particles during the process, damaging the adherence of the coating to the substrate (Pawlowski, 2008). What may have caused this non-significant influence is that the variation of distances used for this alloy was not significant compared to the high velocities of HVOF process. Once the parameter distance deposition shows no significant influence on none of the cases, it can be adopted any of the levels used. However, by the literature it is known that for smaller distances tend to generate more adherent coatings (Davis, 2004), for this cause it would be interesting to adopt the lowest distance value.

Based on the significant effects it is possible to calculate an adjusted model Eq. (1) and Eq. (2), with which can be estimated, in accordance with the levels chosen, the oxide and pore contents. The adjusted model is the sum of the mean values of porosity (3.123 %) and oxidation (2.847 %) with the effects of the significant parameters divided by the number of replicates. It can be seen that for the higher levels (1 for the parameters B and C) is obtained the lowest estimated contents for both the porosity Yest=1,09 %, and oxidation Yest=2,131%. The estimated value for the oxide content was very close to BC treatment that had 2.35 % of oxides and the content of pores was even lower, only 0.64 %, showing that with these parameters it is possible to obtain a Bond Coat with low porosity, as well low oxide contents.

$$\mathbf{Y_{est}=3{,}123-1{,}005B-1{,}028C} \quad \textbf{(Porosity content estimated)} \tag{1}$$
$$\mathbf{Y_{est}=2{,}847-0{,}448B-0{,}268C} \quad \textbf{(Oxide content estimated)} \tag{2}$$

## 4. CONCLUSION

With the observed data can be concluded that:

- Through the analysis of variance was verified that the only parameters that were significant were the Carrier gas pressure and Powder Feed Rate.
- For the highest pressure (150 psi) and higher feed rate (40 g/min) were obtained the smallest values of oxides and pores, and as reported by the literature, Bond Coats with low oxide and pore contents have high resistance to oxidation at elevated temperatures.
- Contrary to expectations, distance deposition showed a non-significant influence on porosity and oxide content of the deposited coatings. This non-significant influence may be due to the differences between the distances used (150 and 250 mm) are small compared to the very high velocities of the HVOF process.

- Since the objective of this work is to produce coatings with a low porosity and oxides, they were chosen the highest levels for feed rate and carrier gas pressure, which produce coatings with low porosity and oxide content.

## 5. ACKNOWLEDGEMENTS

The authors are grateful to Thermal Spray and Special Welding Laboratory (LABATS) from UFPR in which the depositions were made. To LACTEC for the assistance provided. To CAPES-REUNI, PRH24 ,PGMEC and PIPE for the encouragement and support to develop this work .

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