Influence of the Process Parameters on the Properties of Diamax Deposits Obtained by Flame Thermal Spray

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Abstract. The paper aims to determine the influence of the process parameters, namely: C2H2 gas flow rate and inclination angle of the spraying gun on physico and mechanical properties of the hardalloyed layers of Diamax 10999 Eutalloy, on steel support - obtained by flame thermal spray process. For this purpose, the two technological parameters varied on three levels and in each case were evaluated the deposits properties. Investigations conducted by electronical microscopy SEM, X-ray, micro-hardness and by adherence evaluation and of the deposits porosity allowed the establishment of the performant deposit. Thus it was found that at the decreasing of the spraying distance, the deposit porosity decreases; in layer appear the phenomena of overheating, issue that determine the adherence reducing in average of 22%, and also the modification of chemical composition. The results recorded have afforded the obtaining of an optimum domain of variation of the process parameters.

1. Introduction
The thermal spray process in oxygase flame, at low pressures, is one of the oldest spraying processes, being considered also the cheapest process for layers obtaining, which use for melting the input material the heat developed by burning of a gaseous fuel mixture (usually acetylene - C2H2 in combination with oxygen - O2), [1-3]. The process uses, as added materials, metals (pure metal or alloys based on Al, Zn, Fe, Ni, Co, etc.) as non-metallic materials in the form of powders of different micrometric sizes or wire, [4,5]. Flame-spray coatings have often been used to protect against corrosion and/or wear protection of various tracks, to dimensional reconstruction, to obtain new properties or to obtain surfaces with a decorative aspect, [6]. Studies, in the field of thermal spraying, [7,8] have demonstrated that a pulverized particle is strongly attached to the substrates of a substrate if its velocity and temperature is sufficiently high, [9, 11]. In thermal flame spraying, the temperature and particle velocity at the moment of impact with the surface of the substrate is dependent on the C2H2/O2 ratio, the impact angle and respectevelly on the spray distance, [7,8]. Obviously the
modification of the spray jet temperature - by increasing it, will have a positive effect on the temperature of the particles and implicitly on the deposition properties.

The Diamax deposits, which mainly containing tungsten (W) and nickel (Ni), are widespread due to their combination of unique properties: hardness and resilience, offered by tungsten carbide (WC) - the compound which is formed by spraying and provides excellent wear resistance. Generally, the metal matrix composite coatings on the basis of WC (MMC) [12-13], use special deposition processes that require high temperatures which induce secondary effects, such as the formation of undesirable brittle phases (W2C and the amorphous binder) [14-15] and increase the porosity of deposits, [16].

The present researches are focused on the study of the process parameters influence, respectively: of C2H2 flow rate and the angle of inclination of the spray gun against the surface of the substrate (α), on the physical, chemical and mechanical properties of the Diamax 10999 Castolin deposits, produced by flame spray.

2. Experimental procedures

2.1. Materials and process parameters

In the researches, it was deposited Diamax 10999 Eutalloy, produced by Castolin Switzerland - containing 15% W - 30% W - as 45% Ni, 15% Cr, 5% Co and Fe - which ensure the wear resistance at high temperatures, [23]. For the deposits obtaining, a SuperJet -S-Eutalloy type torch, manufactured by Castolin Switzerland, was used.

The deposits were carried out on a frontal flat surface of some samples, Ø40×50mm – EN 582, made of C15-DIN 17210-1652 steel, with the following composition C—0.14, Mn—0.43, Cr—0.3, Ni—0.3, Si—0.15, P—0.04, and S—0.04 (wt.%).

The frontal flat surface of the cylindrical samples was cleaned by grit-blasting of Al2O3 (grade 24), which are driven by a compressed air jet, at a pressure of 4bar. The parameters of the flame spraying process are presented in Table 1.

The coating process used was the manual one which uses the support for the spray gun. Therefore, the amount of material deposited is a characteristic of the system. However, the test conditions of each passes may be considered in the range of 50 to 80 μm of thickness The final coating varied between 250–480 μm of thickness.

Table 1 - Deposition parameters.

| Parameters                  | Value    |
|-----------------------------|----------|
| C2H2 gas flow rate (l/min)  | 20/22/24 |
| O2 gas flow rate (l/min)    | 38       |
| Spray angle (°)             | 60/75/90 |
| Spraying distance (mm)      | 80       |
| Number of spraying passes   | 6        |
| Scan speed of the gun, (m/s)| 0.16     |

2.2. Characterization methods

The coatings obtained by flame spraying were examined with stereomicroscope Optika SZM 2 — made in Italy, for the purpose of excluding the coatings which contain defects.

The microstructural investigations realized in the cross-section of the samples, were done with a optical microscope Optika coupled with a scanning electronic SEM microscope, of the Vega Tescan LMH II type, (made by Czech Republic). The image analysis was carried out with software ImagePro, (product by Media Cybernetics, Bethesda, MD, USA) and allowed the estimate the WC content, the determining the porosity of the cross section of the polished coatings and the mean free path between the reinforcing particles. About ten images of each coating system were used to determine the average
WC content (in vol.%). For determination the mean free path between the reinforcing particles was used the count/size function in the ImagePro software and about twelve images of each coating. On the images was drawn a random line and was recorded the number of times that the line intersected WC particles. Because ImagePro presents the length of the line in pixels, was used a line to measure the scale bar to convert pixels in to micrometres. The mean free path was determined used following equation: 

$$\lambda = \frac{1}{V_p N_i}$$

where: \(\lambda\) is mean free path, \(V_p\) is volume fraction of the reinforcing particles and \(N_i\) – is the number of particle intersected per unit length of test line.

The micro-hardness HV was determined using digital microdurimeter CV - 400D, manufactured by CV Instruments - made by Japan. Measurements were made using a load of 300 g for 10 s. On each sample were carried out twelve random measurements, accordance standard E384, [18]. Indentations were spaced at a distance of at least four times the diagonal of the previous indent as per ASTM C1327 to avoid strain-hardening effects and possible cracking of the WC particles [19].

Qualitative analysis phase was performed using X-ray diffractometer, X'PERT PRO MRD type, product by PANalytical, Holland, which works in 2θ angle range from 0° to 90° and a High Score Plus software.

The adherence of the obtained deposits was determined by tensile test of the coatings deposed on the frontal flat surface of the samples (for the determination of tensile adherence strength \(R_H\)) in accordance with EN 582. Twelve tests were performed on each sample using an Instron machine (Instron Corp., Canton, MA) at a crosshead speed of 0.075 cm/min.

The wear resistance of the coated bars was evaluated using an RFT-III (Kyowa R/D Ltd., Japan) reciprocating friction and wear tester at room temperature without lubrication. Tests were performed under loads of 200N for 5h, at rotation speed 200rpm and at room temperature of 23°C and relative humidity of 26%. Three tests were performed for each condition for repeatability and the averages of the three test results are given in the paper.

3. Results and discussions

Figure 2 shows the microstructure of the deposit were made from mixtures of powders with chemically stable, based on the W and Ni. In the SEM images, it is observed that in each layer there are the following distinct phases: a light phase containing W bonded in the form of carbides (WC) and a darker phase with a high Ni content. Black areas were identified as pores. It can be seen that the degree of homogeneity of the structure increases with the increase in C2H2 flow. This is explained by the fact that as the gas flow increases, it increases the temperature of the spray jet and maintains the molten state of the particles at the moment of impact with the surface of the substrate.

**Figure 1.** SEM micrographics of Diamax 10999 Castolin coatings deposited using flame spraying, inclination angle \(\alpha = 90^\circ\), at C2H2 gas flow rate: 20 l/min (a); 22 l/min (b) and 24 l/min (c).

Table 2 shows that the porosity of WC deposits decreased as the inclination angle (\(\alpha\)) increased. This phenomenon was also noticeable by P.C. Du and colab [17].
Table 2. Porosity values of Diamax 10999 coatings obtained in different technological conditions.

| Nr Crt | Materials                  | Inclination angle, α [°] | C\textsubscript{2}H\textsubscript{2} gas flow rate, [l/min] | Porosity, [%] | Mean free Path, [μm] |
|--------|---------------------------|--------------------------|-------------------------------------------------------------|---------------|----------------------|
| 1      |                           | 60                       | 20                                                          | 18.8          | 84.3                 |
| 2      |                           |                          | 22                                                          | 15.6          | 62.4                 |
| 3      |                           |                          | 24                                                          | 11.3          | 37.5                 |
| 4      | Diamax 10999 Castolin     | 75                       | 20                                                          | 13.6          | 46.8                 |
| 5      |                           |                          | 22                                                          | 8.8           | 28.4                 |
| 6      |                           |                          | 24                                                          | 7.2           | 24.6                 |
| 7      |                           |                          | 20                                                          | 12.6          | 32.4                 |
| 8      |                           | 90                       | 22                                                          | 5.3           | 21.5                 |
| 9      |                           |                          | 24                                                          | 4.5           | 9.2                  |

The amount of Diamax deposition obtained by flame spraying was determined by X-ray diffraction (XRD) analysis. In Figure 4 there are presented XRD profiles of the deposits made at different flows of C\textsubscript{2}H\textsubscript{2}. It can be seen that XRD profiles contain mainly the quantities of phase: WC and Ni. The WC and Ni drops are preponderant at angles of 360, 480 and respectively 400. Figure 4 shows that when the C\textsubscript{2}H\textsubscript{2} flow increases, there is a relative increase in the primary peak of the WC, compared to the peak corresponding to the Ni content in the layer. It can be asserted that by increasing the temperature of the gas jet it is allowed to fix of a larger amount of WC in the layer, aspect which contributes to the increase of the wear resistance of the Diamax 10999 layers.

![Figure 2. XRD profile of the Diamax 10999 Castolin coatings obtained using α = 90° and C\textsubscript{2}H\textsubscript{2} gas flow rate: 20 l/min (a); 22 l/min (b) and 24 l/min (c).](image)

Vickers’ microhardness indentation determinations performed on Diamax 10999 deposits allowed a first estimate of how will behave the depositions at erosion. Due to the used indentation load, relatively low (300gf), the depths produced were located both in areas with a high contain of WC and in the areas of matrix existence of Ni, which led to large variations of HV microhardness. Figure 3 is
represented the effect of the C\textsubscript{2}H\textsubscript{2} flow rate on the microhardness of the coating layer. Figure 3 also shows that as the C\textsubscript{2}H\textsubscript{2} flow rate increased, the microhardness HV also increased. Diamax powders produced the hardest coatings with a hardness of 509 HV\textsubscript{0.3} when a flow rate of 24 l/min of C\textsubscript{2}H\textsubscript{2} was used. This is to be expected because the hardness of the layer should increase when the particles hit the molten substrate and spread evenly therein. The microdurality values recorded in areas with a high Ni content were relatively low, around 112 HV\textsubscript{0.3}, being lower than in the areas rich in WC. The microhardness values recorded in areas with a high Ni content were relatively low, around 112 HV\textsubscript{0.3}, being lower than in the areas rich in WC. In addition, the high porosity of the Ni matrix, deposited by flame spray, allows an easy fixation of the hard particles (WC), due to the decrease of the resistance at deformation of the previous formed layer. This phenomenon was also observed in other studies involving WC, [20]. Although microhardness is not the only indicator of wear resistance of a coating, it is the simplest way to characterize it.

![Figure 3](image1.png)

**Figure 3.** The variation of the micro hardness of Diamax 10999 Castolin coatings obtained using different C\textsubscript{2}H\textsubscript{2} gas flow rate and spraying angles: α = 60° (a); α = 75° (b); and α = 90° (c).

![Figure 4](image2.png)

**Figure 4.** Specific wear rate of Diamax 10999 Castolin coatings deposited at the different spray angles and C2H2 gas flow rate: 20 l/min (a); 22 l/min (b) and 24 l/min (c).
Figure 4 represents the wear velocity, specific to the coatings obtained at different spray angles. All the layers obtained have a relatively low wear rate (below $10^{-7}$ mm$^3$/Nm), although a slight decrease in the wear rate is observed, with about 12% by increasing the spraying angle. These conclusions have been obtained also by other researchers and can be explained both by increasing the deposition porosity, [21] and by the low deposition rate, [22] - recorded at low angle of inclination.

The C$_2$H$_2$ flow also influences significantly the adherence of the Diamax 10999 deposits, as shown in Figure 5. It was found that the values of the stresses $R_H$ increase by more than 12%, with the increase of the compressed air flow from 20 l/min to 24 l/min. This can be explained by the fact that the increasing flow results in the increase of the jet temperature consequently, of the sprayed particles temperature, which contributes to the increasing number of particles attached to the substrate. It can be stated that the increase of sprayed particles velocity and temperature by the increase of jet temperature was achieved - appearance also encountered by other researchers, [4]. Also, it can be observed that with the decrease of the spray angle, the adhesion of the deposition decreases. Thus, breaking strength - $R_H$, decreases by over 18% when decreasing the spray angle from 90° to 60° . This aspect can be explained by the fact that by the inclination of the spray gun, the spray jet degrades the surface previously created by overheating.

![Figure 5](image)

**Figure 5.** The variation of the adherence of Diamax 10999 Castolin coatings obtained using different C2H2 gas flow rate and spraying angles: $\alpha = 600$ (a); $\alpha = 750$ (b); and $\alpha = 900$ (c).

4. Conclusions

The Diamax depositions, made by oxy-acetylene flame spraying, using a cold spray installation, at low pressure and various process parameters, were investigated to identify the optimal process parameters needed to obtain wear adherent layers and resistant. Thus, it was found that:

- By increasing the flow rate of C$_2$H$_2$ homogeneous layers are obtained - Figure 1, the quantity of pores in the layer decreases - Table 2, increases the adherence of the coating to the substrate - Figure 5.

- The X-ray analysis showed that by increasing the flow rate of C$_2$H$_2$ in the layer, a larger quantity of WC is fixed, thus recording a significantly growth of the depositions microhardness - Figure 2.

- The decrease of the spraying angle caused an increase of the wear rate by approximately 12% - v. Figure 4 and the decrease by more than 17% of the adherence of the Diamax deposits.

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