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TANTALUM COATING FROM THE REDUCTION OF THE ALUMINOTHERMIC TA_2O_5 IN PLASMA SPRAY

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Abstract. *Plasma spray been used in recent years to coat metallic materials used in diverse applications such: metallic implants, cutting tools, and in metallurgical processes, as well as in the aerospace industry and so on. In the present study carried out spraying of a mixture of Ta2O5 (10g) + Al (2.14 g) using an atmospheric plasma torch with a power of 7.5 kW. The coatings deposited on stainless steel substrate 304 without rotation (0 to 6000 rpm) with distances of 30 and 50 mm. To ensure total reduction of Ta2O5 through the aluminothermic process used was a ratio of the mass of the powder with 5% excess Al in the analysis of X-ray diffraction (DRX) revealed Ta pure and impregnated Al and oxides of Ta and Al. It was possible to check the surface morphology of the coating through the analysis of MEV. The analyses of DRX confirm the formation and production phase Ta oxides in all conditions studied, as well as residues of Al, showing that there was no overall consumption of aluminium in the process.*

Keywords: *atmospheric plasma, aluminothermy, tantalum, SEM, XRD.*

1. INTRODUCTION

Tantalum is a material very attractive to the industry in general by adding very interesting properties, such as high melting temperature, high ductility, corrosion resistance and thermal and electrical conductivity significant [1], and to combine excellent mechanical properties. The most important applications of the use of tantalum capacitors in manufacturing occur electrolytic [2], in coating of cutting tools (TaC) [3-4], catalysis [5] in medical applications [6] in glass special high refractive index for optical use [7], and alloys corrosion-resistant [8].

Tantalum powders are principally used for the production of electrolytic capacitors, by having a thin oxide layer formed on the surface of the particles of tantalum with excellent dielectric properties [9]. Ta metal can be obtain by reduction of their oxides of elements such as C, Si, Ca, Mg and Al [10], [11] and [9]. In the case of Al, the reaction is so-called aluminothermic reduction. The commercial process for the production of tantalum reduced to K_2TaF_7 [11].

In aluminothermy, conventional aluminium reacts with Ta_2O_5 with the product Ta and Al_2O_3. The reaction occurs with an external source of heat, but the spread is strongly exothermic [12]. The main difference between the method of spraying for plasma torch and conventional methods is that in the plasma, each particle of the mixture of (Ta_2O_5 + Al) heated individually by energetic collisions of the specimens present in the plasma. In the conventional method, the reaction propagates in the mass layer and then all the powder material melted.

In this paper, we present a variant of the aluminothermic reduction process which uses an atmospheric plasma torch with a power of 7.5 kW with a plasma gas flow and entrainment of the powder respectively equal to 20 l / min and 6 I / min as a source of exothermic heat. We used a mixture of Ta_2O_5 and Al (in a stoichiometric ratio of mass extrapolating the amount of aluminium in 5%) to ensure total reduction of Ta_2O_5. The powder mixture was sprayed at distances of 30 and 50 mm with the substrate at rest and rotating (6000 RPM), in order to obtain coatings with greater cohesion and uniformity. The characterization studies were performed using the techniques of scanning electron microscopy (SEM) to observe the morphology of the coatings and X-ray diffractometer (XRD) for the identification of crystal phases.

2. MATERIALS AND METHODS

Were used in this work a mixture of powders of aluminium (Al) and tantalum pentoxide (Ta_2O_5) in a mass ratio with a stoichiometric excess of 5% of Al This powder mixture was milled for 6 hours in a mill High energy, Fritsch model Pulverisette 7. Were used as substrate disks 304 stainless steel with dimensions of 19 mm diameter and 1 mm thick.

Before spraying by plasma torch, the substrates were previously sandblasted with particles of SiO2 with an average diameter of 450 microns was performed immediately after cleaning the surface of the substrate 304 with absolute alcohol in ultrasonic agitation to remove organic contaminants surface. An initial roughness Ra = 2.3 mM was obtained on the substrate determined with the aid of a model surtronic profilometer Taylor Hobson of the mark 25. The resulting powder mixture of Ta_2O5 (10g) + Al (2.14 g) was asperjido by atmospheric plasma torch, which power was 7.5 kW and the flow of the plasma gas and carrier powder were respectively 20 l / min 6 l / min Fig. 1.

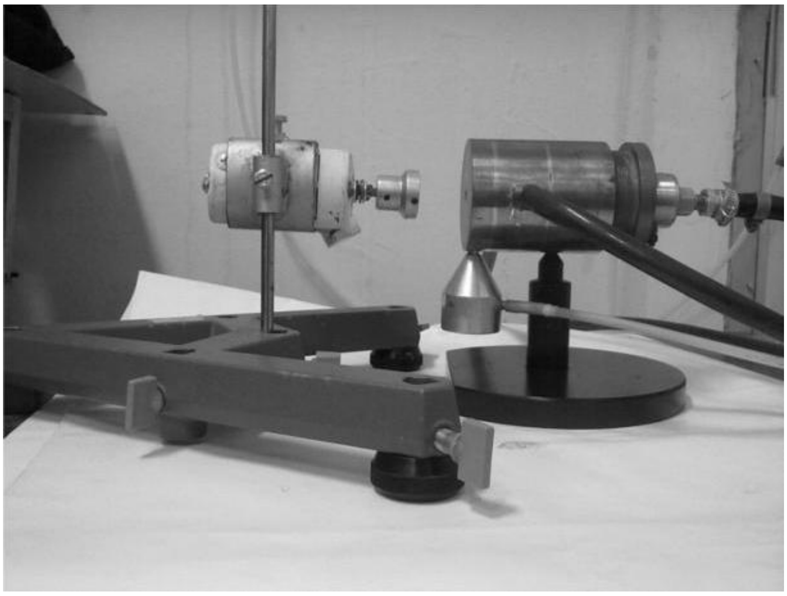

Figure 1 - Experimental apparatus used for coating of Ta_2O_5 + Al

For the deposition of coatings obtained from the plasma torch was used 0.5 g of powder mixture with the substrate distances of 30 mm and 50 mm and with rotating the substrate and at rest. Table 1 shows the conditions used in this work.

Table 1 - Conditions projection of Ta_2O_5 + Al powders

Distance (mm)	Substrate Temperature (°C)	Rotating substrate (RPM)
30	600	standby
30	600	6000
50	600	standby
50	600	6000

The substrate temperature was obtained through a pre-heating with the plasma torch. To measure the temperature used a Chromel-alumel thermocouple inserted into the bottom of the substrate with the sample port at rest. The substrate was coupled to the shaft of a motor 6000 rpm.

The surface morphology of the coating was examined using a scanning electron microscope Philips model XL 30 ESEM. For the characterization of crystalline phases was performed using a X-ray diffractometer Shimadzu XRD - 6000 model with CuKα radiation. The diffraction was performed with a scanning angle, θ - 2θ between 20° - 90° with a speed of 2 ° / min.

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3. RESULTS.

The micrograph of the powder mixture of Ta2O5 with Al milled for 6 hours in high-energy mill, shows that the larger particles are about 36 micrometre (Fig. 2). This value is the arithmetic average of the diameters of the larger particles shown in fig. 2, obtained using the image analysis software, Image-Pro Plus.

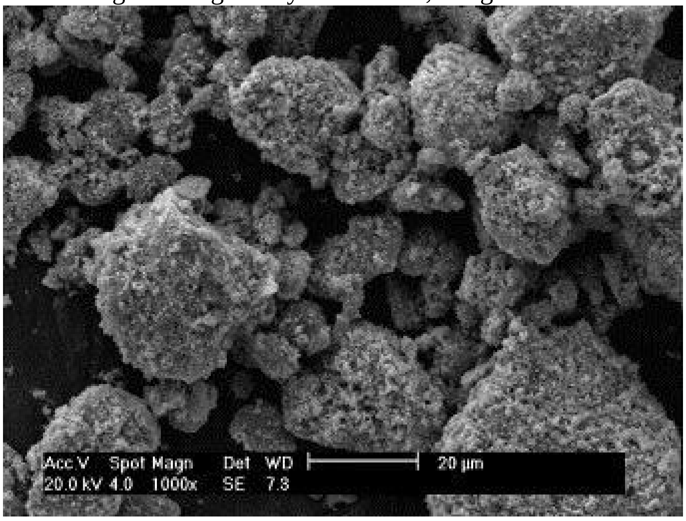

Figure 2 - micrograph of Ta_2O_5 + Al - milled for

The surface morphology of the coatings obtained under the different experimental conditions shown in the micrographs Fig. 3.

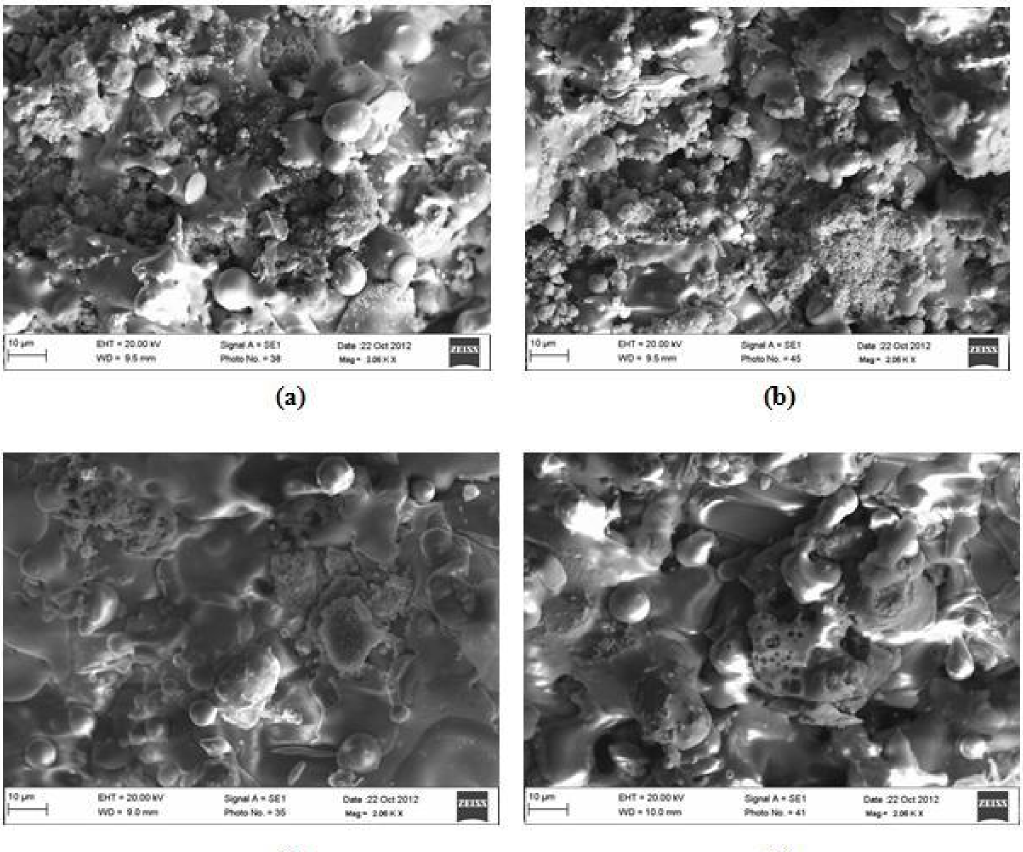

Figure 3 - Electron micrograph of the coatings produced in different conditions. (a) Fixed - 30mm, (b) Fixed - 50mm, (c) rotating - 30mm and (d) rotating - 50mm.

In a more detailed observation can be seen that the samples in rotation-fused particles had a higher scattering in the samples at rest, as indicated by the micrographs of Figure 3 (c) and 3 (d). However, there is no significant differences related to the preheating. These differences have only been observed with analysis by X-ray diffraction, as shown in Fig. 4, where there is a greater tendency for phase tantalum (Ta) samples with the rotation of the substrate.

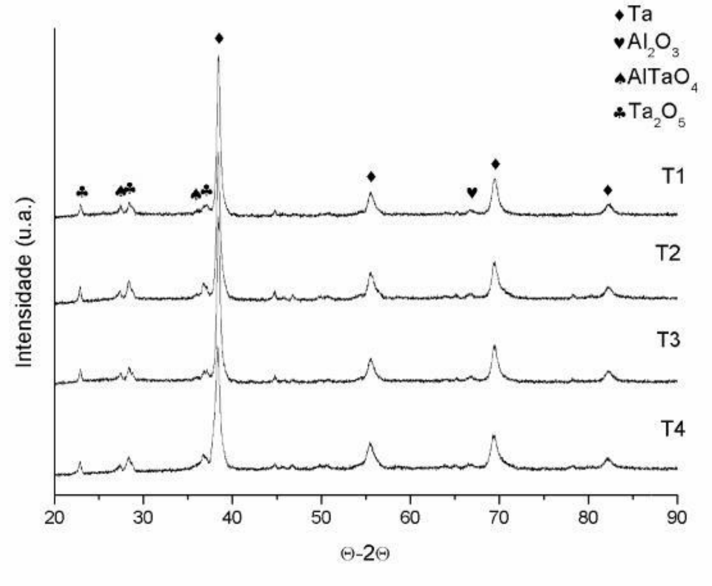

Figure 4: Analysis of X-ray diffraction of the samples sprayed. T1 - rotating 30 mm, T2 - Fixed to 30 mm; T3 - rotating 50 mm; T4 - fixed 50 mm

The analysis of X-ray diffraction (XRD) carried out (fig. 4) shows the diffraction patterns of the four conditions used in this work. It can be observed the presence of aluminium oxide, Al_2O_3 resulting from aluminothermic process. The tantalum pentoxide this shows that not all of it was reduced resulting residue oxides. However, most of the structure is composed of pure tantalum metal. This analysis was similar to that obtained by Mendes [13].

4. CONCLUSION.

1. The milling produced particles containing both reagents, forming a large contact area. This increases the reactivity of the mixture and facilitates the spread of the reaction. Each particle reacts separately.

2. Atmospheric plasma torch may be used to promote the aluminothermic reduction of tantalum oxide.

3. The SEM analysis shows that the rotation substrates, were used with a higher level of cast Al and impregnated RT.

4. The identification of the crystalline phases performed by X-ray diffraction (XRD) was similar, or may be different for quantitative analyses.

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