# EVALUATION OF CERAMICS TITANIA COVERAGE DEPOSITED BY THERMAL SPRAY AT PLASMA ON GRADE FIVE TITANIUM ALLOY

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***Abstract.*** *The grade five characteristics of titanium alloy are, good corrosion resistance in biological environment combined with an excellent degree of biocompatibility, made it an interesting material used as biomaterials in the fields of dentistry and orthopedics. The long-term biological effects of metal ions due to leaching of the implants are not completely understood. It is known that the titanium ions are considered carcinogenic chemicals agents, aluminum ions cause neurological disorder and vanadium ions are associated with enzymatic disorders, among other problems. The coating of titanium alloys for titanium oxide (titania) can act as a chemical barrier for the ions leached from the metal alloy, besides being a good osseoindutor. The mechanical strength of a ceramic roof made of biomaterials by plasma thermal spraying includes the grip between the substrate (adhesion) as well as the cohesion between the deposited particles. This study evaluates the strength of a ceramic roof titania bioinert in a metallic substrate (titanium alloy), deposited by atmospheric plasma thermal spraying, through adhesion testing, bending and scanning electron microscopy. Coverages were obtained in good quality with low porosity, low non-fused particles and with good adhesion.*

***Keywords****: adhesion, the plasma thermal spray, ceramic roof, titanium alloy.*

## 1. INTRODUCTION

Coatings can be prepared by various physical and chemical methods, these methods among the atmospheric plasma thermal spraying is a technique of coating the dry successful and versatile in the range of materials that allow to be deposited, such as metals, ceramics and polymers. The thermal spray process is widely used in applying coatings of oxides. Since this process occurs at high temperatures, where cast particles or half cast one adhere to the substrate can occur localized melting. It is also recognized for its low environmental impact compared to wet processes (Lima, 2001).

The appropriate characterization of thermal sprayed coatings is of great importance for its choice and development. This characterization can be accomplished by several tests that seek to determine the adhesion of the deposited layer to the substrate and its wear resistance, its ability to propagate cracks or retain brokens, their microstructure, hardness and others. The best knowledge of the coverage is necessarily go through their characterization (Lee, 2004). Among the forms of analysis to highlight the adhesion assays relatively common in scientific studies of thermal spray (Lima and Trevisan, 1999).

In this work tests were used scanning electron microscopy, bending and adhesion of the coating of titanium oxide deposited by thermal spray process to atmospheric plasma in titanium followed by vacuum heat treatment in order to create and characterize a chemical barrier that prevent the leaching of ions from the substrate at the same time facilitates the osseointegration. Thus, the problem in question is of significant scientific importance, deserving therefore be studied scientifically, since the conclusions inferred from this study may come to be applied in the implants area.

## 2. MATERIALS AND METHODS

The flowchart in fig. 1 presents the stages of the experimental procedure adopted in this paper.

### 2.1. Powder and specimen preparation

The material used as the substrate was Ti-6Al-4V from the market and characterized by XRF to verify the chemical composition. The substrates were basically two formats as a requirement of the standard:

***Flat rectangular samples*** - made according to ASTM C1161-02, ie, small plates of approximately 3.0 x 5.0 x 50.00 mm, with deposition being performed in the largest surface;

***Cylindrical samples*** - made according to ASTM C633-08, or 25.4 mm (1 inch) diameter and 25.4 mm in length with the deposit being made on a flat part.

Substrates suffered deposition of titania ($TiO2$) commercial Metco 102, supplied by Sulzer Metco (USA). The particle size of this powder, marketed for applications in the plasma spraying, is in the range 7.8 to 88 micrometers. Before the thermal spray powder was characterized by XRF, and microscopy (SEM), to make sure its composition, particle size and morphology.

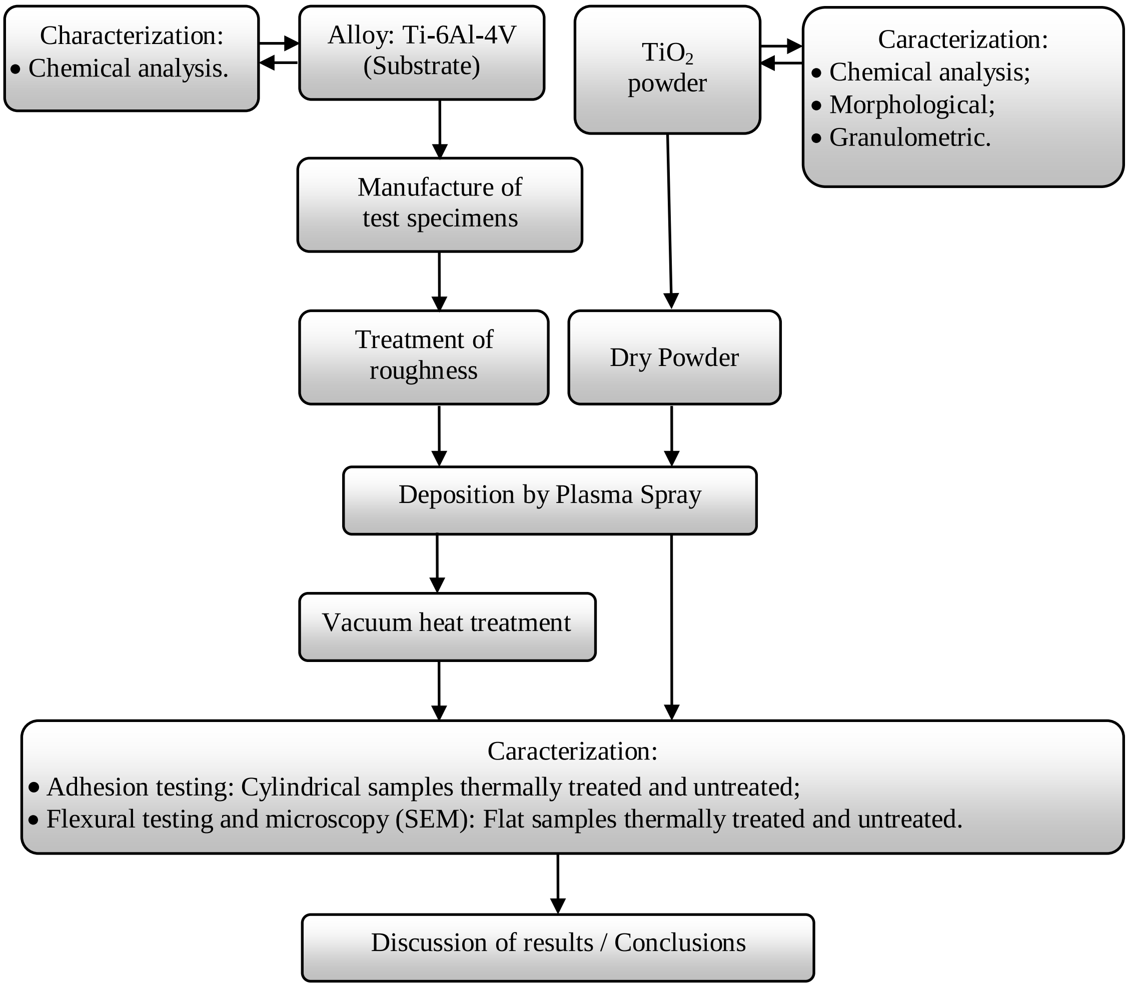

Figure 1. Flowchart of procedures

### 2.2. Deposition and heat treatament

In the deposition of titania powder was used for thermal spraying equipment plasma (Plasma-Spray) to produce coatings that were later characterized. These coatings were performed at the Laboratory of Thermal Spraying Plasma Department of Materials Engineering (DEMA), Faculty of Mechanical Engineering (FEM), UNICAMP, and the machine gun thermal spray plasma METCO 3MBII model. The coatings were applied manually. The gun has radial supply of powder and works with low to medium power levels, reaching up to 40 KW (500 A and 80 V).

For thisEquipment the current range used of operation is 200-500 A. Preliminary studies showed that the use of a stream near the limit of the equipment (ie 500 A) led to the deterioration of the electrodes and the premature loss of the torch nozzle, especially at the anode (Cu). This limits the current practice of using around 400 A. On the other side for currents below 250 A deposition does not occur, due to the low heat generated. Thus the current range to be examined was 300 to 400 A [Silva, 1998].

Before proceeding to the deposition, the substrates were sandblasted with particles of alumina ($Al_2O_3$) between 2 and 38µm at a pressure of 75 psi, 90 ° angle to provide the substrate roughness, eliminating thus the oxides that could interfere with coating adhesion and facilitating the mechanical anchorage of $TiO_2$. After blasting, all samples were cleaned with compressed air to remove the alumina sprayed on the surface, followed by the deposition of $TiO_2$.

The deposition time was 30 seconds to preheat the substrate and the coating for 2 minutes. After each deposition the system coating / substrate was allowed to cool to room temperature.

It was decided, in this work due to selection of parameters provided by METCO, which has already studied these values and recommends its customers.

The coating thickness was controlled by deposition time, since the samples were too small. With the parameters presented a deposition time of 2 minutes (3 passes), assured a thickness of 120µm.

Before the characterization of the coating portion of the samples were annealed in an oven consisting of a high vacuum system that reaches 10-6 Torr. The part to be treated is immersed in an argon plasma to reach the desired temperature, which can range from room temperature to about 2,000 degrees. The plasma is established through a high voltage DC negative, which can reach up to 10,000 volts. The system (furnace) was designed and built at the Physics Institute of Unicamp (IFGW). The heat treatment took place at 850C for 3h. Fig. 2 shows images of the treatment of high heat.

Figure 2. System vacuum heat treatment.

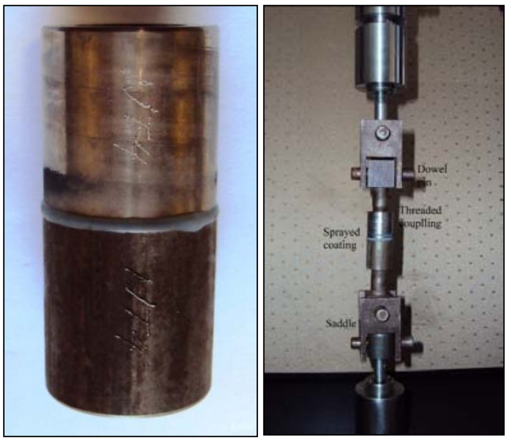

Figure 3. Image of adhesion testing.

## 2.3. Adhesion testing

The adhesion testing was performed according to ASTM C633-08. This essay is the most commonly used in coating thermal spray. It is a simple assay where the samples are cylindrical with a diameter of 25.4 mm and 25.4 mm in length. The cover is placed on a flat face, pasted on the cover and a body-of-proof similar to that used as substrate. Glue was used in an epoxy resin Scotch-Weld 3M 2214, high adhesion power and given the conditions of the standard. The assembly is mounted on a self-aligning device, to avoid shear stress during the test. This set and then pulled into a machine of conventional tensile tests, verifying that the failure charge occurs, the examination of the bodies-of-prove indicates that type of failure occurred, adhesive or cohesive. Fig. 3 shows a picture of the overall adhesion assays.

## 2.4. Bending test

The flexion assay was conducted according to ASTM C 1161-02, wherein the prove-of-bodies are thin and can take cross-section circular, rectangular or square, rectangular section is that it was used in this work. It is of a low-cost and fast execution, but has the disadvantage of large variations in the level of resistance as a function of specimen dimensions and prove-of-the mode of loading. We decided to evaluate tension of flexion rupture, the test on four points because it further extension of the element being tested is subjected to maximum bending moment. Fig. 4 shows the bending test.

Figure 4. Picture of the bending test.

## 3. RESULTS AND DISCUSSION

### 3.1. Chemical composition by X-ray fluorescence

The Table 1 shows the amount of each element in the substrate, where it is observed the presence of titanium, aluminum and vanadium, very small amounts of iron, silicon, sulfur and phosphorus, which together account for 0.23% alloy composition called impurities.

Table 1. Chemical composition of Ti-6Al-4V by x-ray fluorescence.

| Composition | Weight % | Composition | Weight % |
|---|---|---|---|
| Ti | 91.2858 | Si | 0.0475 |
| Al | 4.4712 | S | 0.0059 |
| V | 4.0109 | P | 0.0053 |
| Fe | 0.1735 | --- | -------- |

The Table 2 shows the amount of the composition of each element of the powder coating used in this work. This table (Table 2) can be seen that although the presence of other chemicals, or impurities present in the powder coating (TiO2). The titanium and oxygen, which are components present in the titania, more numerous, amounting to a percentage of 97.40%, while the remaining elements, ie the impurities totaling 2.60%.

Table 2. Chemical composition of titania by x-ray fluorescence.

| Composition | Weight % | Composition | Weight % |
|---|---|---|---|
| Ti | 55.5173 | Al | 0.0448 |
| O | 41.8794 | Na | 0.0214 |
| C | 1.4612 | Ca | 0.0182 |
| V | 0.2928 | W | 0.0165 |
| Nb | 0.2864 | Ni | 0.0087 |
| K | 0.1707 | Zr | 0.0066 |
| P | 0.1042 | S | 0.0053 |
| Fe | 0.0913 | Cu | 0.0035 |
| Si | 0,0717 | ---- | --------- |

### 3.2. Scanning electron microscopy

Fig. 5 shows the Metco 102 powder, fine powder is a hard and brittle, obtained by melting and grinding, with more than 97% composition, the remainder being made up of impurities. The particles are angular, with sharp corners well, have a smooth surface and very low porosity, particle size of about 50µm. In Fig. 6 there is the aspect of coverage with lamellar particles fully melted and the presence of cracks in small quantities.

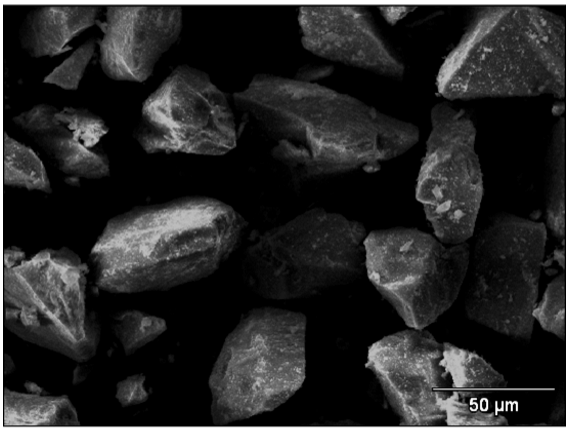

Figure 5. Micrograph (SEM) of powder titania.

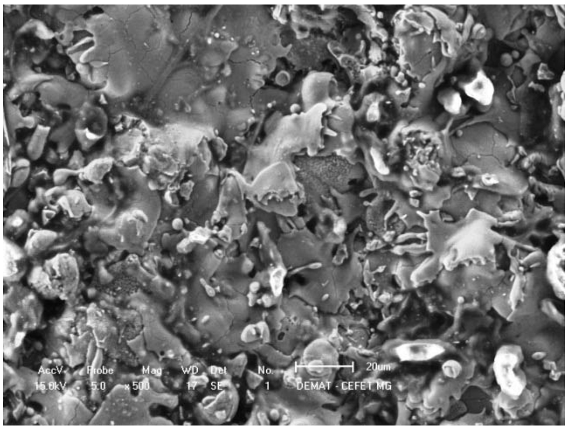

Figure 6. Micrograph (SEM) of coverege..

### 3.3. Adhesion testing

Figure 7 presents the results of adhesion testing, it is observed that the heat treated samples have higher resistance to fracture those not treated, and the sample number 5 its value had doubled, the resistance increase is due to diffusion of alloying elements of the substrate for coating.

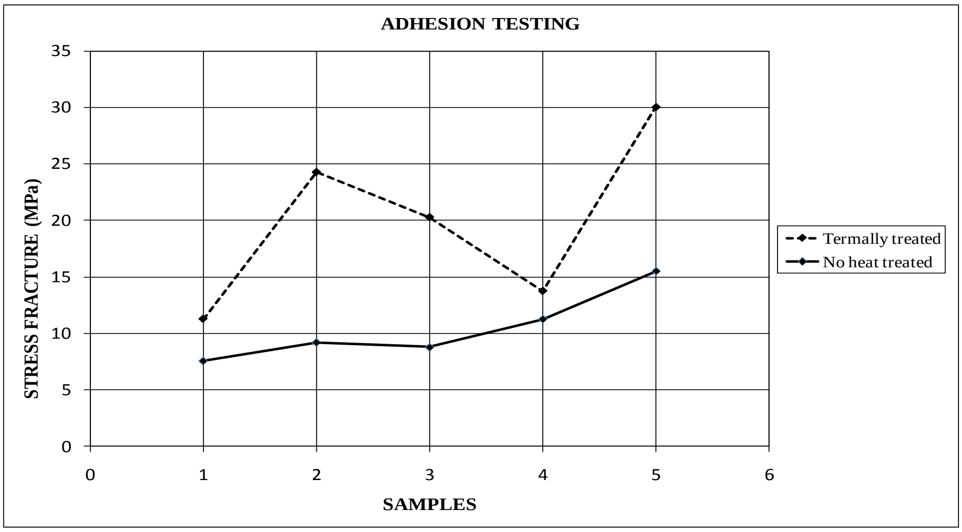

Figure 7. Results of adhesion testing.

### 3.4. Bending test

Figure 8 shows the results of bending test, in which the values of fracture stress for the treated samples are higher than the untreated is presented to reinforce the adhesion testing.

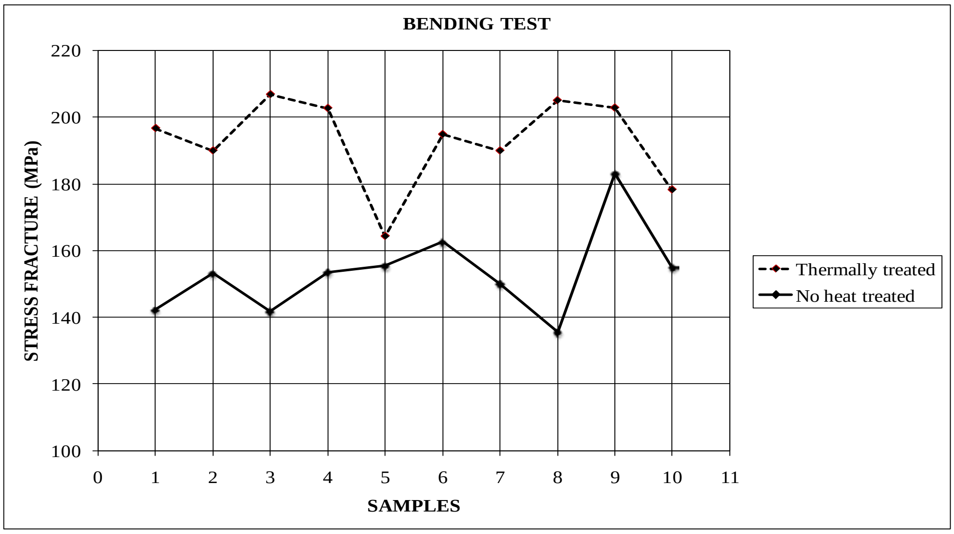

Figure 8. Results of bending test

## 4. CONCLUSIONS

Samples that have not undergone heat treatment after the coating had values of fracture stress lower the heat treated samples in the tensile test and bending, probably due to diffusion coating on the substrate and the partial relief of residual thermal stresses on the coating. The heat treatment after the coating is necessary in this type of material, it increases the resistance to fracture and relieves the residual stresses of the coating. The study shows that these two types of mechanical tests are very important in the evaluation of ceramic coatings on metallic substrates by plasma Sray therefore express resistance to certain types of efforts.

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