

## **PROPERTIES OF TITANIUM NITRIDE LAYERS DEPOSITED BY PLASMA THERMAL SPRAYING AND HVOF METHOD**

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### **Abstract**

The paper presents the results of titanium nitride (TiN) deposited layers on titanium alloy substrate by plasma thermal spraying (APS) and by high-speed oxy-fuel thermal spraying (HVOF). The thickness of the deposited layers have values between 45 and 60  $\mu\text{m}$  for plasma thermal spraying process respectively between 35-51 $\mu\text{m}$  for HVOF method. After the scanning electronic microscopy analysis it was observed that the titanium nitride layers deposited by HVOF method processes present not thermal spraying defects in comparison with the layers deposited by plasma thermal spraying where it was observed defects on the surface of the samples as cracks and microcracks.

### **1. Introduction**

Thermal spraying domain is used in many engineering areas due to the wide range of materials which can be used and to the benefits provided to the components (wear and corrosion resistance, aesthetic). Thermal spraying coatings technologies of composite, ceramic or metal-ceramic layers are relatively new, with related problems due to incompatibility between the deposition layer and the base material (substrate).

The thermal spraying coatings produces benefits to the rolled or cast materials due to the surfaces porosity obtained which acts as a reservoir of lubricant and of the amorphous phases that leads to increased wear resistance. Remarkable results are also obtained in layers subjected to thermal shock by using ceramic materials deposited by thermal spraying [1]. Due to high hardness, dielectric properties, resistance to high temperature, ceramic materials are used in many domains: mechanical engineering, aeronautics, chemical, deposition of biocompatible materials in medicine.

Titanium alloys have good corrosion resistance and good mechanical properties but their wear resistance is low. Usually, components made of titanium alloys subject to wear and corrosion are coated with ceramic materials with high mechanical properties, produced by different methods (laser, thermal spraying). Titanium nitride (TiN) is a ceramic material used in various applications solicited to wear and corrosion due to its high mechanical properties [2]. Titanium nitride is deposited on the active edges of cutting tools, increasing their lifetime at least three times.

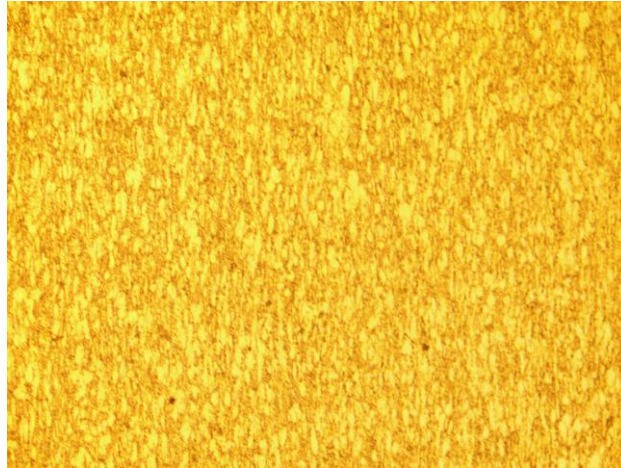
Due to the similar color of gold, titanium nitride is also used to cover some parts of cars. Also, titanium nitride is not toxic, it is approved by FDA (Food and Drugs Administration) to be used at manufacture of medical devices (covering hip joints, knees, dental implants) [3]. Typically, titanium nitride is deposited by PVD process, but realizing with high cost and thin layers [4]. The paper presents the experimental results on depositing titanium nitride (TiN) by two thermal spraying methods: APS (Atmospheric Plasma Spraying) and HVOF (High Velocity Oxy-Fuel).

### **2. Materials used**

For experiments was used titanium nitride powder with the dimension of the particles between 5 and 15  $\mu\text{m}$  and titanium alloy substrate (Ti6Al4V). Table 1 presents the chemical composition of titanium alloy and in Figure 1 is showed the microstructure.

**Table 1. Chemical analysis of Ti6Al4V alloy**

Chemical element	Ti	Al	V	Mn	Fe	W
	88.79	6.50	4.17	0.23	0.11	0.08



**Figure 1. Microscopic image of the titanium alloy**

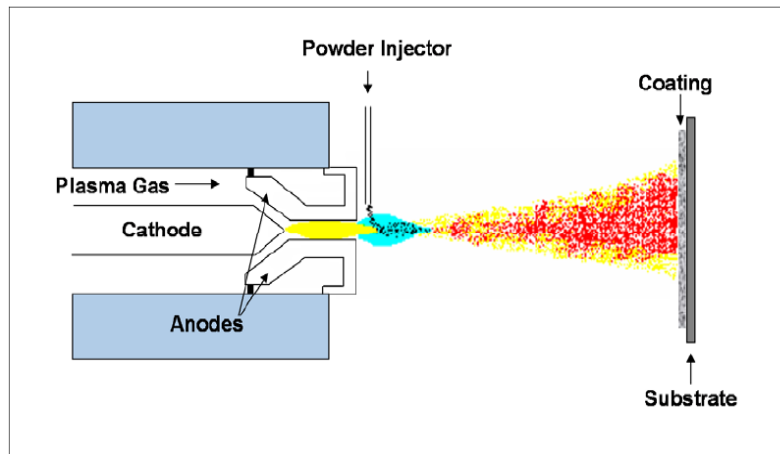
#### *Sample preparation*

A thermal sprayed layer is characterized by: adhesion, structure, density or porosity. The adherence is one of the basic characteristics of the deposited layers which confer strength, durability and protection of the piece that is applied. It is defined as a complex of factors which contributes to the connections between the deposited layer and the base metal.

The sample preparation deposition of thin layers by thermal spraying is an important operation in obtaining of adequate coatings with high adhesion on substrate. Before thermal spraying process the samples are blasted with electrocorindum with the dimension of the particles between 0.8 and 2 mm at a pressure of 5 bars and the blasting distance by 50-60 mm. After blasting the samples were cleaned and degreased with alcohol.

### **3. Deposition of titanium nitride by plasma thermal spraying**

Plasma thermal spraying principle (Figure 2) consist in insertion of a material, usually in powder form, in the plasma jet generated by the electric arc induced between the tungsten electrode and the copper nozzle of the plasma generator. Due to high temperature ( $\approx 15000^{\circ}\text{C}$ ), the material melts and is driven by the gas stream to the workpiece. Particles reach the surface of the piece in plastic or melted state and adhere to it by specific mechanisms [5].



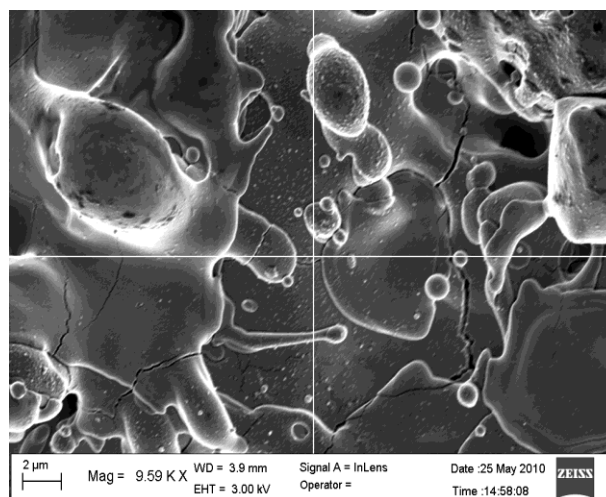
**Figure 2. Plasma thermal spraying principle [5]**

For the experiments it was used Sulzer-Metco thermal spraying equipment from National Research and Development Institute for Welding and Material Testing, ISIM Timișoara. The parameters used for the deposition of titanium nitride layers by plasma thermal spraying are inserted in table 1.

**Table 2. Parameters used for the plasma thermal spraying deposition process**

$I_p$ [A]	$U_a$ [V]	$Q_p$ [l/min]	$Q_{tr}$ [l/min]	$m_p$ [g/min]	$d_p$ [mm]	No passes
300-330	80-100	40-45	6-7	15-17	150±5	3

The thickness of the deposited layers by plasma thermal spraying method determined with the Easy Check device has values between 45 and 60  $\mu\text{m}$ . The average roughness (Ra) determined by SurfTest 201 (SJ-201) device has values between 3.80 and 4.72  $\mu\text{m}$ . In figure is presented the SEM image of titanium nitride layers deposited by plasma thermal spraying method.



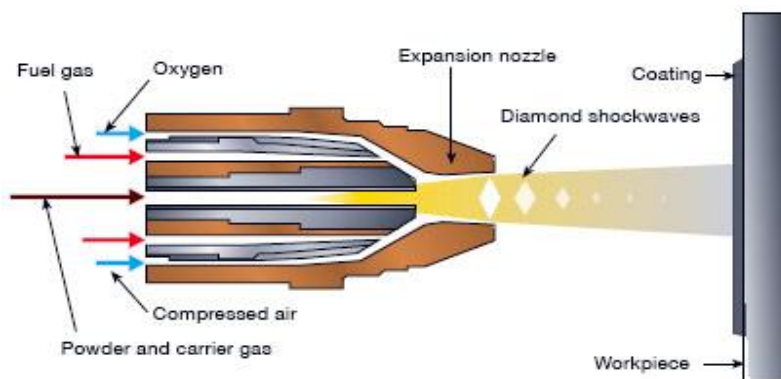
**Figure 3. SEM image of titanium nitride layers deposited by plasma thermal spraying method**

The microscopic analysis of the titanium nitride layer deposited by plasma thermal spraying shows that the coatings presents defects such as cracks and microcracks. After the deposition of titanium nitride layers on titanium alloy substrates (Ti6Al4V) it was

observed that the layers have a low adhesion to the substrate and show a high degree of oxidation. This is due to high temperatures during the thermal spraying process ( $\approx 15\ 000^{\circ}\text{C}$ ).

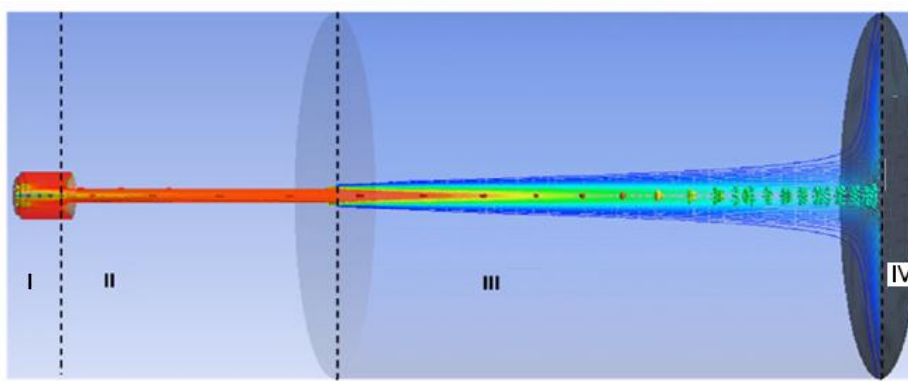
#### 4. Deposition of titanium nitride tin layers by HVOF thermal spraying method

HVOF (High Velocity Oxy-Fuel) is a modern thermal spraying method which has been developed to produce very high speeds of the jet. Gas stream having a temperature of  $2800^{\circ}\text{C}$  and speeds of  $2500\ \text{m/s}$  accelerates and plastify the metal powder particles up to  $800\ \text{m/s}$ , the powder being projected on the substrate. For experiments was used the HVOF thermal spray equipment and thermal spraying gun, ID Cool Flow, gun providing a lower temperature than other flame spray guns, resulting a lower level of oxides. In Figure 4 is showed schematically the HVOF thermal spray gun.



**Figure 4. Principle of HVOF thermal spraying gun [6]**

Due to the temperatures developed during the thermal spraying process, the powder reaches in plastic or melts state and is driven at high speeds by the gas jet directly to the substrate. As powder carrier gas was used nitrogen. HVOF thermal spraying process has four main physico-chemical steps which occur in heat flow (Figure 5) [7].



**Figure 5. Main physico-chemical steps which occur at HVOF method [7]**

In step one the chemical energy is transformed into heat by oxidation of the fuel into the combustion chamber. In stage two is converted the heat energy into kinetic energy and transferred to the particles, the gas stream pressure is obtained in step 3. The lamellar particle shape is due to the kinetic and thermal energy conversion phenomenon (step 4) where the spherical particles are deformed after the contact with the substrate surface.

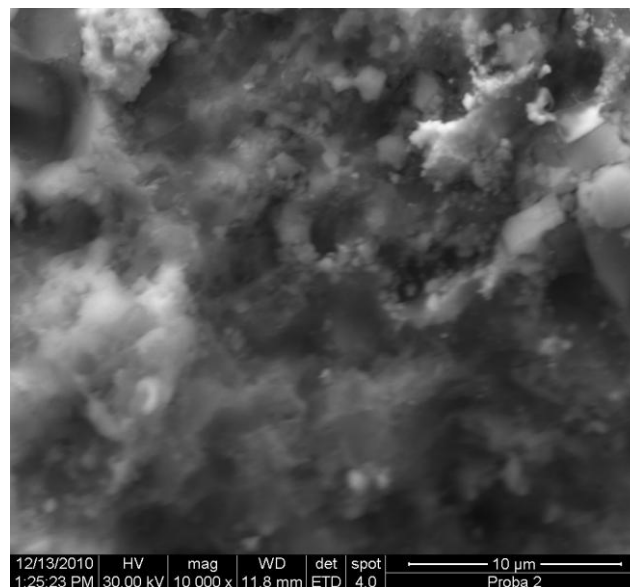
For the deposition of biocompatible hydroxyapatite layers it was used the HVOF thermal spraying equipment from the Politehnica University of Timisoara. The parameters used for the deposition of titanium nitride layers are inserted in table 3.

**Table 3. Thermal spraying parameters**

Oxygen l/min	Hydrogen l/min	Kerosene l/h	Transport gas l/min	Deposition rate g/min	Spraying distance mm
300-320	90-95	2.8-3	15-20	15	70-80

The thickness of the layers deposited by HVOF thermal spraying method has values between 35 and 51  $\mu\text{m}$  and the average roughness ( $R_a$ ) has values between 3,38 and 4.42  $\mu\text{m}$ .

Is Figure 6 is showed the scanning electron microscope analysis of the titanium nitride layer deposited by HVOF thermal spraying method.



**Figure 6. SEM analysis of the titanium nitride layer deposited by HVOF thermal spraying method**

After the scanning electron microscopy analysis it is observed that the deposited layers by HVOF method presents not defects such as cracks or microcracks, specific to thermal spraying processes. This is due to relatively low temperatures of HVOF method ( $\approx 2800^\circ\text{C}$ ), the particles reaches in plastic state and due to high speeds of the gas stream during the spraying process are obtained dense and compact layers, without cracks.

## 5. Conclusions

5.1 In the paper are presented the experimental results obtained by depositing titanium nitride layers by two thermal spraying methods (plasma thermal spraying and high velocity oxy-fuel)

5.2 Titanium nitride is a ceramic material with high mechanical properties, which is used successfully to cover components subject to wear and corrosion conditions

5.3 The scanning microscopic analysis shows that titanium nitride layers deposited by plasma thermal spraying showed defects as cracks due to high the temperatures during the thermal spraying process

5.4 Due to relatively low temperatures of HVOF thermal spraying process used, were successfully deposited layers of titanium nitride on titanium alloy, with relatively low level of oxides, the microscopic analysis shows that the deposited layers present not defects as cracks or exfoliation attesting that this method is optimal for deposition of titanium nitride layers

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