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Assessment of Hard Thin Layers Deposited by Plasma Spray on Hydroboration

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Abstract. Chemical mixers used in pharmaceutic industry are mostly constructed from a series of iron, chrome and nickel alloys. These mixers are used for mixing corrosive chemicals (acid or base) that often contain solid particles acting as the abrasive particles on the mixer blades. In this paper, martensitic stainless steel AISI 415 is used as base material and powder based on tungsten carbide is used for deposition of hard thin film resistant at common action of abrasive particles. The properties of metallic materials can be improved by deposition of layers with special material by plasma spraying method. Microstructure of thin films made of tungsten carbide and base material was performed with Scanning Electron Microscopy (SEM). In order to determine the micro hardness of the base material and the deposited layer, Vickers micro hardness testing have carried out. For determination of roughness of the deposited layer, tests were carried out using the equipment Mitutoyo SJ 301.

1. Introduction
Along classical technologies for obtaining special thin films [1-2], modern techniques to realize thin films by chemical and physical procedures [3-6] were perfected and developed, assuring high hardness and adhesion.

The base material is martensitic stainless steel AISI 415 which has low resistance to hydro abrasive wear. In industry, corrosion and wear are responsible for the maintenance and loss of productivity resulting from the shortened life of components, and the catastrophic failure of structures leading to massive costs of replacement and litigation [7-9].

This type of steel is used for realization of blades from mixers for fluids used in chemical, petrochemical and pharmaceutical industries, of blades from mixers from biogas installation in order to maintain the homogeneity of the substrate, so that the gas shall be released equally from the processed mass.

Erosive wear is caused by hard particles entrained in a fluid or a gas stream striking the surface [10]. Therefore, use of wear resistant materials in the form of coating/cladding is a pragmatic solution to combat wear [11-15].
2. Materials and methods

The base material has been cropped from a chemical mixer’s blade from pharmaceutical industry using Metacut M250 equipment in order to obtain specimens for deposition, roughness and microhardness tests. The specimens have 25 mm width, 25 mm length and 5mm thickness. The chemical composition of martensitic stainless steel AISI 415 was determined with Foundry Master Spectrometry in order to achieve the best precision and is Fe – 80.6%; C – 0.10%; Cr - 13%; Ni – 4.38%; Mn – 0.63%; others – 1.8%.

The analysis by SEM were carried out in order to analyses the surface and the microstructure of the base material. The SEM image is presented in Figure 1 showing elongated grains of Fα with acicular martensitic structure.

![Figure 1. SEM analysis of AISI 415 stainless steel specimen.](image)

Tungsten carbide powder is used for deposition because are compact, with average roughness, high hardness and wear resistant. These depositions have high resistance to hydro abrasion and friction. Chemical composition of the powder is: Co ≈13.0%; C ≈ 4%; W ≈ 83%. Due to cobalt percent over 12%, the depositions are more ductile but with higher hardness and good resistance to abrasion and friction.

![Figure 2. SEM analysis of WC powders and their dimensions.](image)
The plasma spraying methods is carried out using Sulzer Metco 9 MCE equipment from the Faculty of Mechanical Engineering, Technical University Gh. Asachi Iasi. Due to high temperature of plasma jet, between 10000-16000°C, the tungsten is melted well realizing compact surface and due to high speed of plasma jet over 3000 m/s, compact layers can be obtained [16].

3. Results and discussions

In this paper, thin films based on tungsten carbide powder were deposited by plasma spraying on a stainless steel AISI 415.

In order to increase the lifetime of chemical mixer’s blades [18-20] made from AISI 415 stainless steel used in pharmaceutical industry, hard thin films were deposited by plasma spraying to resist to hydroabrasion. Roughness tests, adherence and micro hardness tests were carried out to determine how these parameters influence the hydro abrasive wear of chemical mixer’s blades.

SEM analysis of the deposited layer has been carried out using VEGA II LMH microscope produced by TESCAN having an EDX module – TAX QX2 type with which the microstructure, the thickness of deposited film as well as the area of droplets formed on the deposited surface were determined.

![Figure 3](image)

**Figure 3.** EDX analysis of the surface area on which the distribution of chemical elements in the sample deposited with tungsten carbide (WC) powder by plasma spray method has been determined: (a) the sample area; (b) distribution of elements.

From SEM image, it can be observed that the aspects are not very rough surface profile, having few detachments, micro cracks and un-melted powders.

| Elements   | %   |
|------------|-----|
| Wolfram    | 76.37 |
| Carbon     | 9.84  |
| Cobalt     | 6.97  |
| Oxygen     | 4.40  |
| other      | 2.42  |

**Table 1.** Chemical composition of the sample deposited by plasma spray methods using WC based powder determined by EDX.
The analysis by EDX presented in Figure 3 emphasizes a compact deposition of tungsten carbide. On the surface, large areas with oxides (dents) and flattened droplets of cobalt appears, offering information about the fact that the powder contains separately particles of tungsten carbide and cobalt [21-25].

The plasma spraying method create no transitional structures of melted material layer-substrate but only structures formed by flattened droplets due to the dynamic of spraying which are soldered to the surface of the specimen. In Figure 4 is presented the cross section of deposited film.

![SEM analysis of thin film thicknesses deposited with tungsten carbide by plasma spraying method: 1 - layer, 2 - a substrate.](image)

**Figure 4.** SEM analysis of thin film thicknesses deposited with tungsten carbide by plasma spraying method: 1 - layer, 2 - a substrate.

From the profile analysis made with Scan Explorer Nanofocus Profilometer, on the sample deposited with tungsten carbide powder by plasma spraying, presented in Figure 5, it can be observed that the deposition has not very rough surface, having few peaks of maximum roughness, closed as magnitude.

![Roughness analysis of film deposited with tungsten carbide (WC) by plasma jet spraying.](image)

**Figure 5.** Roughness analysis of film deposited with tungsten carbide (WC) by plasma jet spraying.
The small value of roughness Ra of 3.86μm is due to the powders of tungsten carbides that have small grains dimensions and which can be deposited as very fine surface, reducing or eliminating the finishing operations.

4. Conclusions
The SEM analysis of layers deposited with powder WC on the basis material AISI 415 present a surface with fine granular aspects with micro particles non-melted embedded into the surface without micro exfoliating and evident micro adherence.

The roughness at deposition with plasma jet is influenced most often by the powder granulation. At deposition with powders based on WC, small roughness have been obtained (Ra= 3.86μm), because the grains of WC powders are very small (i.e. 8µm).

From the SEM and optical microscopy analysis of cross section, it can be observed that the specimens with deposition by plasma jet method presents intermetallic compounds, almost compact between the layer and substrate, leading to a good adherence of the coating.

By plasma jet spraying, hard layers can be deposited, at different dimensions, depending on the number of plasma jet passing over the surface to be deposited. The deposition process is automatic, obtaining large deposited surface in short time.

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