Properties of Al₂O₃ and NiAlSi coatings obtained by atmospheric plasma spraying on 34CrNiMo₆ substrate

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In military and mining industry are many machine elements which are subjected to thermal and structural analyses. Idlers must have a high wear resistance, because the functionality of the entire transmissions systems depends on them. Since the maintenance costs of the machines are very high, here we propose to improve the performances of the machine elements and also improve the physical, mechanical and chemical proprieties of those elements. We have chosen the steel type 34CrNiMo₆, MoCN class, STAS 691-880 as basic material and coated it with NiAlSi and Al₂O₃ powders. The prepared samples are analyzed by XRD, EDS and SEM. The NiAlSi and Al₂O₃ coating deposited on steel substrate, is dense, but on SEM analysis we discovered that it has some fine cracks on NiAlSi deposition, and good elasticity of the layer, but also we could consider that the layer has no cracks because of the good deposition process has resulted for Al₂O₃. The deposition process should have been made by heating the substrate, to expand, in order to return to original shape simultaneously to the NiAlSi and Al₂O₃ molten particles solidification.

Keywords: Coatings, Atmospheric plasma spraying, Scanning electron microscope, XRD

For increasing machine elements durability, we made a study of their damage, which led to finding solutions for the surface modification. After a detailed analysis of the materials used in machine elements technologies, we considered that their properties could be improved if we cover them with thin layers but with high properties. In this paper, we present the plasma spraying deposition on idlers and conductive wheels which are parts of the tracking system of a tank or military machinery, or any kind of construction equipment which are using those. The equipment is comprised typically from two driving/conductive rollers and rollers guide/idlers with different forms and sizes, starting from tenths of millimetres up to about 12 m and is used in all fields.

Driving conductive rollers and rollers guide/idlers are machine elements which have on their periphery a contact area subjected to contact pressure, corrosion, fatigue, friction, bending, called the surface of revolution.

The continuous process of contact between the rollers of system machinery, in order to ensure continuous movement of the two driving and guide rollers/idlers need to have good physical, mechanical and chemical proprieties, that is why we choose two alloys and also two types of special deposition powders, and did all the preparations, plasma deposition process, analyses, comparisons between the results. We also purposed that all our experiments may have a useful outcome which could be used by the military industry. Considering that all those machines are operating in special conditions, the driving and guiding rollers can be constructed in a wide variety of materials depending on the tasks which require the contacts, the total time of operation of the gear unit and the guide, the speed and precision, and further requirements may be imposed on certain gear (resistance to temperature, corrosion, explosion and so on).

From the variety of alloyed steels we selected steel type 34CrNiMo₆, which is the most used, because of its high properties. For coating, we have chosen NiAlSi and Al₂O₃. Thermal spray process in plasma deposition is achieved by melting powder material, which is sprayed on the covering surface with a jet of plasma. Material particles, molten reach the substrate surface and solidify, but due to different elasticity of the two materials, cracks may occur in the deposited layer, caused by the phenomenon of contraction.

Experimental Procedure

Plasma spraying process is made with a gun which is the most important element of the facility where we did all the coatings. The gun consists of a copper anode...
(nozzle) and a tungsten cathode and inert gas (plasma) through the nozzle and near the cathode. Between anode and cathode, by high voltage discharge (10000 V) it forms a continuous current arc that ionizes plasma gas (inert), leading to good electrical conductivity thereof. When gas passes through continuous current arc between anode and cathode inside the gun, it dissociates and ionizes to form plasma and is used as a source of concentrated heat. At the exit of the nozzle gas ions recombines, yielding the energy absorbed in a very short time, leading to the formation of a plasma jet with a temperature of 10000-16000°C. The powder is injected directly into the plasma jet which accelerates it to surface area. When it solidifies on the surface, we obtain the coating. The coating thickness can be various if we are doing many passes. The operating principle consists in spraying the powder with a high speed to a substrate. The powder is heated and brought near to the melting point using the plasma created by the combustion gases and driven with high speed to the substrate by the transporter gas. The installation can be used for both micro production as well as for research.

The plasma SPRAYWIZARD-9 MCE plasma spraying deposition facility is shown in Fig. 1.

Other endowments associated:
- Ultrasonic bath UC-50: Capacity 600 mL; Frequency: 46 kHz
- Analytical balance AGN 200 - Assure accurate weighing, stable and fast, with automatic internal calibration: LCD graphic display, alphanumeric tastature, interface RS232, maximum amount of weights 200 g, precision of 0.0001 g, turntable diameter: 90 mm.

The plasma spraying deposition process is shown in Fig. 2.

Powder types used in plasma deposition process

The powder is the material, composed from particles of pure metal or alloys, the dimensions of those particles are between 0.1 - 1000 µm. The size of the particles used to produce the alloys on the industrial scale are a lot more reduced, between 1 - 400 µm.

Metal powders are characterized by a number of physical and chemical properties, which are largely, determine the final properties of the products obtained by aggregation of those powders. There are the following important physical properties:

Shape of the particles: It is estimated from the ratio between the three dimensions of the particles, distinguished three main groups:

- fibrous or acicular particles whose length far exceeds the other two dimensions \( l > b = h \);
- particle beams, plate whose two dimensions are of the same order of magnitude and far exceed the third \( l = b > h \);
- equiaxed particles (spheroidal, polyhedral) in which all three dimensions are almost equal \( l = b = h \).

Deposition of thin films, used as consumable powders, which according to their chemical composition, provides to the layers a certain properties. Anticorrosive coatings: nickel, chromium, titanium, etc., are compact, tight and basically are not changing the geometry of the protected base material. Tough webs: titanium nitride, carbon nitride, chromium nitride compounds extremely tough, ensures high resistance to all types of use under extreme conditions of temperature and chemically aggressive environment.

We have realized samples with rectangular geometry and dimensions 25x10x2. After we obtained samples geometries related facilities tried, we cleaned and degreased to be covered. Consulting specifications and manufacturers catalogs we found that NiAlSi and Al₂O₃ generates very good coatings. NiAlSi and Al₂O₃ coatings are recommended for applications with sliding wear, where they require a low coefficient of friction relative to...
Steel. NiAlSi and Al₂O₃ coating provides good abrasion resistance and high resistance to abrasion and scuffing.

Thermal deposition is a thermal process improvement using filler surfaces. According to deposition parameters, we can modify the properties of layers coated. The thermal depositions process was made under normal conditions of temperature, pressure and humidity. The deposition parameters are variable, and influence the quality of the coatings and mechanical properties as well.

**Results and Discussion**

The qualitative analysis has been made by scanning electron microscope (SEM), which produces images by detecting low energy of secondary electrons, emitted from the sample surface due to its excitation by the primary electron beam. In SEM analysis, the electron beam hit the sample surface and the detectors building an image by mapping the detected signal of beam position. The Quanta 200 3D is equipped with an EDAX acquisition system provided, and is able to make qualitative and quantitative elemental chemical analysis by EDX (energy dispersive X-ray) and crystallography analysis.

Quanta 200 3D Dual Beam electronic microscope, has two beams, electrons and ions. With the electron beams we can obtain images of more than 2 millions magnifications, and also the chemical composition, by the EDAX module. With the ions beam, we can obtain rapid and accurate grinding by various geometries (at µm) of sample material, revealing sub-surface structure, obtaining sections, deposition of layers, etc. Ion system also offers high resolution images. The chemical composition and SEM images of Al₂O₃ and NiAlSi powders are shown in Figs 3 (a,b) and 4 (a,b) respectively.

![Fig. 3- (a) The EDS spectrum and chemical composition and (b) SEM images (1000X) of Al₂O₃ powder](image)

![Fig. 4- (a) The EDS spectrum and chemical composition and (b) SEM images (1000X) of NiAlSi powders](image)
In Fig. 5a, it can be observed that coating has been deposited on the substrate with a constant thickness of 530 µm. The interface between the materials has no defects due to bond stresses from the coating. The layer has been coated uniformly on the substrate so we can approximate good behaviour in operating of the parts. Figure 5b shows a detail of coating microstructure overview, which is representative, for the experiment because we can observe that the coating has a high porosity, but there are no defects in layer. There are no inclusions, gaps or unmelted particles so we can estimate that the process has been correctly performed. During the deposition process the Al$_2$O$_3$ has a high temperature, but when getting in contact with the cold substrate, it solidifies very fast. During the solidification process arise the contraction phenomenon, which leads to the cracks appearance. The sample wasn’t subjected to any solicitation, and the layer has no cracks so we could approximate a good elasticity of the layer but also we could consider that the layer has no cracks because of the good deposition process.

Figure 6a shows a dense structure with a high porosity. On topographic surface, by measuring the roughness could be observed that the sample has a rough surface but has no imperfections. To increase tribological properties it is necessary to perform slight grinding on the surface, in order to decrease friction coefficient, and this operation is possible because the sample has no defects. Figure 6b shows Al$_2$O$_3$ coating chemical composition. Iron is present in the coating of the substrate and from this analysis we can conclude that there was a diffusion of both materials atoms during plasma spraying process.

XRD analysis was accomplished by X’PERT PRO MRD Diffractometer. With this facility we can determine the materials phases, the structural constituents, crystals orientation, lattice Miller indices, etc. The results are interpreted by X’PERT PRO MRD software, and finally we can obtain the material’s diffractogram. Figure 7a shows coating
lattice parameters which demonstrates that the most stable phase is \( \text{Al}_2\text{O}_3 \), and Fig. 7b shows that the same phase has the highest percentage in the coating composition. From this observation we could estimate that the coating has good bond stresses between the particles and its behaviour in operating is satisfactory.

In Fig. 8a, it could be observed that coating has been deposited on the substrate with a variable thickness of 500 µm. The interface between the materials consists of some unmelted particles. This problem occurred during plasma spraying process. Powder’s heating wasn’t sufficient in order to be melt during deposition process. We could estimate that in operating the samples will not have such a good behaviour due to unmelted particles, because could be possible to detach. Also could be possible that the bond stresses between the coating and substrate not to be efficient, because there are some gaps in coating. Figure 8b shows a detail of coating microstructure overview, which shows that are present unmelted particles and gaps, which confers a high porosity to the coating. There are no inclusions in the structure but gaps and unmelted particles disadvantage the coating quality. The sample has no cracks so we could approximate a good elasticity of the NiAlSi coating but due to unmelted particles and gaps we could not consider that in operating will have a better behaviour than \( \text{Al}_2\text{O}_3 \) coating\(^1\).

Figure 9a shows a dense structure with a lower porosity than \( \text{Al}_2\text{O}_3 \) coating, so we can consider that NiAlSi coating could have good tribological properties. Figure 9b shows NiAlSi coating chemical composition. Iron is present also in this coating so we can conclude that due to atoms diffusion there was an alloy of both materials at their interface. Chemical elements distribution is uniform but with the same gaps and in these gaps we discovered the oxygen. If oxygen was deposited over the NiAlSi, we could conclude that the sample has been oxidized after the deposition process in the environment. But the oxygen was found through the atoms of the NiAlSi coating that is why we could conclude that the material oxidized during the deposition process.
Figure 10a shows coating lattice parameters which demonstrates that the most stable phases are Al and Ni-Si, and Fig. 10b show that the same phase has the highest percentage in the coating composition. Also is present Al₂O₃ phase which appeared due to coating oxidization during deposition process.

Conclusions

The Al₂O₃ coating deposited on steel substrate is dense, but on SEM analysis we discovered that the interface between the materials has no defects, it means that we accomplished the experiment in good conditions and respecting the specified protocol, as per Sultzer Metco specifications. Coating covered uniformly the substrate, has a high porosity, but there are not present any defects in layer. There are no inclusions, gaps, unmelted particles or cracks, so we can estimate that the process has been correctly performed and the coating has a great quality and properties. The high roughness requires a slight grinding on the surface in order to improve tribological properties. Chemical composition analysis performed shown that there was a diffusion between both materials during deposition process, because it was found iron presence, this shows that the layer surface and the based material has become one.

The most stabile phase is Al₂O₃, and it has the highest percentage in the coating composition. We can conclude that the coating has good bond stresses between the particles because they are from the same type and its behaviour in operating will be more than satisfactory.

NiAlSi coating has a variable thickness of 10 µm. The coating consists of some unmelted particles and some gaps, which confers a high porosity to the coating. There are no inclusions in the structure but gaps and unmelted particles disadvantage the coating quality. The sample has no cracks so we could approximate a good elasticity of the NiAlSi coating but due to unmelted particles and gaps we could not consider that in operating will have a better behaviour than Al₂O₃ coating, but some of the proprieties can be considered as improved. In coating structure is present Al₂O₃ phase which appeared due to coating oxidization during deposition process. From these analyses we could estimate that Al₂O₃ coating has a better quality than NiAlSi and in operating will have a better behavior.

References