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An Assessment Study on the Quality of Ni-Cr Coating Before and After Heat Treatment

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ABSTRACT: This paper presents the results of research to assess coating quality before and after heat treatment. The spraying material used here is a mixture of Ni-Cr powder with 80% Ni and 20% Cr. By theoretical analyzes, the combustion of a powder mixture, the movement of liquid metal particles on the metal surface and the adhesion process of liquid metal particles on the metal surface have been determined. Experimental results show that after performing the spraying process on the surface of the part, forming a metal layer of spraying coating on the surface. In addition, if the coating is pre-heat treated on a metal surface, there will be a boundary layer separating the metal coating from the base metal. After carrying out the heat treatment process by diffusion annealing method, there has been diffusion between spraying layer and base metal, creating a link between coating metal and base metal; In addition, the appearance of carbides by diffusing carbon from the base metal to the outside creates the carbides to increase the abrasion resistance for coating. The structural assessments are concretely demonstrated by the results of mechanical evaluation: If before the process of heat treatment, the hardness value of the spray coat was only 191HV, after spraying it could reach the average value of 340HV; There are positions of carbides that can reach 980HV. The adhesion value of the coating can reach 29Mpa after heat treatment and the abrasion intensity of the coating is 1.89x109kg / Nm which is only about one third of the coating without heat treatment.

KEYWORDS: Ni-Cr coating; heat treatment; spraying coating; metal

INTRODUCTION

Metal coating technology was invented by a Swiss engineer named Max Ulrich Schoop from the early years of the 20th century [1]. The principle of this technology is to use the heat source (arc, burning gas, plasma) to melt metal. After that, the liquid metal is blown by the strong stream of compressed air into small particles (fog), then shoot at the prepared surface (cleaning, roughening) to create a metal coating of the required thickness, in which metal particles overlap each layer. At first, metal coating was only for decorative purposes. By the time of the Second World War, this technology had begun to be used on a large scale in most European countries and increasingly proved to be superior in areas such as surface protection, restoration and decorating instead of precious metals. By the 1980s metal coating had become a separate field of science and technology manifested as a surface treatment technology, on the other hand it was like a new manufacturing technology method in manufacturing. To evaluate the strong development of metal coating technology, it can be based on the development of equipment and application scope in the field of industry and life [2]. Metal coating technology is increasingly concerned because it is important and decisive to the properties of the coating material because it creates a surface layer capable of meeting working conditions such as abrasion resistance, corrosion resistance, heat resistance ... Metal coating technology is also used in many fields with different purposes such as: Protection against rust and corrosion in the atmosphere, soil and water environment; Create a conductive layer on non-conductive surface, used for decorating works; Recovering worn parts; Repair defects for castings or defects occurring during mechanical processing, saving precious metals [3]. In the process of developing thermal coating technology, many scientists have formulated the theory of coating formation, in which the theories play an important role including: The theory of Pospisil-Sehyl, Schoop, Karg, Katsch, Reininger and Schenk has been cited by the authors [4,5]. The theory of Pospisil - Sehyl said that the coating is formed by the liquid metal droplets blown by compressed air stream at a very high speed (on average about 200 m/s). These droplets are broken down into very small particles of mist, which are spherical or uneven, diverse. Metal oxidation actually starts from the flow process and continues during the flight of particles until it hits the surface of the spray, more or less amount of oxide is the factor that affects the quality of the coating. According to this theory metal particle at the moment of impact on the surface of the spray are liquid [6]. Frey theory said that compressed air provides energy to metal particles when impacted on the sprayed surface with a change of heat [7]. When leaving the nozzle began to cool and solidify very quickly due to the effect of compressed air. At the time of impact, they will deform plastic so they link together into pretty solid bonding layers. The temperature of the metal ray is reduced very low to about 50^0C to 100^0C. So, it is possible to cover them with combustible materials without burning the base material. The theory of Moridi, Guagliano, Dao put forward the view: The metal cools and cools due to the kinetic energy and compressed air [8]. On the other hand, in the process of flying from the nozzle, the particles are in a cold state so that no plastic deformation when impacting occurs. Wen's theory offers a different perspective: the author argues that the temperature of the spray particles must be at the melting point for the welding between them [9]. That is, at the time of impact on the sprayed surface, it will be heated to the melting point to make the welding between the spraying elements and the base metal, but not really [10]. From the theoretical views of the formation of the coating of scientists in the world, it is shown that there were different views about the state of the spray particles at the time of impact, but according to the author [3] described the formation of a coating characterized by four stages: (1) the heating and melting phase of the spraying material, (2) the dispersion phase of forming liquid metal droplets, (3) the flight phase of metal droplets, (4) the impact phase of metal droplets on the metal substrate surface to form a coating, by image is modeled as Figure 1 and is analyzed as follows:

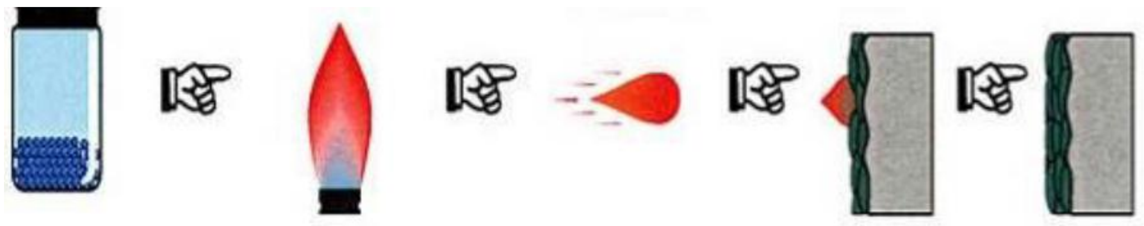


Figure 1. Stages of thermal coating process

The stage of heating and melting molten material: acetylene combustible fuel gas, oxygen and compressed air are fed into the spray gun at the spray gun combustion chamber. They are mixed together to form a mixture of flammable gases with high temperature and pressure. Powder injection material, mixed with nitrogen gas (called carrier gas), is drawn into the combustion chamber after each explosive pulse of the material is heated to a flowing state and compressed with high pressure, moving along the flammable gas flow at supersonic speed and beyond sound thanks to Laval tube structure [11].

Scattering phase of forming liquid metal droplets: when the gas stream carrying molten material flows out of the nozzle, high-pressure melting droplets come into contact with the low-pressure atmosphere,causing the pressure inside the molten droplets to drop suddenly and they expand into many small mist-shaped particles, according to the principle of the spraying. The dispersion of metal droplets under the influence of gas pressure and temperature depends on the combustion air pressure and the nozzle diameter [12].

Flying phase of metal droplets: The flight speed of particles increases gradually from the burning zone to the nozzle, but when leaving the nozzle, the speed decreases gradually due to air resistance. High-temperature molten metal droplets disperse into small particle beams that interact chemically physically with their surroundings, the spraying material oxidized and dissolved gas. Oxidation depends on the composition of the combustion gas. Gas solubility depends on the surface tension of metal droplets, the attraction of atomic atom atoms, the coefficient of thermal conductivity of the material. During the flight, due to the difference in pressure inside the particle and the ambient pressure, the particles continue to break apart into many small particles. The funnel flow is shaped like a funnel, the flaring angle of the funnel depends on the flaring angle of the Laval pipe, the spray speed and the nozzle diameter [13].

The impact phase of metal droplets on the substrate metal surface to form a coating: The coating material particles with kinetic energy and heat fly to the surface. Upon interaction, the kinetic energy of the particle becomes heat energy, energy transfer to the substrate surface increases the surface temperature, when in contact with the substrate surface, the liquid metal particles are deformed from round too flat and firm on the rough surface of the base metal. As a result of the heat transfer, the particles are deformed and cooled, together with the reduced volume, most of the particles adhere to the substrate, a small part is shot out of the surface. Depending on the displacement of the nozzle or the object being sprayed, a beam of particles is fired on the substrate surface in layers, superimposed to form the required coating. The distribution of the aerosol particles at the destination of the injection stream and the superposition spraying of layers 2 and the aerosol contacting with the substrate surface is shown in Figure 2.

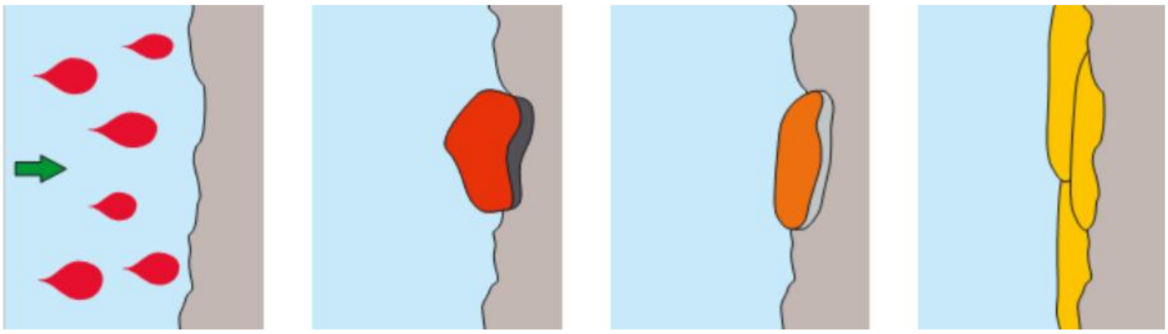
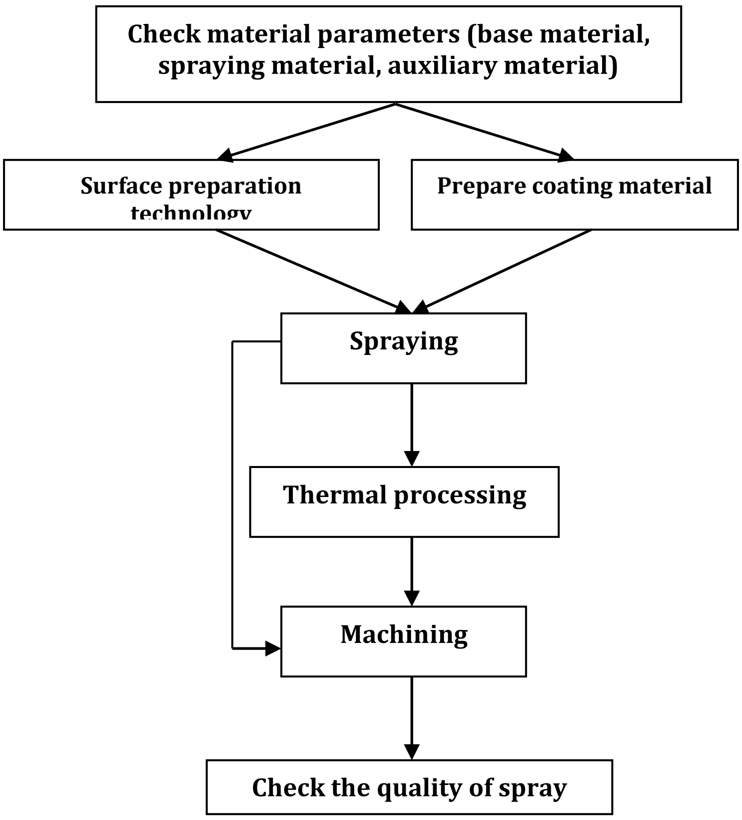


Figure 2. Flow chart of spraying material forming the coating [14]

When the liquid metal droplets come hitting the surface of the substrate, they will cling and solidify, successively in groups of particles, layers and coatings. The essence of the coating is the adhesion of the metal layer to the base metal. The process of forming the link between the coating and the base metal layer is the process of adhesion and diffusion. In order to create a diffuse layer to increase the level of connection between the coating and the base metal, appropriate heat treatment must be applied. Currently, the research on spraying mainly focuses on spraying technology, and studies on the role of post-coating heat treatment have not been studied much. In this paper presents the initial research results on the formation and quality of coating before



and after heat treatment.

MATERIALS AND EXPERIMENTAL SET-UP

Figure 3. Metal coating technology

Coating materials

With the coating spraying technology used here, it is suitable to use as a powder with the following ingredients.

*Metal powder: With powder spray we check the main parameters such as particle size, shape, flow. Particle size is assessed by wire-passing sieve, microscopic examination shape, flow determined according to standards.

Table 1. Chemical composition of spray powder

Powder	**Chemical composition**		
	Ni	Cr	C
Ni-20Cr	75-68	22-23	< 0,15

Spraying powders must be prepared in advance. The preparation of powder consists of the following steps: drying powder and gradation of particles. Drying powder: Typically, sprayed metal powder is usually dried at a temperature of 120-150ºC for a period of 1-3 hours. For ceramic powder, the drying temperature is higher than 700-800ºC and drying time is from 4-5 hours. Grading of spray powder often uses wire sieves with sizes smaller than 100mm.

Gas-fired spraying equipment

Figure 4. Gas-fired spraying equipment

Equipment for checking structure of coatings

Axiovert 25 CA optical microscope - Laboratory of metallurgy and heat treatment-Hanoi University of Science and Technology.

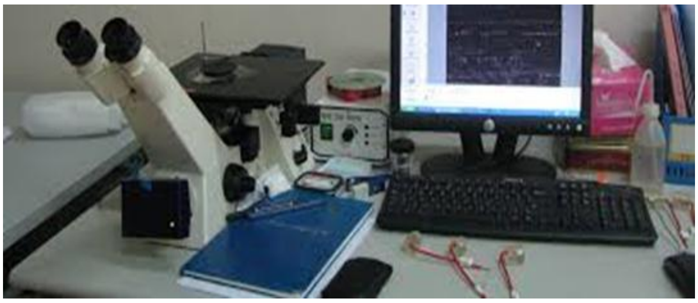


Figure 5. Optical microscope

Scanning electron microscope (SEM) method: Identification of alloy organizations at a high magnification level; Observe the organization and the resulting phases that are not visible under conventional optical microscopy. The experiment was performed on the scanning electron microscope FESEM Jeol 7600 – Laboratory of microelectronics and micro-analysis - Advanced Institute of Science and Technology - Hanoi University of Science and Technology. The machine performs high resolution; the magnification ability reaches 300,000 times.

Figure 6. Scanning electron microscope FESEM Jeol 7600

EDS method: Determine the distribution of alloying elements in the grain and at the grain boundary by point. The level of impurities in the grain and the grain boundaries according to points. Analysis on EDS device - Laboratory of electron microscopy and micro-analysis - Hanoi University of Science and Technology. This device uses X-rays to analyze the chemical composition of materials, observe the organization of materials on surfaces and at separate points in a small area. Thereby determining the distribution of alloying elements on the grain boundary and in the grain. The thesis performed chemical composition analysis at the grain boundary and inside the grain on alloy samples, analyzing EDS distribution of elements inside and outside the grain boundary.

Mapping method: Determine the distribution of alloying elements in particles and at the grain boundaries by scanning the surface distribution. Studying the distribution of elements on the image of mapping organization is done on scanning electron microscopes at the Laboratory of Electron Microscopy and Micro Analysis - Hanoi University of Science and Technology.

Figure 7. Hardness tester

Evaluate the adhesion of the coating the combined layer of aluminum coating and Ni-20Cr coating, aluminum coating below, Ni-20Cr coating on top. Therefore, only the aluminum coating is in contact with the steel substrate, while the Ni-20Cr coating is in contact with the Al coating. There are several methods of determining the adhesion of the coating to the substrate: the method of latching, the method of glue ... In this section, to determine the adhesion strength of the Al coating on the steel substrate, use a latch pull method. Fundamentals of the method The sample consists of a cone A and a key plate B with a conical hole and made of steel CT3, a sample of the adhesion measurement model shown in Figure 8.

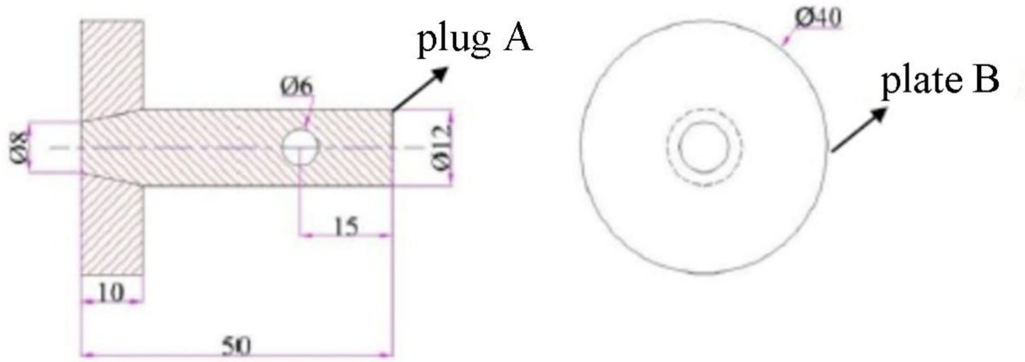


Figure 8. Diagram of adhesion test principle

When installing the pin on the latch disc, the latch surface fits into the flat of the latch disk (the fitting between the conical pin and the latch disk is the tight fitting). After machining the common surface (between the pin and disc latch) we proceed to spray on it. In the process of adhesion testing, we proceed to pull the pin from the latch disc and the ratio of the maximum pulling force on the surface of the latch surface is the adhesion to be found.

Adhesion measurement result: is the ratio between the breaking force and the surface of the test surface, which is typical for the adhesion of the coating to the substrate:

$$\sigma_k = \frac{P}{F} \tag{1}$$

P - pin drag force, kG; F - pin cross section, cm^2
Testing equipment and samples Measuring the adhesion of coatings and steel substrates on DLR equipment (Germany) - Institute of Tropical Technology - Vietnam Academy of Science and Technology. Figure 9 shows the adhesive measurement diagram, in Figure 10 is the sample for measuring the adhesion and the attached jigs.

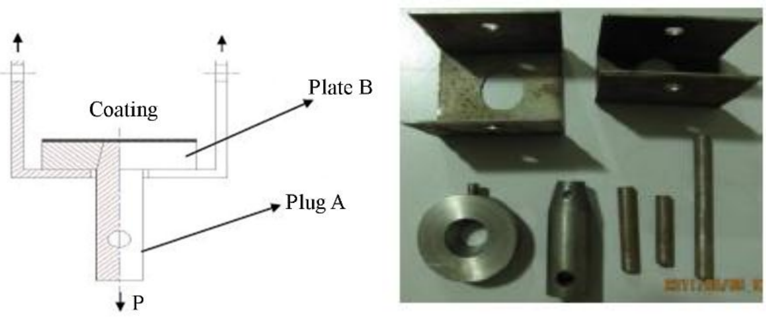


Figure 9. The principle diagram and the test sample

The attachment of the adhesion test consists of a key plate B with a central drilled hole in which plug A is fitted with a tight fitting mode. The two parts A and B are fitted together so that the sprayed faces are on the same plane. After preparation, the sample surface is sprayed with an aluminum coating with a thickness of 1 - 1.5 mm.

Measure the abrasive strength of the coating

Sample preparation

The abrasion test piece is a Ni-20Cr duplex coating on CT3 steel substrate after heat treatment at 1100^{o}C / 8 hours and at the same time test on the untreated sample for comparison.The test sample is made according to Figure 10. The surface of the sample before the abrasion test is ground flat.

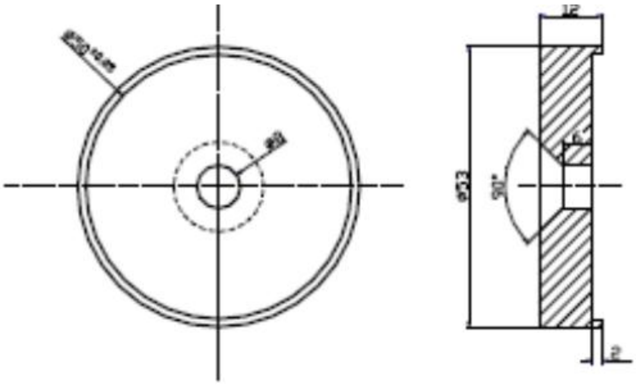


Figure 10. Drawing drawing of the abrasion test piece

Testing method: according to ASTM G99.Let Ni-20Cr - carbon steel friction pairs work with P = const, V = const, do not use lubricant.The intensity of abrasion by weight method is calculated as follows:

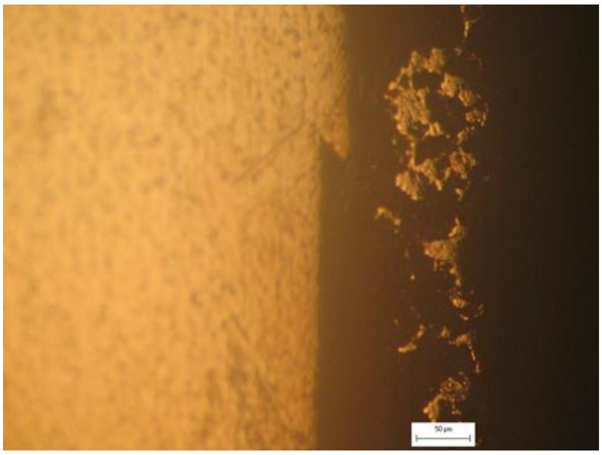
$$I = \Delta M / QxS \qquad (2)$$

Where: I (Kg / Nm): Intensity of wear; ΔM (kg): The loss of sample weight after the experiment; Q (N): Load test; S (m): Sample running distance.With abrasion test parameters: Test load: 30 N; Speed of rotation: 100 rpm; Running time: 600 seconds; Turning diameter: 45 mm.

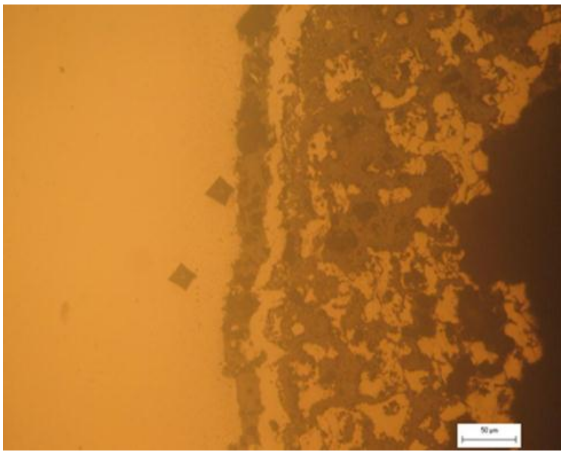
Thermal treatment process of spray coating: Sprayed samples after spraying need to be heat treated with the aim of reducing porous density, increasing the adhesion of metal to the coating metal. Samples covered with metal after being sprayed were conducted diffusion annealing at 1100°C for 08h. After 08h samples are taken, conduct optical analysis; analyze SEM + EDS and determine the properties of the spray coat.

RESULTS AND DISCUSSION

Coating structure

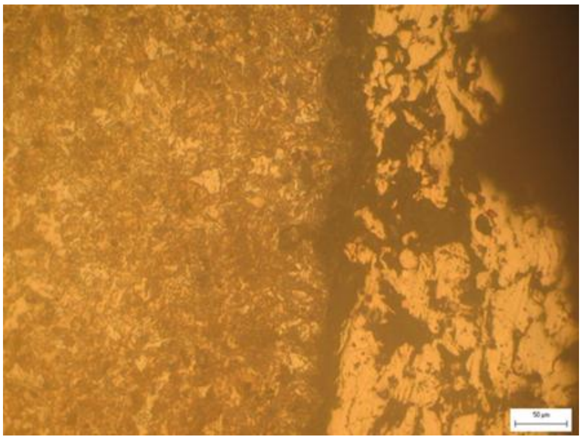


a) Before heat treatment

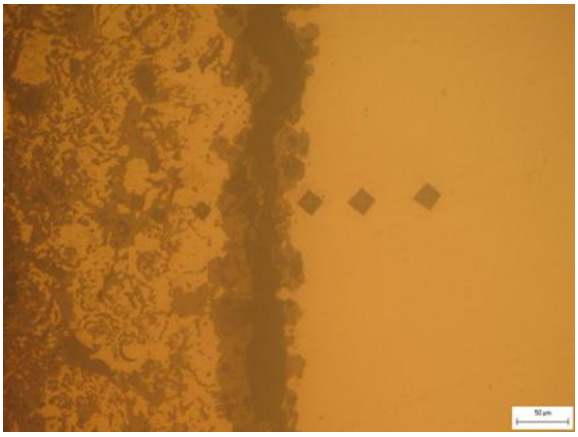


b) After heat treatment

Figure 11. Structure and depth of three-layer coating



a) Before heat treatment

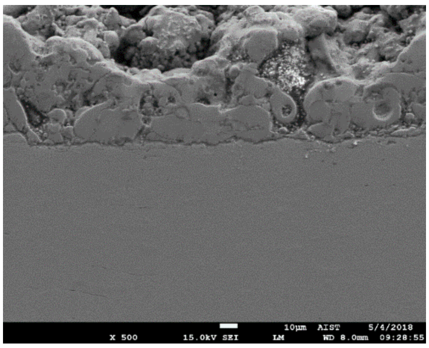


b) After heat treatment

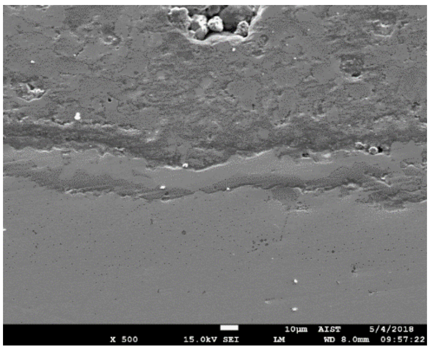
Figure 12. Structure and depth of fully coating

Results of analysis of the coating structure before heat treatment found that: Although the spraying process has a high melting point, the surface of the substrate and the spraying layer does not seem to be tightly organized. The organization at the boundary shows that there is only mechanical connection between the overlay and the foundation. It is only mechanical adhesion that does not diffuse between the coating and the base metal so the hardness value can be high but the adhesion will be poor. In addition, there are pores in the organization that are brought by the spraying process. The appearance of pores will reduce the degree of cohesion of the substrate. Figure 11.a shows the structure of a two- and three-layer coating. When performing two-layer and three-layer coating, the appearance of (mechanically) linked layers to connect the base layer with and the coating layer. Also, Figure 12.a shows a fully coating by calculation mode.The results of analysis after heat treatment in Figure 11.b and Figure 12.b show: The samples after heat treatment coating thickness increased. This can be explained by the diffusion created by the organizational link of the coating with the base metal layer. Moreover, depth analysis of the coating showed an increase compared to the sample without heat treatment.

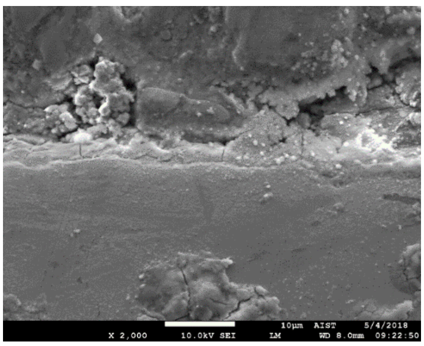
Analysis of SEM + EDS images coated before and after heat treatment



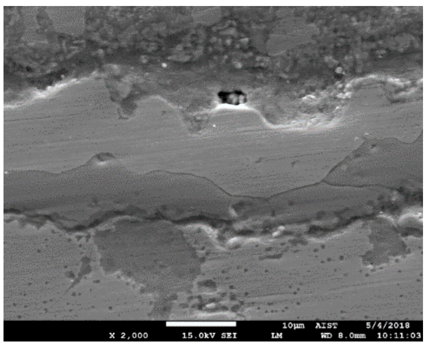
a) Sample before thermal treatment x 500



b) Sample after heat treatment x500



c) Sample before thermal treatment x2000



d) Sample after heat treatment x2000

Figure 13. Analysis of SEM images before and after heat treatment

SEM images show that pre-heat samples have a clear separation between the coating and the base metal. It is this separation that will lead to the coating to peel off when working. The EDS analysis clearly shows the separation between metal coating and base metal. The coating elements concentrate on the entire side without forming a diffuse layer between the coating and the base metal. When performing diffuse composting analysis, microstructure showed that formed a link layer between base metal and coating. EDS analysis shows the clarity of this link layer. The elements are evenly dispersed from the coating side to the associated metal side. It is this diffusion that will contribute to the formation of the link layer and avoid peeling during the work of the part.

Hardness of coating layers: The results of hardness analysis in Figure 14 show: The hardness value of the coating before heat treatment is low. This low value is explained by the fact that it is not really possible to create the metallic phases of Fe with Cr and Ni as well as the carbides of this layer, so the hardness value is low.

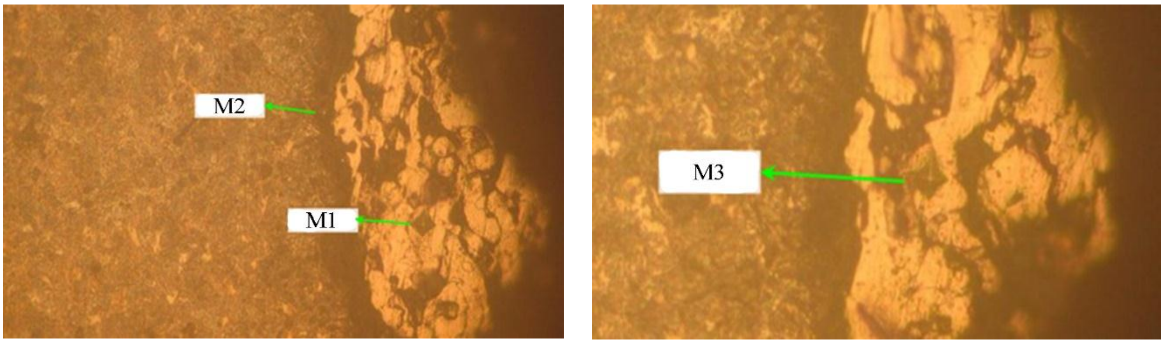


Figure 14. Hardness of coating before heat treatment

The results of the hardness value analysis in Figure 14 show: Except for sample 1, the rest of the samples have a relatively uniform distribution of the coating; Bonding layer and metal background. Moreover, in some samples by the method of measuring micro hardness as well as organizational observations showed the appearance of intermetallic phases. It is this process that makes the part have good adhesion after diffusion; Inside the organization receive the metallurgical phases of Fe; Cr and Ni. This enhances the durable chemical ability and the ability to wear parts.

CONCLUSION

In this study, we focus on studying the nature of the spraying process; establish the process of spraying gas fire and heat treatment regime as well as studying the nature of spray coating. The basic objectives of the study have been achieved include: (1) Determining the nature of the coating and the process of applying Ni-Cr alloy to the metal (sample is CT3 steel); (2) Develop a process of spraying Ni-Cr with steel. Introduce the process of tempering the sample to create a diffuse layer between the coating and the base metal; (3) Studying the connection layer between coating and base metal. Thus, by the results of theoretical and empirical analysis the paper has been determined:Research the nature of the process of spraying Ni-Cr alloy coating on the base metal. Organized and mechanical analysis of samples after heat treatment to increase durable chemical ability for steel. Identify the bond layer between the original coating and metal. By analyzing SEM and EDS images, the layer between the coating and the base metal was identified. Determination of mechanical quality through hardness and grip of spray coating. Determine the amount of wear before and after coating.

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