AN EXPERIMENTAL AND STATISTICAL ANALYSIS OF THE HARDNESS OF NI–P AND NI-P/SIC COMPOSITES

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ABSTRACT: Electrolytically obtained Ni-P and Ni-P/SiC deposits were analyzed and tested by Vickers microhardness. The incorporation of silicon carbide particles significantly increases the hardness compared to raw Ni-P deposits. The hardness decreases with increasing applied load, and at high loads the value obtained is significantly influenced by the substrate, indicating that the measured hardness no longer reflects the intrinsic hardness of the deposited film. The results highlight the efficiency of silicon carbide particles in optimizing the mechanical properties of the Ni-P composite, and statistically generated exponential models confirmed accuracies of over 99.8%.

KEY WORDS: composite material, hardness, statistical analysis, electrodeposition, layers

1. INTRODUCTION

Composite materials are materials made from two or more components with distinct physical or chemical characteristics, combined in such a way that the final product exhibits superior performance compared to each individual component. The two components are called the matrix, which can be polymeric, metallic, or ceramic, and the reinforcement (such as glass fiber, carbon, Kevlar, etc.). Between the matrix and the reinforcement lies the interface**,** which acts as a bonding element between the two and appears as a very thin layer where chemical and mechanical interactions occur between the matrix and the fibers.

Each of these elements has a particular importance in the execution of the composite. Thus, the matrix, which is the basic element, takes over and distributes the efforts, protects the material, helps in the processing of the material, the particles or fibers are incorporated into it, fills the free spaces in the reinforcement, maintains the connection between the structural elements and offers the possibility of modeling complex geometries. Although the matrix is considered the "invisible" constituent, it influences the strength, durability and final performance of the composite material. In addition to all these benefits, the matrix can also present some non-conformities such as the presence of porosities, the existence of microcracks, contractions, is subject to delamination or can oxidize or thermally degrade.

The reinforcement, or reinforcing phase, is a dispersed phase that provides mechanical strength and stiffness to the composite. This component supports most of the mechanical loads, giving the material high tensile and compressive strength (especially continuous carbon or aramid fibers); significant fatigue and impact resistance, stiffness and high elastic modulus.

The interface plays an important role in the efficiency of the composite, as it ensures load transfer, ensures durability because it determines the behavior of the material in corrosive environments (humidity, high temperatures, chemicals); influences delamination resistance and impact resistance. Inside the interface, chemical bonds can form between the matrix and the reinforcement, and the fibers or particles are physically trapped in the matrix (adhesion by roughness or shape). To improve its characteristics, a coating can be applied that is tolerated by the matrix, or the fibers can be chemically treated and the matrix treated with compatibilizing agents.

Composite materials are considered new materials, artificially obtained and whose properties can be modulated by modifying working parameters, environmental conditions, and the manufacturing process [1,2].

Among the most important characteristics are the notable resistance to tension-compression, bending, shock, fatigue, abrasion and scratching. From a chemical point of view, composites are chemically stable and resistant to corrosion, have good thermal, electrical and magnetic properties. At the same time, they can be specially designed for precise applications and can be obtained by various techniques.

There are several criteria for classifying composite materials [eu]. Thus,

a. According to the nature of the matrix, composite materials are divided into:

- Metal matrix composites (MMC), where the matrix is a metal;
- Ceramic matrix composites (CMC), where the matrix is a ceramic;
- Polymer matrix composites (PMC), where the matrix is a polymer;

b. According to the nature of the reinforcement, composite materials are divided into:

- Fiber composites
- Particle composites
- Layered composites (laminates)

c. According to the structure, composite materials are divided into:

- Unidirectional composites
- Bidirectional composites
- Multidirectional / random composites

Composite materials are used in a wide range of fields, due to the various properties they possess such as: the optimal balance between strength and mass, high rigidity, corrosion resistance, etc [3]. We find composite materials in the automotive industry in structural elements, lightweight bodies, panels, etc.; in the aerospace field in internal structures, wings, fuselages, etc.; in nautical, sports and leisure in boat bodies, yachts, seaplanes, skis, tennis rackets, bicycles, etc.; in construction [4] and industrial applications as concrete reinforcement, structural panels, sandwich panels, corrosion-resistant pipes, tanks, wind turbine blades [5] etc; in medicine, in prostheses, implants, housings for medical equipment, light and rigid surgical instruments; in the electronics and telecommunications industry, in housings for phones and laptops, antennas and structural supports.

Composite materials are in many ways superior to conventional materials. Thus, compared to traditional materials, composites are characterized by a very high strength/weight ratio, can be created to provide rigidity or strength in a certain direction, have good fatigue resistance, are chemically resistant, etc. [6, 7]. Heat treatment applied to both conventional materials and composites leads to increased hardness and mechanical strength, reduces internal stresses, increases ductility and toughness [8].

Electrolytic deposition of Ni-P composite material

The first nickel-phosphorus layers were obtained by chemical methods, which, although effective, had certain limitations in terms of uniformity and layer thickness. As electrolytes were developed (Brener, 1947), NiP deposits could be obtained electrolytically, offering the possibility of precisely controlling the composition and characteristics of the deposited layer.

The Ni–P (nickel–phosphorus) composite is defined as an electrolytically deposited layer with remarkable properties, which makes it widely used. These properties are influenced by the phosphorus content in the layer, which determines the structure and behavior of the layer.

The structure of the Ni–P deposit depends on the amount of phosphorus incorporated in the layer. For low P content incorporated in the layer (below 5%), the structure is crystalline; for a content of up to 9% P, the structure is combined, having crystalline parts and amorphous parts, and when the content incorporated in the layer is high, above 10%, the structure is amorphous.

The amount of phosphorus in the layer is an important factor on which several variables depend. For example, corrosion resistance increases with increasing phosphorus content (above 10–12% P, the layer structure becomes amorphous and very chemically stable).

Ni-P material is resistant to abrasion and wear and has a low coefficient of friction. Aesthetically, the composite material is deposited uniformly and the surface is smooth and can be polished.

The properties of Ni-P material are influenced, in addition to the phosphorus content in the layer, by heat treatment. Thus, heat treatment can increase hardness, wear resistance and mechanical stability, but can decrease corrosion resistance and increase brittleness, especially at temperatures exceeding 350–400°C.

Ni-P material hardness

The hardness of a composite material is the property of a material to resist plastic deformation or local penetration when subjected to a concentrated force.

The hardness of composite materials can be performed by various methods, such as the well-known traditional Rockwell, Brinell, Vickers tests and micro- and nano-scale indentation tests for thin composite material coatings.

The hardness of the Ni-P layer depends on the chemical composition, crystal structure, applied heat treatments, deposition technique and the existence of composite particles in the matrix.

Hardness of Ni-P/SiC composite material

The Ni-P/SiC composite consists of a Ni-P alloy metal matrix, with an amorphous or crystalline structure, into which very hard silicon carbide ceramic particles have been incorporated. The inclusion of SiC particles in the Ni-P matrix increases the hardness, improves wear resistance and mechanically stabilizes the material.

The distribution of SiC particles is influenced by the microcrystalline or amorphous structure of the Ni-P alloy.

The hardness of the composite is influenced by several variables: the concentration and size of the SiC particles, the heat treatment and the deposition method.

2. TECHNICAL REQUIREMENTS

The composite material samples were obtained electrolytically, in a special installation, with a bath containing the following elements: $NiSO_4$, $6H_2O$, $NiCl_2$, $6H_2O$, H_3PO_3, H_3PO_4, with well-determined concentrations, using a current whose intensity varied, so that deposits with variable thicknesses were obtained.

Vickers hardness tests were performed on samples of composite material of the type PxSy-z, where x represents the amount of H3PO3 in the electrolyte, and x is the concentration of SiC particles using variable loads (15g, 25g, 50g, 100g, 200g, 300g, 500g, 1000g, 2000g), applied perpendicular to the surface of the layer, and z is the measured thickness of the layer.

The samples are of two types: the first was obtained with 20 g/l H_3PO_3 content, without SiC particles suspended in the electrolyte; in this case the measured thickness was set at 29 µm; and the second sample was obtained from electrolyte containing 20 g/l H_3PO_3 and 80 g/l SiC and the measured thickness was 28 µm.

3. EXPERIMENTAL RESULTS

The dependence between the hardness obtained when applying variable loads, for samples P20S0-29 and P20S80-28 is given in Figure 1.

Analyzing figure 1, it is observed that for both samples the hardness decreases with increasing applied load, and the hardness of the P20S80-28 deposit is higher than the hardness of the P20S0-29 sample, which shows that the hardness is influenced by the incorporation of particles into the layer.

As expected, the introduction of silicon particles causes a hardening of the layer, which leads to an increase in the hardness of the layer.

The STATISTICA program was used, through which the hardnesses obtained experimentally for the two samples were compared with the hardnesses issued by this program.

The curves in figure 2 represent the characteristic curve of the variation of the measured microhardnesses as a function of the applied load and the microhardness curve imposed by the program (which is generated

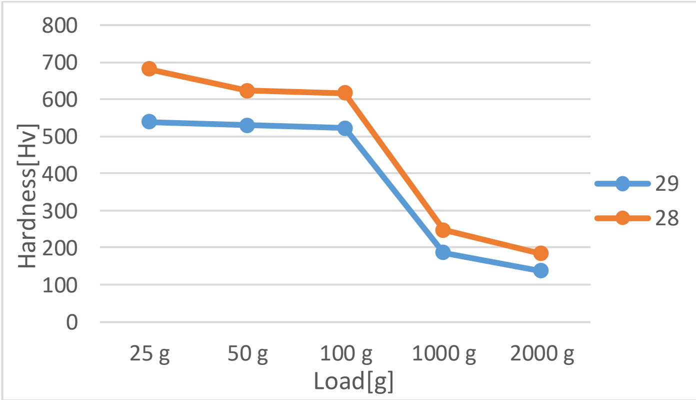

Figure 1. Variation of hardness as a function of applied load and deposition thickness

by an exponential equation) for the P20S0-29 deposition. It is observed that the shape of the graphs is similar, and the measured hardness value and the predictive value have almost identical values, which determined an error of 99.99%.

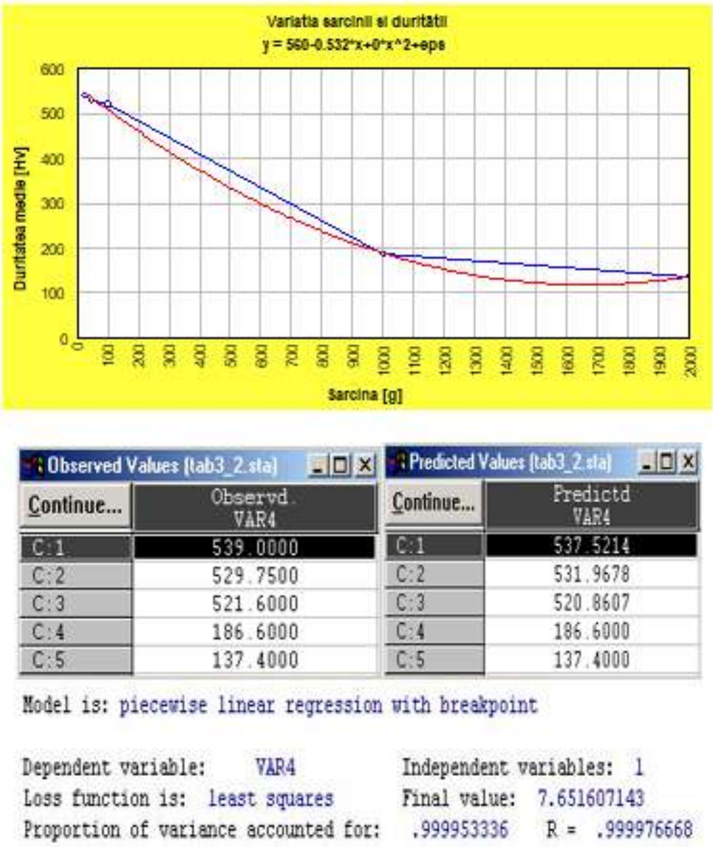

Figure 2: Variation of hardness as a function of applied load for P20S0-29µm deposition

The program was also applied to the sample that has incorporated Silicon Carbide particles,
P20S80-28, obtaining the curves in figure 3. The equation according to which the curve for the P20S80-28 deposition is drawn is an exponential equation, the shape of the graphs being similar, and there is not a very large difference between the measured microhardness values and those imposed, so that the error given by the program is 99.80% (figure 3).

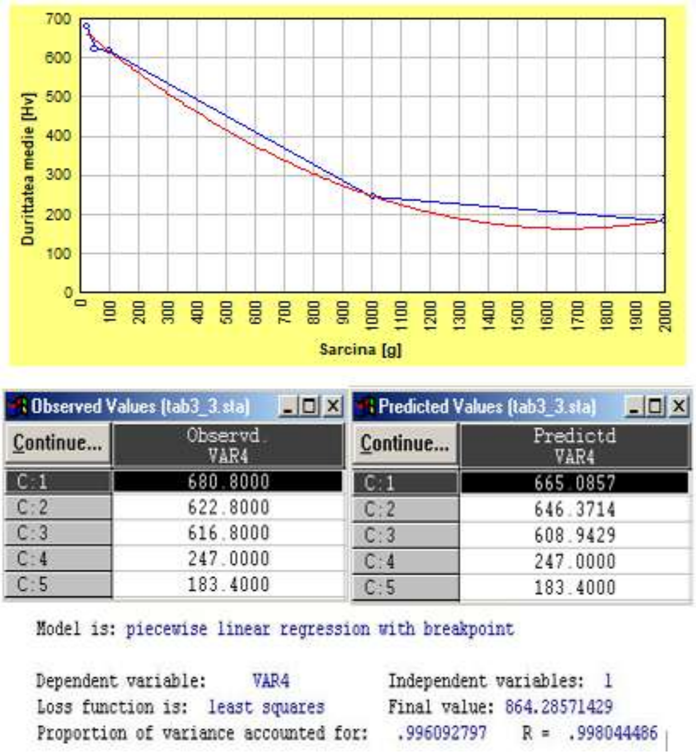

Figure 3. Variation of hardness depending on the applied load for the deposition of P20S80-28

The equation according to which the curve for the P20S80-28 deposition is drawn is an exponential equation, the shape of the graphs being similar, and there is not a very large difference between the measured microhardness values and the imposed ones, so that the error given by the program is 99.80% (figure 3). Another important observation shows that, in both cases, the hardness tests performed with loads of 1000 g and 2000 g showed a significant decrease in the hardness of the layer, which makes the support increasingly influence the value obtained.

Under these conditions, the measured hardness no longer corresponds to the real hardness of the layer, but reflects the hardness of a composite assembly formed by the substrate and the deposit.

3. CONCLUSION

Composite materials highlight the functional interaction between the matrix, reinforcement and interface, each component contributing specifically to the optimization of the mechanical, physical and chemical properties of the structure. This multi-phase system allows for superior performance compared to conventional materials.

The matrix, which is the continuous phase responsible for the transfer and distribution of mechanical stresses, determines the overall behavior of the composite material, and the reinforcement provides the main mechanical strength.

The interface regulates the efficiency of load transfer and structural stability in aggressive environments.

Ni-P and Ni-P/SiC electrolytic deposits present different mechanical behaviors determined by the composition of the layer and its structure and the presence of SiC ceramic particles significantly increases the mechanical performance.

The incorporation of silicon carbide particles into the Ni-P matrix determines a notable increase in hardness, highlighting their major role in optimizing the wear resistance and mechanical stability of the layer.

The hardness of both types of deposits constantly decreases with increasing applied load, which shows a pronounced sensitivity of the measurements to load variation.

When applying high loads (1000–2000 g), the results obtained are strongly influenced by the substrate, which means that the values obtained no longer reflect the deposited layer, but the layer-substrate ensemble.

The experimental analysis of Vickers hardness, performed with variable loads, indicates a downward behavior of the measured values with increasing applied load, a phenomenon correlated with the influence of the substrate at high loads.

The comparison of the experimental data with the predictive models generated by the STATISTICA software revealed a very high concordance, highlighting the correctness of the mathematical modeling used Statistical analysis given in the form of an exponential model confirmed an excellent correspondence between the experimental and theoretical values, with a concordance of over 99.8%, thus validating the reliability of the analysis method.

The Ni-P/SiC composite demonstrated superior performance compared to simple Ni-P deposition, which recommends it for applications requiring high hardness, wear resistance and stable mechanical behavior.

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