THE TIME PREPARATION INFLUENCE ON THE ELECTRODEPOSITION HARDNESS OF THE COMPOSITE MATERIAL PART I - Ni-P alloys

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Abstract: One of the used techniques to obtain composite deposits is electrolytic deposition. On the electrodeposited Ni-P alloy layers obtained by varying the time of preparation, toughness is influenced by both the thickness layer and applied load. Hardness tests conducted with perpendicular loads applied to the surface layer show that the application of small tasks for the same film thickness, hardness obtained decreases as the applied load increases and when the layer is thicker the hardness is greater.

1. INTRODUCTION

Composite deposits are part of advanced materials with a wide applicability in industry. However, a particular need to use composite coatings in various economic fields is because these materials have a higher life and a better resistance to the mechanical and corrosion. A particular importance have the composite layers from the composite materials which are systems composed of two or more materials aimed to improving their properties comparison with the separate properties of materials that are formed.
Composite materials consist of two phases [1]:
- Phase I, called the matrix, is present as crystalline or amorphous state,
- Phase II, called dispersed, consisting of one or more phases dispersed in the matrix in any state of aggregation. Depending on the components, can be discontinuous when the components are dispersed particles of different sizes and continuous when components are fibers. Composite layers can be obtained by several methods (electrochemical codeposition, chemical, in polymer matrix, CVD, ECVD, plasma spraying, sputtering in vacuum), which leads to different properties. One of the techniques used to obtain composite deposits with a special appearance at the surface while providing protection from atmospheric corrosion and to that produced by some chemical and mechanical damage due to friction, abrasion, etc. is electrolytic deposition. The electrolyte development is a relatively simple process and layers so obtained can be controlled in terms of composition, appearance and mechanical properties. It is a process that takes place at low temperature electrolysis cells having a relatively high life and low cost.

2. HARDNESS DETERMINATION OF Ni-P ELECTRODEPOSITIONS OBTAINED BY VARYING THE TIME

Electrodeposition were obtained in an electrolysis cell where we can get deposits with areas up to 300 cm^2 and thickness of 100 μm. The device consists of a set of cells in which is realized the degreasing, pickling, actual lodging and washing specimens. Codeposition tank has 8 liters capacity and is equipped with a renewal system of the electrolyte suspension through distributors located in the top of the device and which are adaptable to complex geometry parts. This tank contains the injection system of the electrolyte, nickel anode, specimens support, the system providing mechanical agitation and cathode agitation. Electrolyte composition consisted of $NiSO_4x6H_2O$, $NiCl_2x6H_2O$, H_3PO_3,H_3PO_4 [*2*].

Layers were deposited on copper substrate with different thicknesses obtained by varying development time (10-40 min) using a constant electrolyte containing phosphoric acid (20 g/l). Hardness tests were made with variable load (25g, 50g, 200g, 500g, 1000g, 2000g), applied perpendicular to the surface layer. Samples were noted on the time of development: P1 (10min), P2 (20 min), P3 (30 min), P4 (40 min). Since the coating thickness plays an important role on the layer properties were measured thicknesses of these layers (Table 1), and the results obtained from measurements by light microscopy in cross section are averages of 5 measurements.

Table 1. Thickness measured samples

Layer	P1	P2	P3	P4
Measured average thickness (μm)	6,8	8,4	29	59

Because mechanical properties are influenced by working conditions [3], was studied Ni-P alloy hardness variation depending for varying time. Deposits microhardness was measured using a laboratory device "Shimadzu HMV-2", equipped with a pyramid-shaped penetrator square base and top angle $\alpha = 136^0$. Tasks are automatically selected and displayed on a liquid crystal display. Measuring system connected to a computer is supported by a video camera which transfer obtained images to the computer for processing and extracting indentation diagonals to calculate Vickers hardness, which can be loaded with different loads applied perpendicular on the layer surface (figure 1).

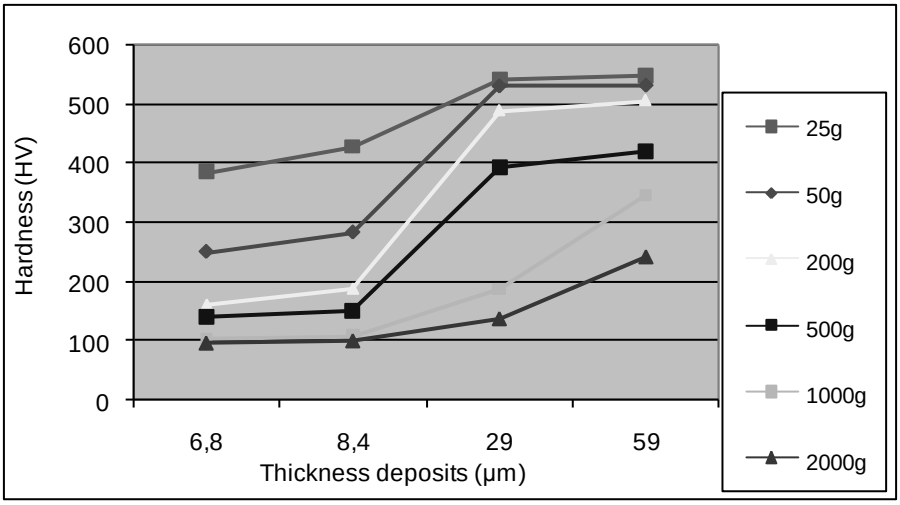

Figure 1: Variation of hardness deposits depending on their thickness and applied load

The figure is observed that the hardness variation evolves three areas, namely:
- For samples obtained during the preparation of 10 min, the hardness shows a small increase;
- For samples obtained during the development of 10-20min, hardness increases significantly;
- For samples obtained during the development of 20-40 min, the hardness has a different pattern application tasks such as: for 25g, 50g, 200g, 500g tasks, hardness remains almost constant, for 1000g and 2000g tasks, hardness increases rapidly.

For loads applied to the same layer thickness, hardness obtained decreases as the applied load increases and, the increased thickness, hardness increase. Differences between the hardness obtained at loads up to 500g are not particularly large, which shows that the substrate hardness influence is zero, so measured hardness is only hardness of Ni-P layer.

For large loads applied (1000g and 2000g), measured hardness shows notable differences from the smaller tasks. It can be seen that measured hardness is not the intrinsic hardness layer, it becomes a composite material hardness, substrate and deposit. With the optical microscope were viewed Vickers intendor impressions left by the application of heavy loads of 1 kg and 2 kg for the studied layers (Figures 2, 3, 4, 5).

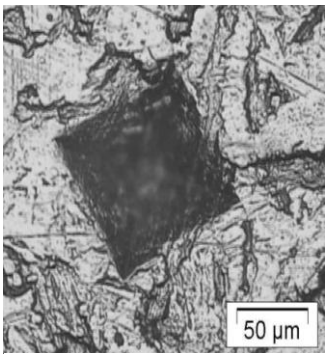

Figure 2: Micrograph fingerprint sample P1 (1kg)

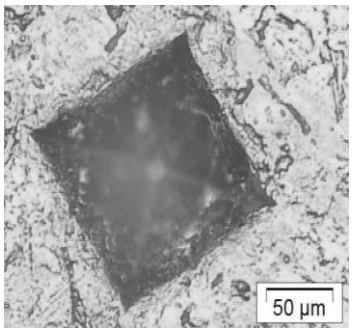

Figure 3: Micrograph fingerprint for sample P2 (2kg)

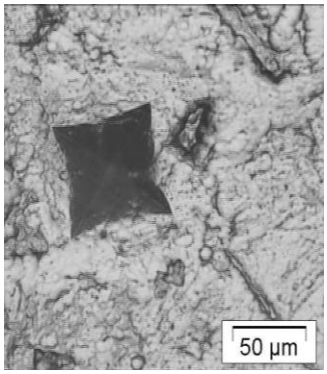

Figure 4: Micrograph fingerprint for sample P3 (1kg)

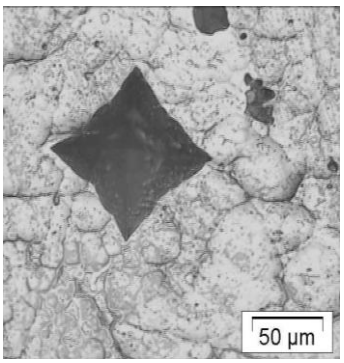

Figure 5: Micrograph fingerprint for sample P4 (2kg)

Thus, micrograph fingerprints of 1kg or 2kg tasks for any sample application shows that heavy loads do not cause cracks in the layer.

Determination of the layers hardness allowed the fingerprints diagonals measurement left by the penetrator, and their values are listed in Table 2.

Table 2. Diagonals fingerprint application tasks for deposits P1, P2, P3, P4

Layer	Load [g]					
	25g	50g	200g	500g,	1000g	2000g
	D_{med} (µm)					
P1	12,7	20,4	44,4	82,6	134,3	196,1

Note that there is a very big difference between the sizes of the diagonals fingerprints up to the task of 500g and dimensions of the diagonals fingerprints obtained with 1000g and 2000g task. Because tasks were applied perpendicular to the surface layer, we apply the relationships established between diagonal fingerprint left by the penetrator from these tasks and thickness:

$$h = \frac{d}{2} 0{,}374 = 0{,}187d; \quad (1)$$

$$h_s \geq \frac{0,187}{0,85} \cdot d_a \quad (2)$$

$$h_{admis} \leq 0,85 \cdot h_{s,} \quad (3)$$

h_s - layer thickness

$$h_s \geq \frac{h_a}{0,85} \quad (4)$$

$$d \leq \frac{0,85 h_s}{0,187}; \quad (5)$$

$$d \leq 4,55 \cdot h_{s.}\ ;\ d = d_a \text{ – diagonal fingerprint} \quad (6)$$

$$h_s \geq 0,22 \cdot d_a \quad (7)$$

These relationships, together with diagonal fingerprints size experimentally obtained can be used to apply the mathematical model Hays-Kendall [4] and analytical models Buckle [5] and Jönsson - Hogmark [6]. The Hays-Kendall tries to reconcile the effect of load dependence of microhardness (DSM) penetration, the law Kick admitting that measured hardness must be independent of applied load penetrator [7]. The basic idea for determining an intrinsic microhardness of a coating is to use only a part of the total pressing force F of the penetrator which respects Kick law ($F_2 = ad^2$, the force is dependent from diagonal footprint square), law which implies that the microhardness is constant for a given substance. Buckle analytical models [6] and Jönsson - Hogmark determining the intrinsic hardness of deposits using the results of hardness testing. Each model uses one formula of hardness, and the results of these calculations are compared with experimental results.

3. CONCLUSIONS

On the electrodeposited Ni-P alloy layers obtained by varying the time of preparation, toughness is influenced by both the thickness and applied load. Hardness test, conducted with loads applied perpendicular to the surface layers, shows that the application of small tasks for the same film thickness, hardness decreases as the applied load increases and the hardness of the thicker layer is greater. Attempts made with 1kg and 2kg shows that hardness decreases significantly and the support increasingly higher in the measured hardness value. It may say so, that measured hardness is not measured intrinsic hardness of the layer, it becomes the hardness of the composite material substrate and deposit. The obtained fingerprints micrographs for 1kg and 2kg tasks show that the layers have no cracking.
By measuring the diagonals fingerprint and by the relationship that characterize the task application perpendicular to the surface layer ($hs \cdot d \leq 4.55$) can be checked Hays-Kendall model, which agree the microhardness dependence load effect (DSM) penetration, with Kick's law admitting that measured hardness must be independent of applied load of penetrator. Also, fingerprints measured in the hardness tests can be used to the analytical

Buck and Jönsson – Hogmark tests too, for determining the intrinsic hardness of the deposits using experimental data obtained from the hardness test.

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