THE PROPERTIES OF ELECTROCHEMICAL DEPOSITION
NI-P/SiC BY TREATMENT TO 300°C

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Abstract: In this study, attempt has been made to investigate the hardness of Ni–SiC composites manufactured by electro-codeposition process, which is significantly correlated to the vol.% SiC in the matrix. The electrodeposition Ni-P/SiC coatings were obtained by the variation of the H₃PO₃ content and by the treatment heat of 300°C. The properties of the coating were evaluated by Vickers microhardness tests using variables load (15 up to 500 gram. force). The microhardness depends on the H₃PO₃ content and the composition varies according to three fields.

1. INTRODUCTION
Electroplating or electrolytic deposition of a metal on a support material in conditions able to produce a definite structure and a smooth surface finish of the deposit is mainly used in decorative and anticorrosion purposes. A different approach, adopted in this paper, for increasing the hardness of Ni/SiC coatings consists in increasing the hardness of metallic matrix of the coating by introducing phosphorus in Ni during the electroplating process.

Applying heat treatment produces increased hardness in layers.
The heat treatment at 190°C produced the desorption of the hydrogen incorporated in the coating during the electroplating process as a result of the proton reduction at the cathode. Such dehydrogenation heat treatment are frequently applied in there is a risk of hydrogen embrittlement in electroplated coatings.
The heat treatment at 420°C produce in the coatings structure the precipitation hardening phase transformation. The first exothermic peak is around 300°C and indicate the incipient precipitation of metastable NiₓPᵧ compounds and the second peak indicates the coalescence of equilibrium Ni₃P phosphide.

2. EXPERIMENTAL RESULTS
A series of Ni-P/SiC composite coatings electroplated on a mild steel support containing 0,17 %C and 1,4 %Mn have been investigated. These coatings have been obtained by procedure described in a previous paper [2].

Vickers microhardness tests have been performed at various applied loads F from 15 to 500 gram force for a three material composites Ni-P/SiC. The size d of the indentation diagonal was taken as the average of ten measured values for each material composites Ni-P/SiC and each applied load F.
The coatings have been symbolized PₓSᵧ where x is the amount of H₃PO₃ dissolved in the electrolyte (in g/litre) and y is the amount of SiC particles in suspension in the electrolyte (in g/litre).
Four Ni-P/SiC coatings have been tested for the hardness measurement. The Ni-P/SiC composite have been called P0S40, P5S40, P10S40 and P20S40.
Because of the small thickness of the coatings was in range of few tens micrometers, the hardness was characterized by a microhardness indentation method.
The microhardness apparatus equipped with a Vickers diamond indenter of square pyramidal shape having a 136° apex angle was used.
Various loads have been applied on the indenter, namely 15g, 25g, 50g, 100g, 200g, 500g. All indentations were made by applying the load F perpendicular to the smooth free surface of coatings.

Ni-P/SiC composite coatings were produced in the electrolysis cell shown in figure no.1, by varying the phosphoric acid from the electrolyte. To these deposits were applied an electrolytic heat treatment of 300°C.

Hardness tests were performed by applying loads between 15 and 500 g. Following these tests have been obtained the curves from figure no. 2 which give the dependence between the applied loads and measured hardness.

For these samples is found that the measured microhardness changes depending phosphoric acid composition of three areas.

For low content of phosphorous acid in the electrolyte ($H_3PO_3 < 5 \text{ g} / \text{L}$ - field of composition), hardness increases with the growing deposits of phosphorus acid content;
The content of $H_3PO_3$ between 5-10g/L ($2^{nd}$ field of composition), the hardness decreases.

From $H_3PO_3 = 10g / l$ ($3^{rd}$ field), the hardness presents higher values. It also notes that, since an applied load is bigger the hardness decreases. For loads up to 200g hardness varies not very high (especially in $1^{st}$ and $2^{nd}$). At 300g load the hardness begins to decrease, and decreases more and more for 500g load.

Heat treatments on Ni-P/SiC layers increases the hardness (figure no.3). This is due to the structural changes occurring with temperature changes.

![Fig. 3. Hardness variation depending on the phosphorus content and heat treatment for 500g applied load](image)

The high test value was obtained when 16 %at. P has been introduced in the Ni matrix of the composite and when the best precipitation hardening has occurred. Indeed the highest value promoted by P and by the heat treatment is four times higher than for the pure Ni composite coating. This impressive increase points to role played by P in SiC/Ni based composite coatings. By comparing in Fig. 3 the hardness value in non-treated and in heat treated condition another striking feature becomes obvious. Indeed the mere introduction of P in the Ni based supersaturated solid solution (non-heat treated condition) result in a noticeable increase in hardness but only up to a certain amount of phosphorus, above which the true hardness seems to remain constant. On the contrary in heat treated condition the true hardness are far higher and more dependent on the P content in coating.

Both series of coatings have been given a precipitation hardening heat treatment at $420^0C$ in view of strengthening the Ni matrix by precipitated submicronic particles consisting in nickel phosphides. The temperature of heat treatment has been selected in agreement with the position of exotermic peaks on the differential scanning calorimetry spectra as reported in [4].

3. CONCLUSIONS
Application of heat treatment at $300^0C$ lead to the hardness increasing after three fields of composition.
Heat treatments at various temperatures cause structural changes that are responsible for increasing hardness layers. The high test value was obtained when 16 % at. P has been introduced in the Ni matrix of the composite and when the best precipitation hardening has occurred. Indeed the highest value promoted by P and by the heat treatment is four times higher than for the pure Ni composite coating.

REFERENCES