

Elaboration and structural characterisation of NiP/talc layers

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The NiP layers which contain diverse particles have attracted considerable attention lately, especially because of their resistance to wear and improved tribological properties. The aim of this paper is to obtain and characterize NiP coatings with talc particles incorporated. Composite NiP deposits of talc particles were made to develop deposits for lubricant tribological applications.

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1. Introduction

The NiP layers which contain diverse particles have attracted considerable attention lately, especially because of their resistance to wear and improved tribological properties. The composite coatings can be prepared by different process from which the electrodeposition or autocatalytic (electroless) deposition are the most adaptable. The processes consist of incorporating particles to the metallic matrix from an electrolyte containing particles in suspension [1]. In some applications such as bearing or pipe lining, the Ni-P coatings serve as a wear resistant function layer in the corrosive environment. It has been shown that the synergistic effect under tribocorrosion, such as the interactive chemical-electrochemical – mechanical process of materials in sliding or rolling contacts immersed in a corrosive environment, can be complicated [2].

Therefore, the aim of this paper is to obtain and characterize NiP coatings with talc particles incorporated. Composite NiP deposits of talc particles were made to develop deposits for lubricant tribological applications. The process consists of inserting micrometer particles of talc in the form of particulate matter in a chemical purity nickel bath. Talc is a natural magnesium silicate with the formula $Mg_3Si_4O_{10}$ easy hydroxy (OH)₂, formed by superposition of nanometer layers (0.9 nm) [3]. Weak interactions between these layers allow their fall in relation to each other, under the effect of shear stress. On a macroscopic scale, this behavior makes talc has a very low coefficient of friction and lubricating properties when particles are present at the interface between two bodies in motion. The advantage compared to materials such as talc Teflon (PTFE), graphite or molybdenum disulphide (MoS₂) is high thermal stability, being stable in air up to 900°C, which allows us to consider the use of such deposits for applications high temperature mechanical [4].

2. Materials and experimental methods

Evidence for deposition of composite surfaces were polished, cleaned and degreased, being kept in acetone for

1 minute. Next, they were rinsed in distilled water at room temperature. To achieve a particularly cleaning these surfaces, the samples were immersed in an etching solution of hydrochloric acid (concentration 50 g/l), heated to 50°C, after which they were rinsed in distilled water at room temperature and then dried. The deposits on 35NiCrMo16 steel samples were carried out in baths industrial, low-phosphorus. Spectral analysis of selected steel as the substrate (35NiCrMo16) revealed the following chemical composition: 0.33% C, 0.30% Si, 0.40% Mn, 1.75% Cr, 0.45% Mo, 4.00 % Ni, Fe rest. The samples were heat treated before or after filing the composite layer. The type substrate microstructure is therefore ferrite-perlitic. The talc is a hydrophobic substance; therefore putting it in solution is quite difficult as talc particles tend to foam and crowding in contact with the liquid. To avoid such problems, talc particles were pretreated with thus improving their wetting ability, while decreasing their speed rearrangement. To achieve composite deposit, the amount of talc particles is fed into the nickel bath agitated by a mechanical shaker to 1000 rev / min, and then continues with a stirring speed of 500 min⁻¹ for 30 minutes. The stirred suspension is heated to 88° C, checking the pH from time to time. Talc used contains no chlorine or other minerals, is so very pure composition very close to that standard.

3. Results and discussions

3.1. Composition of deposited layers

The phosphorus content determined by X-ray spectrometry with energy dispersion (EDX) ranged between 4 ... 6% by mass; the values measured are presented in Table 1. The table shows that as the concentration increases talc composition electrolytic bath, increases the concentration of phosphorus in the composite layer deposited.

Table 1. The phosphorus content of the deposited layers with different concentrations of talc

Type of deposit	P % mass	P% atomic
NiP 0 g/l talc	3,96	5,62
NiP 40 g/l talc	3,89	6,03
NiP 120g/l talc	5,65	8,55

Figs. 1-3 present results spectrometric analysis (EDX method) to determine the initial chemical composition of the layers of composite NiP no added talc (Fig. 1), 40 g / l talc (Fig. 2) and 120 g / l talc Fig. 3).

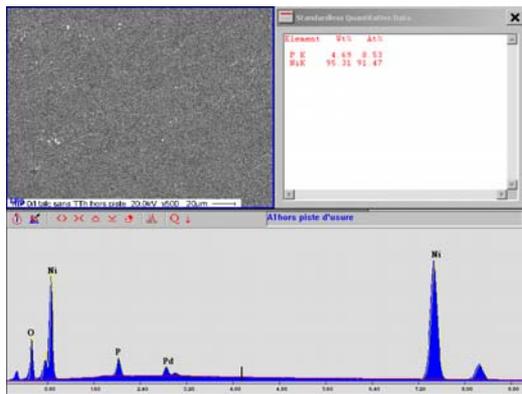


Fig. 1. EDX diagram showing the chemical composition of the composite layer deposited (NiP without talc)

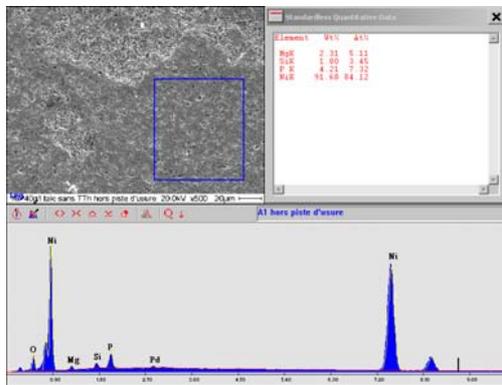


Fig. 2. EDX diagram showing the chemical composition of the composite layer deposited (NiP + 40 g / l talc)

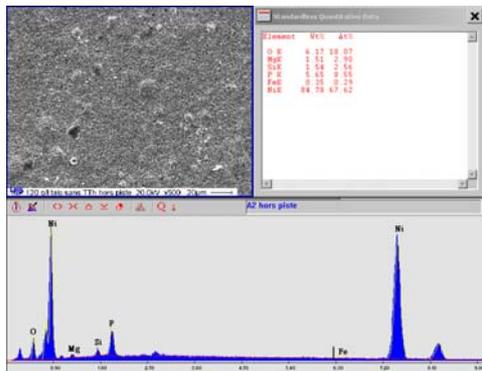


Fig. 3. EDX diagram showing the chemical composition of the composite layer deposited (NiP + 120 g / l talc)

3.2. Topography of deposited layers

Composite layers deposited surface topographies were made in white light interferometry method using an interferometer optical roughness type Veeco WYKO NT 1100. Analyzed surfaces allow lateral resolution of 1µm. Analysis of the deposited layer depth may reach 1 mm, without nanometer vertical resolution may be affected. Topographical observations were performed on three samples, one for each type of submission: chemical NiP, NiP 40g/l talc and NiP120g/l talc. Results are presented in Figures 4. By comparing values of the roughness, adding talc particles significantly increases the values of all the parameters that characterize the deposited layer roughness (R_a , R_z , R_q and R_t).

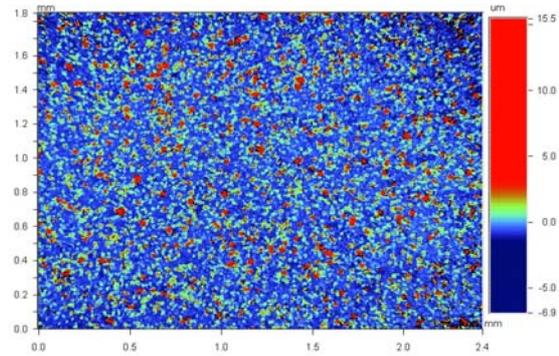
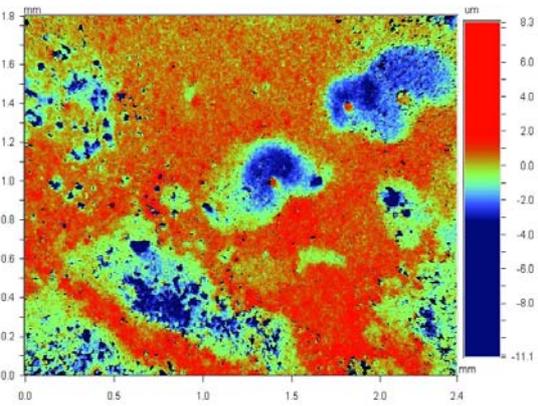
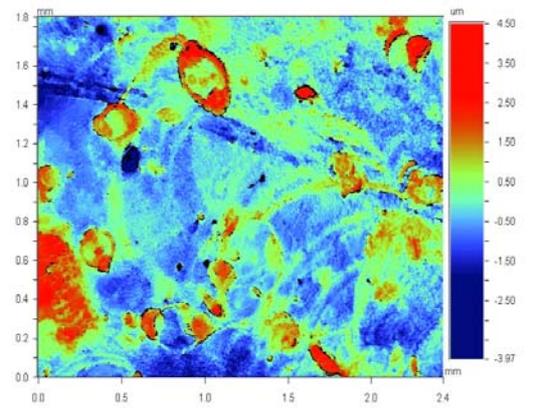


Fig. 4. a) NiP composite coating raw and with b) 40g / l talc, c) 120g / l talc

The values of roughness parameters are presented in table. 2.

Table 2. The roughness characteristics

SAMPLE	R_a (nm)	R_q (μm)	R_z (μm)	R_i (μm)
NiP	430	0.841	7.32	8.47
NiP/40g/l talc	893.71	1.21	14.74	19.43
NiP/120g/l talc	809.88	1.13	12.9	22.35

The variation of R_a parameter function of talc composition is presented in Fig. 5.

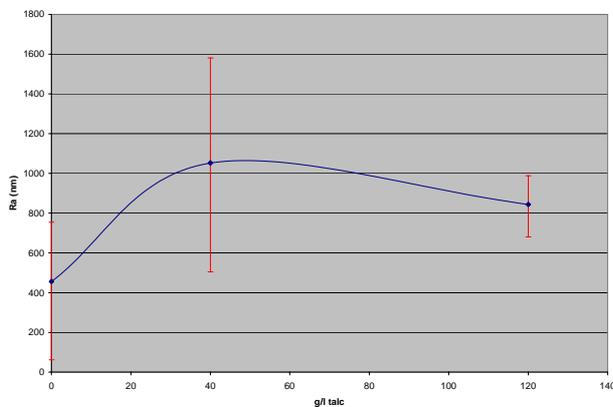


Fig. 5. Variation of R_a

The evolution of media roughness of deposited layers growth from NiP 0g/talc to NiP 40 g/l talc deposit and decrease again for NiP 120 g/l talc layer.

3.3. Morphology of the deposited layers

The surface morphology is highly influenced by the insertion of talc. In Figure 5 are shown micrographs deposited layers taken with a scanning electron microscope PHILIPS SEM 515, samples were prepared by palladium metallization.

Image analysis shows the influence of the presence of talc layer composite mass. If in absence of talc the surface layer is very smooth, in the presence of talc surface has been observed some bumps (nodules), due to agglomeration of talc particles in some areas. It can be seen especially in Figure 5 that these nodules increase as concentration increases talc in the composite layer deposition.

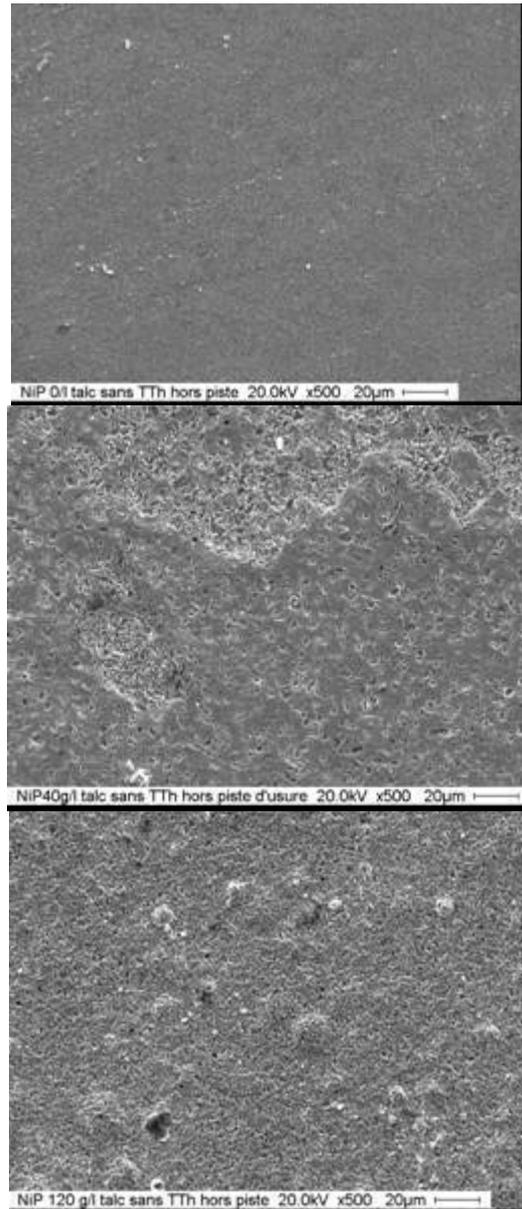


Fig. 5. Different deposit morphologies (SEM micrographs, magnification $\times 500$): a - NiP without talc, b - NiP + 40 g/l talc, c - NiP + 120 g/l talc

4. Conclusions

The interferometry analyses show that the layers are characterizing by an irregular dispersion of talc particles for 40g/l talc. The irregularity is minimalizing for 120 g/l talc layer due to the uniform dispersion of talc particles. The morphologies put in evidence that at surface the layer present various irregularities due to talc because the talc is a hydrophobic substance. Therefore putting it in solution is quite difficult as talc particles tend to foam and crowding in contact with the liquid, affecting the final properties.

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References

- [1] B. Etchevery, J. Alexis, J.Y.Paris, J.P. Bonino, J.Denape, J.A. Petit, Propriétés tribologiques de revêtements composites NiP/talc codéposés chimiquement, *Tribologie et couplages multiphysiques*, 22 – 24 mai 2006, 159 – 168, Lille, France.
- [2] B. Etchevery, Adhérence, mécanique et tribologie des revêtements composites NiP – Talc multifonctionnels à empreinte écologique réduite, thèse pour obtenir le grade de Docteur, soutenu à l'I.N.P. de Toulouse, France, en decembre 2006.
- [3] M.T. Popescu, J.Alexis, Recherche concernant le comportement des aciers revêtus au frottement sec et à haute température, *Ingenieria Automobilului*, nr. 14/martie 2010, București, România, 9 – 11.
- [4] M.T.Popescu, J.Alexis, Recherche concernant le comportement des aciers revêtus au frottement sec et à haute température, (2-eme partie), *Traitements & Matériaux*, no. 404-405/mars 2010, Paris, France.

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