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Research Article

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Use of full factorial design to study the effects of experimental factors in the electrode position of composite Ni-P + TiO_2 coating

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ABSTRACT

The composite Ni–P+TiO_2 coatings was prepared by code position of Ni–P with TiO_2 powder (anatase) on a copper substrate from the nickel-plating bath in which titanium dioxide particles were held in suspension. The code position was carried out under galvanostatic conditions. To optimize the production conditions of the Ni–P coatings modified with TiO_2 by the method of mathematical statistics, the full factorial design of experiments was used. The relationship between the percentage content in the electrodeposited composite Ni–P+TiO_2 coatings (y) and the electrodeposition parameters like cathodic current density (j dep), bath pH (pH) as well as content of TiO_2 powder suspended in the galvanic bath (c), has been described by the adequate polynomial equation and illustrated graphically. The results of the full factorial design showed that the most important effects are Concentration of TiO_2 in the bath, the interaction of TiO_2 concentration / pH and, to a lesser extent, deposition current density and pH. Such electrochemical code position method may be a good alternative in the field of composite coatings.

Key words: Electrode position, full factorial design, Ni-P-TiO_2coatings, composite materials

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INTRODUCTION

Coatings Ni-P-TiO_2, form a large series of composite materials with good mechanical and physical properties as well as good resistance to corrosion [1-2]. The multiplicity of work on this type of alloy, attests to their interest.

Composite coatings belong to the specific type of materials consisting of a crystalline or amorphous matrix and the other solid phase dispersed within it [3, 4, 5–6]. Amongst many metal matrix used, Ni and its alloys with W, Co, Fe, Mo or P, has featured strongly. The dispersed phase most often are carbides (WC, TiC, SiC), nitrides (Si_3N_4, AlN), sulfides (MoS_2), metal oxides (Al_2O_3, Al, Ti, Mo, V, Co), intermetallic (RuAl, NiAl), silicon, organic compounds as polytetrafluoro ethylene (PTFE) or polyethylene (PE), and even liquid-containing microcapsules. The presence of such phases in the Ni-based matrix permits improve the corrosion or wear resistance of the appropriate composite coatings. However, as it was reported in Ref. [6], where nickel and nickel–phosphorous matrix composite coatings reinforced by TiO_2, SiC and WC particles were produced under direct and pulse current conditions from an additive-free Watts' type bath, the embedding of ceramic particles modifies in various ways the nickel electrocrystallisation process while the Ni–Pamorphous matrix is not affected by the occlusion of these particles. It is assumed that the nickel ions adsorption on solid phase particles suspended in the solution is of a key importance in the composite electrodeposition. It has been reported that into the Ni–P matrix greater amounts of non-agglomerated particles of the solid phase can be embedded as compared to the polycrystalline Ni matrix [6]. This fact was confirmed also in the earlier study on the composite nickel–phosphorous matrix composite coatings containing TiO_2 and/or Ti micro-particles [5, 7-8].

Composite electrocoatings can be obtained by chemical, thermal or electrolytic methods [9–10]. The kind of matrix and the type of embedded solid particles determine the properties of the composite material. Due to the interaction between the matrix and dispersed substance a complex with properties different from the component's features is created.

In the presented work, it was resolved how far a change of the electrodeposition parameters values (which is easy for realization in practice of electrolytic production of composite coatings) influences the change of composition of the Ni–P+TiO_2 coatings. The choice of the matrix type and the component was based on earlier tests]. Three parameters of electroplating were chosen as in dependent variables, namely, pH of the bath (X_1), TiO2 content in the bath (X_2) and cathodic current density (X_3). The percentage content of Titane in the Ni–P+TiO2 coating (Y), was established as a criterion of the electrodeposition course.

EXPERIMENTAL SECTION

Coating deposition

Copper sheets of size 20 mm × 10 mm × 1 mm were used as the substrate material for the electrodeposition of composite Ni-P-TiO_2 coating. Before electrodeposition, the copper substrates were polished mechanically to mirror finish with 1200 grit SiC paper, degreased in a 30% ethanol solution for 5 min, washed with distilled water, and dried in air. All specimens were etched in a 10% sulfuric acid solution for 2 min and then rinsed with deionized water. The bath composition and operating conditions used for preparing composite Ni-P-TiO_2 coatings were given in Table 1.

Table 1: Bath composition and electrodeposition conditions for the composite Ni–P + TiO_2coatings

Type of the coating	Ni–P+TiO_2
Bath composition	$NiSO_4\cdot 7H_2O$ – 51g.dm^{-3} +NH_4Cl –10.7g.dm−3 +$NaH_2PO_2\cdot H_2O$ –29g.dm−3 +CH_3COONa – 10g.dm^{-3} +H_3BO_3 – 8g.dm^{-3}
TiO_2 (anatase) powder	100- 200 g.dm^{-3}
pH	5-7 (adjusted with NaOH)
Temperature	Ambiante
Deposition current density	8-15 mA.cm^{-2}
Plating time	30 min
Substrate	copper
Weight of the coating	0.18-0.22g.cm^{-2}
Adhesion	Very good
Colour of the coating	Mat-grey, white velvet-like tarnish on the surface

Table 2: Selected values of the independent variables

Level of value	Value in the coding notation	PH of the bath X_1= PH	TiO_2 content in the bath X_2=C (g.dm^{-3})	Current density of deposition X_3=jdep (mA.cm^{-3})
Lower	-1	5	100	8
Upper	1	7	200	15

The nine column in Table 3 presents the calculated values by way of the modelling procedure. The values obtained by the model (Y calculed) are compared with those of experimental data (Y experimental).

Table 3: Experimental design matrix and measured values of the considered responses (Yexp= percentage content of Titanium in the Ni–P+TiO_2 coatings),Ycal=quantity calculated on the base of the evaluated polynomial equation and residue

Run	X_1	X_2	X_3	jdep (mA.cm^{-2})	C (g.dm^{-3})	pH	Y(exp)	Y(cal)	Residue
1	-	-	-	8	100	5	3,95	3,996	0,046
2	+	-	-	15	100	5	4,85	4,804	-0,046
3	-	+	-	8	200	5	5,29	5,244	-0,046
4	+	+	-	15	200	5	6,89	6,936	0,046
5	-	-	+	8	100	7	3,8	3,754	-0,046
6	+	-	+	15	100	7	4,53	4,578	0,048
7	-	+	+	8	200	7	7,72	7,766	0,046
8	+	+	+	15	200	7	9,52	9,474	-0,046

The relation between a percentage content of titanium dioxide in the Ni–P+TiO_2 coatings (Y) and the coded variables: X_1, X_2 and X_3, which are related to the natural variables, namely jdep, C and pH, was expressed as [11,12]:

$$Y=b_0+b_1X_1+b_2X_2+b_3X_3+b_{12}(X_{12}) +b_{13}(X_{13}) +b_{23}(X_{23}) +b_{123}(X_{123}).$$

The statistical calculations and multiple regressions were performed using Nemrodw software [13]. Regression analysis was performed to fit the response function (percentage content of Titanium in theNi–P+TiO_2 coatings) with the experimental data. The values of regression coefficients obtained are given in Table 4. The final regression equation, after putting values of all coefficients, is as follows:

$$Y=5.819+0.629X_1+1.536X_2+0.574X_3+0.221(X_{12}) +0.004(X_{13}) +0.691(X_{23}) +0.046(X_{123}).$$

Table 4: Values of model coefficients.

Nom	Coefficient	F.Inflation
b_0	5.819	-
b_1	0.629	1.00
b_2	1.536	1.00
b_3	0.574	1.00
b_{12}	0.221	1.00
b_{13}	0.004	1.00
b_{23}	0.691	1.00
b_{123}	0.046	1.00

RESULTS AND DISCUSSION

1. Effects of the processing factors on the studied properties

The calculated values of the coefficients of the polynomial equations of (Y) are given in Fig.1.The comparison of the representative bars of b_1, b_2 and b_3 showed that:

Considering the algebraic value of b_2(The concentration of TiO_2effect), increasing the concentration of TiO_2 in the bath of the deposit increases the atomic weight of titanium in the Ni-P-TiO_2 coating.

Positive effects have also been observed by increasing the pH and the deposition current but were relatively less important.

The effects of interaction between factors on the properties of Ni-P-TiO_2 coating were examined on the basis of values bijFig.1 and the interaction diagrams of Fig.2.

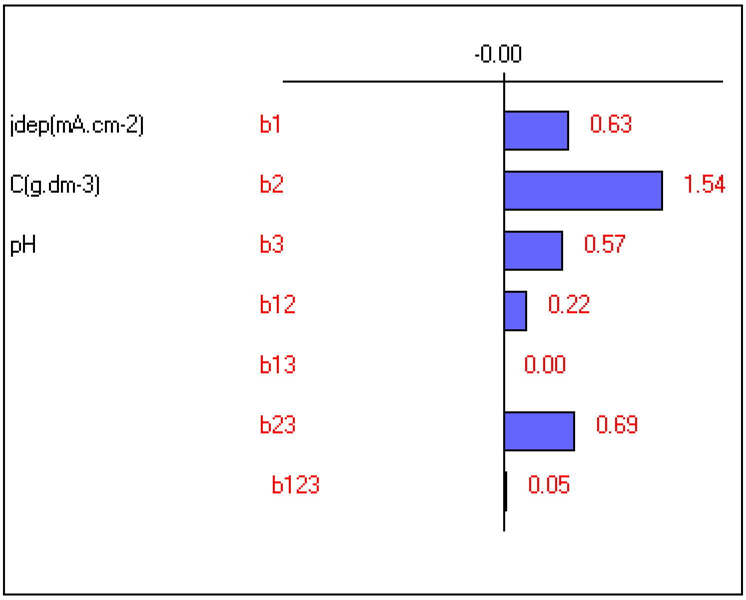


Fig.1. Values of the linear and quadratic coefficients of the mathematical equations expressing the variations of the percentage content of Titanium in Ni-P-TiO_2 coating (Y) versus coded parameters associated to the studied operating factors

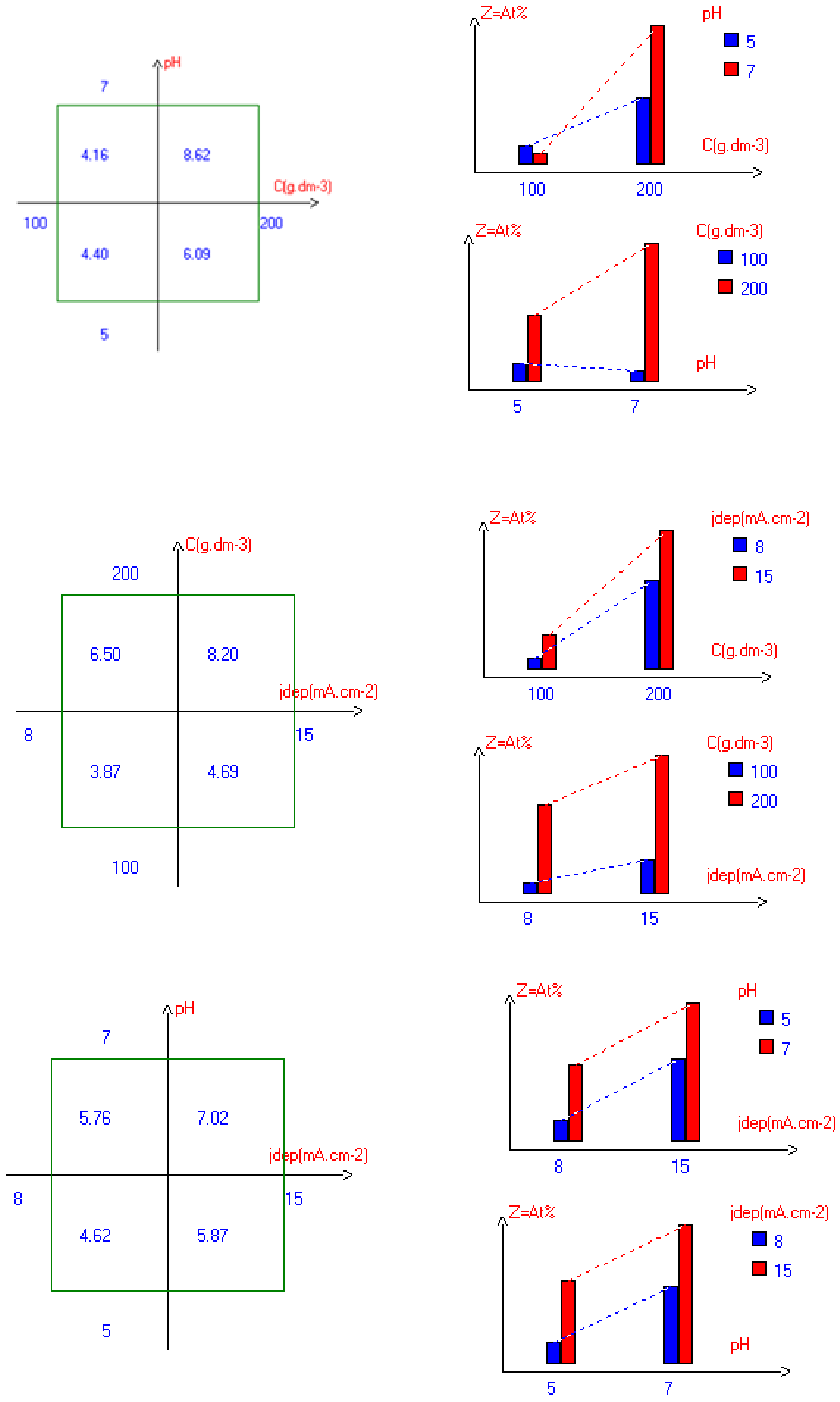


Fig.2. Diagrams representing the effects of interactions between the studied factors on the percentage content of Titanium in Ni-P-TiO_2 coating (Y)

The most important effects: Concentration of TiO_2 in the bath, the interaction of TiO_2 concentration / pH, to a lesser extent, deposition current density and pH were Somewhat comparable impacts and Their Were similar to That the interaction of TiO_2 concentration / pH

The concentration of TiO_2 seems to have an important effect that must be associated with a strong interaction concentration of TiO_2 / pH. The effect of the concentration of TiO_2 is even more marked in the neutral electrolyte (pH = 7) than in the acidic (pH = 5).

The highest level of deposition current density has the effect of increasing the performance about 1.2 units on average.

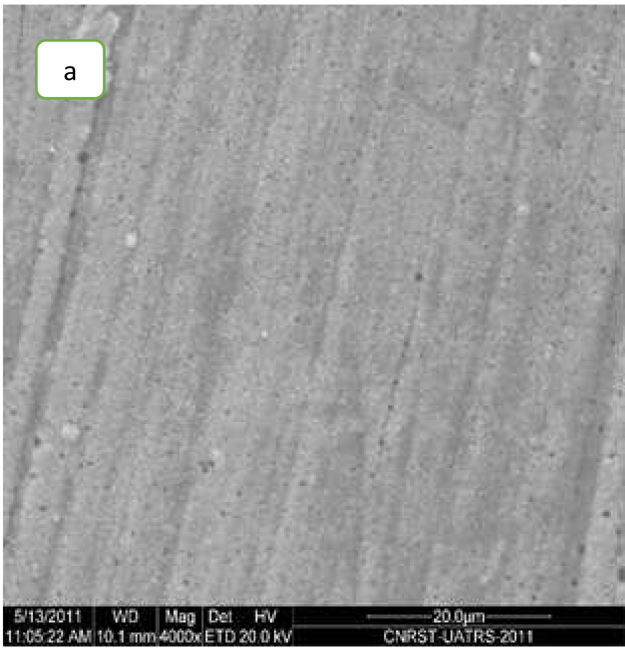
2. Chemical and physical characterization of the composite Ni–P + TiO2 coating obtained under the optimized conditions of electrodeposition.

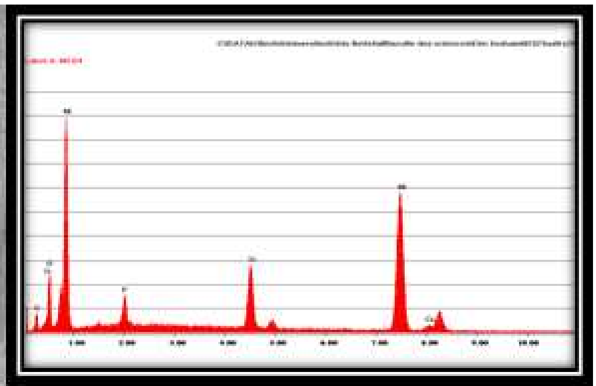
The surface morphology of the as-received Ni–P + TiO_2 (200g/l) and comparable Ni–P+ TiO_2 (100g/l) coatings obtained under the optimal parameters of the elctrodeposition process is shown in Fig. 3a and b, respectively.

Both deposits show a good adhesion to the substrate from the Cu plate. One can see that the surface morphology of the Ni-P+TiO_2 (100g/l) coating (Fig. 3b) is smooth, however, cracks formed during the electrodeposition process are visible. This coating shows silvery-grey colour and it contains (at %): Ni-69.16, P-6.64, Ti-4.53 and O-19.67. The modification of the Ni–P system by embedding of TiO_2 (200g/l) particles into the metallic matrix results in the compact and non- cracked Ni–P + TiO_2 (200g/l) coating production (Fig. 3a).

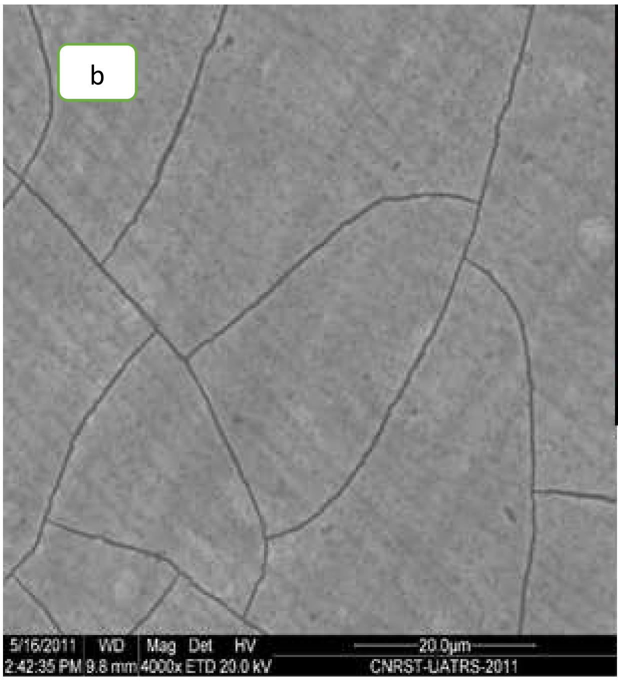
The composite Ni–P + TiO_2 (200g/l) coating has a mat-grey, rough metallic surface showthat this deposit contain (at %): Ni-54.18, P-5.26, Ti-9.52 and O-31.04.

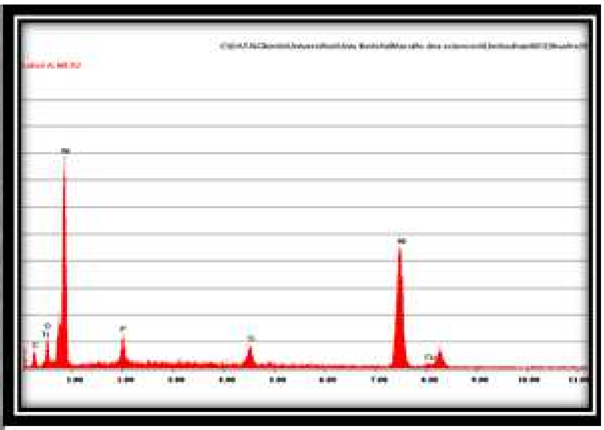
In this respect it was concluded that the increase of the amount of titanium dioxide in the electrodeposition bath had a large and positive effect on the properties measured.





Elément	Wt%	At%
O K	11.56	31.04
P K	3.79	5.26
Ti k	10.61	9.52
Ni K	74.04	54.18





Elément	Wt%	At%
OK	6.56	19.67
PK	4.29	6.64
Ti k	4.52	4.53
Ni K	84.63	69.16

Fig. 3. Surface morphology coupled with EDX of the composite Ni–P + TiO_2 coating obtained under the optimized conditions of electrodeposition (pH=7, I=15 mAcm-2 and [TiO2] =200g.dm^{-3}) (a) and comparable Ni–P-TiO_2 coating (pH=7, I=15 $mAcm^{-2}$ And [TiO_2] =100gdm^{-3}) (b)

CONCLUSION

The full factorial design was proven a useful tool for evaluating the effects of the electrodeposition current density, bath pH and content of TiO_2 powder suspended in the bath on the morpho-structural of deposits.

Adding a quantity of titanium dioxide ***[TiO₂] =200g.dm***$^{-3}$ in the electrodeposition bath, any pH=7adjustingand applying a current density jdép=15mA.cm^{-3} provides:

- The codepositedTiO_2 nanoparticles were uniformly distributed in the Ni-P matrix.
- Non- cracked coating production.

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