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### **RESEARCH ARTICLE**

# Deposition of Ni- CO/TiO<sub>2</sub> Nanocomposite Coating by Electroplating

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Abstract
$Ni - CO$ alloys have been prepared by codeposition with different contents of $TiO_2$ particles onto copper plate in a nickel plating bath. The influences of
$TiO_2$ particles concentration, current density, temperature, PH and additives on the composition of coatings were investigated. The morphology and chemical composition of composite coatings were studied by (SEM) coupled
with (EDS) energy dispersive spectrometer and examine the coatings microhardness. The results reveal TiO <sub>2</sub> particles were uniformly distributed
into Ni-CO alloy and significantly improve the microhardness of the coatings.
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## 1. Introduction

Electrodeposition of metal matrix layers is generally applied to modify the substrate surface in order to produce a wide range of useful materials with improved mechanical, decorative, electrochemical, electrical, magnetic or optical properties. Metal alloy coatings electrodeposition is more complex process than a single metal one and involves the control of several chemical and operational parameters [1-3].Research on nano-composite electrodeposited coatings has been attention directed towards the determination of optimum conditions for their production, i.e., current density, temperature, particle concentration and bath composition [3- 6]. Ni– CO alloy has been widely used as the recording head materials for computed hard drive industries [4]. The aim of this paper is to understand the electrochemical behavior of Ni-CO/TiO<sub>2</sub>coating with effect of bath parameters.

## 2. Experimental procedure

The average size of TiO<sub>2</sub> particles was 5µm. copper plate (10mm×10mm) used was as a substrate and platinum plate (40mm×40mm) as anode in the electrodeposition experiments. The electrolyte contained: NiSO<sub>4</sub> (200g/l), NiCl<sub>2</sub> (40g/l), CoSO<sub>4</sub> (40g/l), Na<sub>3</sub>C<sub>6</sub>H<sub>5</sub>O<sub>7</sub> (50g/l), H<sub>3</sub>BO<sub>3</sub> (30g/l), TiO<sub>2</sub> (5 – 20g/l) and saccharine (2.5 – 5g/l). Prior to plating, the TiO<sub>2</sub> particles of 5µm were dispersed in the electrolyte in the presence of saccharine. The substrates were sequent ultrasonically cleaned in ethanol, acetone and distilled water and then immersed immediately in the plating bath to allow the electrodeposition of the target nanocomposite coatings. The PH of solution was (5–8). The temperature was controlled (40 – 60 °C). The current density was varied between (5 – 30 A / mm<sup>2</sup>). All experiments were sufficiently wetted and remained suspended uniformly in the electrolyte. At the end of the experiments, the weight of the remaining TiO<sub>2</sub> in the electrolyte solution was calculated by vacuum filtering of the TiO<sub>2</sub> particles. The different between the final and initial weight of the TiO<sub>2</sub> in the solution produced the weight of TiO<sub>2</sub> in the coatings cross sections were observed using a scanning electronic microscopic (SEM) (JEOL JSM 840 A) and the composition of the coating was determined using an (EDS) coupled with the (SEM). The hardness of the composite coatings with a thickness about (5 – 25 µm) was measured by a

Vickers microhardness instruments at an applied of (50 g) for 10s. All tests were performed under unlubricated condition at room temperature and in ambient air.

#### 3. Results and discussion

Many researchers have been conducted to explain the mechanism of codeposition of fine particles with metal from electrolytic solutions. In a simple way, the particle incorporation in a metal matrix could be described as a four steps process: as shown in figure 1.

- 1. Formation of surface charge on particles in suspension,
- 2. Particle mass transfer from the bulk of the suspension to the electrode surface,
- 3. Particle-electrode interaction,
- 4. Particle incorporation and irreversible entrapment simultaneously with growing metal layer [7].



Fig. 1. Some of the steps involved in the metallic matrix composite electrodeposition.

To study the effect of TiO<sub>2</sub> particles concentration, figure 2 shows the relationship between the amount of codeposited TiO<sub>2</sub> and the concentration of TiO<sub>2</sub> particles in the plating bath at the fixed current density of  $30A/mm^2$  at 45 °C.



Fig. 2. Effect of amount of TiO<sub>2</sub> in the bath on the weight percentage of TiO<sub>2</sub> in the composite coating at 30 A /mm<sup>2</sup> and 45 °C.

In figure 2 exhibited an increase in the particle incorporation with increasing particle content of the electrolyte. The maximum amount of codeposited  $TiO_2$  particles was achieved at the concentration of 20g/l of  $TiO_2$ . Then, further increase in  $TiO_2$  concentration has no effect on wt% of codeposited  $TiO_2$ . The embedding of  $TiO_2$  can be attributed to the adsorption of suspended particles on the cathode surface as suggested by Guglielmi's two – step adsorption model [8]. After (25g/l) bath load of  $TiO_2$  resulted in a slight decrease in the weight of the  $TiO_2$  in the deposited and indicated that the adsorption of  $TiO_2$  particles in the coating has reached the saturated state [9].



Fig.3. Effect of amount of TiO<sub>2</sub> in the bath on the percentage weight of particles with different current densities.

Figure 3 shows the relation between the bath load in the deposit and the current density at various  $TiO_2$  concentrations in the electroplating bath. According to the stockes mode, the increasing in the current density accelerates the electrophoretic velocity of  $TiO_2$  particles and increases the columbic force between  $Ni^{2+} - CO^{3+}$  adsorbed on particles and the cathode which is accordance increase the  $TiO_2$  content in the Ni– CO deposit [10]. From figure 4, it is shown that the thickness of the deposit is increased with increasing the current density up to  $30A/mm^2$  and the after that decreased gradually. This decreasing in the thickness may be because that the increasing in the current density result in more rapid deposition of metal alloy matrix and fewer particles are embedded in the coating [11].



Fig. 4. Effect of current density on thickness of the compositesat 45 °C, PH 6.5 and 20 g / 1 TiO<sub>2</sub> concentration.



Fig. 5. Effect of PH on thickness of the composites with 30 A /mm<sup>2</sup>, 45 °C and 20 g / 1 TiO<sub>2</sub> concentration

The effect of PH on the thickness of coating is illustrated in figure 5. It is clear from these results that the thickness is increased with increasing PH up to 6, after that the thickness is decreased due to the nickel hydroxide precipitation. From figure 6, it is shown the effect of one of the important parameter in the electroplating is the bath temperature . It shown that the thickness of coating is increased with the temperature increasing up to  $60C^{\circ}$ , after that the hydrogen evolution hinders the composite deposition.



Fig.6. Effect of temperature on thickness of the composites with 30 A  $/mm^2$ , PH 6.5 and 20 g / 1 TiO<sub>2</sub> concentration.



Fig. 7. The variation of microhardness with TiO<sub>2</sub> concentration and different current densities at PH 6.5 and 45 °C.

The incorporation of  $\text{TiO}_2$  in the coating increased the microhardness, the microhardness of Ni– CO/ TiO<sub>2</sub> composite coatings with the different contents of TiO<sub>2</sub> particles and different current densities are shown in the figure 7. From the figure 7, the enhancement in the microhardness of Ni– CO/ TiO<sub>2</sub> composite coatings was due to the strengthening effect caused by the dispersion of TiO<sub>2</sub> particles in the composite coating, which impeded the motion of dislocation in the metallic matrix [12]. The SEM of the Ni– CO/ TiO<sub>2</sub> coatings is presented in figure 8. The addition of titania particles to the bath obviously affects the surface morphology of the coatings. The obtained composite layers have a mat gray metallic surface with white Ni– CO spots visible to the naked eye. Ni– CO/ TiO<sub>2</sub> composite coating was more compact and consisted of smaller and spherical – sized grains.



Fig.8. SEM micrograph of sample codeposition at 30 A / mm<sup>2</sup>, PH 6.5 and 45 °C with:

- a.  $10 \text{ g/l TiO}_2$  bath concentration without saccharine.
- b.  $15 \text{ g/l TiO}_2$  bath concentration with 3 g/l saccharine.
- c.  $20 \text{ g/l TiO}_2$  bath concentration with 5 g /l saccharine

### 4. Conclusion

- 1. Ni– CO/ TiO<sub>2</sub> composite were successfully prepared by electrodepostion process in the Ni– CO plating bath.
- 2. It is recommended to prepare the Ni– CO/ TiO<sub>2</sub> composite coatings of the largest weight percentage of TiO<sub>2</sub> particles by plating in the bath containing (20 g/l) TiO<sub>2</sub> particles at temperature 45 °C, current density of 30 A/mm<sup>2</sup> and PH of ( $6 \pm 0.5$ ).
- 3. TiO<sub>2</sub> particles reinforced Ni– CO composite coating revealed a much smoother surface microstructure with excellent mechanical properties

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