

# Influence of pH on the electrochemical deposition of composite coatings in copper matrix with TiO<sub>2</sub> nanoparticles

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**Abstract:** Cu-nanoTiO<sub>2</sub> composite coatings electroplated from copper sulfate bath were obtained and characterized. The paper presents the influence of the different pH of Cu–TiO<sub>2</sub> coatings obtained at DC current density. The pH were 0.5 and 2 in electroplating bath. The surface morphology and composition of layers were studied by scanning electron microscopy (SEM) and EDX analysis. The influences of pH parameter on the thickness, grain size and surface morphology of coating were studied.

**Keywords:** Optic, Surface, SEM, TiO<sub>2</sub>

## 1. Introduction

At present, nano-materials have many applications due to their unique characteristics. have been used in order to fabricate different types of composite materials Co–Al<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>–Al, and Cu–SiC [1–3]. It is expected that electrochemical techniques could play an increasingly significant role in nano-technology. Metal–matrix composite electrodeposition refers to electrolysis in which nanoparticles are suspended in the electrolyte and are subsequently embedded in the electro-formed solid phase. The resulting composite possesses properties that differ from the bulk, depends on the degree and type of particle incorporation [4–5]. The properties of nano composite materials depend not only on the properties of their individual parents, but also on their morphology and interfacial characteristics [6–7]. Experimental work it has generally shown that virtually all types and classes of nanocomposite materials lead to new and improved properties, when compared to their macrocomposite counterparts. Therefore, nanocomposites promise new applications in many fields such as mechanically reinforced lightweight components, non-linear optics, battery cathodes and ionics, nanowires, photo catalyst activity, field effect transistors (as dielectric gate), UV detectors (as photo absorber), dye-sensitized solar cells (DSSC),

electrochromic displays sensors and other systems [8–10]. Due to relation of optical properties and photo responsively of TiO<sub>2</sub> nanoparticles to nanoparticle size, surface area and morphology, optimization of these parameters in order to having efficient response have crucial importance [10].

The paper presents an investigation on copper coatings obtained with TiO<sub>2</sub> nanoparticles by electrocodeposition.

Two different copper electrolytes which cover almost the complete pH range were investigated. The results are compared with copper coatings without oxide nanoparticles to understand the effects of dispersed phase on the properties of composite coatings.

## 2. Experimental

Copper–TiO<sub>2</sub> nano composites were deposited from an acidic copper sulfate bath. The basic composition of electrolyte and deposition parameters is given in Table 1. For the deposition of Copper–TiO<sub>2</sub> composite coating, copper was selected as substrate. The approximate dimensions of the substrates were and prior to the electrocodeposition were ground using 1000, 1500, 2000, 3000, 5000 grades silicon carbide papers. Copper plates of 10mm×20mm×0.8mm area were used as anode. The surfaces of cathodes were activated in 10% sulfuric acid solution. Before deposition the solution was allowed for

Ultrasonication for 25 minutes and magnetic stirring for 30 minutes for homogenous dispersion of the TiO<sub>2</sub> powder in the solution. The pH plating parameters were changed according to Table 2.

**Table 1.** Electrolyte composition and deposition conditions.

Electrolyte (Acidic copper sulfate bath)	Copper Sulfate (CuSO <sub>4</sub> .5H <sub>2</sub> O) : 0.8 (M) Sulfuric acid (H <sub>2</sub> SO <sub>4</sub> )
Current density	50 $\frac{mA}{cm^2}$
Temperature	27 °C
Plating time	20 minutes
Dispersion	TiO <sub>2</sub> : 1gr

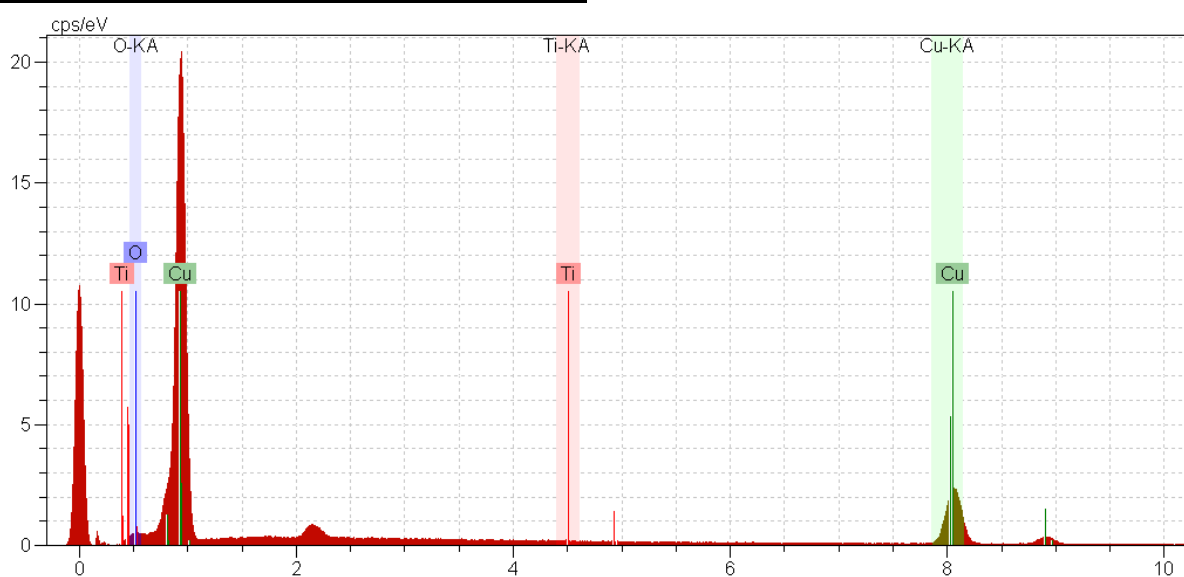
**Table 2.** pH plating Composite Cu–TiO<sub>2</sub>

sample	pH
1	2
2	0.5

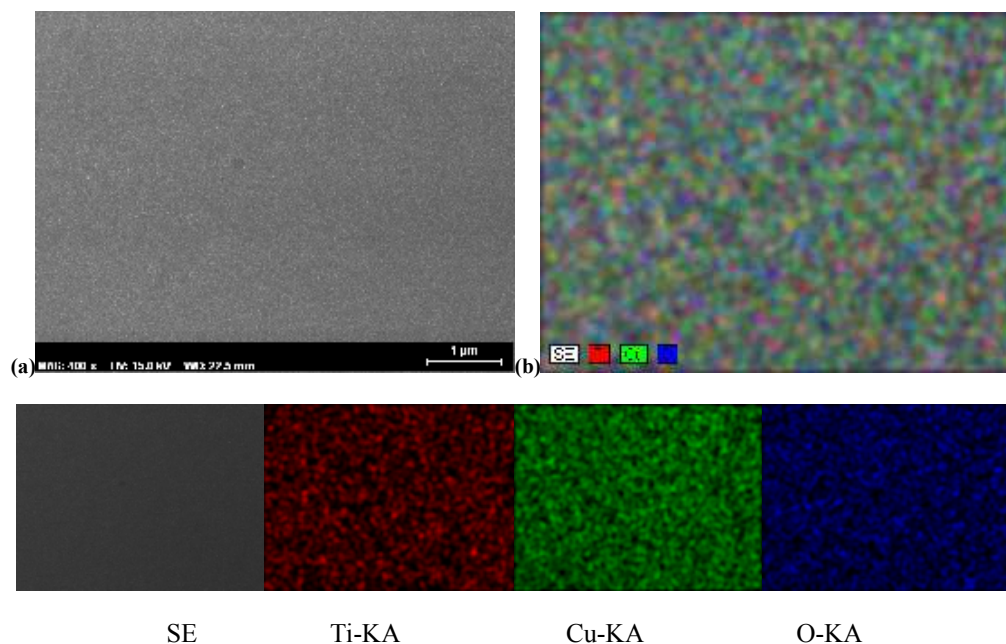
Composite Cu–TiO<sub>2</sub> coatings produced by electroplating method were examined by EDX and the surface morphology and particle distribution of the electrodeposited composite coatings were performed by using SEM.

### 3. Results and Discussion

Fig. 1. showed that the presence of nano-TiO<sub>2</sub> particles in composite coatings was evidenced by energy dispersive analysis, EDX Composite and Cu–TiO<sub>2</sub> coatings produced by electroplating method were examined by EDX, as presented in Figs. 2.

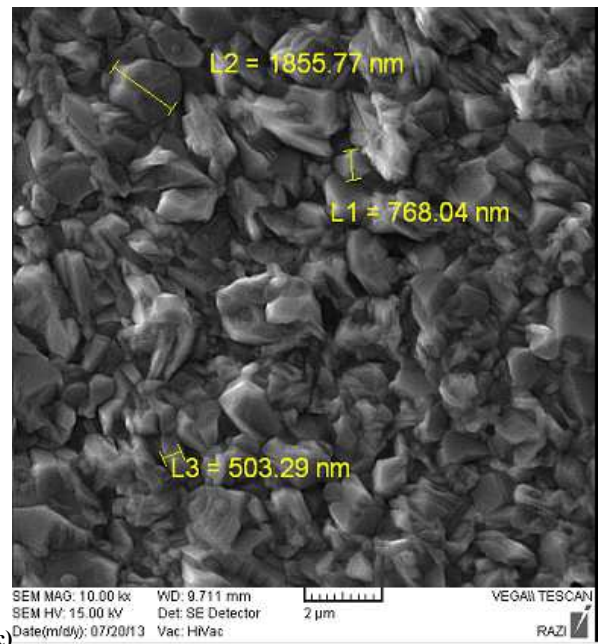
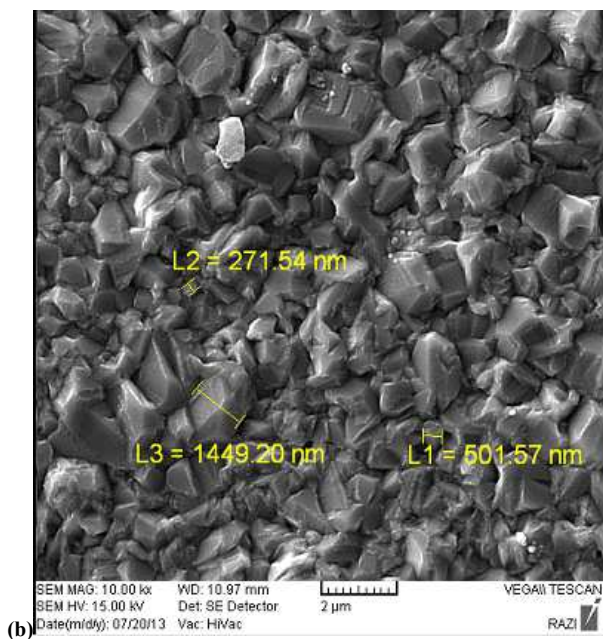
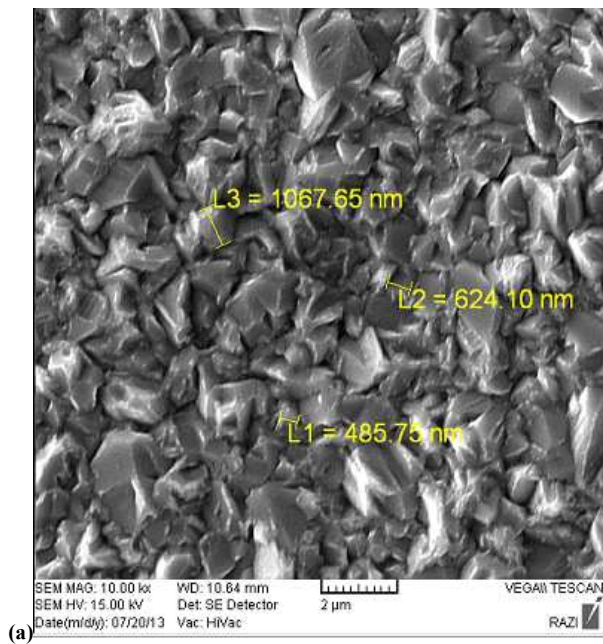


**Fig 1.** EDX spectrum of Cu/nano-TiO<sub>2</sub> composite coatings



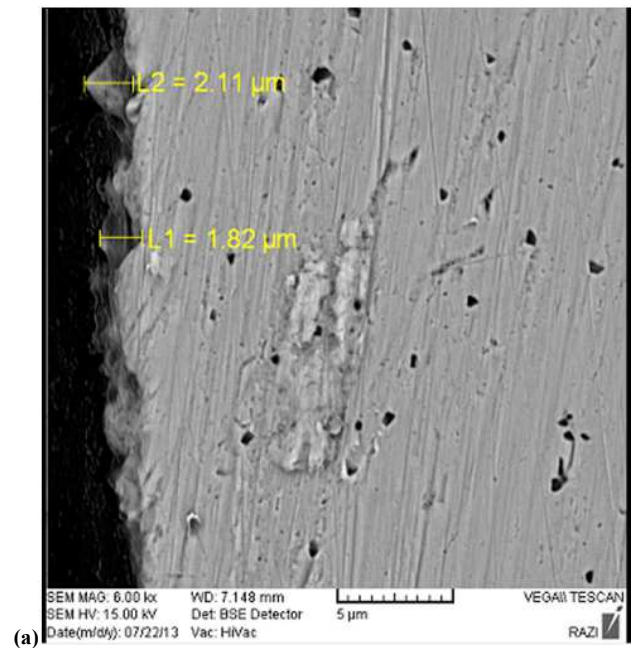
**Fig 2.** EDX analysis (map data) of Cu–TiO<sub>2</sub> composite coating on copper substrate, pH is 2, a) SEM image and b) Map data.

Fig. 3. shows the SEM surface morphology of pH plated coatings, with and without nano- $\text{TiO}_2$  particles. The effect of nano- $\text{TiO}_2$  particles with different pH on the coating grain size was considerable. The average grain size of about 725.83nm in copper non-composite coating, about 740.77 nm in pH=2 at Cu- $\text{TiO}_2$  composite and was increased to 1042.36 nm due to the pH=2at Cu- $\text{TiO}_2$  composite. In contrast, the effect of nano-  $\text{TiO}_2$  particles seems to be different in coatings plated by different pH. The distribution of  $\text{TiO}_2$  particles in pH=2 seemed to be more homogeneous as compared with composite pH=0.5 plated coating.

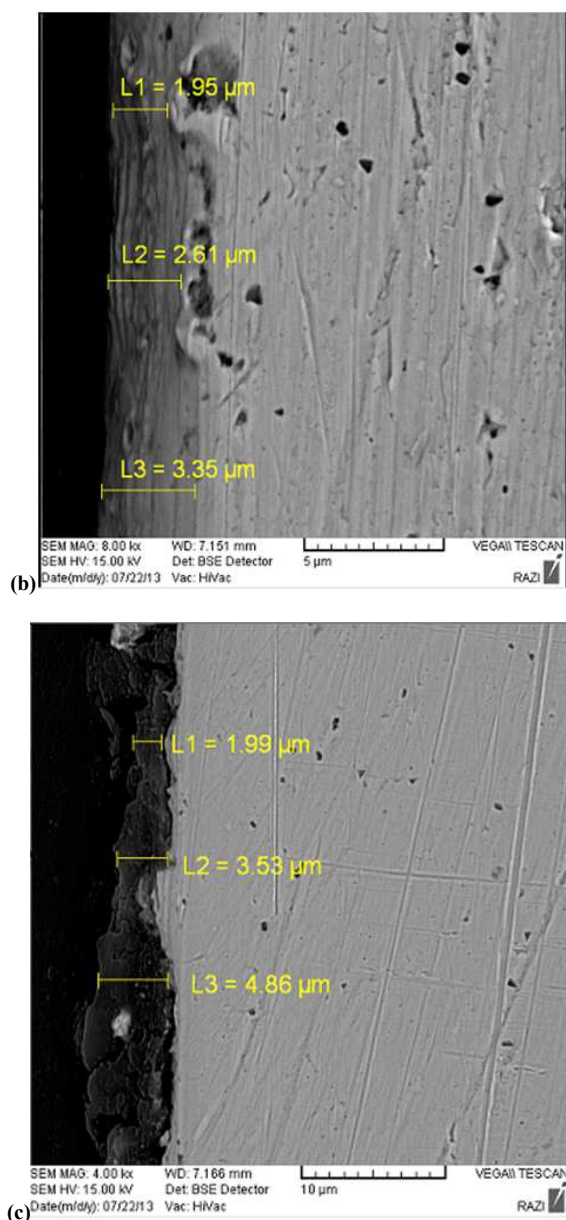


**Fig 3.** Surface morphologies of coatings applied by DC electroplating ( $\times 2000$ ), (a) non-composite copper coating, (b) composite copper nano- $\text{TiO}_2$  coating with pH=2, (c) pH=0.5.

The cross-sectional SEM micrographs of coatings are shown in Fig 4. The composite coating thickness applied by pure copper layers and different PH of Cu- $\text{TiO}_2$  composite. As it is seen, the thickness of coatings applied by pure copper layers is about 1.97  $\mu\text{m}$ , whereas the coating thickness of pH=2 plating coating is about 2.63  $\mu\text{m}$  and pH=0.5 plating coating is about 3.38  $\mu\text{m}$ .







**Fig 4.** Cross-sectional scanning electron micrographs of, (a) pure copper coatings, (b) composite coatings with nano-TiO<sub>2</sub> pH=2 and (c) pH=0.5 in electrolyte.

From the above Figure 4.8 it can be observed that smooth coating surfaces was observed on the coating surfaces in pH=0.5 and also small pits were observed at pH=0.5 this is attributed to evolution of hydrogen at the cathode surface.

## 4. Conclusion

In the present study, Cu-TiO<sub>2</sub> nanocomposite coatings were developed successfully by using Electrodeposition process on the Copper substrate from copper sulfate bath. From the detailed investigation of the results obtained, the

following conclusions can be drawn:

The Scanning Electron Microscope (SEM) showed that the presence of nano-TiO<sub>2</sub> particles in different pH affected the grain size and homogeneity of the coating morphology. It led to bigger grain size in pH=0.5 plated coatings.

Good quality deposits (finer grain size and more homogeneous) can be obtained at rather pH. For example at pH=2.

Cross-sectional micrographs showed that the lower pH plated composite coatings were more coherent as compared to rather pH and the maximum obtained thickness of the pH=0.5 plated.

Cross-sectional micrographs surface morphologies obtained the coating surfaces observed at pH=0.5 deposition parameters were smooth.

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