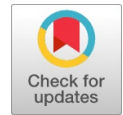# Nano SiC Reinforced Copper Nanocomposite by a Simple Electrodeposition Method

S. Ramalingam

Abstract: The Cu and Cu-SiCnanocomposite coatings were prepared by a simple electrodeposition technique from acidic copper sulphate electrolyte using SiC nanoparticles with an average particles size of 50 nm under optimized bath composition and preparation conditions for possible electrical contact material applications such as electrical switch and wiring boards where higher electrical conductivity and thermal conductivity is required. The scope of the present study is to enhance the strength, mechanical, corrosion and wear resistance properties of Cu-SiCnanocomposite compared to pure Cu coatings. The as prepared Cu and Cu-SiCnanocomposites were characterized for structural, mechanical and corrosion resistance properties by EDX, XRD, FESEM, Vickers microhardness tester and AC-impedance and Tafel polarization techniques. The elemental composition (wt% of Cu and SiC nanoparticles) of Cu-SiCnanocomposites was analyzed by EDX coupled with FESEM analysis confirms the presence of nanoSiC in the copper matrix and they were 89wt% of Cu and 11 wt% of SiC nanoparticles respectively. The surface morphology of Cu and Cu-SiCnanocomposites was studied by FESEM analysis shows that SiC nanoparticles were uniformly dispersed in the surface of the copper matrix compared to pure copper. The crystallite structure and grain size of the Cu and Cu-SiCnanocomposite electrodeposits was measured by XRD analysis. From the XRD results, the grain size calculated using Debye-Scherrer's formula was ~35 nm for pure Cu and ~33 nm Cu-SiCnanocomposite. The crystallite structure of Cu and Cu-SiCnanocomposites was fcc (face centered cubic) and the preferred orientation of the plane was (220). The microhardness of Cu-SiCnanocomposite coating increased with increasing SiC nanoparticles concentration in the bath compared to pure Cu coating. The corrosion resistance measurements were performed for the pure Cu and Cu-SiCnanocomposite coatings by electrochemical impedance spectroscopy (EIS) and Tafel polarization techniques. It shows that, Cu-SiCnanocomposites has high corrosion resistance than pure Cu in 3.5wt% NaCl solution.

Keywords: Electrodeposition, Cu-SiCnanocomposite, SiC nanoparticles, Corrosion resistance, Microhardness.

I. INTRODUCTION

In the recent years, on the basis of production and utilization, the copper and copper based alloys possess third position after iron & steel and aluminum, due to its exceptional electrical conductivity, thermal conductivity and good corrosion resistance [1-2].Though copper is an excellent electrical and thermal conductor material, it possesses low wear resistance and mechanical strength. In order to increase the mechanical strength, corrosion and wear resistance, the second phase nanoparticles reinforced metal matrix nanocomposites has been prepared and reported in the earlier are $Cu-TiO_2$, $Cu-CeO_2$, $Cu-SiO_2$, $Cu-ZrO_2$, $Cu-Al_2O_3$, $Cu-Si_3N_4$, Cu-CNTs etc. [3-12].These second phase nanoparticles reinforced metal matrix nanocomposites showed increased hardness and better corrosion and wear resistance compared to particle free metal electrodeposits [13-22]. The advantages of electrodeposition is an excellent low cost and low temperature technique compared to the conventional methods such as powder metallurgy, sputtering, spin coating, thermal spraying, centrifugal casting etc., which is used to coat over the complicated conductive surfaces. Electrodeposition is an industrially viable and economically preferable technique for the deposition of metals and alloys and metal matrix nanocomposites (MMNCs) [23-32].

The SiCnanoparticlesare very hard materials and possess high strength and high thermal conductivity, good corrosion and wear resistance and low cost material. This property of SiC nanoparticles makes them useful in ceramic applications such as ceramic bearing, textile ceramics, high frequency ceramics and ceramic engine parts. Also, SiC nanoparticles are resistant to oxidation at high temperatures and has very high melting point of 2730°C. In order to increase the strength, corrosion resistance, wear resistance and heat resistance of composite materials, currently the SiC nanoparticles can be used.

Presently, the production of copper matrix nanocomposites by various methods has increased due to their potential industrial applications such as electrical contacts and connector materials in electrical devices [1- 2][33-38]. The aim and scope of the present work is to synthesize nanoSiC reinforced Cu matrix nanocomposites by simple electrodeposition method using acid copper sulphate electrolytes with optimized bath composition and process conditions.

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The synthesized Cu-nanoSiC composites were characterized for structural, mechanical and corrosion resistance properties by EDX, FESEM, XRD, Vickers hardness tester and Electrochemical AC impedance and Tafel polarization techniques and the results were compared with the pure copper electrodeposit.

II. EXPERIMENTAL METHODS

A. *Electrodeposition of Copper and Cu-SiCNanocomposites*

The chemicals, copper sulphatepentahydrate ($CuSO_4$. $5H_2O$), sulphuric acid (H_2SO_4), gelatin and SiC nanoparticles (size 50 nm) used for the electrodeposition of copper and Cu-SiCnanocomposites are pure analytical grade and purchased from Merck and Sisco research laboratories (SRL).

The electroplating bath was prepared by weighing the chemicals as per the bath composition and dissolved in deionized water and kept in magnetic stirrer for 10 minutes to ensure the complete dissolution of all the chemicals. The electrolytic grade pure copper anode and mechanically polished copper cathodes are used in the deposition process. First, the copper cathodes are cut in the machine to the required size (4 cm^2 areas) and then polished with course and fine emery sheets and degreased in acetone to remove oil and grease from the surface of the cathodes. Then it is dipped in dilute HCl solution and then washed with distilled water and air dried to use in the deposition bath for the preparation of the composite coatings.

The copper sulphatepentahydrate ($CuSO_4.H_2O$) (0.3M) and sulphuric acid (H_2SO_4) (1.3M) solution is prepared with de-ionized water in 500 ml beaker. To this 10 g/l , 20 g/l, 30 g/l and 40 g/l of nanoSiC particles (size 50 nm) are dispersed with gelatin additive (1 g/l) separately and this solution is kept agitated for 6 to 8 hr to ensure complete and uniform dispersion of nanoSiC in the electrolytic copper sulphate bath. Then, the pure copper anode and polished copper cathodes are immersed in the electrolytic bath and connected to the DC regulated power supply. The electrodeposition was carried out with the current density of 5 A/dm^2 for the duration of 60 minutes at 30ºC [3][5][8][11].The pH of the bath was maintained at ~1 throughout the process. The electroplating bath composition and preparation conditions are given in the Table1. After the completion of the deposition process, the samples were taken out from the bath, washed with distilled water and air dried. The particle free pure copper is also electroplated using the same bath composition without the addition of nanoSiC particles. The pore free, uniform and smooth surface samples were obtained with the thickness of ~65 µm by electrodeposition technique [3][5][8][11].

Table1: Bath composition and conditionsfor the electrodeposition of copper and Cu-SiCnanocomposites

Bath composition and operating parameters	
$CuSO_4.5H_2O$	0.3 M
H_2SO_4	1.3M
SiC nanoparticles (50 nm)	0, 10 g/l , 20 g/l, 30 g/l, & 40 g/l
Gelatin additive	1 g/l

Current Density	5 A/dm^2
pH	~1
Temperature	30°C
Deposition Time	60 minutes

B. *Characterization of Cu-SiCNanocomposites*

The electrodeposits of pure Cu and Cu-SiCnanocomposites were characterized for structural, mechanical and anti-corrosion properties. The crystallite size and structure was measured by X-ray diffractometer (XRD) (Bruker D8 Advance Eco) with CuKα radiation source of wavelength λ = 1.5406 A° operating at 40 kV to 25 mA. The grain size of pure Cu and Cu-SiCnanocomposites was measured by using the Debye-sherrer equation;

$$D = \frac{0.9\lambda}{\beta \, Cos\theta}$$

Where D is the crystallite size and λ-is the wavelength of the radiation, β-is the full width at half maximum height (FWHM) of the peak. The microstructure and elemental composition of the Cu-SiCnanocomposites was examined by FESEM coupled with EDX (Quanta FEG 250) analysis. The microhardness of the electrodeposited pure Cu and Cu-SiCnanocomposites was measured by Vickers microhardness tester.

The corrosion resistance of the electrodeposits of pure Cu and Cu-SiCnanocomposites was measured by electrochemical impedance spectroscopy (EIS) and Potentiodynamic polarization techniques. The corrosion rate (CR) was calculated using i_{corr}(corrosion current) values by the formula [3][5][8][11];

$$\text{Corrosion rate (CR)} = k \left(\frac{i_{corr}}{\rho}\right) EW$$

Where, Corrosion rate (CR) is given in mm/yr, $k = 3.27 \times 10^{-3}$ mm g / µA cm yr, ρ = Density of Cu (8.96 g/cm^3), EW = Equivalent weight of Cu (31.77).

III. RESULTS AND DISCUSSIONS

A. *EDX Analysis of Cu-SiCNanocomposites*

The weight % of elements present in the Cu-SiCnanocomposites were analyzed by Energy dispersive X-ray spectroscopy (EDX) analysis. The representative EDX spectrum for the electrodeposited Cu-SiC (30 g/l) nanocomposites is shown in Fig. 1.
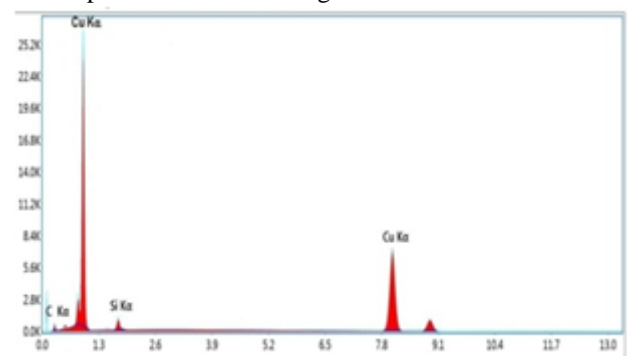

Fig. 1 EDX Spectrum of Cu-SiC (30 g/l) nanocomposite coating.

The weight % of Cu and SiC nanoparticles present in the Cu-SiC (30 g/l) nanocomposite coatings are 89% and 11% respectively, and the corresponding EDX mapping analysis pictures are shown in Fig.2(a-e).

The EDX spectrum (Fig.1) and its mapping analysis pictures (Fig.2) confirm the presence of SiC nanoparticles on the copper matrix by electrodepositiontechnique. The presence of 11 wt % of SiC nanoparticles on the copper matrix enhances the mechanical and anti-corrosion properties of the Cu-SiCnanocomposite coatings.
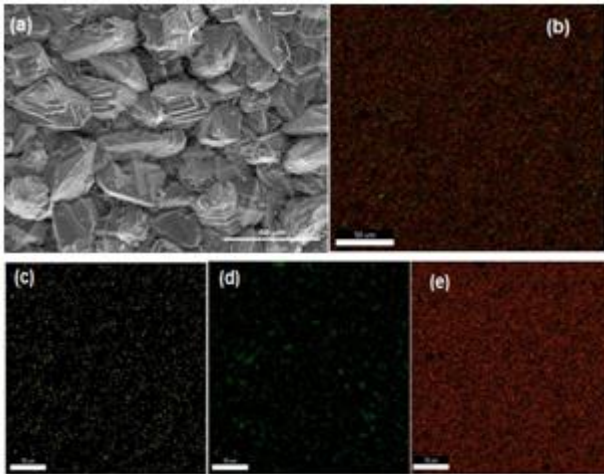

Fig.2(a-e) FESEM image and its EDX mapping pictures.
(a) FESEM image of Cu-SiC (30 g/l) nanocomposite,
(b) EDX mapping picture of Cu-SiC
(30g/l)nanocomposite, (c) presence of C, (d) presence of Si
(e) presence of Cu.

B. FESEM Analysis

FESEM (Field Emission Scanning Electron Microscopy) (Quanta FE-SEM 250) was used to examine the Surface morphology of electrodeposited Cu and Cu-SiCnanocomposites are shown in Fig.3(a&b). The electrodeposited pure copper shows (Fig.3(a)) uniform cauliflower like crystallites with outward growth and the Cu-SiC (30 g/l) nanocomposite shows (Fig.3(b)) uniformly distributed SiC nanoparticles on the surface of copper matrix. FESEM image (Fig.3(b)) confirm the presence of SiC nanoparticles on the surface of copper and these uniformly distributed SiC nanoparticles enhance the mechanical, corrosion and wear resistance properties of the Cu-SiCnanocomposite coatings.
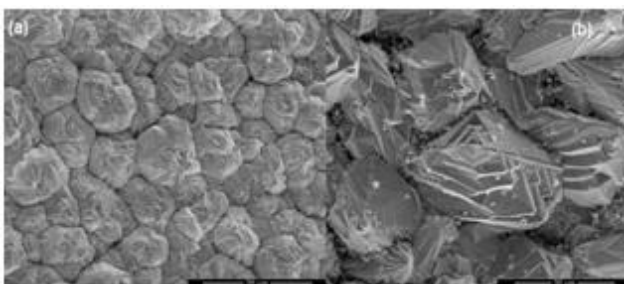

Fig. 3(a&b). FESEM images of (a) Electrodeposited Cu and (b) Cu-SiC (30 g/l) nanocomposite coating.

C. XRD Analysis of Cu and Cu-SiCNanocomposites

The structural analysis was performed for the electrodeposited pure Cu and Cu-SiCnanocomposites by X-ray diffractometer (Bruker D8 Advance Eco) and their XRD pattern is shown in Fig.4. The grain size was calculated using Debye-Sherrer formula for the XRD patterns of Cu and Cu-SiCnanocomposites were ~35 nm for pure Cu and ~33 nm for Cu-SiCnanocomposites. The crystallite structure of electrodeposited Cu and Cu-SiCnanocomposites were crystalline fcc (face centered cubic). It is observed that from the XRD studies, the electrodeposits of pure Cu and Cu-SiCnanocomposites exhibit the preferred orientation along the plane (220) (Fig.4).The XRD patterns of pure Cu and Cu-SiCnanocomposites are matched and confirmed with the standard JCPDS copper reference (JCPDS Card No.040836) [3] [5][8][11].
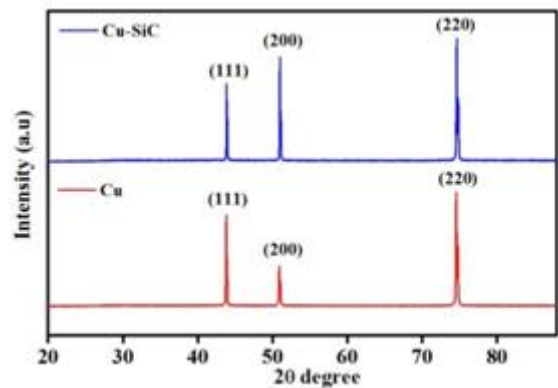

Fig.4. XRD Patterns of electrodeposited pure Cu and Cu-SiC (30 g/l) nanocomposite coatings

D. Effect of SiCNanoparticles Concentration

Fig.5 shows the relationship between the SiC nanoparticles concentrations in the bath and the weight % of codepositedSiC in the electrodeposits. The weight % of codepositedSiC increases with increasing SiC nanoparticles concentrations in the bath from 10 g/l to 30 g/l. Further increasing the SiC nanoparticles concentrations above 30 g/l, the weight % of codepositedSiC decreases for the 40 g/l particles loading; this was confirmed by EDX analysis (Fig.5).
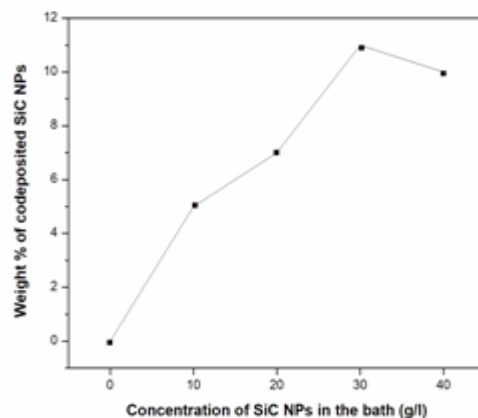

Fig. 5.Effect of concentration SiC NPs in the bath (g/l) andthe weight % of codepositedSiC NPs.

The mechanism of codeposition of SiC nanoparticles on the cathode surface follows the Guglielmi's two step adsorption model [39].

As per the Guglielmi's two step adsorption model, in the first step the SiC nanoparticles are loosely adsorbed and they are in equilibrium with the particles in the suspension. In the second step the SiC nanoparticles are irreversibly adsorbed on the cathode surface to form the Cu-SiCnanocomposite thin films [39].

E. Mechanical Properties

1. Vickers Microhardness

Vickers microhardness was measured for electrodeposits of pure Cu and Cu-SiCnanocomposites are shown in Fig.6. The microhardness of the Cu-SiCnanocomposites increased with increased content of SiC nanoparticles in the composite electrodeposits. The higher microhardness of the Cu-SiCnanocomposite is due to the dispersion strengthening effect caused by the added SiC nanoparticles and the nucleation and grain refinement of SiC nanoparticles during the electrodeposition process [3][5][8]. This enhanced microhardness of the Cu-SiCnanocomposite coatings is also due to the reduction in grain size of Cu-SiCnanocomposite coatings (grain size of Cu-SiCnanocomposite is ~33 nm) obtained by electrodeposition method and confirmed by XRD analysis.
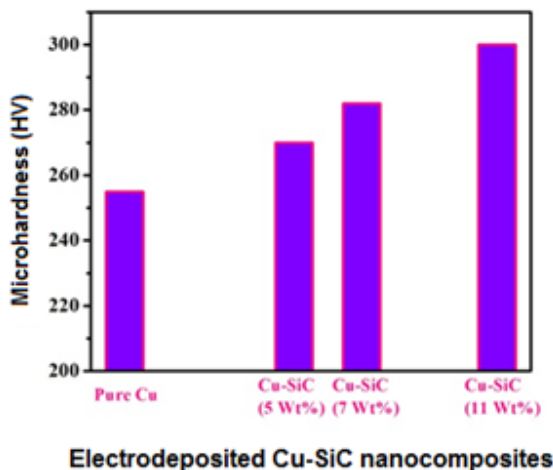

Fig. 6.VickerMicrhardness (HV) of pure Cu and Cu-SiCnanocomposite coatings

F. Corrosion Resistance Studies

Corrosion resistance measurements were performed for the electrodeposited pure Cu and Cu-SiCnanocomposites by electrochemical impedance spectroscopy (EIS) and Tafel polarization techniques and the results were presented in Table(2&3).

1. Electrochemical Impedance Spectroscopy (EIS) Analysis

The electrochemical impedance spectroscopy (EIS) analysis was performed to evaluate the impact of SiC nanoparticles (NPs) on the corrosion resistance of the Cu-SiCnanocomposite coatings is shown in Fig.7.The electrodeposited coatings of 1 cm^{2}area were immersed in 3.5wt% NaCl solutionact as working electrode and the commercial saturated calomel electrode (SCE) was used as

the reference electrode for the measurement of all potentials. The platinum wire was used as auxiliary electrode in the three electrode cell assembly for the measurement of corrosion rate of the Cu and Cu-SiCnanocomposite coatings. The electrochemical impedance nyquist plots were recorded and the charge transfer resistance (R_{ct}) was measured for all the coatings and the results are tabulated (Table2). From the results, it is observed that the charge transfer resistance (R_{ct}) was increased with increasing SiC content in the Cu-SiCnanocomposite coating compared to electrodeposited pure copper coatings. It shows that the Cu-SiCnanocomposite coatings were more corrosion resistance than the electrodeposited pure copper in 3.5wt% NaCl solution.
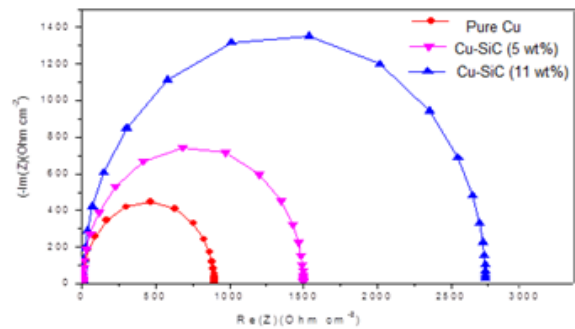

Fig.7.Nyquist plot of electrodeposited Cu and Cu-SiCnanocomposite coatings.

Table2: Impedance parameter values for the Cu and Cu-SiCnanocomposite electrodeposits.

Electrodeposit Details	Weight % of SiC	$R_{ct}(\Omega/cm^2)$
Electrodeposited copper	0	898
Cu-SiCnanocomposite	5	1501
Cu-SiCnanocomposite	11	2733

2. Tafel Polarization Studies

Tafel polarization studies were performed by immersing the working electrode (electrodeposit) of 1 cm^2 area in 3.5wt% NaCl solution in a three electrode cell assembly and the results were presented in Table3(Fig. 8). The corrosion potential (E_{corr}), corrosion current (i_{corr}), and the tafel slopes ba and bc are calculated from the tafel polarization curves (Fig. 8) and are given in the Table3.

Table3:Tafel parameter values for the electrodeposited pure Cu and Cu-SiCnanocomposite coatings.

Electrodeposit Details	Weight % of SiC	E_{corr} (V) vs SCE	I_{corr} (µA/cm^2)	Corrosion rate (mm/year)
Electrodeposited copper	0	-227.769	13.802	0.1599
Cu-SiCnanocomposite	5	-241.891	12.523	0.1451
Cu-SiCnanocomposite	11	-348.165	4.897	0.0567

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From the results, it is observed that the corrosion current (i_{corr}) and the corrosion rate (CR) were decreased for all the Cu-SiCnanocomposites compared to pure Cu coatings (Table3).

It shows that, Cu-SiCnanocomposites were more corrosion resistance than the pure copper coatings (Table3).
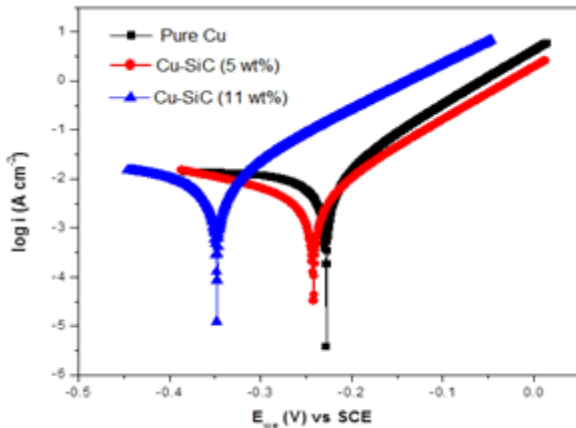

Fig. 8.Tafel plots of electrodeposited Cu and Cu-SiCnanocomposite coatings.

IV. CONCLUSIONS

The Nanostructured Cu and Cu-SiC composite coatings were successfully fabricated by a simple electrodeposition technique and they were characterized by EDX, FESEM, XRD and Vickers Microhardness and Electrochemical AC impedance and Tafel polarization techniques for possible electrical contact material applications. Based on the results, the following conclusions were deduced.

- From the XRD studies, the grain size was measured by using Debye-Sherrer formula for Cu and Cu-SiCnanocomposite coatings and found to be ~35 nm and ~33 nm respectively.
- The crystallite structure of Cu and Cu-SiCnanocomposite coating was fcc (face centered cubic) and the preferred orientation was (220).
- The Vickers microhardness of Cu-SiCnanocomposites increased with increasing the SiC content in the composite coatings compared to pure copper coatings.
- The electrochemical impedance spectroscopy (EIS) and Tafel polarization studies confirmed that Cu-SiCnanocomposite coatings was high corrosion resistance than electrodeposited pure copper in 3.5wt% NaCl solution.

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