Study the Effect Some of Electrodeposition Parameters on Wear Resistance for Cu-MWCNT Composite Coating

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Abstract

A Copper- Mullet walled Carbon Nanotube Cu/MWCNT composite coatings prepared by direct and pulse electrodeposition with various contents of MWCNT particles, using electrodeposition method from acidic sulfate bath. The effect of the current density, pulse frequency and particle loading (PL) of MWCNT particle in the electrolyte on the morphology and volume fraction Vol% of MWCNT in deposit were investigate. The experimental results indicated that the depositing condition affect on the microstructure of deposited copper and MWCNT Vol% in deposit. Also, the Vol% of MWCNT increases in deposit with particle loading (PL) increasing in the bath and increased with increased current density. Increasing MWCNTs in electrolyte produced increased microhardness values and increasing content MWCNT in the electrolyte resulted in decrease wear rate.

Keywords: Multiwalled Carbon Nanotube; copper; Composite; Coatings; Electrodeposition.
1. Introduction

Electrocodeposition is a process by which particles are entrapped during electroplating to produce a metal matrix composite. Small particles are dispersed in the electrolyte bath to be codeposited with the metal matrix film. Other methods for producing particle reinforced metal matrix films include thermal spraying, powder metallurgy, stir casting, etc. A large variety of composite films have been deposited by electrocodeposition. For example, matrix materials such as Au, Cu, Ni, and Co have been plated. Particle reinforcements have included SiC, Al₂O₃, TiO₂, carbon nanotubes, and organic particles [1]. Electrocodeposition has numerous advantages in developing metal matrix composite coatings amongst other coating processes for example, uniform depositions on complexly shaped substrates, low cost, good reproducibility and the reduction of waste [2]. The properties and structure of composite coatings donot only depend on the distribution, concentration, and nature of the reinforced particle, but also indirectly on the kind of electrolyte used and deposition parameters (current density, current deposition mode (Direct and pulse) temperature, pH values, etc.). It is worth mentioning that in case of pulsed deposition; the controlling parameters are more, leading to better control on the coating and its properties when compared with the conventional direct current. Pulse current electrodepositions have higher mechanical and tribological properties than direct current platings [3]. Fatima N. Jaseem and Mofeed A.L. Jaleel obtain Ni/SiC micro-composite coatings with a variety of contents of SiC particles of particle size (10 μm) [4], by using electrodeposition method from nickel watts bath in which the SiC particles suspend Hayashi et al. proposed that the amount of metal ions adsorbed on particles has an important influence on the codeposition behavior. Roos and coworkers also developed a mathematical model for codeposition [5]. Carbon nanotubes (CNTs) show a better combination of mechanical, electrical, optical, thermal and structural properties. (CNTs), in particular, show great advantages as a matrix-reinforcing phase for composite coatings in high wear environment due to their extremely high stiffness, strength and lubricity [6]. In this work, we examine the effects of electrodeposition conditions on the surface morphology and microstructure of Cu/MWCNT composite plating coatings produced using a sulfuric bath. Also, physical properties, such as hardness, wear resistance of the composite coatings were characterized.

2. Experimental process

2.1 Specimen preparation

Specimens with estimated dimensions of (5×5×0.8 cm³) of copper sheet were used as substrate. The surface substrates were ground use SiC paper down to 2000 grade. Only one of the two surfaces with an area of (4×4 cm²) were used as a plating area by dividing other surfaces with suitable polymer adhesive, giving an effective plating area of (16) cm². Then the substrates copper was rinsed in distilled water, rinsed with alcohol, pickled in 10% (H₂SO₄) for 30s, followed by washed with distilled water, dried and prepare to deposition.

2.2 Co depositions

The codeposition of Cu/MWCNT composite coatings were prepared by using electrocodeposition set up with vertical and parallel electrodes have a constant distance of (9) cm for all experiment. The electrolyte was acidic sulfate bath. The composition and the variety of the parameters of the experimental procedure are listed in Table-1. The electrolyte was mixed with MWCNT powder of particle size (10) μm, supplied by (Cheap Tubes Co.). The MWCNTs did not disperse homogeneously in the base bath; therefore, uniform dispersion of MWCNTs was achieved by adding of a poly(acrylic acid PA5000), mean molecular weight of 5000 dispersant to base bath with stirring, and magnetically stirred for 1 h and followed by ultrasonically agitated for 1: 30 h just before electroplating to improve the wettability of particles and to get homogeneous dispersion of reinforcement particles in the electrolyte. During the electrodeposition, a magnetic stirring was done continuously to keep away from settling down of MWCNT. The bath temperatures was maintain by using heating element (filament wire), which was directly curved in bath, and controlled by temperature controller. The plating time was changed according current density to ensure that all coatings was about (50) μm thick.
Table 1-Composition Bath and Operational parameter

<table>
<thead>
<tr>
<th>Electrodeposition parameter Direct Current(DC) Electrodeposition</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Electrolyte (Acidic copper sulfate bath)</td>
<td>200 g/l CuSO₄.5H₂O and 15 ml H₂SO₄</td>
</tr>
<tr>
<td>Current density</td>
<td>(0.01-0.05) A/cm²</td>
</tr>
<tr>
<td>Temperature</td>
<td>30±2 °C</td>
</tr>
<tr>
<td>Multi-wall carbon nano tube (MWCNT)</td>
<td>(0.5, 1, 3, 5) g/l</td>
</tr>
<tr>
<td>Partical loading</td>
<td>1 ±(0.2)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Pulse current(PC) Electrodeposition</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Peak current density</td>
<td>0.03 A/cm²</td>
</tr>
<tr>
<td>Plating time</td>
<td>1:15 min</td>
</tr>
<tr>
<td>Frequency</td>
<td>25, 50, 75 Hz</td>
</tr>
</tbody>
</table>

Result and Discusses:

3.1 Morphological Test

In order to study the morphology of prepared electrocomposite coating and examine the MWCNT particle incorporations in coatings were used scanning electron microscopes (SEM) (INSPECT S50 (FEI) made in Netherlands). The surface imagery in Figure-1 shows that morphology of the (Cu/MWCNT) composite coatings deposition at different current density and constant partials loading 1 g/l, it can be noted that the grain size increase with increase CD. This may be due to the nature of the construction which occurs in the deposit.

![Figure 1-SEM microstructure of Cu/MWCNT composite with different current density, constant PL 1g/L: (a)0.01 A/cm² (b)0.03 A/cm² (c) 0.05 A/cm²](image)

3.2 Vol% of MWCNT in Deposit

The Vol% of MWCNT particles in the copper matrix was assess by dissolution analysis, which include calculation the weight % of MWCNT which situated within the Cu matrix, after dissolve both the substrate and Cu matrix by using solvents do not have an effect on the particles. The MWCNT Vol% reinforced MMCs prepared by electrodeposition technique cannot be only controlled or set for the required Vol% but it form in the simple range which can be controlled the Vol% of MWCNT by controlling the deposition parameter.

Influences of plating parameter on MWCNT vol% in deposit

1. Current Density:

Figure-2 shows that when the CD increases, the vol % of MWCNT particles in deposit increase, the increase current density increases throwing power as well as increasing of polyacrylic acid(PA) leads to increase (ev) of MWCNT and works to attractive positive copper ions on the surface of the
MWCNT leading to the completion of it strongly towards to the cathode which leads to increasing vol% MWCNT in the deposit.

![Figure 2](image2.png)

**Figure 2** - Difference of vol % MWCNT in the deposit with CD, PL= 1g/l

2. Partical Loading (PLs):
The MWCNT Vol % in deposit increases with increasing the PLs in the plating bath as shown in Figure-3. The increasing perhaps due to increase the amount MWCNT particle that attainment the surface of cathode with increasing the concentration of MWCNT particles in plating bath. Only the particles which stay adsorbed on the surface of cathode for a sufficient time are effectively codeposited into the growing copper matrix [7]. As the amount of particles future the surface of cathode increases with increasing the concentration of MWCNT in the plating bath.

![Figure 3](image3.png)

**Figure 3** - Difference of vol. % MWCNT in deposit with particle loading CD= 0.03 A/cm²

The vol % of codeposited MWCNT in coatings, increased with increasing the pulse frequency at constant current density in the deposition as shown in Figure-4. The repeated process in the pulse deposition mode increases the MWCNT Vol% in the composite.
3.3 Microhardness

Microhardness of composite coating (Cu-MWCNT), pure copper coatings and the pure copper substrate was determined by using a LARYEE microhardness tester (model HVS-1000, made in China) with indenter of diamond pyramidal part with square base having an angle of 136° between the opposite faces. After completing indentation process, the two diagonals will be measured. The test was carried out with load 100 gf for 15 seconds to make sure that indentation is up to the coating surface only. The hardness values were taken at 5 different places on the surfaces and average of these values were measured in the results. Figure-5 shows the relationship between the microhardness and PL at constant current density. It can be observed that the rise of the hardness with increasing frequency may be attributed to the reason for increase in the vol% of MWCNT. This can be associated with the fact that an increase in the bath loading provide more number of particles for adsorption at the cathode, leading to a larger number of particles receiving co-deposited, and these results in the increase of the hardness in the composites. In other word the increase in the MWCNT vol% in the deposition rise the value of microhardness as show in Figure-(6a, b)

Figure 5-variation of the micro-hardness with Particle loading for pure and composite coatings by pulse deposition
3.4 Wear Rate

A locally made wear test machine was developed to approve the (Ball-on-disk) mechanism according to the ASTM G99-95a. The developed machine consists of rotating disc of aluminum with four grips in order to fix the plate sample. While the counter part was a hardened steel ball (Ø10mm).

The wear rates for pure Cu and composite coating tested were displayed in Figure-7. Some of the result shown that deposition occurs on the ends of MWCNT leading to voids and thus increases in granular volumes. Although the vol% of MWCNT in the deposit increased but there was a rise in the natural result of the construction that left gaps in the coatings despite the slight increase in the hardness.

Figure 6-Variation of microhardness with Vol % of MWCNT for different (a) current density (b) Partical loading

Figure 7-Variation of wear with Vol % of MWCNT for different current density.

Figure-8 shows that the result decreases in wear rate with pulse current increasing. It was result to increase vol% MWCNT and increase hardness as such notes increase wear at PL low and decrease at PL high. The attributable reason that to action MWCNT on increase threading grains coatings copper resulting to increase energy suspend is there copper of coatings.
Figure 8 - Variation of the wear rate with Particle loading for pure and composite pulse deposition

4. Conclusion

Based on investigational results obtained in this work, it was possible to sketch the following conclusions:

1. The MWCNT incorporation in Cu matrix influenced by deposition parameters, an increase of the MWCNT Vol% in composite deposit is established by increasing in PL and current density.
2. The results revealed that introducing MWCNTs into the electrolyte resulted in increasing the amount of MWCNTs in the copper electrodeposited layer. Increasing MWCNTs in the electrolyte produced increased microhardness values and MWCNT content in electrolyte increasing resulted in decrease the amount of wear rate.

5. References