COPPER MATRIX COMPOSITE LAYERS CO-ELECTRODEPOSITED FROM SULFATE BATH WITH ALUMINA NANOPARTICLES SYNTHETIZED BY SOL-GEL TECHNIQUE

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Abstract

The alumina nanoparticles (Al_2O_3) were synthesized using the sol-gel technique from an inorganic solution. The obtained nanoparticles had a microstructure of corundum after calcination at 1000°C. For characterization of alumina nanoparticles, use the FE-SEM/EDS method with mapping software, XRD, and TEM. The α-Al_2O_3 phase was detected with an average particle size of 100 nm. After synthesis of Al_2O_3 nanoparticles, the various concentrations of particles were added in lab-made sulfate electrolyte (1.0, 3.0, and 5.0 wt. %). The direct current (DC) galvanostatic regime with constant current density (50 mA·cm-2) was chosen for co-electrodeposited free Cu layers and an alumina particles in-corporated copper composite layers. Three layer thicknesses were designed: 2, 22, and 52 μm with the aim of investigating the influence of layer thickness on the change in mechanical (hardness and adhesion), structural (grain size, crystallinity), topography (roughness), and hydrophilic/hydrophobic (water contact angle) properties of the layers. The optimal layer thickness and concentrations of alumina particles were done. After microindentation testing, increased microhardness values were observed: 9.96 % (1.0 wt. % of α-Al_2O_3), 134.1 % (3.0 wt. % of α-Al_2O_3), and 61.9 % (5.0 wt. % of α-Al_2O_3) compared with the Cu layer without alumina reinforcement. The adhesion values of Cu and Cu-Al2O3 layers were evaluated according to micro indentation adhesion method and Chen-Gao mathematical model which used for calculated the critical reduced depth. The best adhesion has a layer of Cu-Al_2O_3 with 1.0 wt. % of alumina particles and 52 μm thickness. The surface roughness parameter of the alumina-reinforced copper layer increased from 3 to 7 times compared to Cu-free layers, according to AFM analysis. By incorporating 3.0 and 5.0 wt. % of α-Al_2O_3, the composite film becomes harder than the brass substrate (HV = 144), and layer hardnesses were 102 HV (1.0 wt. % of α-Al_2O_3), 217 HV (3.0 wt. % of α-Al_2O_3), and 150 HV (5.0 wt. % of α-Al_2O_3) for an optimal layer thickness of 22 μm. The oscillation of microhardness values is consistent with the change in copper grain size. Based on image analysis, the grain size of the copper layer is in the range of 0.7 to 2.5 μm, and for the layer with particles, it is in the range of 0.9 to 4.5 μm. Grain size of copper layes have the same trend as the roughness, shows an increasing character with increasing layer thickness and with the incorporation of alumina particles. Wettability of the layer is better when α-Al_2O_3 nanoparticles are incorporated in the layer, and the measured water contact angles ranged from 66.57 ± 0.94° to 81.42 ± 1.10°.

Keywords: *sol-gel, alumina, copper matrix composite, microhardness, adhesion.*