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Influence of parameters of the pulsating current (PC) regime on morphological, structural and hardness characteristics of copper coatings electrodeposited on Si(111)

Ivana Mladenović¹, Nebojša Nikolić¹,
Jelena Lamovec², Dana Vasiljević Radović¹, Vesna Radojević³

¹ University of Belgrade, Institute of Chemistry, Technology and Metallurgy, Belgrade, Serbia

² University of Criminal Investigation and Police Studies, Belgrade, Serbia

³ University of Belgrade, Faculty of Technology and Metallurgy, Belgrade, Serbia

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Electrodeposition of Cu was performed on Si(111) by the pulsating current (PC) regime in the range of the average current densities (j_{av}) between 15 and 70 mA cm⁻². The selected values of the average current densities were attained by varying either pause duration (t_p : 28.3, 15, 7.5 and 5 ms, i.e. j_{av} : 15, 25, 40 and 50 mA cm⁻² for the constant values of deposition pulse of 5 ms and current density amplitude (j_A) of 100 mA cm⁻²) or amplitude of the current density (j_A : 120 and 140 mA cm⁻², i.e. j_{av} : 60 and 70 mA cm⁻² for the constant values of deposition pulse of 5 ms and pause duration of 5 ms). Morphological and structural characteristics of the obtained coatings were examined by scanning electron microscope (SEM), atomic force microscope (AFM) and X-ray diffraction (XRD), respectively. With increasing the average current density, morphologies of the coatings changed from those with large and well defined crystal grains obtained at j_{av} of 15 mA cm⁻² (the dominant effect of activation control) to fine-grained obtained at j_{av} of 50 mA cm⁻² (the mixed activation-diffusion control) and those with globules when diffusion becomes a dominant process ($j_{av} = 70$ mA cm⁻²). The minimum roughness showed the Cu coating obtained at j_{av} of 50 mA cm⁻². Simultaneously, crystal structure changed from the strong (220) to the strong (111) preferred orientation with increasing average current density. The change of surface morphology was discussed by the effect of applied parameters of the PC regime on the type of electrodeposition control, while change in crystal orientation of produced coatings was explained by various rates of growth on various crystal planes. Hardness analysis of the produced coatings was performed by application of the Chicot-Lesage (C-L) composite hardness model. By application of this model, the relative indentation depth (RID; where $RID = h/d$; h is an indentation depth, and d is a thickness of coating) of 0.14 was established as the limiting value separating the area of the absolute hardness of the Cu coatings ($RID < 0.14$) from the area in which application of the C-L model is necessary for a determination of the absolute hardness of coatings ($RID > 0.14$). For $RID < 0.14$, the measured composite hardness corresponded to the absolute hardness of the coating.

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