

13th Multinational Congress on Microscopy

September 24-29, 2017 in Rovinj, Croatia

BOOK OF ABSTRACTS

Editors

**Andreja Gajović, Igor Weber, Goran Kovačević, Vida Čadež
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The comparative XRD and SEM analysis of electrochemically produced silver nanostructures

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Electrodeposition technique is very suitable way to obtain nanostructures of the desired shapes and dimensions. It is attained by the choice of regimes and parameters of electrolysis, such as the type and composition of electrolyte, temperature of electrolysis, the type of working electrode, the time of electrolysis, the addition of additives, application of periodically changing regimes of electrolysis, etc. (1). Thanking its unique electrical, chemical and optical characteristics, silver nanostructures found wide applications in electronics, optics, catalysis, sensors, etc (2). In this study, we produce silver nanostructures via electrochemical route from the two various types of electrolytes: basic (nitrate) and complex (ammonium) ones. Ag particles were produced by the potentiostatic regime of electrolysis at the room temperature using Pt as the working electrode. Electrodepositions were performed at overpotentials which corresponded to the plateaus of the limiting diffusion current density, i.e. at 90 mV (for the nitrate electrolyte) and 625 mV (for the ammonium electrolyte). Morphologies of the obtained particles were characterized by the technique of scanning electron microscopy (SEM), while the preferred orientation of the particles was evaluated by X-ray diffraction (XRD) analysis by calculation of the „Texture Coefficient“, $TC(hkl)$ and the „Relative Texture Coefficient“, $RTC(hkl)$ (3). The needle-like dendrites, as that shown in Figure 1a, were formed from the



nitrate electrolyte at 90 mV. The X-ray diffraction (XRD) analysis of the needle-like dendrites (Figure 2) showed their strong (111) preferred orientation. On the other hand, very branchy 3D (three dimensional) pine-like dendrites (Figure 1b) were formed by electrodeposition at 625 mV from the ammonium electrolyte. Analysis of the pine-like dendrites showed that they are constructed from corn-cob-like forms with spherical grains as the basic element. The X-ray diffraction analysis of the pine-like dendrites (Figure 2) showed the considerably smaller degree of Ag crystallites oriented in the (111) plane indicating almost random orientation of crystallites in these particles. The strong difference in morphology of the obtained particles is explained by different affiliation of Ag electrodeposition processes from these two electrolytes. Namely, Ag electrodeposition from the nitrate electrolyte belongs to the fast electrochemical processes (the high exchange current density values), and then, Ag is classified into the group of the normal metal (1). On the other hand, when Ag is electrodeposited from the ammonium electrolyte, then Ag is classified into the group of the intermediate metals which the basic characteristics are considerably lower exchange current density values than the normal metals. Simultaneously, the different preferred orientation of the obtained particles can be ascribed to different growth rate on these crystal faces caused by different surface energy values.

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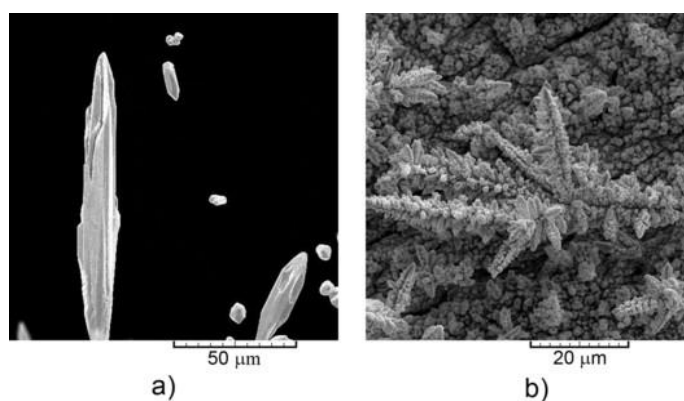




Figure 1. (a) The needle-like dendrite electrodeposited at an overpotential of 90 mV from 0.10 M AgNO₃ in 2.0 M NaNO₃, and (b) The pine-like dendrites electrodeposited at an overpotential of 625 mV from 0.10 M AgNO₃ in 0.50 M (NH₄)₂SO₄ with the addition of NH₃ in excess to dissolve silver sulfate precipitate.