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Controlled gold deposition by pulse electrolysis

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ABSTRACT

Gold deposits were obtained by electrolysis on glassy carbon surface in N,N-Dimethylformamide solution with 0.004 M H₂AuCl₄ and 0.05 M Bu₄NClO₄. Because of a pulse potentiostatic method including voltage region of (0.1–1.6 V), pulse time of 6 ms and pause of 300 ms the deposits were found as nanoparticles. Increasing potential value the particle size became smaller while absolute number of particle became higher. Higher number of pulse cycles resulted to the particle aggregation and eventually might be a beginning of compact thin film. Scanning electron microscopy was used to analyze morphology of the deposits. Reaction mechanism was discussed by Tafel diagram.

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1. Introduction

The best way to get small particles will be favorable factors that cause a high nucleation rate and slow particle growth. It might be achieved by a cyclic voltammetric method [1,2] and pulse electroplating [2,3] in aqueous solutions. Recently obtained silver nanoparticles in acetonitrile [4,5], palladium [6] and palladium–silver [7] in dimethylformamide (DMF) identified a promising non-aqueous approach. DMF is a well known organic aprotic solvent that is chemically not reactive to noble metals while promotes the formation of solvates with d-metal ions, providing solubility of their metallorganic salts. Moreover this organic solvent is stable at elevated temperature that is good advantage for chemical engineering. Being a good donor of electrons DMF demonstrates a high solid surface adsorption that becomes responsible for the size and shape of deposited solid particles. In case of electrolysis DMF might be used as electrolyte with a high electrochemical stability that makes possible metal deposition in range of high potential without electrolyte decomposition.

Gold might be a good example of noble metals to be electrochemically deposited in DMF. Since both gold and DMF are not reactive to each other, they give chance to build a simple system for the detailed study of the electrolysis mechanism. On the other hand a deep understanding of the whole electrochemical process might be a valuable issue to synthesize gold particles (AuPs) on micro- and nanolevel. Obviously, the AuPs characteristics strongly

depend on their preparation procedure. Electrochemical gold deposition is a reliable method to prepare deposits with a certain size, shape and distribution on the surface. The present paper provides experimental results on controlled gold deposition by pulse electrolysis in DMF.

2. Experimental

Organic aprotic solvent – N,N-Dimethylformamide, (HCON(CH₃)₂, 99%, Alfa Aesar); hydrogen tetrachloroaurate (III) trihydrate (HAuCl₄·3·H₂O, 99.99%, Alfa Aesar) additionally dehydrated by ethanol as in [8]; and tetrabutylammonium perchlorate (Bu₄NClO₄, ≥ 99.0%, SigmaAldric) as inert electroconductive additive were used.

IPC-Pro 200 potentiostat was used in all the electrochemical experiments. Gold electrodeposition was carried out in glass thermostated (35 °C) three-electrode cell with the volume of ~50 cm³ in DMF solution with 0.004 M H₂AuCl₄ and 0.05 M Bu₄NClO₄. Glassy carbon (GC) rod of ~3 mm in diameter (SIGRADUR G rods, HTW Hochttemperatur-Werkstoffe GmbH) encapsulated in fluoroplastic cartridge case and gold plate ($S=2.2 \times 10^{-4} \text{ m}^2$) was used as working and counterelectrodes respectively. All the experimental potential values were referred to Ag/AgCl in saturated KCl solution as silver chloride (SCE) reference electrode. Before every electrochemical experiment the GC surface was mechanically polished using ~3; ~1 and ~0.3 μm microfinishing films followed by polishing alumina-based suspension (~0.3 μm, Metallchemie). After that the surface of the working electrode was washed by distilled water and isopropanol in ultrasonic bath and then dried in the argon flow. An

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electrodeposition process was carried out by the pulse potentiostatic method in voltage (E) region of (0.3–1.6 V vs SCE), pulse time (τ_{on}) of 6 ms and pause (τ_{off}) of 300 ms. These parameters were experimentally found in [6] as that of optimal pulse electrolysis to form discrete AuPs uniformly distributed on the support. It was showed that gold deposition at the electrode surface together with refilling of AuCl_4^- ion concentration in the pre-electrode area might be possible at these conditions. Since pulse and pause time is reasonable to maintain constant AuCl_4^- ion concentration the current density (j) remains almost stable during the whole pulse electrolysis. The number of the pulse–pause repetitions (N), so called “pulse cycle”, varied from 8 to

1182 at the selected -1.2 V vs SCE and/or they were theoretically predicted to install a constant amount of electricity at a different pre-set potential.

Obtained gold deposits were washed sequentially in DMF and isopropanol. After that the samples were dried in the argon flow. Particle shape, size and number were studied using a ZEISS EVO 40XVP scanning electron microscope (SEM). All the SEM images were obtained by recording secondary electrons (SEs) while scanning electron beam over the surface. The excitation of SEs was achieved by irradiation of the samples with beam energy of ~ 15 keV. A SMARTSEM software package was used during the

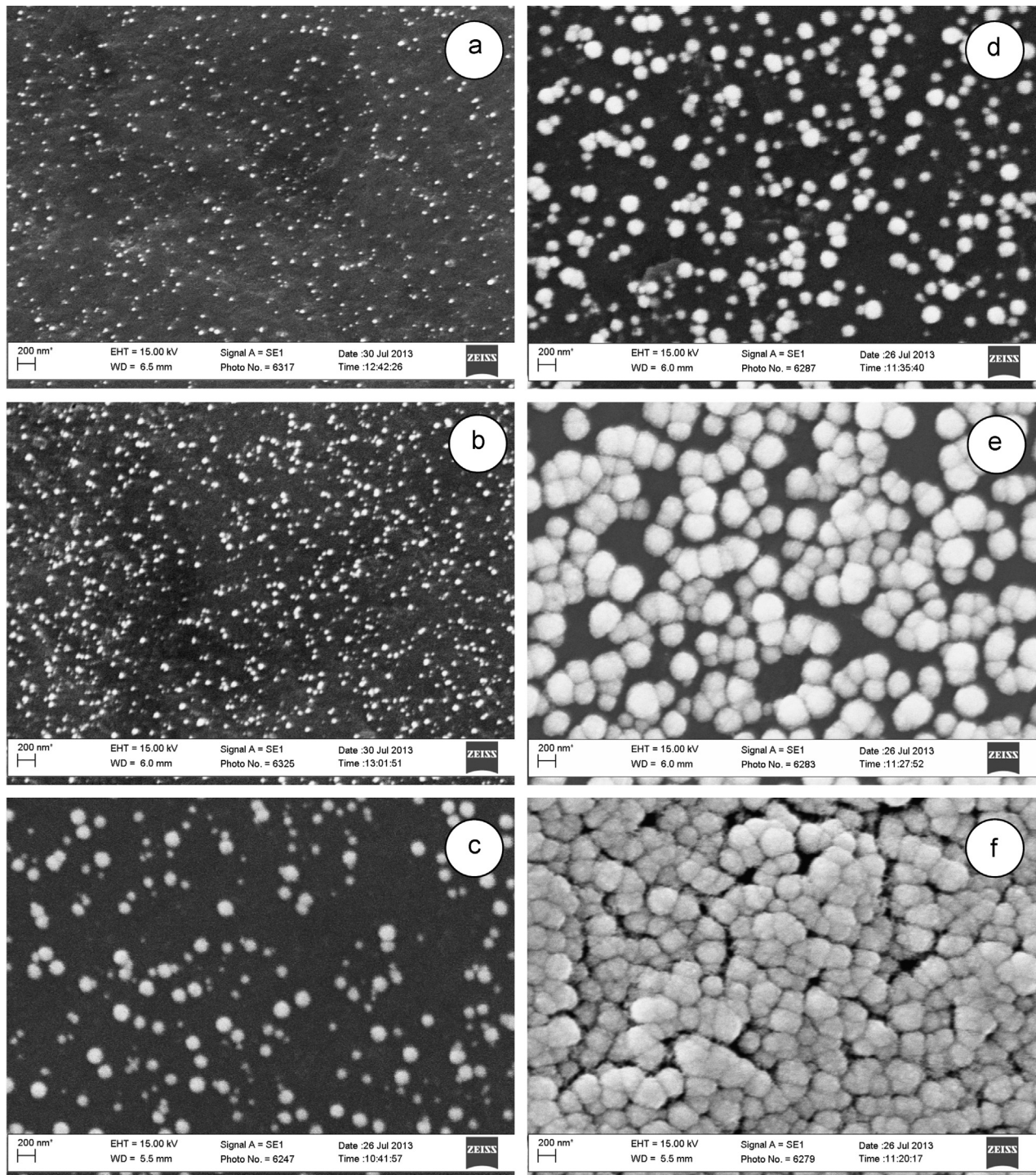


Fig. 1. SEM images of gold deposits obtained by pulse electrolysis on GC surface in DMF solution with 0.004 M HAuCl_4 and 0.05 M Bu_4NClO_4 at -1.2 V vs SCE for 8 (a); 16 (b); 62 (c); 137 (d); 616 (e) and 1182 (f) pulse cycles.

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