Pulse electrodeposition of noble metal nanoparticles on the silicon in DMSO and DMF solution

Shepida M.V., Kuntyi O.I.
Institute of Chemistry, Lviv Polytechnic National University, 12 Bandera Str., Lviv 79013, Ukraine.
E-mail: maryana_shepida@ukr.net

Introduction

Electrochemical deposition of nanoparticles of silver, gold and platinum metals (nano-M) on silicon is one of the modification methods required to create a nanostructured cream and use new resources. Electrolysis supports the ability of controlled origin geometry of nanoparticles and morphologies of nanostructures. Separately, in contrast to physical methods (thermal extraction, electron beam opening and laser treatment), it notes simply the necessary hardware and technology and is effective in the formation of protected MNP/Si systems [1, 2]. Exactly in our work the controlled production of nanoparticles of metals (Ag, Au, Pd, Pt) on reliable silicon among other aprotic workers by a method of the pulse mode of the electric power is established.

Materials and chemical reagents

The metals deposition was carried out by electrochemical deposition on the silicon surface from solutions of their compounds: gold and palladium obtained in DMSO (DMSO, 99%, Alfa Aesar). solution 4 mM $HAuCl_4$ and 4 mM $Pd(NO_3)_2$, silver from 0.1M $(NH_4)[Ag(CN)_2]$ in *N*, *N*-dimethylformamide (DMF, 99%, Alfa Aesar).
For investigations we used n-type Si(100) plates (Crysteco company) with a resistivity of 4.5 Ohm·cm. The silicon plates were divided into squares 1x1 cm^2. The samples were immersed in metal salts solutions and kept under of 25 ^oC for 1–5 min. After applying the metals, the samples were successively washed with corresponding organic aprotic solvent, isopropanol, acetone and dried in the air at 20 ^oC.
The morphology of the resulting deposits on the silicon surface was investigated using ZEISS EVO 40XVP scanning electron microscope. The images of the modified surface were obtained by recording secondary electrons using an electron beam with the energy of 20 kV. The chemical composition of the deposits was investigated using energy dispersion analysis (EDX).

Experimental results

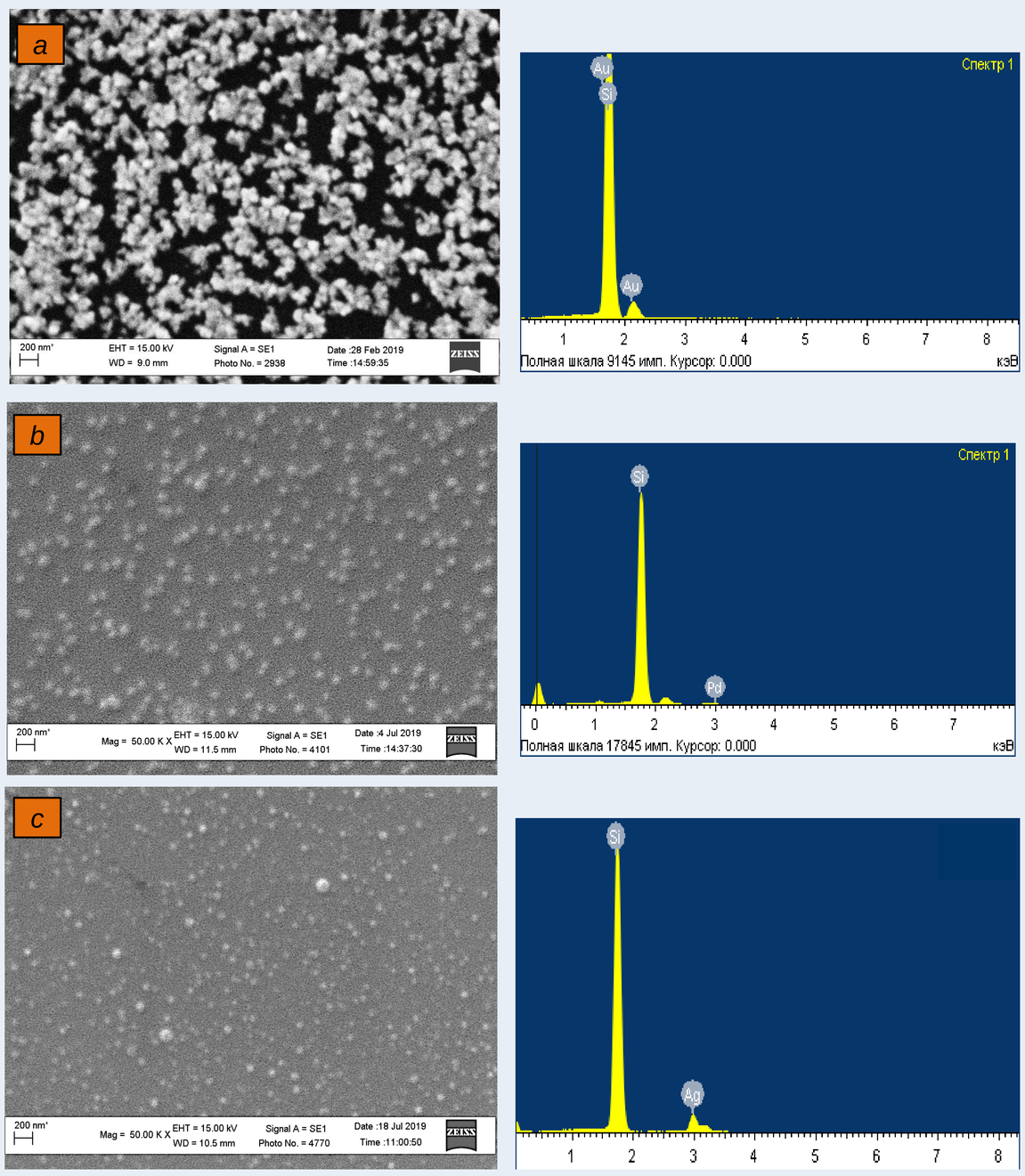

Fig.1 SEM image of the surface of silicon with deposits of gold ***(a)*** and palladium ***(b)***, obtained in DMSO solution 4 mM $HAuCl_4$ and 4 mM $Pd(NO_3)_2$ and silver ***(c)*** obtained in DMF solution 0,1 M $(NH_4)[Ag(CN)_2]$ and EDX precipitates of the corresponding metal

The effect of cathode potential, current density and duration of electrodeposition on the morphology of nanostructured sediments and the size of MNPs was investigated. It was established that gold (Fig. 1a) and palladium nanoparticles (Fig.1b) in the size of 50...250 nm are uniformly distributed over the surface of the silicon. During electrodeposition 2D fill of a surface of silicon occurs - from nanosized discrete particles to formation of nanostructured film it is shown.

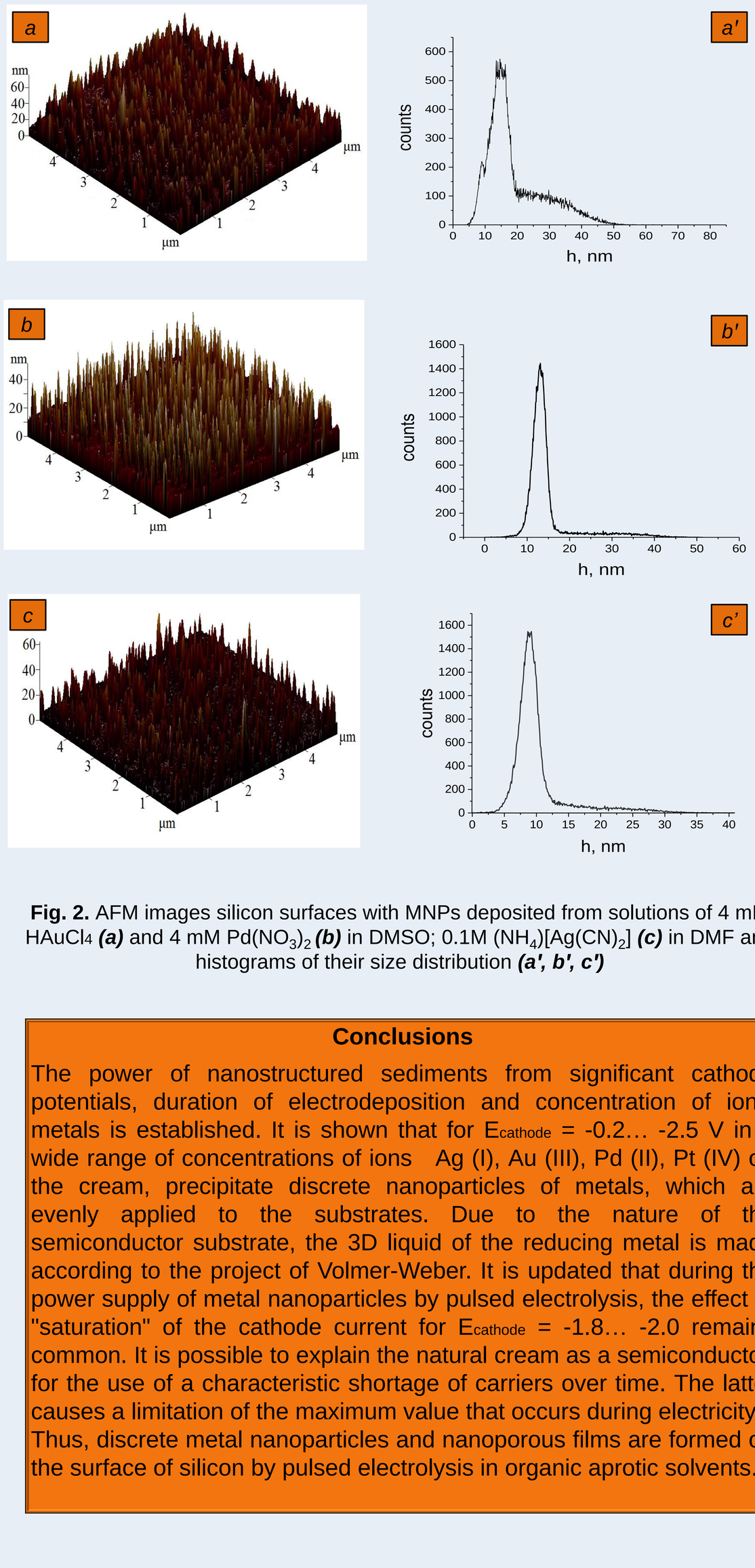

Fig. 2. AFM images silicon surfaces with MNPs deposited from solutions of 4 mM $HAuCl_4$ ***(a)*** and 4 mM $Pd(NO_3)_2$ ***(b)*** in DMSO; 0.1M $(NH_4)[Ag(CN)_2]$ ***(c)*** in DMF and histograms of their size distribution ***(a', b', c')***

Conclusions

The power of nanostructured sediments from significant cathode potentials, duration of electrodeposition and concentration of ionic metals is established. It is shown that for $E_{cathode}$ = -0.2… -2.5 V in a wide range of concentrations of ions Ag (I), Au (III), Pd (II), Pt (IV) on the cream, precipitate discrete nanoparticles of metals, which are evenly applied to the substrates. Due to the nature of the semiconductor substrate, the 3D liquid of the reducing metal is made according to the project of Volmer-Weber. It is updated that during the power supply of metal nanoparticles by pulsed electrolysis, the effect of "saturation" of the cathode current for $E_{cathode}$ = -1.8… -2.0 remains common. It is possible to explain the natural cream as a semiconductor, for the use of a characteristic shortage of carriers over time. The latter causes a limitation of the maximum value that occurs during electricity.
Thus, discrete metal nanoparticles and nanoporous films are formed on the surface of silicon by pulsed electrolysis in organic aprotic solvents.

References

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