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SPM investigation of porous anodic oxides for Ni dots nanometer scale preparation

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Recently nanoscale structures have attracted much interest owing to their potential use in high density magnetic memories, optical devices, solar cells application and others. Formation of the nanostructures based on hexagonally arranged porous alumina as a mask or matrix structure in the lithography processes is cheaper than that based on traditional methods like electron beam lithography [1,2]. As a result of self-organizing effect and followed by electrochemical processing, regular hexagonal cells with controlled parameters may be used as such mask during formation of the arrays of mesoscopic and dotted semiconductor and metal structures. Thus, investigation of self-organizing processes during porous anodic oxides formation is very important and actual. We have investigated physical and chemical processes of spontaneous spatial structuring which take place at the metal-electrolyte interface during controlled electrochemical anodization. Based on this results we have obtained the regular nanometer-scale uniform Ni dots.

Atomic force microscopy (AFM) is one of the most useful techniques of SPM (scanning probe microscopy), which allows to obtain three-dimensional surface topography of materials surface and geometrical parameters of elements with high resolution [3]. Three-dimensional surface topography of investigated samples was obtained with a scanning force microscope SOLVER P47 (NT-MDT, Russia). The data were acquired in the semicontact mode of AFM with silicon cantilevers. The lever parameters of the silicon cantilevers were: length 90.0 μm, width 60.0 μm, thickness 2.0 μm, resonant frequency 350 kHz, force constant 48 N/m. Image Processing Software of this SPM was used by us to make our measurement results more informative.

The samples were prepared using the methods described in [1,2]. In contrast to another deposition techniques such as chemical vapor deposition (CVD), electrochemical deposition, the nanodots growth starts at the top of the Ta_2O_5 pillars preliminarily formed in the alumina pore bottoms area [4,5]. The hexagonally ordered porous alumina host have been prepared by two-step anodization process, which was described in detail earlier [4,5]. After the first

prolonged anodization of regular pores arrangement due to self-organizing and complete chemical dissolution of the first oxide layer, the surface of aluminium film consists of regular hexagonal texture of self-organized pore tips which act as self-assembled mask for the second anodization step. After the second anodization for 1 h an ordered nanopore array is obtained with straight pores from top to the bottom and with the thickness of about 1 μm. The parameters of anodization process were: 4% orthophosphoric acid H_3PO_4 aqueous solution was used as electrolyte, the forming voltage was U_f=80V, and the anodization temperature was T=12^0C. Polycrystalline metal films (Ni, Co, Fe, NiFe) were deposited by a vacuum deposition technique. In our case we used the methods of electron-beam or magnetron sputtering onto fixed substrate or rotating around its axis. This technique presents serious alternative to electrochemical plating into anodic oxide pores [6] because it allows deposition of any magnetic and semiconductor materials and doesn't need in taper of barrier layer in the pore bottom area. The nanodots are formed above the dome-shaped Ta_2O_5 pillars. In fig.2 the SEM micrograph is seen with Ni nanodots in locally formed dielectric holes on Si substrate. This is one of fundamental new results. In fig.1a, b AFM image and Phase image of Ni nanodots array on ceramic substrate is present, respectively. The measured dots diameter was 15 to 20 nm and the inter-dots distance was about 80 to 90 nm. Experimental studies on the inter-dots spacing control and dot size in self-organized three-dimentional arrays of Ni nanodots are presented. An ordered close-packed monolayer of Ni nanodots of 10 to 100 nm diameter was fabricated. In addition, by changing the forming electrolyte voltage, electrochemical anodization with various inter-dot spacing were performed. The results of this study (fig.3) demonstrate the linear relationship between the diameter (1) and inter-dots spacing (2) versus the forming voltage which shows that the inter-dots spacing could be controlled easily by altering the electrochemical processing conditions.

In our method of the Ni nanodots preparation the aspect ratio of interdots distance to diameter was constant and equals about 20. We suppose that this is connected with that in the formation of three-dimentional matrix of Ta_2O_5 pillars embedded into porous Al_2O_3 was involved the self-organization process [7]. An interpore distance and pillar diameter are proportional to the applied cell potential and depends on the electrolyte composition. This allows to obtain nanodots with different geometrical sizes for different application. Presented method may be used for the quantum dots preparation from such materials as semiconductor, metal and others.

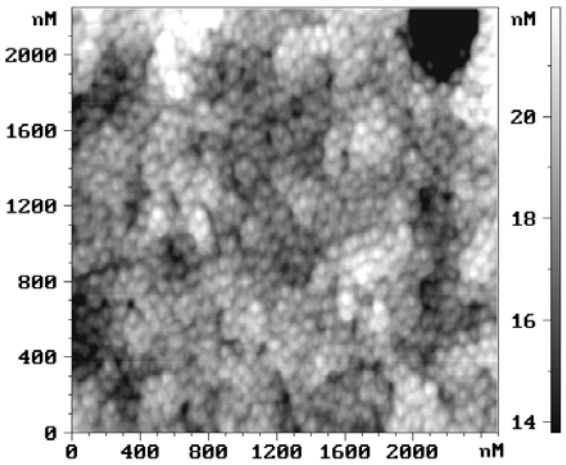

Fig.1a. AFM image of Ni nanodots array on ceramic substrate.

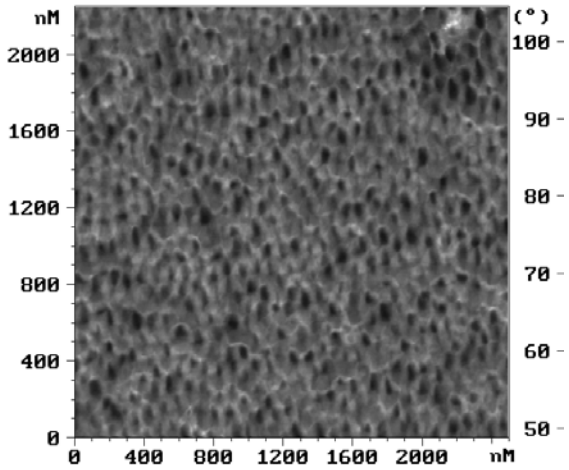

Fig.1b. Phase image of the same sample area.

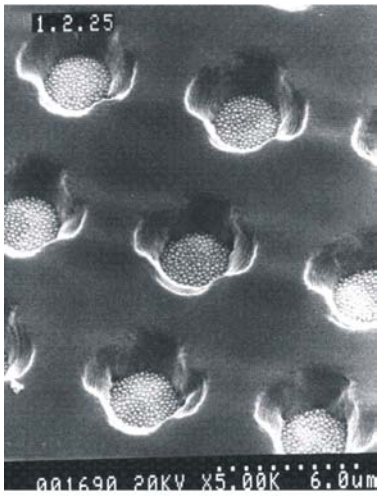

Fig.2. SEM micrograph with Ni nanodots in locally formed dielectric holes on Si substrate.

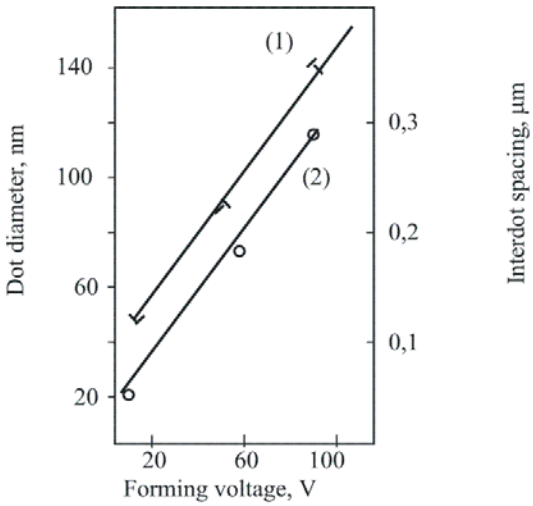

Fig.3. Relationship between the dots diameter (1) and inter-dots spacing (2) versus the forming voltage.

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