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Nanostructures of Si/SiO_2/Metal Systems with Tracks of Fast Heavy Ions

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Abstract—Structures based on the SiO_2/*n*-Si and SiO_2/*p*-Si systems, with nanopores in silicon dioxide layers filled with Cu and Ni nanoparticles, have been prepared and investigated using the fast heavy ion technology, which includes irradiation with $^{197}Au^{26+}$ ions, chemical etching of ion tracks, and subpotential electrochemical deposition. The selectivity of filling nanopores with metals and cluster character of their formation in tracks is shown.

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Currently, the physical properties of low-dimensional systems and nanomaterials are intensively studied with the aim of developing various nanoelectronic devices. The stable increase in the budget of electron industry stimulates search for new (including alternative) technologies in order to decrease the sizes of electronic devices to the nanoscale range. In this context, the interest in the technology of tracks of fast heavy ions has been renewed. This technique makes it possible to form narrow and extended radiation-induced damage regions ("latent ion tracks") by exposing a material to a high-energy ion flux. Subsequent etching of latent tracks allows one to form cylindrical or conical nanopores with characteristic sizes from 10 to 1000 nm, depending on the irradiation and etching conditions and the substrate type [1, 2].

The purpose of this study was to form new structures based on etched ionic tracks in oxidized silicon, in which homogeneous aggregates of nonmagnetic and ferromagnetic nanoparticles are formed by subpotential electrochemical deposition. These structures were formed on the basis of the previously developed TEMPOS (Tunable Electronic Material in Pores in Oxide on Semiconductors) technology [3], which is used to produce MOS devices with nonlinear current–voltage characteristics, including negative differential resistance [3, 4].

The investigations were performed on KDB-4.5 and KEF-4.5 Si(100) wafers (boron-doped *p*-Si and phosphorous-doped *n*-Si, respectively, with a resistivity of 4.5 Ω cm). Silicon dioxide layers were formed on them by thermal oxidation at 1100°C for 10 h in purified oxygen. The oxide layer thickness was 0.7 ± 0.1 μm. Latent ion tracks in the thus obtained SiO_2/*n*-Si and SiO_2/*p*-Si structures were formed by irradiation with 350-MeV $^{197}Au^{26+}$ ions (fluence 5×10^8 cm^{-2}) in the BIBER cyclotron chamber (Center of Ion Beam Technologies, Hann Meytner Institute, Berlin, Germany).

Etching of latent tracks in hydrofluoric acid with concentrations of 1.35 and 2.7% (for 40 and 20 min, respectively) at a temperature of 20 ± 1°C led to the formation of stochastically distributed pores in the silicon dioxide in the form of truncated cones with the diameters of bases 200 nm at the interface with Si and 250 nm on the SiO_2 surface. Their height corresponded to the SiO_2 layer thickness, which decreased to 200 nm after etching. A scanning electron microscopy (SEM) image of etched ionic tracks in SiO_2 is shown in Fig. 1.

Nanopores were filled with metals (copper and nickel) by subpotential electrochemical deposition. A specific feature of this method is that deposition occurs at more positive potentials in comparison with the equilibrium Nernst potential, thus providing interaction between deposited metal and silicon substrate atoms. It is noteworthy that this method makes it possible to form both homogeneous nanostructures and arrays of metal, insulator, and semiconductors nanoclusters and/or their alternating layers. It is characterized by the effective control of the process, including variation in

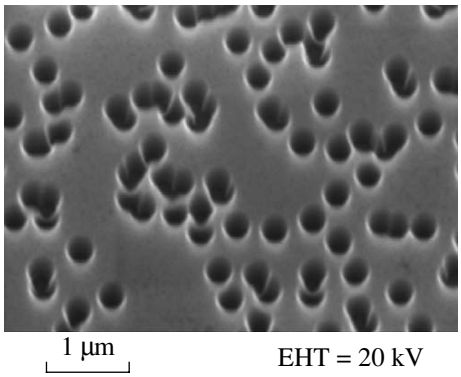

Fig. 1. SEM images of ion tracks, which are conical nanopores after etching.

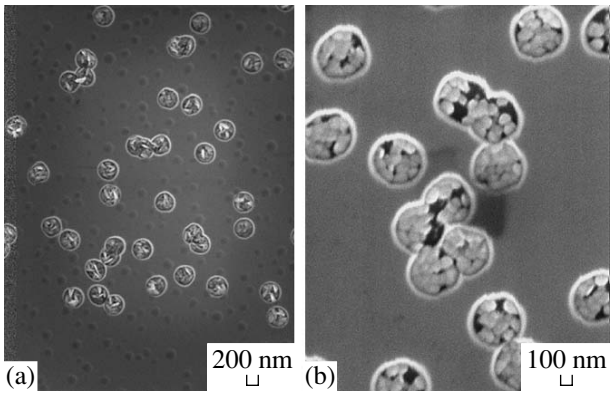

Fig. 2. SEM images of SiO_2/Si structures with (a) Cu and (b) Ni nanoclusters in etched ion tracks in a silicon dioxide layer.

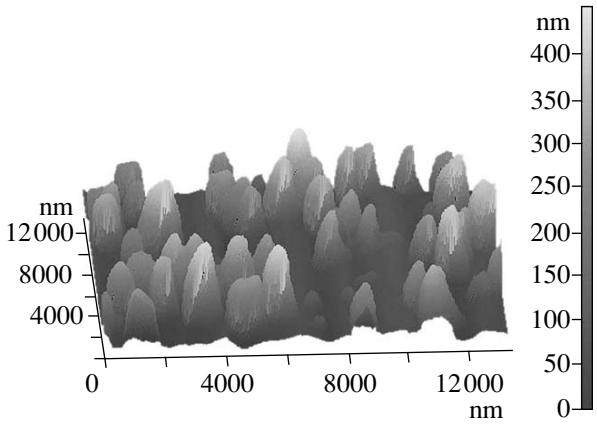

Fig. 3. Isometric image of the SiO_2 surface with etched ion tracks filled with Cu.

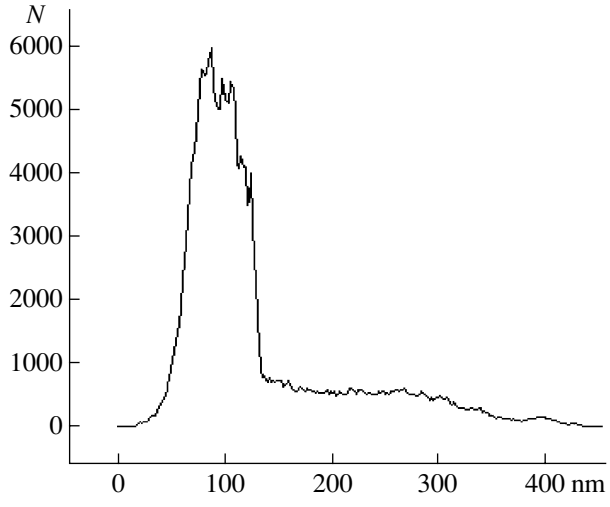

Fig. 4. Height distribution histogram of Cu overgrowths within the scanned area in Fig. 3.

the structural parameters (cluster sizes, layer thickness, sequence of layers, deposited material composition and morphology), by changing the electrode potential [5].

During subpotential metal deposition, the working electrode was the single-crystal silicon substrate in the SiO_2/Si structure with etched tracks. To obtain a uniform potential distribution along the surface, a 0.1-μm-thick Al film was deposited on the rear side on the silicon wafer, to which an electric contact was connected. In addition, In–Ga eutectics was deposited on the electrode in the region contacting with the holder. The surface of the deposited Al film exposed to the electrolyte was protected by a chemically resistant KhSL lacquer. Before deposition of metals into nanopores in silicon dioxide, the electrodes were successively treated in HNO_3 at $T = 80$°C for 30 s and then in HF at 20°C for 10 s with subsequent fixing in 20% NH_4F solution for 5 min. The thus prepared Si surface is hydrophobic and hydrated (contains Si–H bonds).

Metals were deposited using a three-electrode two-chamber electrochemical cell. The electrolytes for copper and nickel deposition were, respectively, 0.5 mol l^{-1} H_3BO_3 + 0.005 mol l^{-1} $CuSO_4$ and 0.5 mol l^{-1} H_3BO_3 + 0.5 mol l^{-1} $NiSO_4$ solutions.

SEM images of the structures obtained by metal deposition in silicon dioxide nanopores are shown in Figs. 2a and 2b for copper and nickel, respectively. It can be seen that the subpotential electrochemical deposition ensures selective filling with metals of only regions with etched ionic tracks, without forming a continuous metal film on the SiO_2 surface. In addition, these images clearly indicate that metals are deposited in nanopores as individual, contacting each other clusters less than 80 nm in size.

These results were confirmed by the surface analysis of the obtained structures using scanning probe microscopy (in the mode of atomic force microscopy) on a Solver-P47 microscope. The data on the character and occupancy of nanopores with a metal (by the example of Cu) are shown in Fig. 3, which presents an isometric surface image of a Si/SiO_2/Cu structure. One can see that, in the chosen modes of subpotential electrochemical deposition, not only ion track channels are filled but also columnar overgrowths are formed above them on the SiO_2 surface. A similar situation occurs when tracks are filled with Ni. These data make it possible to calculate the distribution density of these surface overgrowths, their lateral sizes, and dome shape for each of them. Analysis of this image showed that the maximum height of the metal columns above the insulator surface does not exceed 350–400 nm.

Figure 4 shows the height distribution histogram within the scanned portion in Fig. 3. This distribution indicates that the most likely relief height is 80–100 nm and the overgrowths, from 150 to 400 nm in height, occupy an area an order of magnitude smaller than the relief area. This plot is in fact a distribution of the den-

sity of probability (scale *N*) of finding a particular height within a given area.

On the basis of the results obtained in this study, we can draw the following conclusions:

(i) it is shown that the subpotential method of electrochemical deposition of metals into etched ion tracks is effective for selective deposition of metals, which form nanoclusters in these tracks;

(ii) use of scanning probe microscopy made it possible to reveal the formation of metal overgrowths from track channels above the insulator surface and determine their height distribution probability.

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