MICRO- AND NANOSTRUCTURES FOR CRYOELECTRONICS¹

A. Plecenik^{2,a} M. Gregor^b, T. Plecenik^a, P. Kúš^a, M. Kubinec^a, M. Štefečka^b, V. Jacko^c, V. Gašparík^{a,b}

^a Department of Experimental Physics FMFI Comenius University, Mlynská dolina F2 SK-842 48 Bratislava, Slovakia

^bCyclotron Centre of Slovak Republic, Karloveská 63 SK-842 55 Bratislava 4, Slovakia ^cInstitute of Measurement Science, Slovak Academy of Sciences, Dubravska cesta 9 SK-841 04 Bratislava, Slovakia

Received 11 February 2005, in final form 9 May 2005, accepted 9 May 2005

Preparation of titanium thin film microstructures with dimension of strips down to 5 micrometers, and nanostructures with dimension down to 40 nanometers is discussed. The microstructures were prepared by optical lithography with subsequent Ar^+ ion beam etching. Nanostructures were prepared on microstrips by AFM (Atomic Force Microscope) local oxidation of Ti thin film and etched in acid solution. Typical nanostructures - nanodots and nanostrips are presented.

PACS: 68.37.Ps, 68.55.Jk, 81.07.-b

1 Introduction

The discovery of new phenomena and processes in nanoscale systems opens up opportunity for development of innovative nanosystems and nanostructured materials. New quantum phenomena in nanosystems have been observed and one can expect their various unique applications. Recent huge development of experimental tools and improvements in lithographic techniques allows preparation of atomically controlled structures resulting in line width as small as 10 nm. Local oxidation of surface by Atomic Force Microscopy (AFM) is one of the promising lithographic techniques allowing to pattern the desired thin film nanostructures directly. The AFM lithographic processes are reproducible and exhibit good operation control during the lithographic steps. The study reported here is focused on the lithographic processes for preparation of tens nanometer structures usable for more complex nanodevices, such Superconducting Single Photon Detectors (SSPD) [1] and Single Electron Tunneling Devices (SETD) [2]. The width of the strips for SSPD is limited by the coherence length ξ of superconducting material, e.g. several tens nanometers for MgB₂ superconductors. Determination of size of an island is given by condition for observation of the Coulomb blockade, i.e. in ultra-small tunnel junctions the charging

0323-0465/05 © Institute of Physics, SAS, Bratislava, Slovakia

367

¹Presented at SSSI-IV (Solid State Surfaces and Interfaces IV) Conference, Smolenice, Slovakia, 8–11 Nov. 2004. ²E-mail address: plecenik@fmph.uniba.sk

energy must be larger than the thermal one. It depends on the temperature and capacity of the system $(e^2/(2C_T) \gg k_B T)$, where C_T is the capacity of the system, k_B is the Boltzman constant and T is the temperature), hence several tens nanometer size for tunnel junctions between island and electrodes are required.

In this paper we present thin film technology of micro- and nanostructures preparation usable for SSPD and SETD applications. The width of structures down to 1 μ m and 40 nm, respectively were done. The peculiarities of technological steps are discussed.

2 Experimental

Titanium thin films with thickness 10 nm were deposited on unheated single crystalline Si substrates by dc magnetron sputtering at 0.2 Pa argon working pressure [2]. Topography of the thin films was controlled by AFM Solver P-47, and elemental composition by Auger Electron Spectrometry (AES). Microstructures on titanium thin films were formed using the optical lithography technique and fabricated by Ar⁺ ion beam etching using ion gun Platar, type Klan 53M. Energy of Ar⁺ ions ranged from 200 eV to 600 eV. Ion beam current was 10–25 mA. After stripping the photoresist, microstructures with 10 μ m central strip were obtained.

The nanometer scale nanostructures were fabricated on the central strips of microstructures using AFM local oxidation technique in trapping mode. The voltage between the AFM tip and the surface was below 10 V, the oscillation amplitude of the tip was 2–3 nm, and the writing speed was 100 nm/s. All modifications were done at room temperature and ambient air with relative humidity 50–90 % (special chamber with humidifier was used). The final nanostructures were obtained by selective etching of TiO_x in acid solution (1:60 ratio of HF:water).

3 Results and Discussion

Titanium thin films were optimized in order to achieve the smallest roughness. Under our conditions the roughness of the titanium thin film surfaces strongly depended on the thickness of titanium thin film and altered from about 10 nm for 70 nm thick film to below 1 nm for 10 nm thick film. Typical surface topography of 10 nm thick film is shown in Fig. 1 (left). Composition of such titanium thin film was analyzed by AES (Fig. 1 (right)).

Oxygen was detected in the whole thin film because of high residual pressure in vacuum chamber ($p \ge 5.10^{-4}$ Pa). The increased content of oxygen on the surface, and from the same reason on SiO_x/Ti interface, is brought about by creation of native TiO_x oxide. However, these oxides do not influence function of used method for preparation of nanostructures. The microstructures on such titanium thin films were prepared by optical lithography and Ar⁺ ion beam etching. Typical microstructures with detail of central 10 μ m wide microstrip are shown in Fig. 2. It was necessary to prepare the microstructures before local oxidation because maximum scanning area of AFM is $50 \times 50 \ \mu$ m².

In all samples, both height and width of the nano-oxides increased with both increasing applied voltage to the cantilevers V_g and increasing counting rate [3]. Height of TiO_x increased from about 1 nm at $V_g = 2$ V (threshold voltage for oxidation) to 7 nm at $V_g = 10$ V. On the fresh samples, it was possible to prepare TiO_x strips with diameter down to 100 nm. It was possible to improve the height/width ratio by oxidation of titanium base electrode before AFM



Fig. 1. Topography (left) and depth profile of composition (right) of 10 nm thick titanium film by AFM and AES, respectively.

local oxidation. TiO_x oxide on titanium surface with thickness $1 \div 2$ nm changes the condition in AFM resonant system, and probably changes the attractive Coulomb force between the cantilever and the surface. On such oxidized titanium thin films it was possible to prepare TiO_x strips with diameter down to 40 nm. Similar effects were described e.g. in [4]. where authors covered Ni surface with Al₂O₃. Our typical result is shown in Fig. 3 (left). TiO_x lines with diameter 40 nm were prepared on partially oxidized titanium films before AFM local oxidation. After optimization of oxidation process for maximal high it was possible to prepare nanodots with height of TiO_x up to 8 nm (Fig. 3 (right).

Final nanostructures were prepared by selective wet etching of TiO_x lines in 1:60 ratio of



Fig. 2. Design of microstructure (left) prepared on titanium thin film and detail of 10 μ m central strip (right).



Fig. 3. TiO_x nanolines (left) with dimension down to 40 nm and nanodots with dimension down to 100 nm on titanium thin film.

HF:water solution. Duration of etching was about 20 seconds. The etching process was uncontrolled for higher concentration of HF, because titanium film was removed also. After wet etching, the surfaces as well as lines exhibit some residual contamination (Fig. 4 (left)). After Ar^+ ion beam etching (15 minutes at ion energy 500 eV and current density of ions 15 mA) the final structure was obtained (Fig. 3 (right)).

Preparation of nanostructures by AFM local oxidation of titanium thin films was studied. Microstructures, as precursors for AFM local oxidation, were prepared by optical lithography and Ar^+ etching. AFM local oxidation of titanium thin films was optimized for preparation of nanolines with width down to 40 nm.



Fig. 4. Nanolines after wet etching in HF+water solution (left) and after following etching in Ar⁺ion beam.

Acknowledgement: The authors thanks to the Ministry of Education of the Slovak Republic for support of the grant "Nanostructures for nanometrology, sensors and cryoelectronics". This work was also partially supported by the VEGA project No. 1/9177/02 and APVT project No. APVT-20-011804.

References

- G. N. Gol'tsman, O. Okunev, G. Chulkova, A. Lipatov, A. Semenov, K. Smirnov, B. Voronov, A. Dzardanov, C. Williams, Roman Sobolewski: *Appl. Phys. Lett.* 79 (2001) 705
- [2] M. Gregor, A. Plecenik: Metrology and testing 1 (2003) 3
- [3] M. Gregor, A. Pleceník, A. Zeleňák, M. Štefečka, T. Plecenik, P. Kúš, V. Gašparík: Int. conference ION 2004, (Eds. J. Zuk, J. Filiks). Poland 2004, ISBN 83-227-2273-7
- [4] K. Watanabe, Y. Takemura, Y. Shimazu, J. Shirakashi: Nanotechnology 15 (2004) S566