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Fabrication of micro- and nanostructures by scanning probe microscopy. Local anodic oxidation

K. KOLANEK[1*], T. GOTSZALK[1], M. ZIELONY[1], P. GRABIEC[2]

[1]Wrocław University of Technology, Faculty of Microsystem Electronics and Photonics, ul. Janiszewskiego 11/17, 50-372 Wrocłlaw, Poland

[2]Institute of Electron Technology, al. Lotników 32/46, 02-668 Warsaw, Poland

Atomic force microscopy (AFM) is a high resolution imaging technique in which a cantilever with a very sharp tip is scanned over a sample surface. AFM technique can also be used to fabricate micro- and nanostructures on metallic or semiconductor surfaces. Nanolithography by local anodic oxidation or by noncontact atomic force microscopy (NC-AFM) has strong potential to pattern the surface with a well defined feature size at the nanometer regime. In the paper, the growth rate of nanostructures produced by local anodic oxidation process has been investigated. Mechanisms of nanooxidation have been studied and dependences of its rate and resolution on the voltage applied between the tip and a sample surface, tip speed, and ambient humidity.

Key words: *local anodic oxidation; nanolithography; nanotechnology; scanning probe microscopy; atomic force microscope*

1. Introduction

Scanning probe microscopy (SPM) is a branch of advanced techniques used to examine properties of surfaces, in which interactions between a near field probe – sharp tip at the end of a cantilever – and the surface are examined [1]. Figure 1 shows experimental setup used in one of the most reliable SPM methods – atomic force microscopy (AFM). In this method, the microtip scans surface and by measuring the interactions with dedicated electronics one may map surface properties.

After developing first scanning probe techniques it became clear that these methods are also capable of changing surface properties with a very high spatial resolution. First experiments showing possibilities of transferring patterns on the surface were conducted in 1990 [2]. It was proven that fabrication of micro- and nanostruc-

*Corresponding author, e-mail: krzysztof.kolanek@pwr.wroc.pl

tures by scanning probe techniques may compete with well known photolithographic methods.

Use of local anodic oxidation technique combined with scanning probe microscopy gives new perspectives to achieve fine resolution below 30 nm with inexpensive equipment needed. One of the main advantages of the technique is the ability to fabricate and examine created micro- and nanostructures at almost the same time.

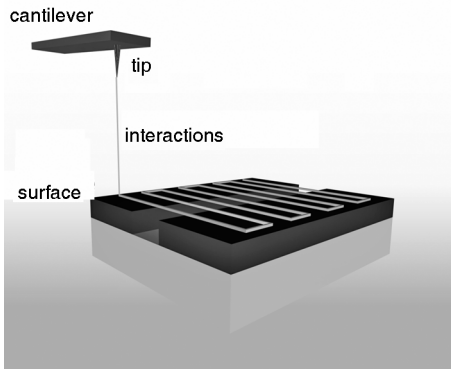

Fig. 1. Idea of the atomic force microscopy

The process of classical anodic oxidation is significantly shrunk when combined with atomic force microscopy. In this method, the conductive tip is approached to the surface to be modified. With the tip radius ca. 10 nm and distance between tip and surface approximately well below a couple of nanometers, very fine patterns may be transferred on the surface. When humidity of the process is controlled in the microscope environmental chamber, the tip of the cantilever is immersed in water acting as an electrolyte covering the whole surface. Oxidation process in this case emerges very locally – only below the conductive tip.

In the paper, experimental investigation has been described conducted at the Faculty of Microsystem Electronics and Photonics of the Wrocław University of Technology. The influence of main parameters on the local anodic oxidation process has been determined. The algorithm responsible for transferring patterns on the surface was explained. In addition, the equipment for the combined surface measurement and nanolithography processes has been shown.

2. Experimental

Local anodic oxidation. Local anodic oxidation proceeds in an environmental chamber of the noncontact atomic force microscope which ensures, together with a bubbler and a nitrogen source, a constant humidity. The developed system for the anodic oxidation of silicon surfaces is shown in Fig. 2.

Due to the control of humidity, a few monolayers of water are on the surface. The water layer is needed to perform oxidation and acts as an electrolyte. The semiconduc-

tor surface to be oxidized is connected as an anode and together with a conductive tip is immersed in the electrolyte [3].

Strong electric field induced by a sharp conductive tip causes dissociation of water and assures presence of hydroxide groups, reacting with the silicon surface according to the following equations [4, 5]:

$$2H_2O + 2e^- \rightarrow H_2 + 2OH^- \quad (1)$$

$$Si + 4h^+ + 2OH^- \rightarrow SiO_2 + 2H^+ \quad (2)$$

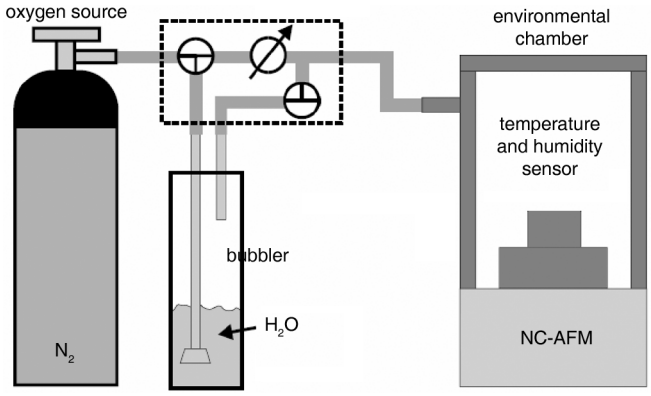

Fig. 2. System maintaining a constant humidity during local anodic oxidation

In order to perform the AFM nanolithography process, the silicon substrate should be preprocessed. It was cleaned with deionized water and dried with pure nitrogen. Then native oxide was removed by 5% dip in HF solution, and the silicon surface was rinsed with deionized water and dried with pure nitrogen. The bath in hydrofluoric acid additionally passivated the surface.

Algorithm of local anodic oxidation. Local anodic oxidation algorithm is controlled by a signal processor DSP Adwin-Pro Keithley based system [3]. This approach makes it possible to control executing algorithm with 1 μs time precision. The signal processor executes the program code which enables establishing key factors of local anodic oxidation parameters, such as:

- amplitude of voltage applied between the surface and a conductive tip,
- frequency of voltage modulation,
- rate of movement of the tip over the surface,
- time of approach to anodization point,
- time of anodization on a given point.

The pattern which should be fabricated on the surface is created in especially designed graphic editor. Local anodic oxidation of the silicon surface is performed in a two step procedure [6]. Before oxidation, the line to be transferred on the surface is scanned and topography information is stored in a DSP processor. Then the scanner moves to the beginning of the line and with a feedback loop turned off and based on stored topography information the line is oxidized.

Measurement set-up. Experiments were conducted on a surface of n-type Si(100) with a resistivity of 12 Ω·cm. An atomic force microscope operated in a noncontact mode was equipped with conductive cantilevers. The average spring constant and resonance frequency were ca. 1.4 N/m and 58 kHz, respectively. The cantilever was excited at its resonance frequency by a piezoactuator and a function generator Stanford Research Systems DS340. The position of the sample with respect to the tip was derived from measurements of the oscillation amplitude and deflection of the cantilever. The optical signal reflected from the back of the oscillating cantilever illuminated a four-section photodiode. Electrical signal converted by photodiodes was then detected by a lock-in amplifier Stanford Research Systems SR530 and was acquired in a real time by a signal processor. After surface preparation, the sample was closed in an environmental chamber of the atomic force microscope. The environmental chamber was equipped with inlets for dry and H_2O saturated nitrogen. The relative humidity RH, was stabilized at a constant value of 50%. The microscope tip was oscillating at a fixed distance of a few nanometers above the sample surface and a voltage pulse was applied between the tip grounded and the sample. The applied voltage induced the formation of a water bridge between the tip and the sample whenever the voltage strength was above a certain threshold voltage [5]. During the application of the voltage pulse, the electrostatic force induced deflection of the cantilever. It also reduced the oscillation amplitude, however, the tip never got in contact with the surface.

3. Results and discussion

In the initial phase of the experiments, the micro- and nanostructure heights as well as the width of the fabricated silicon oxide were examined in function of voltage amplitude

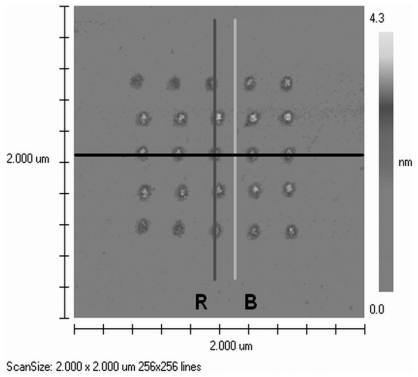

Fig. 3. NC-AFM image of Si(100) surface; 25 dots 80 nm wide and 2 nm high have been written; to each dot, a voltage pulse of 20 V and 10 s is applied between the sample and the tip

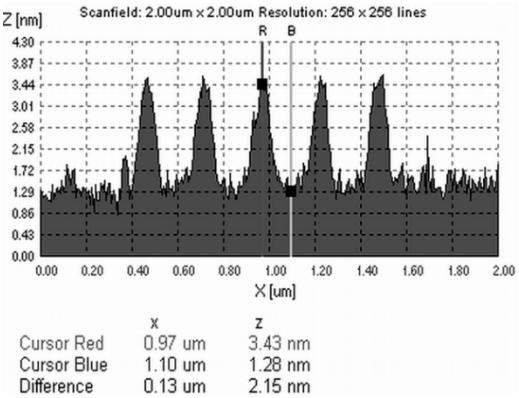

Fig. 4. NC-AFM cross section of fabricated nanostructures produced by static voltage; pulse parameters were 20 V, 10 s

applied between the tip and the surface as well as in function of the duration of the voltage pulse. Figure 3 shows an NC-AFM image of the silicon surface after local anodic oxidation process and Fig. 4 displays the cross-sections of the oxide dots.

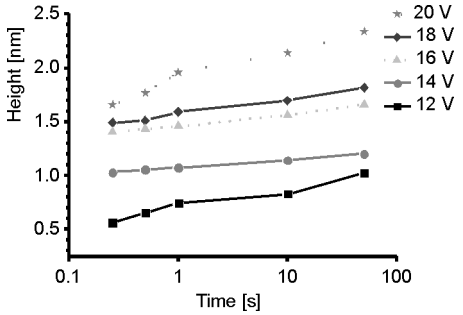

Fig. 5. Dependence of silicon oxides height on pulse duration and static voltage amplitude

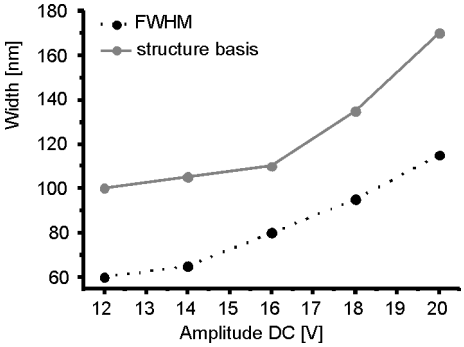

Fig. 6. Dependence of silicon oxide width on static voltage pulse amplitude; pulse duration 250 ms

The results of the whole series of measurements are presented in Figs. 5 and 6. The results shown in Fig. 5 indicate that increasing time of the voltage pulse duration does not significantly influence the height of the produced nanostructures manifesting the logarithmic character. Increase in the static voltage amplitude causes the enlargement not only of the height but also of the width of produced nanostructures as shown in Fig. 6. This is connected with increasing intensity of electric field facilitating formation of water bridges joining the tip and silicon surface [7]. It is possible to fabricate recurrent examples of nanostructures with an appropriate control of static voltage amplitude and pulse duration.

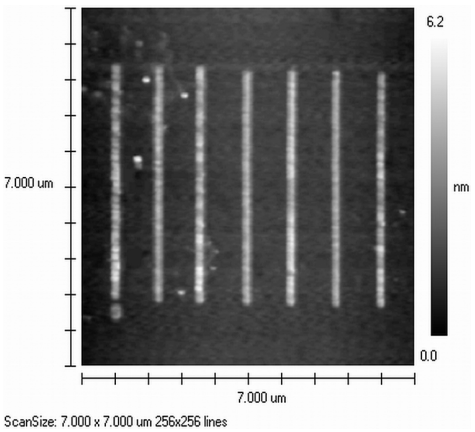

Fig. 7. NC-AFM image of Si(100) surface with fabricated oxide lines; tip speed – 2 μm/s, voltage amplitude – 15 V_{ac}, modulation frequency – 25 Hz and relative humidity 45% RH

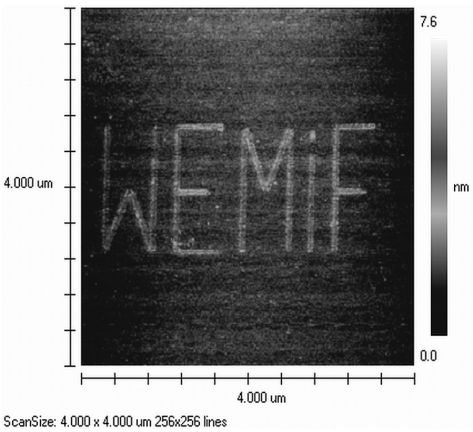

Fig. 8. NC-AFM image of Si(100) surface after local anodic oxidation as an example of more compiled pattern – logo of the Faculty of Microsystem Electronics and Photonics

The next step in the research was to examine possibility to create lines of silicon oxide. In this case, voltage modulation has been used instead of DC voltage. The parameters of the process were tip speed, voltage amplitude and ambient humidity in the microscope environmental chamber. Figures 7 and 8 show NC-AFM exemplary results of the experiments.

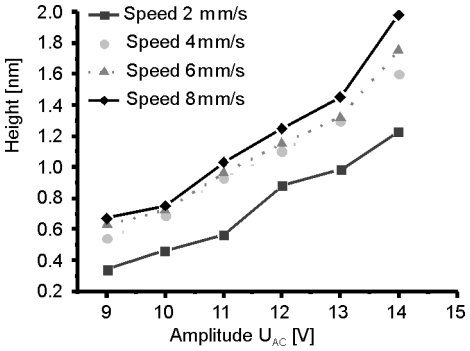

Fig. 9. Dependence of silicon oxide height on voltage amplitude and tip speed; modulation frequency 25 Hz

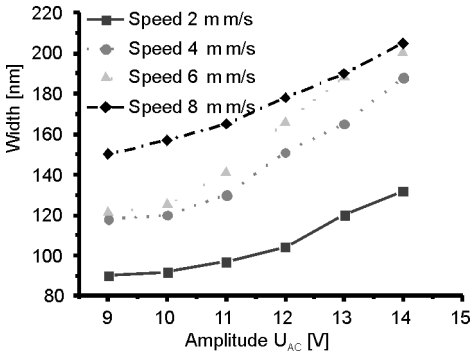

Fig. 10. Dependence of silicon oxide width on voltage amplitude and tip speed; modulation frequency 25 Hz

Figures 9 and 10 show that an increasing voltage modulation amplitude causes approximately linear growth of the width and height of fabricated oxide lines. Reducing the scanning rate also enlarges the height and width of the oxides. Modulation of the voltage applied between the tip and a sample causes removal of space-charge on the oxide surface in initial stage of local anodic oxidation process [8]. As a consequence we observe enhancement of the growth rate and the produced nanostructures are higher.

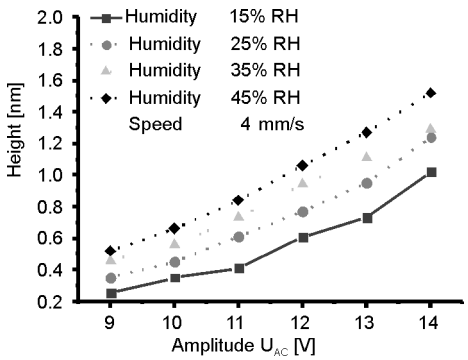

Fig. 11. Dependence of silicon oxide height on voltage amplitude and tip speed; modulation frequency 25 Hz

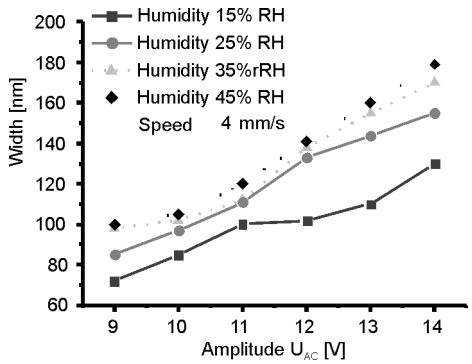

Fig. 12. Dependence of silicon oxide width on voltage amplitude and tip speed; modulation frequency 25 Hz

Humidity in the environmental chamber plays a crucial role in the local anodic oxidation process as shown in Figs. 11 and 12. Decreasing humidity causes the

local anodic process to receive reduced amounts of hydroxide species and thus height and width of the fabricated oxide lines is reduced. A lower humidity also results in difficulty of water bridge formation which also reduces fabricated oxide line volume [9].

4. Conclusions

A very precise characterization of the system parameters is required in order to perform reliable nanostructure fabrication on a silicon surface. The most important factors that should be taken into account are: amplitude of the voltage applied between a tip and a sample surface, tip scanning speed and humidity. There are also parameters, verification of which may be problematical, first of all: tip radius, speed of wear of the tip, angle of tip inclination to the surface, the height of tip suspension over the surface, quality of surface as well as moisture in environmental chamber of the atomic force microscope. Additionally, for suitable conditions of local anodic oxidation, the fabricated nanostructures have their height comparable with roughness of the surface, which can result in a misinterpretation of the obtained results.

A well designed and calibrated atomic force microscope is capable of turning local anodic oxidation technique into a method suitable of creation fine structures on a semiconductor surface in the nanometer regime. The spatial resolution can be optimized and the nature of local anodic oxidation phenomenon permits fabrication of features below 100 nm. The method seems considerably superior over many photolithographic techniques. Preliminary tests show that oxide patterns written using the scanning probe microscopy may also serve as a mask for other microelectronics techniques like wet chemical etching. Future optimization of the described technology will be connected with the application of arbitrary voltage shape pulses and application of cantilevers with higher force constants. An advanced system designed for fabrication of micro- and nanostructures by local anodic oxidation has been developed at the Faculty of Microsystem Electronics and Photonics, Wrocław University of Technology.

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