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Probe Modification for Scanning-Probe Microscopy by the Focused Ion Beam Method

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Abstract—The paper presents the results of experimental investigations into probe modification for atomic-force microscopy (AFM) and scanning tunneling microscopy (STM) by etching the point of AFM cantilevers and tungsten STM probes by applying the method of focused ion beams (FIBs). It is shown that the use of etching by the IB method allows one to obtain the probes with rounding that is less than 10 nm and with an aspect ratio of 1 : 50. The application of these probes increases the resolution and the reliability of measuring by the AFM and STM methods. The obtained results can be used for developing the technological processes of production and modification of sensor probes for AFM and STM, as well as the methods for diagnostics of the structures of microelectronics, nanoelectronics and the microsystem and nanosystem technologies.

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INTRODUCTION

Currently, the method of scanning-probe microscopy (SPM) holds the greatest promise for surface diagnostics. The use of SPM allows one to study the local geometric, electrical, and mechanical properties of the substrate surface and form nanodimensional structures on the surface of solids [1–6]. The resolution of SPM methods is defined by many factors, most of which are geomentical properties, specifically, the rounding radius and aspect ratio of the point sides [6].

The silicon cantilevers with typical values of a point radius on the order of 10 nm and an aspect ratio of 1 : 3 are applied as probes in atomic-force microscopy [7].

When interpreting the AFM results, the actual geometric parameters of objects are difficult to determine since the obtained images of the surface morphology are the superposition of the actual surface shape and the probe profile of the applied cantilever. The topical problem consequently involves the development of probe forming methods with parameters (rounding radius of the point and its aspect ratio) that allow one to minimize the morphology distortion of the substrate surface when using the AFM methods.

A metal wire of diameter from 100 to 500 μm is used as a probe for STM. The point is normally formed by the electrochemical etching (ECE) method at the wire end. The spatial resolution of the STM method is specified by the effective transverse size of the region of tunnel current percolation between the probe and the substrate. The STM probe must then have the minimum radius of point rounding to increase the reliability of the results.

The method of focused ion beams [1, 8, 9] is one of the most promising in terms of obtaining the minimum values of the point rounding radius and a high aspect ratio. This method lies in the local ion-beam modification of the solid surface under high vacuum conditions. The key feature of FIBs is the high resolution which is provided by the application of a gallium ion beam 10 nm in diameter, as well as by the possibility of varying the impact of the parameters over wide limits [10].

The purpose of the present study is to design the methods of probe modification for atomic-force microscopy and scanning tunneling microscopy using the etching by focused ion beams.

EXPERIMENTAL

The modification and investigation of probe characteristics were carried out using the ultra-high vacuum of focused ion beams (UHV FIB) of th emulti-purpose nanotechnological complex NANOFAB NTF-9 (produced by Nanotechnologia-MDT JSC, Zelenograd) and the scanning electron microscope Nova NanoLab 600 (FEI Company, the Netherlands).

At the first stage of research, the probes in the form of silicon cantilevers NSG 10 were modified [7]. The cantilevers were put into the vacuum chamber of the UHV FIB module of NANOFAB NTF-9 so that the probe point was guided upwards, which means in the direction of the ion source. The operating vacuum was maintained at the level of $2–3 \times 10^{-4}$ Pa under ion-beam treatment. The following parameters of this treatment were used in forming the samples of AFM cantilevers: the accelerating voltage of ion beam was 30 keV; the current of the ion beam was 0.3 nA; and the impact time of the ion beam varied from 500 ns to 4.6 μs.

Figure 1 presents the schematic sketch of two procedures that were developed for modification of cantilevers in terms of the FIB method. The etching by the

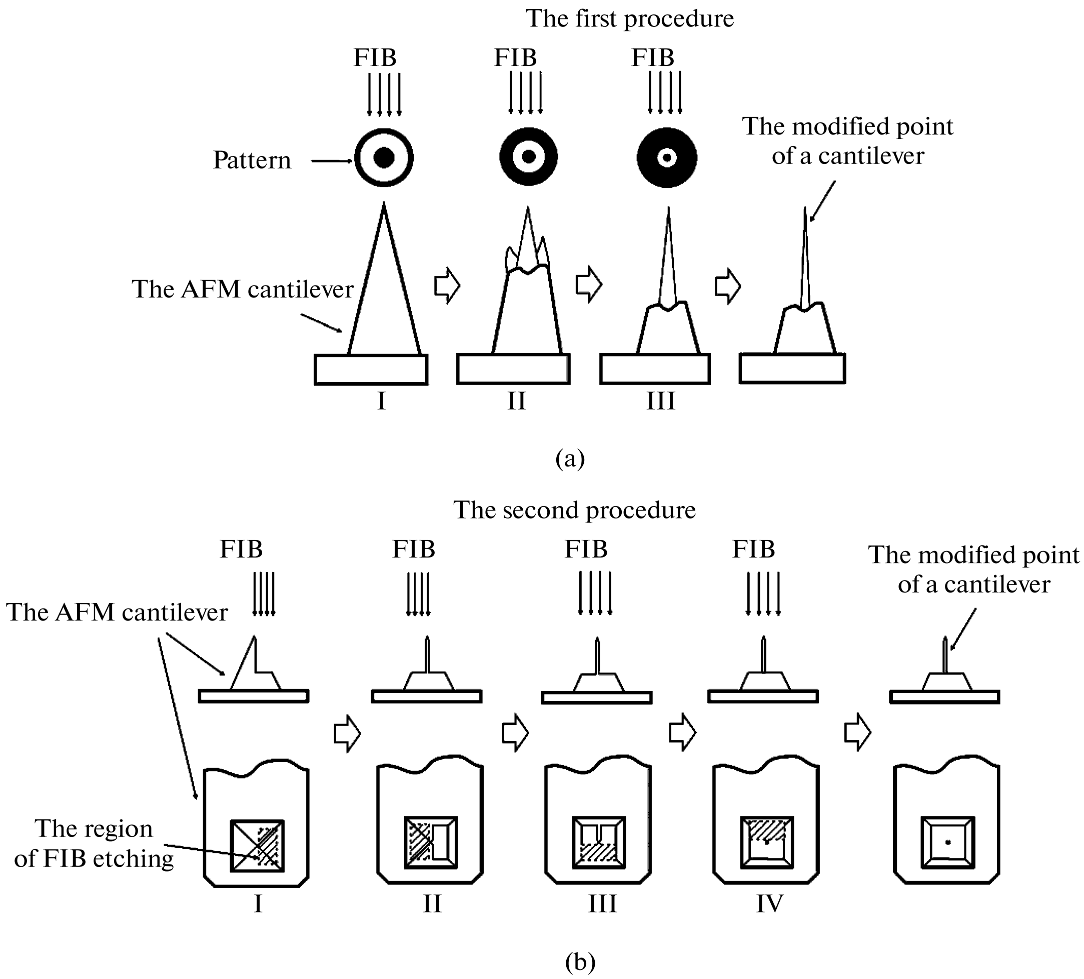

Fig. 1. A schematic sketch of procedures for the modification of the probe point by the FIB method.

first procedure was performed under controlling the spatial distribution of intensity of ion flow which was specified by the complex of bitmap pattern—*.bmp (Fig. 1a).

The surface of the cantilever point was examined after modification by using the scanning electron microscopy (SEM). The analysis of SEM images (Fig. 2) shows that a cantilever with a point rounding radius of about 5 nm and aspect ratio 1 : 30 (Fig. 2b) was obtained at the final stage of modification by the first procedure (stage III in Fig. 1a).

The modification in terms of the second procedure was carried out using the standard means for controlling the ion beam of the control program of the UHV FIB module of NANOFAB NTF-9. At each stage, an operator manually relocated the region of the FIB etching, which was specified by the rectangle-type pattern, through 90° about the probe axis (Fig. 1b). Figure 3a gives the SEM images of the cantilever that was modified using the second method at different stages of FIB etching. At the final stage (stage IV, Fig. 1b), the cantilever with a point rounding radius of about 9 nm and an aspect ratio of 1 : 50 was obtained (Fig. 3b).

When studying the probe modification for scanning tunneling microscopy, initial probe samples were manufactured using the method of electrochemical etching in 8% KOH solution of the pattern made of tungsten wire 100 μm in diameter. The probes were then moved into the vacuum chamber of the UHV FIB module of NANOFAB NTF-9, where the modification of the point of STM probes was performed by the second procedure (Fig. 1b). The main parameters of FIB etching are the following: the current of focused ion beam is 100 pA, the accelerating voltage is 30 keV, and the time of ion beam treatment at a point is 1 μs.

Figure 4 presents the SEM images of the initial STM probes obtained by ECE, as well as the STM probes modified by the FIB method. The analysis of the SEM images shows that the radius of point rounding of the initial STM probe decreases from 146 nm to 7 nm as a result of the modification by the FIB method.

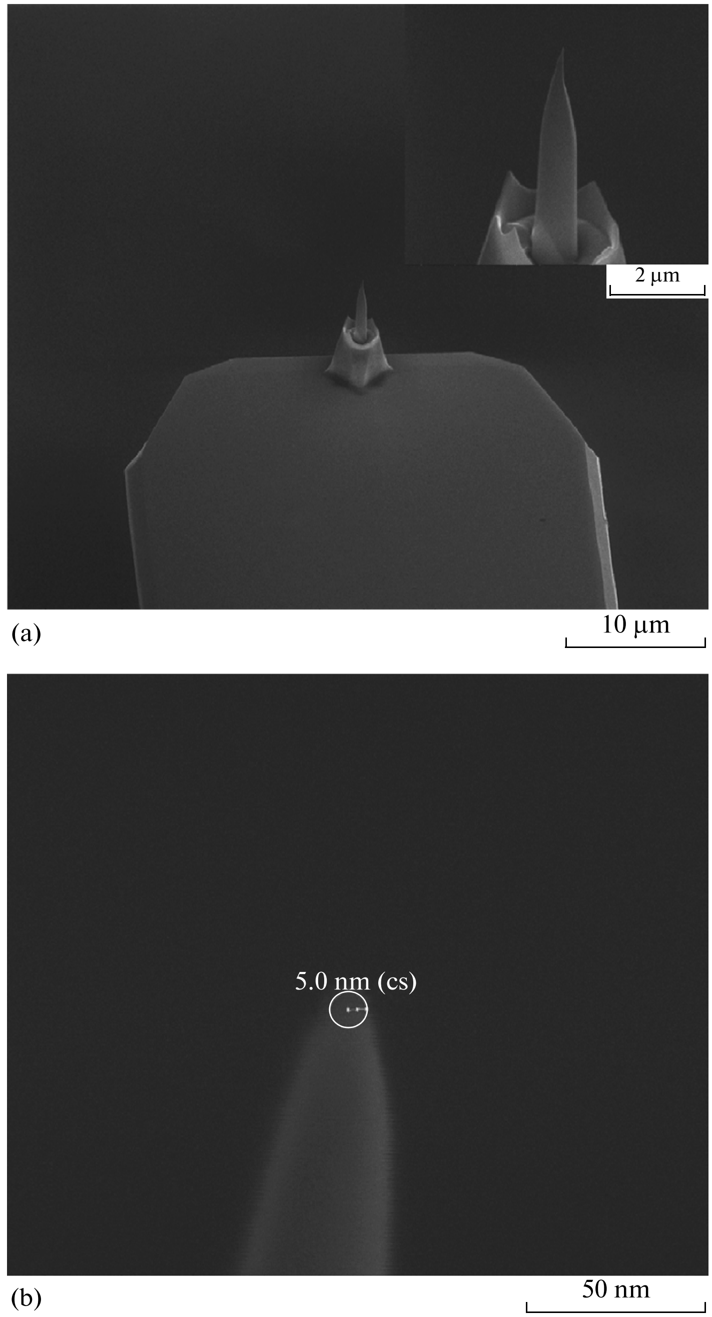

Fig. 2. SEM images of the probe of an AFM cantilever modified in terms of the FIB method by the first procedure: (a) 5000×; (b) 1 300 000×.

EXPERIMENTAL

The experimental investigations on the influence of the modification of the sensor probe on the resolution and reliability of the results of AFM measurements was carried out on the NTEGRA Vita probe nanolaboratory (Nanotechnologia-MDT JSC, Zelenograd) by scanning the surface of test structures: TGZ3 relief height measurements [7] and custom-made integrated microcircuits (IMC). The scanning of test structures was conducted in the semicontact AFM mode.

Figure 5 portrays the AFM images of the surface of the TGZ3 relief height measure. The analysis of AFM images was carried out to define the characteristic dimensions of the TGZ3 relief height measure by using the Image Analysis 3.5 program package [7].

In order to investigate the characteristics of cantilevers modified by the first procedure, the surface of

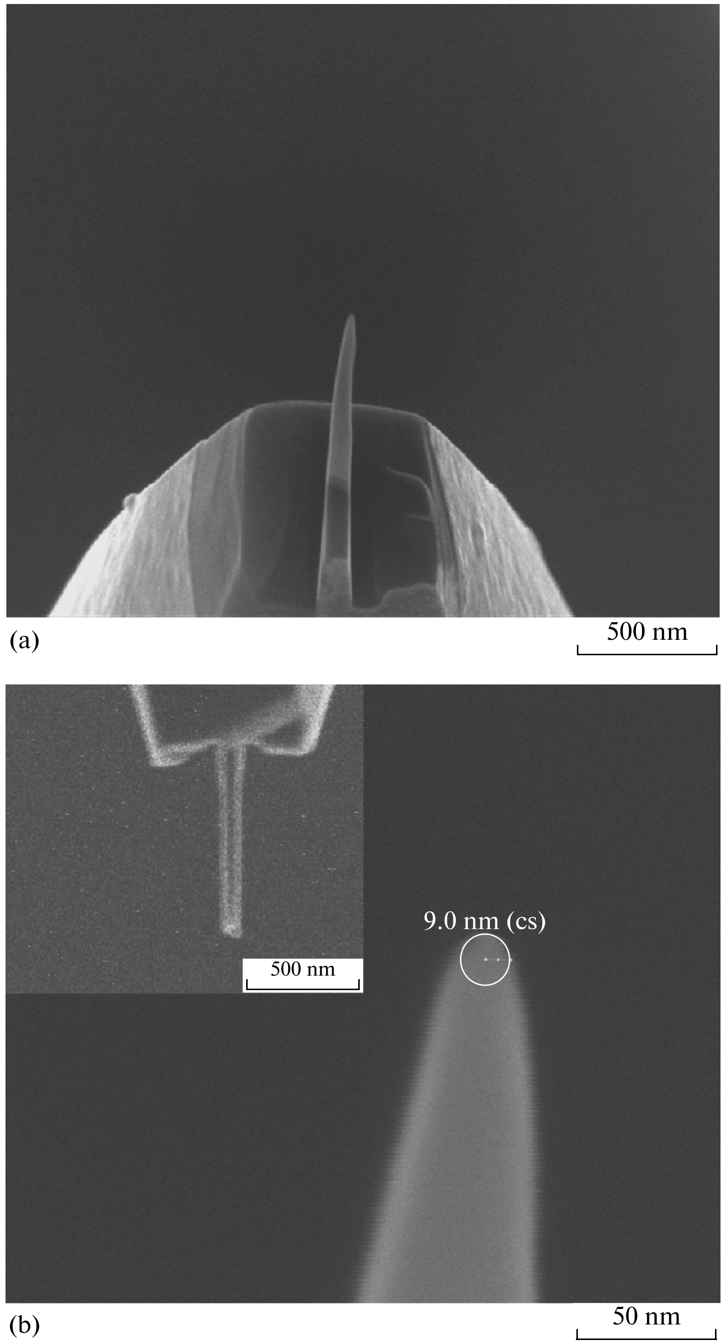

Fig. 3. SEM images of the probe of an AFM cantilever, modified in terms of the FIB method by the second procedure: (a) after stage II; (b) after stage IV.

the IMC fragment with a submicron topological norm, which contains the field-effect transistor, was studied (Fig. 6).

Figure 7 presents the AFM images of the surface of a custom-made IMC. They were obtained by the standard cantilever and modified cantilever in the second procedure.

The effective radius of the STM probe point was defined on the surface of the highly oriented pyrolitic graphite (HOPG) in the mode of STM spectroscopy. This results in obtaining the relations between the tunnel current and the probe, thesubstrate distance ($I(z)$ relations), as shown in Fig. 8.

The resolution of the STM probes was examined by scanning the HOPG surface under constant height conditions. The STM image of the HOPG surface with atomic resolution which is presented in Fig. 9 was obtained by using the modified probe.

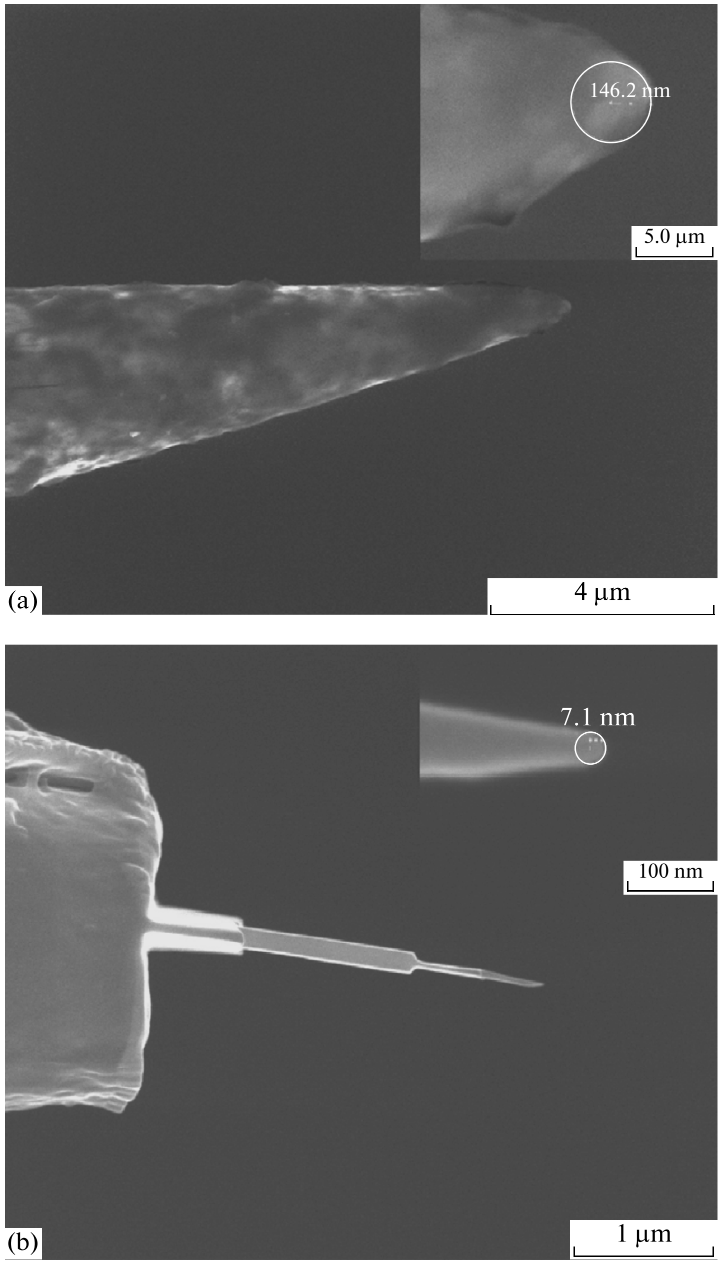

Fig. 4. SEM images of tungsten STM probes: (a) initial probes; (b) modified by the FIB method.

RESULTS AND DISCUSSION

The analysis of AFM images (Fig. 5) shows that the shape of the structures of TGZ3 relief measure of the height, which are obtained using standard cantilevers (Fig. 5a), contains artefacts that probably came into existence due to the contribution of the cosine angle of cantilever point (~22 [7]) to the distortion of the shape and literal geometric dimension. The artefacts were absent on the AFM image in scanning the TGZ3 relief height measure by the modified cantilever (Fig. 5b). The table presents the geometric parameters of the TGZ3 relief height measure, which were obtained in terms of AFM image analysis. It was found that geometric parameters from AFM images obtained by the modified cantilever fit the certified data well [7]. The geometric parameters from AFM images obtained by the modified cantilever do not fit the certified data and have a large dispersion.

The analysis of the AFM images of the IMC surface fragment with a submicron topologocal norm in Fig. 6 shows that the application of a modified canti-

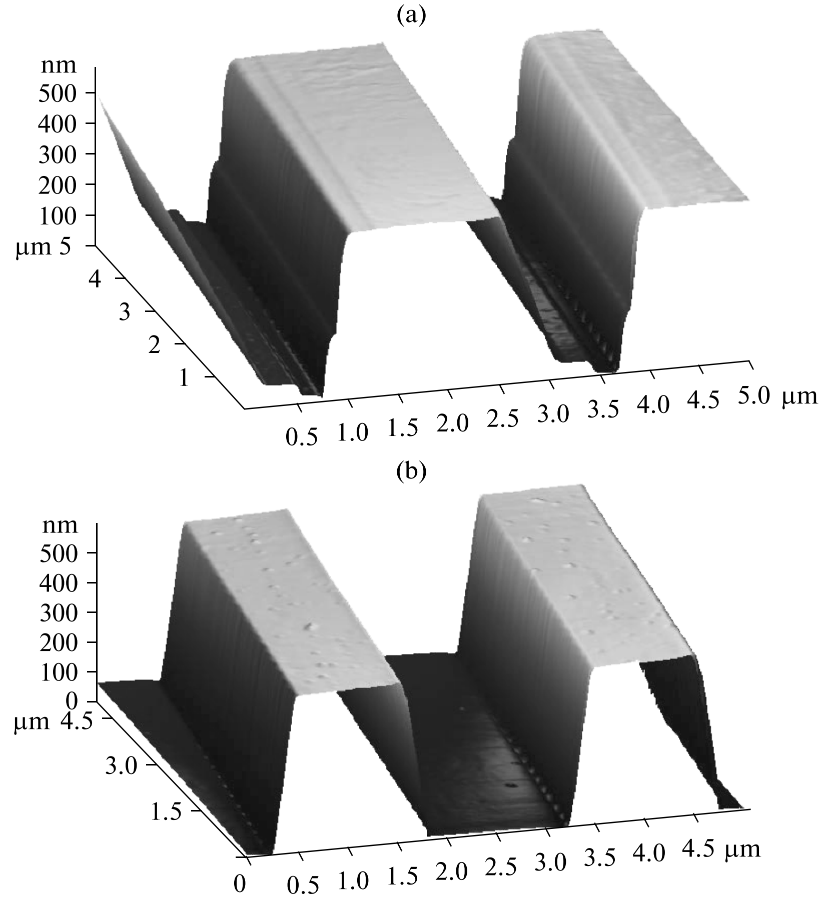

Fig. 5. AFM images of the relief measure surface of the TGZ3 period and the height, which are obtained by (a) the initial cantilever and by (b) a cantilever modified by the FIB method.

lever allows one to reveal the structure of the IMC surface with certainty. Figure 6b portrays the AFM image of the IMC transistor structure, in which the contact with the gate region of the field-effect transistor 600 nm wide and with a height of 140 nm. This correlates well with the data of SEM (Fig. 6c). The contact in scanning by the initial cantilever was not explicitly expressed, and the determination of its geometric parameters failed (Fig. 6a).

It follows form the analysis of the AFM images of the custom-made IMC surface (Fig. 7) that the use of a modified cantilever allows one to increase the resolution and reliability of measuring the geometric parameters of IMC elements. The measured width of the conducting path of the IMS is 4 μm (Fig. 7d), whereas in the width measured by the standard cantilever is on the order of 6 μm (Fig. 7a, b). A significant difference between the measurement results may be

Geometric parameters of the TGZ3 relief height measure

Data	Parameters of the TGZ3 measure		
	The width of the step, μm	The period, nm	The height, μm
Certified [7]	1.5 ± 0.01	3 ± 0.01	500 ± 2.5
Measured by a standard cantilever	2.3 ± 0.2	2.9 ± 1.7	511 ± 7.5
Measured by a modified cantilever	1.5 ± 0.07	3 ± 0.5	500 ± 3.1

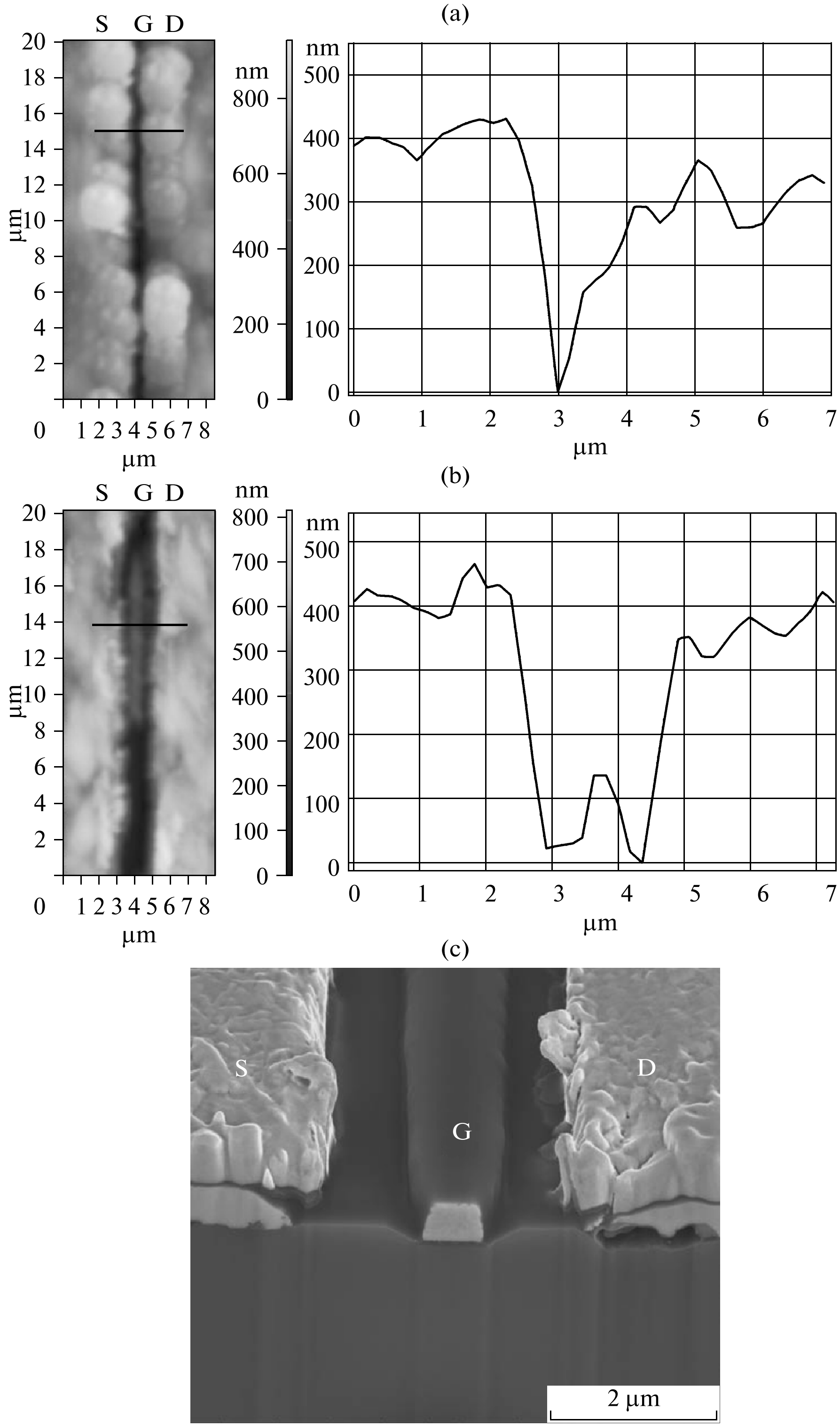

Fig. 6. The IMC fragment: (a) AFM image and profilogram of a field-effect transistor surface, which are obtained by the initial cantilever; (b) AFM image and profilogram of a field-effect transistor surface, which are obtained by the modified cantilever; (c) SEM image of the cross section of the field-effect transistor surface.

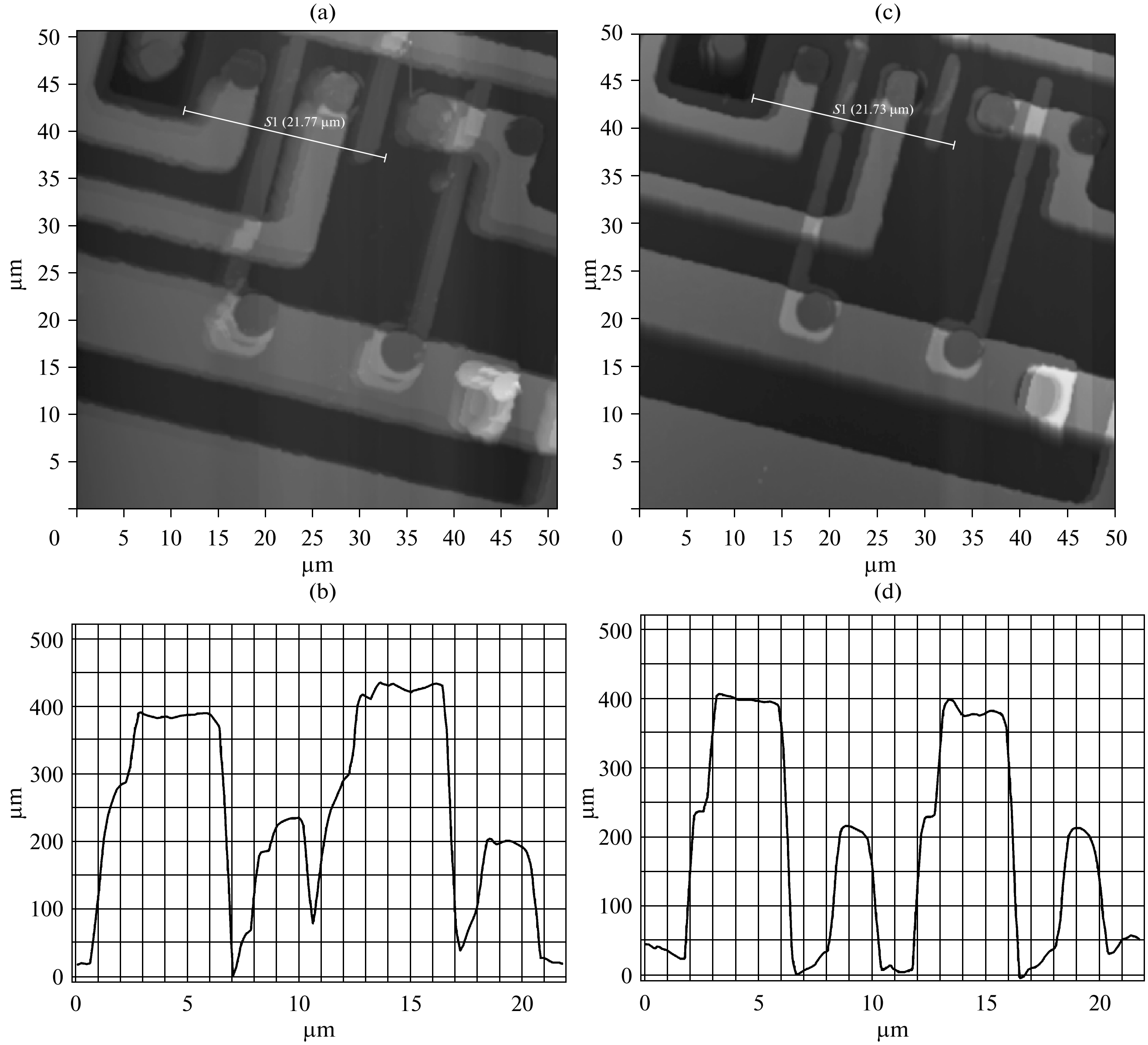

Fig. 7. AFM images and profilograms of the IMC surface obtained by (a), (b) initial cantilevers and by (c), (d) modified cantilever in terms of the FIB method.

related to the various values of aspect ratios between the point sides of the initial and modified cantilevers.

The analysis of relations between the tunnel current and the probe–substrate distance in Fig. 8 shows that the effective radius of point of the modified STM probe is ~0.4 nm and the radius of the initial probe is ~3 nm. Moreover, the $I(z)$ relation of the modified probe has a higher stability of the tunnel current in comparison to the probe obtained by ECE. This characterizes the higher resolution of the modified probe.

The STM image with atomic resolution, which was obtained by the modified probe in Fig. 9, allows one to reveal the features of the thin structure of two-dimensional electron gas on the surface of highly oriented pyrolitic graphite. The atomic structure on the STM image of HOPG surface was not revealed at scanning by the probe. This is due to the effective radius of the point not being sufficiently small.

CONCLUSIONS

The procedures for the modification of the probe point for atomic-force microscopy and scanning tunneling microscopy in terms of etching by focused ion beams. Samples of modified AFM cantilevers and tungsten STM probes were produced. It is shown that

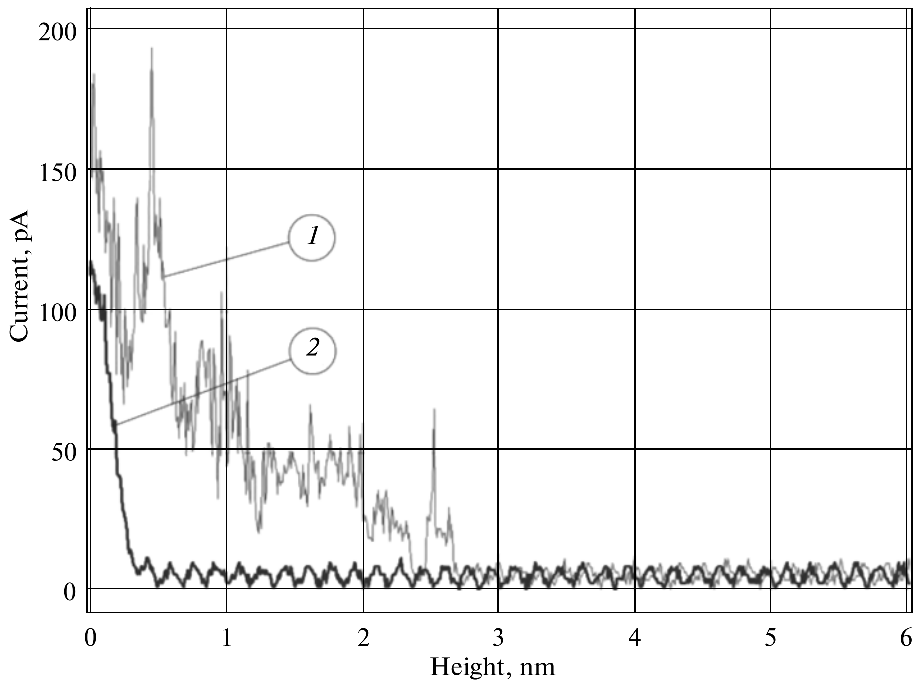

Fig. 8. $I(z)$ characteristics on the HOPG surface, which are obtained using STM probes in terms of the following methods: *1*—ECE; *2*—FIB.

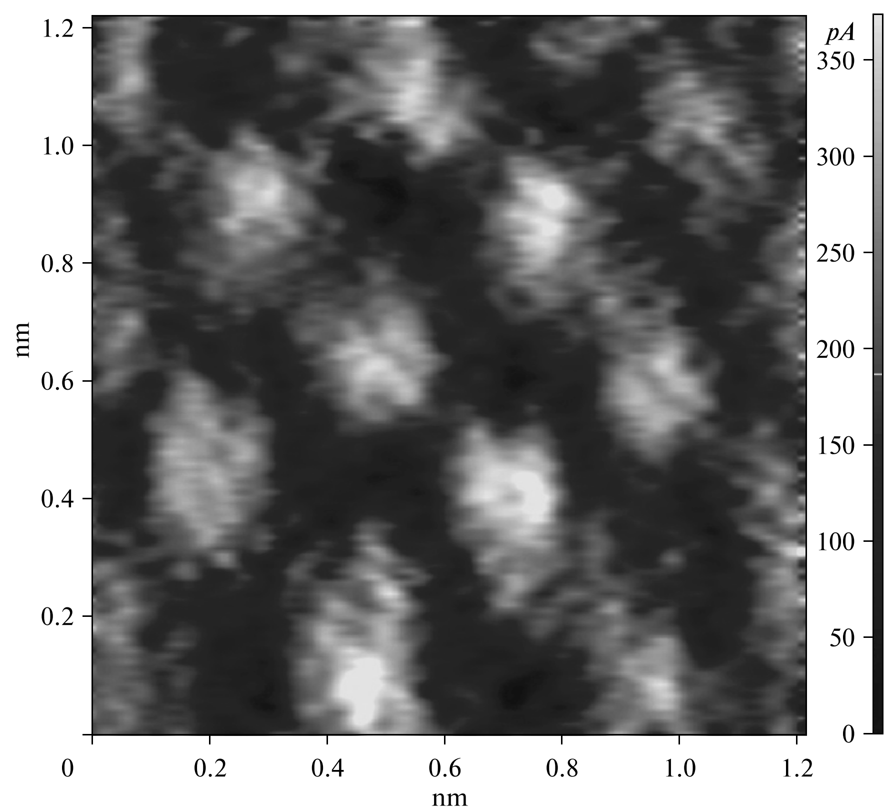

Fig. 9. STM image of the HOPG surface that is obtained by the probe modified by FIB method.

the FIB method provides the formation of the probes with the radius of tip rounding less than 10 nm and aspect ratio 1 : 50. It is shown that the use of modified cantilevers for the diagnostics of submicron structures allows one to minimize the artefacts of SPM images, as well as to increase the resolution and the reliability of the obtained results. The paper demonstrated the promising prospects of a modified cantilever application when studying IMC structures with a submicron topological norm.

The way of using the modified tungsten probes for STM images of conducting solid structures with an atomic resolution is also demonstrated.

The obtained results can be used to develop the technological processes of the production and modification of sensor probes for AFM and STM and the methods of diagnosing the structures of microelectronics and nanoelectronics, as well as microsystem and nanosystem technologies.

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