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Analysis of Modes of Nanoscale Profiling during Ion-Stimulated Deposition of W and Pt Using the Method of Focused Ion Beams

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Abstract—In this work the results obtained in experimental studies of conditions of the nanoscale profiling of a silicon substrate surface under the ion stimulation of W and Pt deposition by a Ga^+ ion beam are represented. It is shown that, according to combinations of process conditions, deposition, or etching processes, conditions of the formation of transition structures can also be implemented. It is found that the rate of ion-stimulated deposition of W and Pt averages 8 nm/min and 50 nm/min for ion-beam currents of 2.3 pA and 7.9 pA, respectively, and the rate of ion-beam etching of a silicon substrate is 6 nm/min and 55 nm/min for ion-beam currents of 2.3 pA and 111.4 pA respectively. With the use of these results, the modes are determined and a prototype of sensing element of tunnel accelerometer is formed using focused ion beams (FIBs). The results can be used to develop manufacturing methods of generating patterns of nano- and microelectronics and nano- and microsystem engineering on the basis of FIBs.

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INTRODUCTION

The problem of forming nanoscale structures is of immediate interest for the creation of modern circuit technology for nanoelectronics and nanosystem engineering [1]. To solve this problem, methods of local surface patterning with the use of various actions (optical lithography, electron-beam lithography, ion-beam lithography, scanning probe microscopy lithography, and methods of epitaxial and atomic layer deposition) become a frequent practice.

The focused ion beam (FIB) method opens up new opportunities for the creation of nanostructures with controlled variables. The FIB method makes it possible to perform the local ion-stimulated vapor deposition of materials, ion-beam etching, and ion implantation [1–4] with high resolution. The noted possibilities make it possible to use the FIB method for solving problems of nanolithography, nanometer-scale patterning, and surface modification [4, 5].

In particular, the nanoscale structures of Pt and W deposited by the FIB method are used to form MEMS and NEMS structures, to make electric interconnections during the reengineering of VLSI circuits, to protect structures during sample preparation in TEM, and for electrostatic charge compensation in SEM. The ion-stimulated deposition of tungsten is also used during the formation of various micromechanical structures [1, 2, 5–8]. However, the widespread application of the method is slowed down by insufficient previous studies of the processes of ion-stimulated deposition of materials under the influence of FIBs [1–3].

The purpose of this work is to experimentally research modes of ion-beam action on the surface of the silicon substrate to determine common laws of the local ion-stimulated vapor deposition of platinum and tungsten by the FIB method to form elements of micro- and nanoelectromechanical systems.

EXPERIMENTAL PROCEDURE

Experimental research were carried out using a NANOFAB NTC-9 FIB CVD module ultra-high-vacuum nanotechnological facility (NT-MDT, Russia) equipped with a C31U ion gun and a gas-injection system (Orsay Physics, France). The SEM investigation into the obtained structures was carried out with a Nova NanoLab 600 scanning electron microscope (FEI Company, the Netherlands), and AFM using a ultra-high-vacuum module of the scanned probe microscopy of the NANOFAB NTC-9 facility. N-type silicon substrates, with resistivity of 4.5 Ohm·cm, were used in the experiments.

A liquid-metal gallium ion source forming the Ga^+ ion beam up to 10 nm in diameter and up to 30 keV of energy with an ion-beam current from 1 to 500 pA is used in the FIB CVD module. For the ion-stimulated deposition of tungsten, carbonyl $W(CO)_6$ is used, and for the ion-stimulated deposition of platinum,

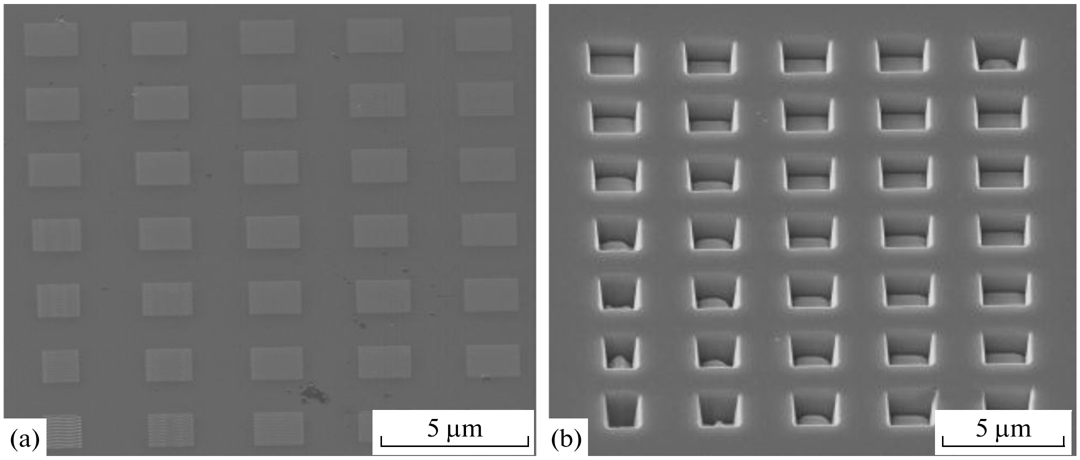

Fig. 1. SEM images of Pt structures arrays formed at (a) I = 2.3 pA and (b) I = 511.4 pA.

its organometallic compound $Pt(C_2H_5C_5H_4)(CH_3)_3$ [6, 7] is used.

Ion-stimulated deposition was carried out according to the procedure that is standard for that method; during this procedure, gas compounds are injected locally into the area of exposure of ion beam by means of the gas-injection system through fine-bored nozzles that are brought to the surface of the substrate [1, 2]. The process of ion-stimulated deposition results from the degradation of the entering gas compound under the action of accelerated focused ion flow to release the compound component deposited to the substrate. According to [9], the basic processes of dissociation of gas molecules take place in the adsorbed monolayer on the surface of the substrate during local heating under the action of the ion flow. The volatile reaction products are exhausted by a vacuum system [2].

The severity of the ion-beam exposure to the surface of a substrate is defined by combinations of process conditions: ion-beam current (I), number of passes of the ion beam (N), region of overlap of the next points of ion beam exposure (RS), effective diameter of the ion beam (PS), single-pass duration of ion beam (ST), exposure time of ion beam at single point (PT), and cumulative exposure of ion beam by a specified raster-type pattern (T).

The exploration of common laws of ion-stimulated deposition was carried out. For this purpose, the raster-type pattern in the form of an array consisting of 35 test structures of 2 × 2 μm in size was made in the FIB CVD module control program. Ion-beam scanning by the pattern was carried out at RS = 0%, T = 90 s, parameter N varied from 1 to 1 million, depending on the combinations of PT and T; the variation range for PT and PS is shown in Fig. 1a. The ion-beam current was determined as 2.3, 7.9, 33.6, 111.4, and 511.4 pA for each array respectively. By so doing, five arrays in 35s test structures in every array studied by the SEM method were obtained.

At the second stage, exploring the dependence of the rate of ion-beam etching of silicon substrate on the number of passes of ion beam by the raster-type pattern was carried out. For this purpose, the raster-type pattern in the form of an array consisting of 15 test structures 2 × 2 μm in size was created in a FIB CVD module control program. The process conditions are represented in the table. After treatment by an ion beam at ST = 10 ms, the surface of the substrate was studied by the AFM method. In so doing, several test structures arrays formed at the ion beam currents from 2.3 to 111.4 pA were studied.

At the third stage, experimental research of common laws of ion-stimulated deposition of Pt and W were carried out. For this purpose, a raster-type pattern in the form of array consisting of 15 test structures 4 × 4 μm in size was created in a FIB CVD module control program. The process conditions are represented in the table. The structures formed at ST_1 = 10 ms and ST_2 = 50 ms were studied by the AFM method. In so doing, the test-structure array formed at the current of 7.9 pA on the substrate was studied.

Condensed values of process conditions

	N				
RS = 0%; PS = 5 nm	10	20	30	40	50
	100	200	300	400	500
	1000	2000	3000	4000	5000

RESULTS AND DISCUSSION

Analysis of the SEM-images obtained at the first stage of experimental research showed that, as a result of ion-beam action on the surface of the silicon substrate during Pt and W deposition by the FIB method,

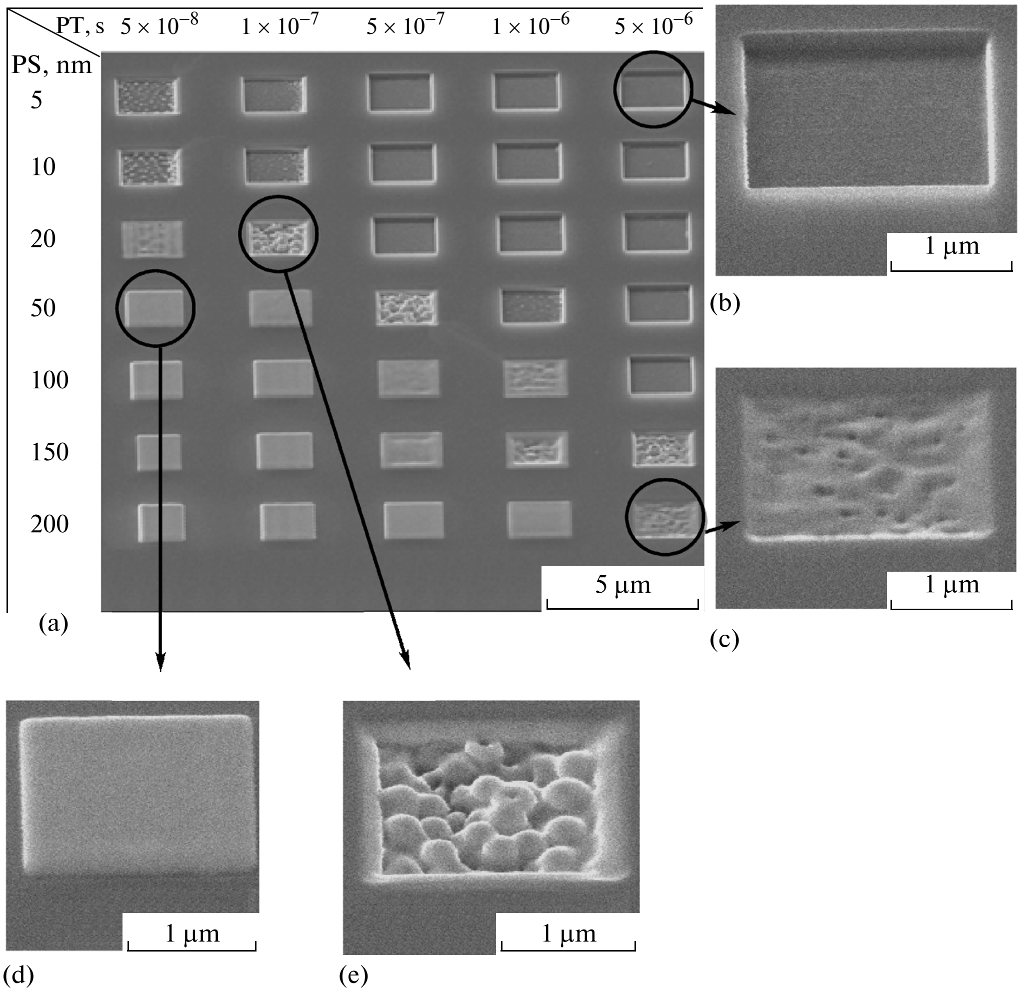

Fig. 2. SEM images of test-structure arrays obtained by the ion-stimulated deposition of Pt at an ion beam current of 7.9 pA.

depending on the combinations of process conditions, various processes of nanoscale profiling of the surface of the substrate can be implemented.

When the ion-beam current was 2.3 pA, the processes of ion-stimulated deposition of materials (Fig. 1a) were implemented on the whole array of the Pt test structures; when the ion-beam current was 511.4 pA, the processes of ion-beam etching of the substrate were implemented (Fig. 1b). Figure 2 shows the array of Pt test structures formed by ion-stimulated deposition at an ion-beam current of 7.9 pA, on which all the processes observed in the experiment are implemented.

Implementation of the ion-beam etching processes can be explained by the influence of combinations of the process conditions wherein the next dose of chemically active precursor gas has no time to enter the ion-beam exposure area from the gas injection system, and the layer of material deposited during the preceding ion beam passes is etched by the ion beam together with a near-surface layer of the substrate (Figs. 1b, 2b).

Intermediate values of ion-beam currents resulting in the implementation of interleaved modes wherein both material deposition and etching of substrate are observed (Figs. 1b, 1d) for the test structures array. When the of ion-beam current increases, the quantity of deposited structures decreases and the quantity of etched structures increases.

A similar tendency is observed at W structure deposition.

The common laws of implementation of the noted processes are influenced by physicochemical properties of chemically active precursor gases [6, 7, 9] such as the adsorption coefficient, density of monolayer of adsorbed molecules, and temperature dependency of desorption rate. For the studied process conditions, the ion-stimulated deposition of Pt test structures changes to etching when the ion-beam current is 33.6 pA, and of W, when the ion-beam current is

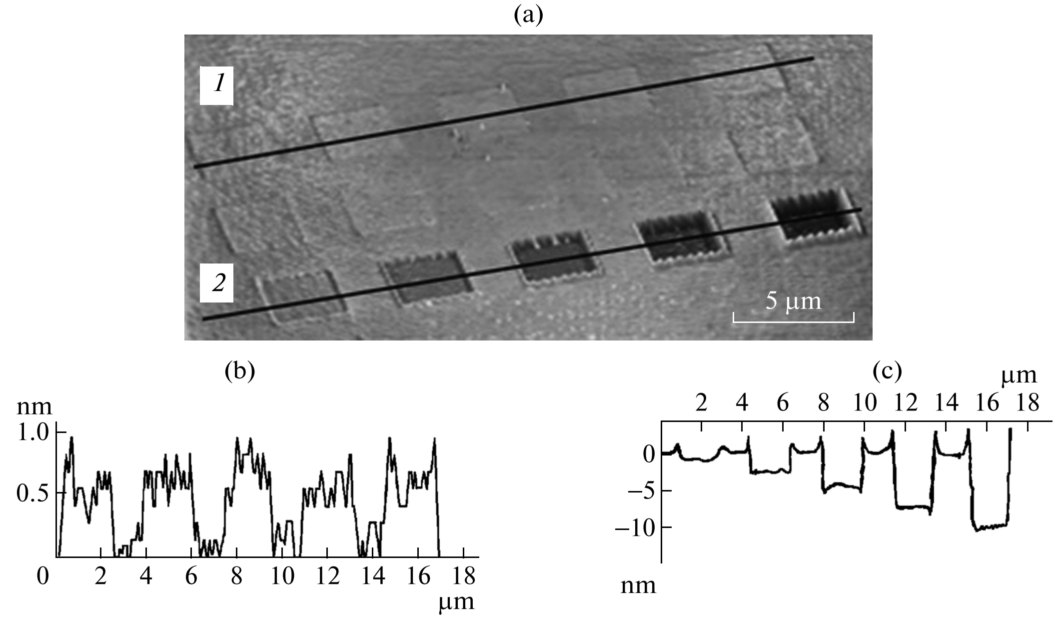

Fig. 3. Array of test structures obtained by ion-beam etching for I = 2.3 pA: (a) AFM image, (b) profilogram along the line *1*, and (c) profilogram along line *2*.

511.4 pA. Supposedly the adsorption coefficient of $W(CO)_6$ is higher than of $Pt(C_2H_5C_5H_4)(CH_3)_3$; thereafter the complete desorption of its molecules and the discontinuity of the deposition process takes place under higher temperatures corresponding to higher FIB currents.

Some interleaved modes implement the criteria of a complicated balance between the competitive processes of ion-beam etching and ion-stimulated deposition, resulting in the formation of transition structures with complex profile of bottom (Fig. 1e) or membrane-type structures (Fig. 1c).

As can be seen from the above, the process conditions of FIB wherein processes of ion-beam etching or ion-stimulated deposition are implemented, whose common laws were studied at the second and the third stages respectively, were determined at the first stage of experimental research.

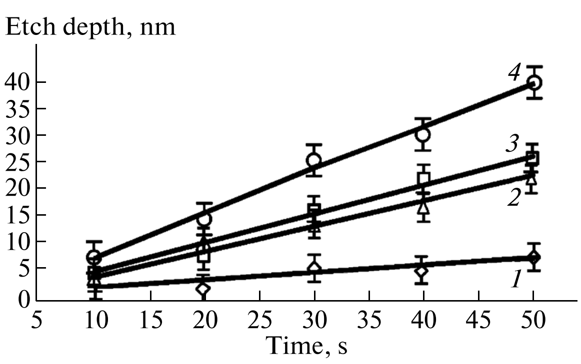

Fig. 4. Dependence of the etch depth of silicon on the exposure time for ion-beam currents: (*1*) 2.3, (*2*) 7.9, (*3*) 33.6, and (*4*) 111.4 pA.

An analysis of the results obtained in studies of the processes of ion-beam etching showed that a local increase in volume of the near-surface layer exposed by ions (Fig. 2b) takes place at modes with a limited number of passes, as well as at a low ion-beam current. This phenomenon can occur due to radiation-induced pore-formation (implantation swelling) [10].

On the basis of the results of processing statistical data of the second stage of the experimental research, dependences of the etch depth of silicon on the exposure time for various ion beam currents (Fig. 3) were obtained. Analysis shows that the rate of etching of silicon by Ga^+ ion beam is constant and depends only on the ion-beam current (Fig. 4) within the range of treatment durations covered.

The experimental results associate well with the dependences represented before in [11], representative of the influence of the FIB parameters on the characteriscics of substrate topography under etching (Fig. 5).

In carrying out the third stage of experimental research, arrays of 15 Pt test structures 4 × 4 μm in size (Fig. 6) were obtained. After W deposition, structures similar to the Pt ones in appearance and behavior of variation were obtained.

On the basis of analysis of the results of the third stage of the experimental research, the dependences of

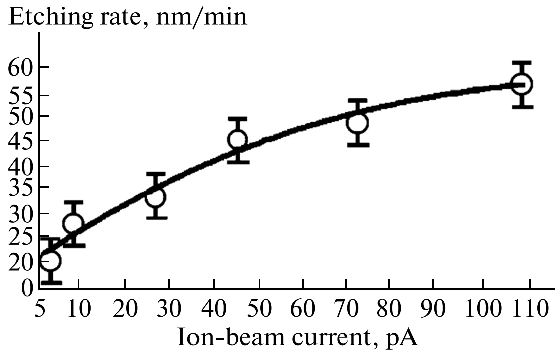

Fig. 5. Dependence of the rate of ion-beam etching of silicon on an ion-beam current.

thickness of the deposited layers of Pt and W on ion beam exposure time (Fig. 7), where $ST_1 = 10$ ms and $ST_2 = 50$ ms, were obtained.

An analysis of the obtained dependences shows that the rates of ion-stimulated deposition of Pt and W for the investigated process conditions are not very dependent on exposure time and reach 52 nm/min and 14 nm/min at $ST_1 = 10$ ms and 48 nm/min and 3 nm/min at $ST_2 = 50$ ms, respectively.

The results obtained in experimental studies were used to develop design and manufacturing technology of a prototype of sensing element of the accelerometer (Fig. 8), a distinctiveness of which manufacturing technology carries out of all manufacturing operations in single total fabrication cycle in an ultrahigh vacuum. The development was implemented by a NANOFAB NTC-9 cluster type nanotechnological facility.

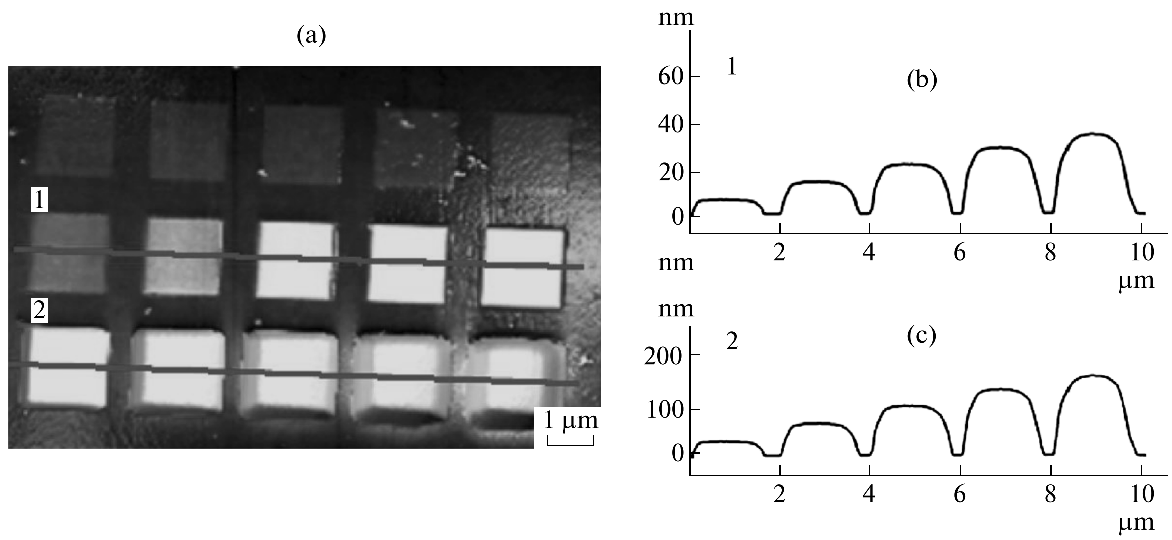

Fig. 6. An array of test structures obtained by the ion-stimulated deposition of Pt at I = 7.9 pA: (a) AFM image; (b) profilograms.

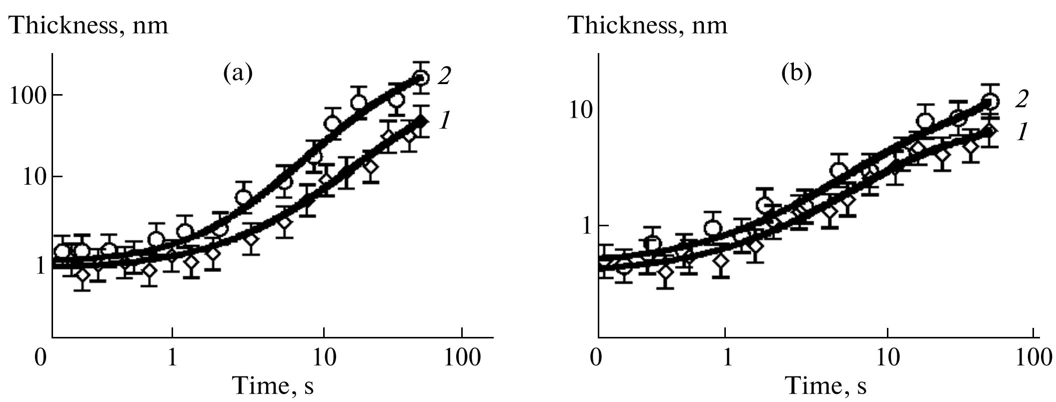

Fig. 7. Dependences of the thickness of the deposited layer on the ion-beam exposure time: (a) Pt and (b) W; (*1*) ST = 10 ms; (*2*) ST = 50 ms.

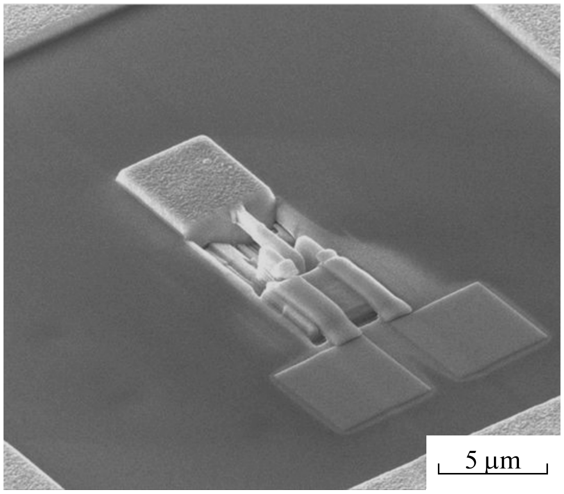

Fig. 8. SEM image of a prototype of a sensing element of the sensor accelerometer obtained on the basis of using the FIB method.

CONCLUSIONS

Experimental research of modes of nanoscale profiling of the surface of silicon substrate during the ion-stimulated deposition of Pt and W was carried out. It is found that the implementation of processes of etching and deposition of materials, or transition processes, can be achieved by varying parameters of action on a substrate by ions. Rates of ion-stimulated deposition of W and Pt that average 8 nm/min and 50 nm/min at the currents of ion beam of 2.3 pA nd 7.9 pA respectively, and rates of ion-beam etching of silicon substrate reaching 6 nm/min and 55 nm/min at currents of ion beams of 2.3 pA and 111.4 pA were determined.

As a matter of record of the modes of the ion-stimulated deposition of W (Figs. 1a, 2d), the ion-beam etching of substrate (Figs. 4, 5), and the time dependence of thickness of deposited structures (Fig. 7b), the prototype of sensing the element of the accelerometer was formed on a $Ti/SiO_2/Si$ structure by the FIB method, in a single total fabrication cycle, with the use of etching and deposition operations. An operating principle of the sensing element is bending of the cantilever, in dependence on acceleration direction, in a plane parallel to the plane of the substrate, and its closing with one of the electrodes formed by the ion-stimulated deposition of W and isolated from the substrate. Sensing elements of this type can be used as aspect sensors; as movement control sensors in navigation systems; in industrial vibration-based diagnostics; and in systems of nondestructive testing, technical diagnostics, and protection.

Our results can be used in the future for the development of methods of manufacturing functional parts and devices of nanoelectronics and nanosystem engineering, in particular, on the basis of using the NANOFAB NTC-9 cluster type nanotechnological facility.

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