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NANOTECHNOLOGY

Study of Modification Methods of Probes for Critical-Dimension Atomic-Force Microscopy by the Deposition of Carbon Nanotubes

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Abstract—The results of an experimental study of the modification of probes for critical-dimension atomic-force microscopy (CD-AFM) by the deposition of carbon nanotubes (CNTs) to improve the accuracy with which the surface roughness of vertical walls is determined in submicrometer structures are presented. Methods of the deposition of an individual CNT onto the tip of an AFM probe via mechanical and electrostatic interaction between the probe and an array of vertically aligned carbon nanotubes (VACNTs) are studied. It is shown that, when the distance between the AFM tip and a VACNT array is 1 nm and the applied voltage is within the range 20–30 V, an individual carbon nanotube is deposited onto the tip. On the basis of the results obtained in the study, a probe with a carbon nanotube on its tip (CNT probe) with a radius of 7 nm and an aspect ratio of 1:15 is formed. Analysis of the CNT probe demonstrates that its use improves the resolution and accuracy of AFM measurements, compared with the commercial probe, and also makes it possible to determine the roughness of the vertical walls of high-aspect structures by CD-AFM. The results obtained can be used to develop technological processes for the fabrication and reconditioning of special AFM probes, including those for CD-AFM, and procedures for the interoperational express monitoring of technological process parameters in the manufacturing of elements for micro- and nanoelectronics and micro- and nanosystem engineering.

Keywords: nanotechnology, atomic-force microscopy, carbon nanotubes, CD-AFM, focused ion beams.

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INTRODUCTION

Increasing the degree of very-large-scale integrated circuit (VLSIC) integration results in topological standards of less than 90 nm, with the influence exerted on the parameters and working capacity of VLSICs by the deviation of element sizes from given values dramatically increasing. In particular, the roughness of the vertical wall of the gate greatly affects the threshold voltage of metal–oxide–semiconductor (MOS) transistors in VLSICs [1]. Here, an important task is the development of procedures and technical means for determining the geometric parameters and the roughness of vertical walls in high-aspect nanostructures.

Critical-dimension atomic-force microscopy (CD-AFM) is a promising method that can effectively solve problems associated with the inter-op express monitoring of the technological process during the manufacturing of VLSICs by examining the morphology and local surface properties of high-aspect structures with a high spatial resolution [1].

The resolvability of an AFM microscope is determined by the radius of curvature and aspect ratio of the probe-tip sides [2–6]. Therefore, the development of methods for the fabrication of probes with parameters that make it possible to minimize distortions of the surface morphology of the sample surface in an AFM investigation is a topical issue. There exist several technological methods for the fabrication of AFM probes. Commercially available probes are fabricated from silicon by microelectronic methods, with the result that a pyramidal tip with a radius of 10–100 nm is formed on the cantilever [2]. Surfaces containing high-aspect structures are studied by the AFM technique with cantilevers which have probes whose tip is modified by the method of focused ion beams (FIB), with a probe-tip radius of about 9 nm [6–8]. A small radius of curvature and high aspect ratio between sides of the probe tip are obtained by modification with local electron-beam and ion-stimulated deposition methods [2, 9].

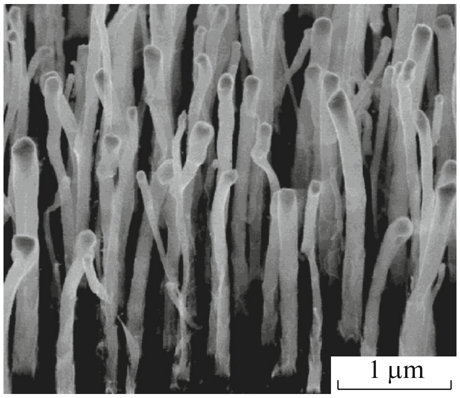

Fig. 1. SEM image of a VACNT array.

A promising way to form high-aspect AFM probes is by positioning a carbon nanotube (CNT) to the probe tip [10–14]. Due to the small diameter, high rigidity, and the elastic properties of CNTs, probes of this kind differ from silicon probes by their high aspect ratio (>1:10) and small tip radius (<10 nm), and from those modified by the FIB method by enhanced wear resistance. CNT probes can be used to determine the roughness of vertical walls in high-aspect structures by the CD-AFM method [15].

The most widely employed among all methods for the fabrication of AFM probes are the local growth of CNTs onto a probe tip by chemical vapor deposition and the deposition of a CNT onto a probe tip under the action of ac electric field via dielectrophoresis and under the action of dc voltage (~60 V) [10–14]. However, these techniques are unsuitable for mass production because the technological process is complicated and, as a consequence, rather expensive. In addition, the result obtained upon the deposition of a CNT onto a probe tip under the action of an electric field heavily depends on the probe radius [16], which is due to the dependence of the region with maximum electric-field strength on the probe-tip radius. For example, at a probe-tip radius of less than 50 nm, the probability of CNT deposition is 5%; with the tip radius increasing to 150–250 nm, the probability of deposition increases to 70% [16]. In this case, an increase in the tip radius of an AFM probe simultaneously leads to an increase in the number of CNTs deposited onto the probe during a single pulse of applied voltage. Use of this pattern requires the preliminary formation of a probe of certain radius, which additionally complicates the fabrication technology of AFM probes.

It should be noted that the Russian company NT-MDT manufactures commercially available AFM probes with carbon nanotubes at the tip [17]. However, the probes fabricated by NT-MDT are expensive, which restricts their application.

In the present study, we suggest the use of AFM probes that have undergone wear as a result of intensive studies of the surface of samples. In this case, the deposition of CNTs onto the tip of probes of this kind enables their reconditioning for further use in the precision surface studies of materials and also in determining the parameters of high-aspect structures.

The goal of our study is to examine methods for the modification and reconditioning of AFM probes via the deposition of a carbon nanotube onto a probe tip under the action of mechanical and electrostatic interactions between the AFM probe and an array of vertically aligned carbon nanotubes (VACNTs) to form CNT probes. Also, the characteristics of AFM probes in analyzing high-aspect structures via the CD-AFM method are examined.

EXPERIMENTAL

An experimental sample with an array of VACNTs was grown in a unit for the plasma-enhanced chemical-vapor deposition (PECVD) of a NANOFAB NTC-9 multipurpose nanotechnology complex (NT-MDT, Russia) [18]. A silicon wafer, on whose surface a double-layer structure constituted by a 20-nm-thick catalytic layer of titanium and a 10-nm-thick adhesive layer of nickel was formed, served as the substrate. The height and density of VACNTs in the array, determined by scanning electron microscopy (SEM) using a Nova NanoLab 600 scanning electron microscope (FEI Company, The Netherlands), were about 2 μm and 8 μm^{-2}, respectively; the diameter of nanotubes in the array varied from 12 to 75 nm (Fig. 1).

Nanotubes were deposited from the array of VACNTs onto the tip of an AFM probe using an Ntegra probe nanolaboratory (NT-MDT). NSG10 commercial silicon cantilevers [17], with a platinum coating and a tip radius of 80 nm which underwent wear as a result of intensive use, were used as probes. Deposition under mechanical interaction of the AFM probe with the VACNT array was performed via force AFM lithography in the vector mode with the probe pressed against the array surface with a force of 0.5 μN (Fig. 2a).

Deposition under electrostatic interaction of the AFM probe with the VACNT array was performed by the AFM method in the current-spectroscopy mode upon the application of a voltage pulse with an amplitude of 10 to 40 V and a width of 1 s to the probe–sample system. This was carried out upon mechanical contact between the probe and the surface of the VACNT array (Fig. 2b) and at distances of 1 and 2 nm between the AFM probe and the surface of the VACNT array (Fig. 2c). No voltage was applied when the AFM probe was removed from the surface of the VACNT array; the carbon nanotubes held in place on the surface of the AFM probe only by Van der Waals forces [19].

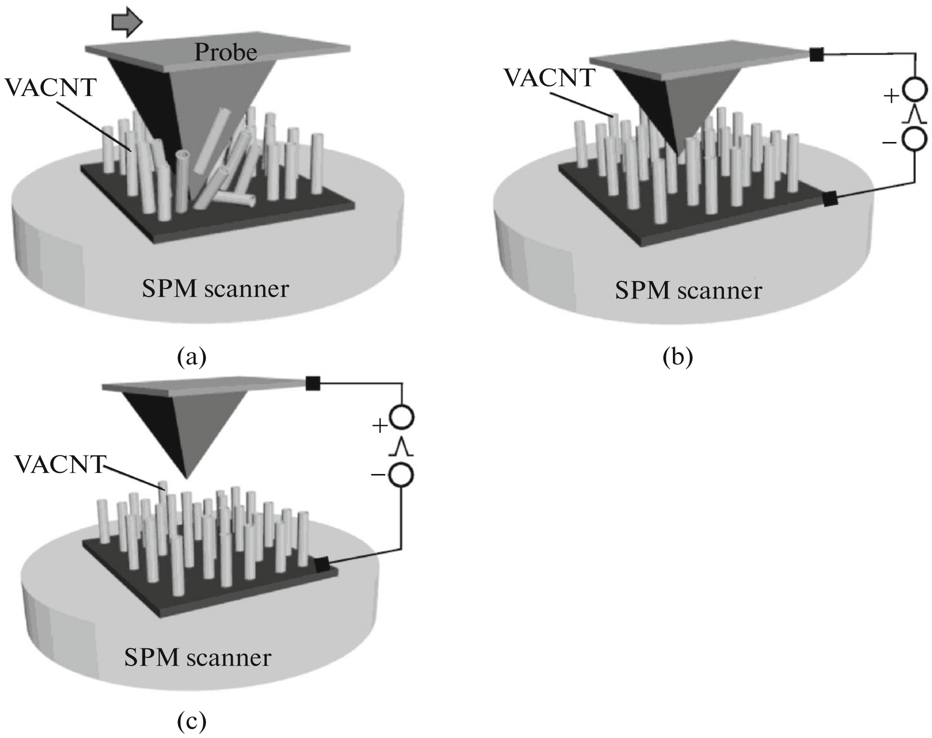

Fig. 2. Schematic of the deposition of CNTs onto the tip of an AFM probe: (a) by force lithography, (b) in the current-spectroscopy mode in the case of mechanical contact between the probe and CNTs, and (c) in the current-spectroscopy mode with a gap between the probe and CNTs.

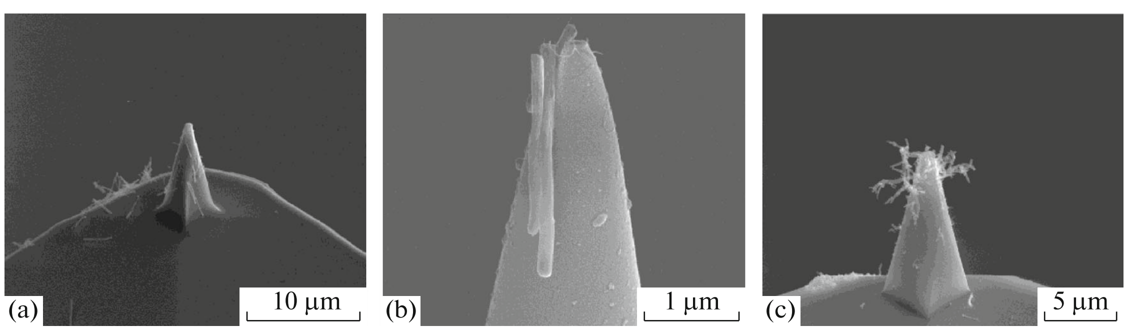

Fig. 3. AFM probe upon the deposition of CNTs by various methods: (a) by force lithography, (b) in the current-spectroscopy mode in the case of mechanical contact between the probe and VACNTs and a probe voltage of 20 V, and (c) in the current-spectroscopy mode at a distance of 1 nm and a probe voltage of 40 V.

The results of VACNT deposition onto the tip of the AFM probe were examined by the SEM method (Fig. 3).

An individual nanotube deposited onto the probe tip was fixed via local ion-stimulated deposition by the FIB method using the Nova NanoLab 600 electron microscope (Fig. 4a). The procedure in which the CNT is fixed to the surface of the probe tip is based on the deposition of carbon for 20 s by the FIB method using a graphical template of the form of a circle of given diameter. A probe tip with a CNT fixed by the FIB method is shown in Figs. 4b and 4c.

The resolvability of the resulting probe was tested for reliability of AFM measurements usin the Ntegra probe nanolaboratory by scanning the surface of a TGZ2 profile height standard [17] with a standard NSG-10 cantilever and the CNT probe. The standard was scanned in the tapping mode of AFM. Figure 5 shows AFM surface images of the TGZ2 profile standard, obtained using different cantilevers. Figure 6 shows a 3D AFM image of the TGZ2 profile standard and a profilogram of its vertical wall. The AFM images were analyzed to determine the geometric parameters of the TGZ2 standard using Image Analysis 3.5 and MathCad 14 software packages.

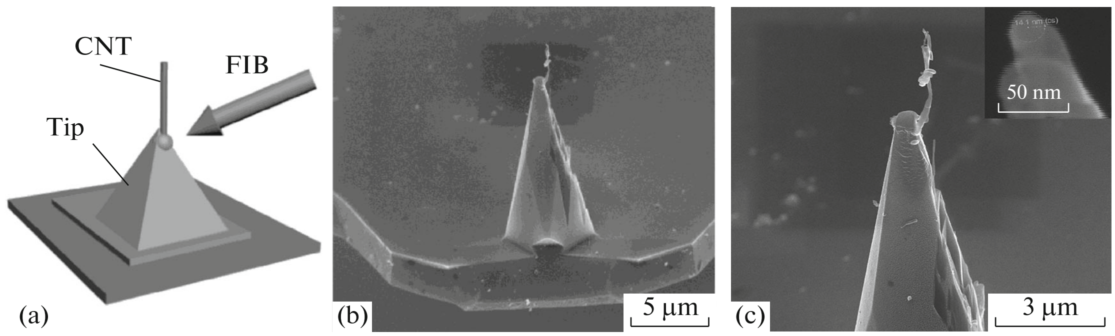

Fig. 4. CNT probe produced by the current-spectroscopy method at a distance of 1 nm and voltage of 30 V: (a) schematic of the fixation of a CNT by the FIB method and (b, c) SEM images with magnifications of ×10000 and ×25000, respectively; the diameter of a nanotube is 14.1 nm.

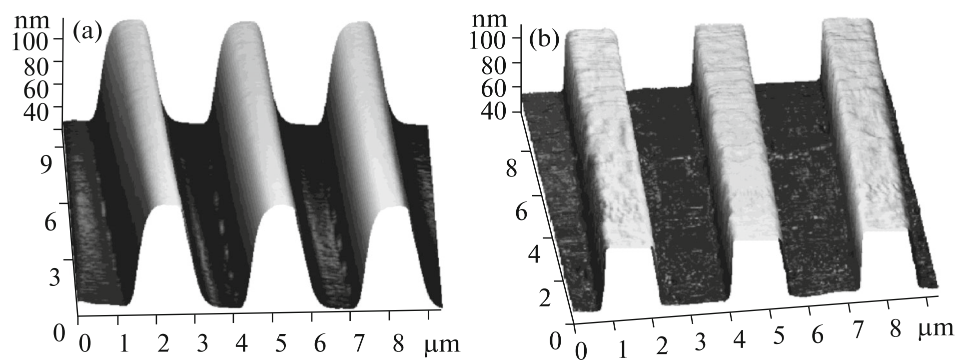

Fig. 5. AFM image of the surface of the TGZ2 profile standard, obtained using (a) a commercial probe and (b) a CNT probe.

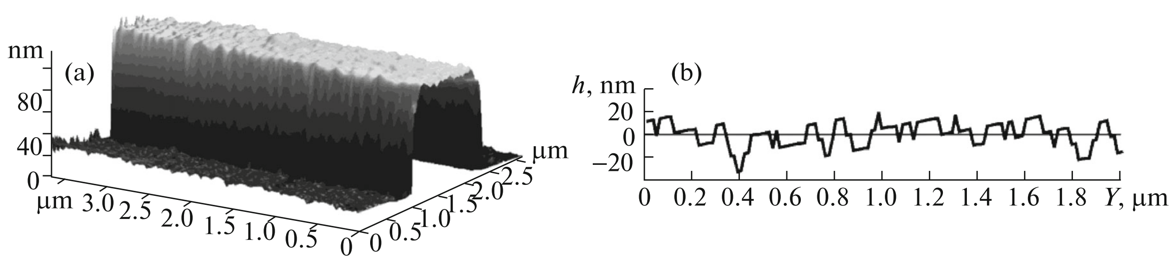

Fig. 6. Element of the TGZ2 profile standard: (a) 3D ACM image and (b) profilogram of a vertical wall.

RESULTS AND DISCUSSION

Upon performing force lithography of the VACNT array, we found that, upon hard contact with the AFM probe, carbon nanotubes are detached from the substrate [20] and then, under the action of Van der Waals forces, adhere to the lateral surface of the probe and the cantilever beam (see Fig. 3a). According to estimates, the Van der Waals forces between the silicon cantilever and the VACNT array may attain a value of 284 N/cm^2 for an individual nanotube [19], which is sufficient for strong bonding of a CNT to the probe surface. However, it is difficult in this case to control the number of deposited nanotubes and their arrangement on the probe surface.

We used the AFM method in the current-spectroscopy mode for the precise deposition of carbon nanotubes onto the apex of the probe tip. An external voltage was applied between the AFM probe and the VACNT array, which resulted in the formation of a nonuniform electric field with maximum strength near the probe tip. Under electrostatic interaction between the AFM probe and the VACNT array, carbon nanotubes are detached from the substrate without any mechanical action, transferred to the probe, and localized near its tip.

An analysis of the influence exerted by the applied voltage in the case of mechanical contact between a AFM probe and the VACNT array (see Fig. 2b)

Geometric parameters of the TGZ2 profiled standard

Data	Parameters of TGZ2 standard		
	Period, μm	Height, nm	Angle of deviation of the vertical wall from the normal, deg
Datasheet specification	3.0 ± 0.01	112 ± 2	~0
Measured with NSG10 probe	2.9 ± 0.02	110 ± 2	11 ± 1
Measured with CNT probe	3.05 ± 0.04	112 ± 0.5	3 ± 1

demonstrated that no CNTs are deposited onto the probe-tip surface at voltages lower than 10 V. This is due to insufficient electric-field strength for the detachment of VACNTs from the substrate. At voltages in the range of 10–20 V, single CNTs are deposited. However, these results are poorly reproducible, which is probably due to nanotube breakage at possible places of structural defects, rather than to the detachment of a CNT from the substrate, i.e., the probability of CNT deposition onto the surface of an AFM probe tip upon the application of a voltage lower than 20 V depends on the possible presence of defects in the CNT being deposited. When a voltage of 20 to 30 V is applied, two to three CNTs are deposited mainly on the lateral surface of the probe (Fig. 3b). This is possibly due to partial penetration of the probe deep into the VACNT array when the probe is brought to its surface.

To tackle this problem, carbon nanotubes were deposited at distances of 1 and 2 nm between the AFM probe and the surface of the VACNT array (see Fig. 2c). As a result, it was found that a large number (>10) of CNTs are deposited near the probe apex at a distance of 2 nm between the probe and the VACNT array and voltages in the range 20–40 V (Fig. 3c). A probable reason for this is that the area of interaction of the electric field created around the apex of the probe tip with the surface of the VACNT array increases as the probe is moved away from the sample. At a distance of about 1 nm between the probe and the VACNT array and voltages of 20 to 30 V, the reproducible deposition of one or two nanotubes is observed near the apex of the AFM probe. Figure 4b shows the SEM image of a CNT deposited onto a probe at a voltage of 30 V. As the voltage is raised to 40 V, the number of deposited VACNTs increases.

It was found that the optimal parameters of CNT deposition onto the tip of an NSG10/Pt cantilever from an experimental VACNT array is a voltage in the range 20–30 V and a distance of about 1 nm between the AFM probe and the VACNT array. Using these parameters, we fabricated a CNT probe with a tip radius of about 7 nm and an aspect ratio of 1:15 between the sides of the tip whose SEM image is presented in Fig. 4c.

Analysis of AFM images of the TGZ2 profile standard, obtained using the NSG 10 cantilever (see Fig. 5a), demonstrated the presence of scanning artifacts whose appearance is due to the contribution from the cone angle of the probe tip. In scanning of the TGZ2 profile standard with the CNT probe, there are no artifacts in the AFM image (see Fig. 5b).

The height and the period of structures in the TGZ2 standard, determined with the CNT probe, were 112 nm and 3 μm, respectively, which is in good correlation with the datasheet specifications (see table). In addition, comparison of the AFM images of the TGZ2 standard, obtained using the NSG10 commercial cantilever (see Fig. 5a) and the CNT probe (see Fig. 5b), demonstrated that the verticality of the side walls of the structure in the standard was better displayed on account of the high aspect ratio between the sides of the modified CNT probe.

An analysis of the vertical wall of the TGZ2 profile standard (see Fig. 6) demonstrated that the roughness of the side wall [average of the maximum heights of surface irregularities in conformity with GOST (State Standard) 7016–82)] was 9.1 nm.

CONCLUSION

The results obtained in the experimental study of methods used to modify probes for atomic-force microscopy by the deposition of CNTs onto the tip of a worn probe under the action of mechanical and electrostatic interaction between an AFM probe and a VACNT array demonstrated the following. Upon the force nanolithography of a VACNT array, nanotubes detached from the substrate adhere to the probe surface due to Van der Waals forces, but there is no localization at the probe tip. When a voltage pulse is applied between the AFM probe and the VACNT array, local deposition of CNTs at the probe tip is possible, with its result dependent on the pulse amplitude and distance between the probe and the VACNT surface.

It was found that, at a voltage of 20–40 V, VACNTs detach from the substrate and then a CNT is deposited onto the surface of the AFM probe. In the case of mechanical contact of the probe with the VACNT array, deposition occurs under the action of an electric field on the lateral surface of the probe. Carbon nanotubes are deposited onto the tip of the AFM probe at a distance exceeding 1 nm. As, however, the distance between the probe and the surface of the VACNT array increases (to more than 2 nm), a large number of nanotubes are deposited, which hinders the fabrication of a CNT probe. Thus, it was experimentally demonstrated that one or two carbon nanotubes are deposited onto the apex of the AFM probe from an experimentally obtained array of VACNTs at a voltage of 20–30 V and distance of 1 nm.

A CNT probe with a radius of about 7 nm and an aspect ratio of 1:15 between the tip sides was fabricated on the basis of the results obtained. Investigation of the surface of a TGZ2 profile standard with a CNT probe demonstrated better lateral and vertical resolvability, compared with the commercial probe, and the possibility of using CNT probes to examine the vertical walls of structures by the CD-AFM method.

The results obtained in the study can be used in the development of technological processes for the fabrication and reconditioning of special probes for atomic-force microscopy, including CD-AFM probes, and also in the development of procedures for the inter-op express monitoring of parameters of the technological process for manufacturing elements for micro- and nanoelectronics and micro- and nanosystem engineering.

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