Development and approbation of nanoscalpels based probes for atomic force microscopy in the field of plasmonics

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Abstract. The formation and approbation of the specialized nanoscalpel (NS) probes were carried out. The possibility of producing diffraction gratings on gold substrates for plasmonics applications was shown. The optimal lithography parameters were revealed by using NS probes. It was shown that NS probes allow improving the lithography results such as the depth and width of the incisions as compared to standard probes.

1. Introduction

In recent years, the probes based on single crystalline or amorphous whiskers are very promising to become one of the main instruments to study samples with developed topological structures by atomic force microscopy (AFM). Unlike nanotubes, whisker structures can be formed directly in the place of the electron beam exposure, they are relatively cheap and easy to manufacture, and besides, such probes have different structures due to usage of various precursor gases and have different forms [1, 2].

In the literature, there are a lot of works devoted to producing functionalized probe sensors based on whisker structures, which are designed to perform specific tasks, such as carrying out the lithography with high resolution and cell nanosurgery [3], as well as the manipulation of objects at the nanoscale level by atomic force microscopy [4]. One of the most interesting fields of such research is plasmonics.

The investigations devoted to excitation, propagation and subsequent detection of surface waves (plasmon polaritons) on the modified metal substrates are of great interest nowadays. This phenomenon is widely used in nanosensorics for studying and characterization of particles of nanometer size. Development of plasmon sources and simulations of effects with surface waves by interacting with the objects is actively investigated [5].

Herewith, scanning near-field optical microscopy, in particular, the photon scanning tunneling microscopy, is basically used for the detection of plasmon waves, while nanomodified periodic structures on metal substrates, such as periodically structured channels [6] and the diffraction gratings [7], for the formation of plasmonic waves.

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In this article, we consider the possibility of creating sources of plasmon waves (nanoscale gratings) by a high-precision surface modification using the atomic-force lithography with specialized probes based on nanoscalpel-formed whisker structures.

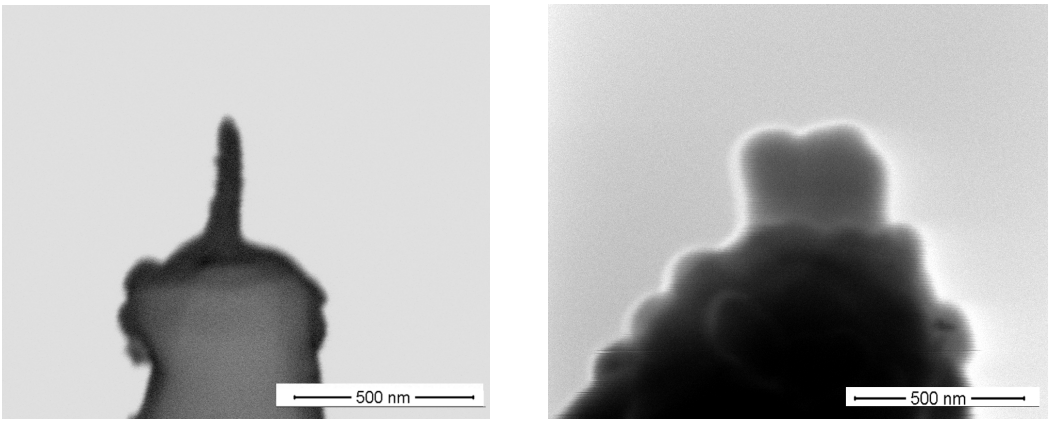
2. Experimental setup

Creation of the NS structures on the tops of probes and control of their parameters were performed by means of a scanning electron microscope (SEM) Inspect (FEI, USA). Studies of the features of NS probes were carried out by means of a scanning probe microscope (SPM) Ntegra Aura (NT-MDT, Russia) in the contact and semi-contact modes. Samples of polycarbonate and gold substrates were investigated and modified to create diffraction gratings with a fixed period. The SPM measurements were performed for both specialized NS and standard probes.

3. Results and discussion

NS is a 2D carbon nanostructure grown by vacuum deposition with a focused electron beam imitating the shape of a blade or a scalpel. This geometry defines the high stability of this type of probes in force impacts directed along the long axes of the nanostructures. NS probes can be used for visualization of samples of relief, as well as for their local modification.

Parameters of a NS grown on the pyramid tops of the probes can be varied within wide limits by changing the accelerating voltage of the electron beam, its diameter, the path of the beam and duration of deposition. It was revealed that the optimum NS width is 300–400 nm, the thickness is 60–80 nm, and the length does not exceed 400 nm while a NS operates in a force lithography mode. The single NS was formed in the vacuum chamber of a microscope at a pressure of about 10^{-3} Pa, an acceleration voltage of 30 kV, and an electron beam diameter of (1–3) nm.



(a) (b)

Figure 1. SEM image of the NS probe after 3 scans and lithography of a 10 nm gold film: a front view (a) and a side view (b).

For calculating the critical force (F_{cr}) that impacts the NS during operation, it is possible to use the approximation of longitudinal critical force for a rectangular thin plate [8] presented by the following equation:

$$F_{cr} \approx \frac{4Eba^3}{3l^2\sqrt{8(1+\sigma)}}, \quad (1)$$

where E is the Young's modulus, l is the NS length, a is the NS thickness, b is the NS width, σ is the Poisson's ratio.

Inserting typical values $E \approx 0.6 \cdot 10^{11}$ N/m^2, $l \approx 4 \cdot 10^{-7}$ m, $a \approx 6 \cdot 10^{-8}$ m, $b \approx 3 \cdot 10^{-7}$ m, $\sigma \approx 0.5$ [9], we obtain a value of F_{cr} of about 9 μN. Since the typical values of the "probe–sample" interaction forces in atomic force lithography are in the range 0.1–8 μN, NS probes are applicable to high-precision lithography on samples with different stiffness. According to the experimental data, the NS probe maintains about 2–3 cycles (20–30 patterns) of lithography (figure 1) retaining its structure and stiffness, which is comparable with the stability of the standard probes.

Semi-contact probes with a hardness of the order of 4.5–5.5 N/m were used during the atomic force lithography. A thin film of 10 nm gold sputtered on the surface of polycarbonate was used as a working substrate. The film of gold was chosen since this material is applied in the fabrication of nano-sized local sources of plasma waves such as diffraction gratings in plasmonics.

Below we consider the variation in the lithography process by altering the clamping (impact) forces (figure 2). Changing the time of probe standing at the same point had no effect on the structure, and it was chosen to be about t = 300 ms, the recommended lithography speed was about 2 μm/s. At a lower speed, there is a significant broadening of the grooves without improvement in depth, whereas at a high speed, reducing the penetration depth occurs without substantially reducing the width of the grooves. When the lithography strength is more than 3 μN, a noticeable shift of the material at the starting point of each line was observed. A possible effect of sticking a probe and contamination of the surface by shifting of the material can occur while increasing the impact forces.

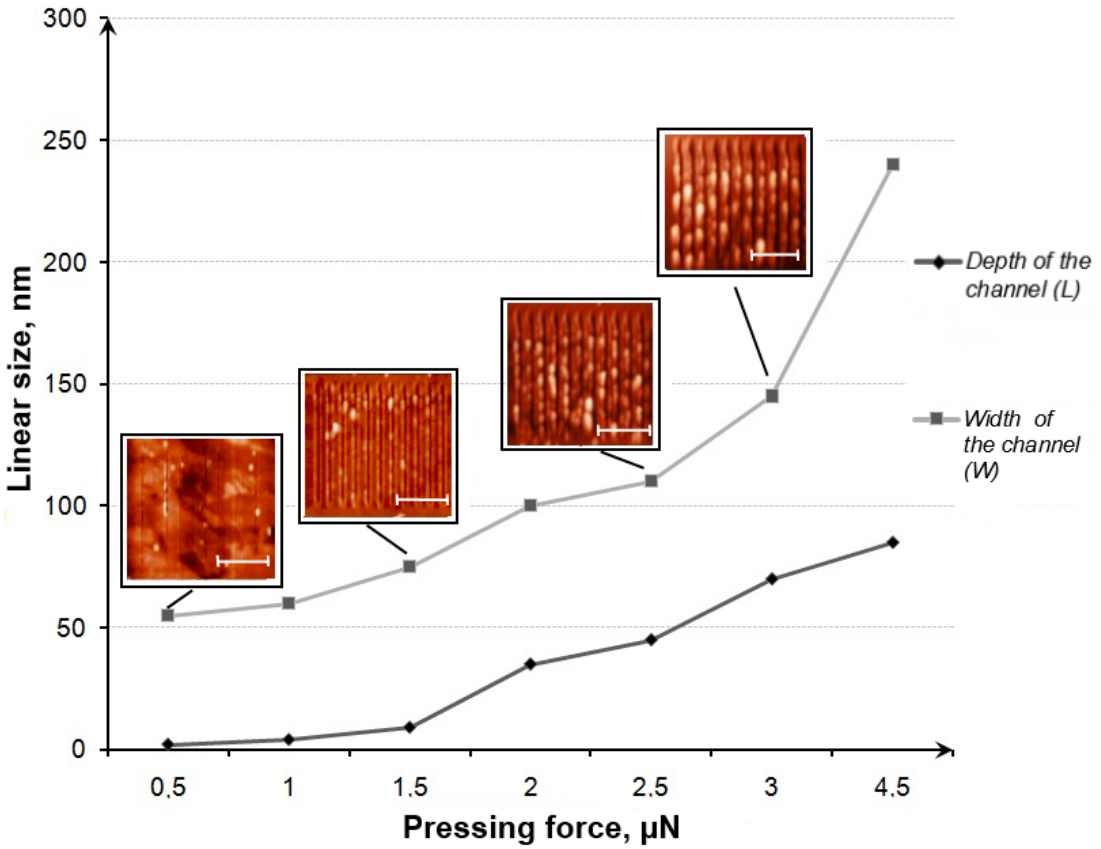


Figure 2. The graph of average features of diffraction gratings (*L, W*) with AFM insertions fabricated by a NS probe at different impact forces (the scale bar is about 1 μm).

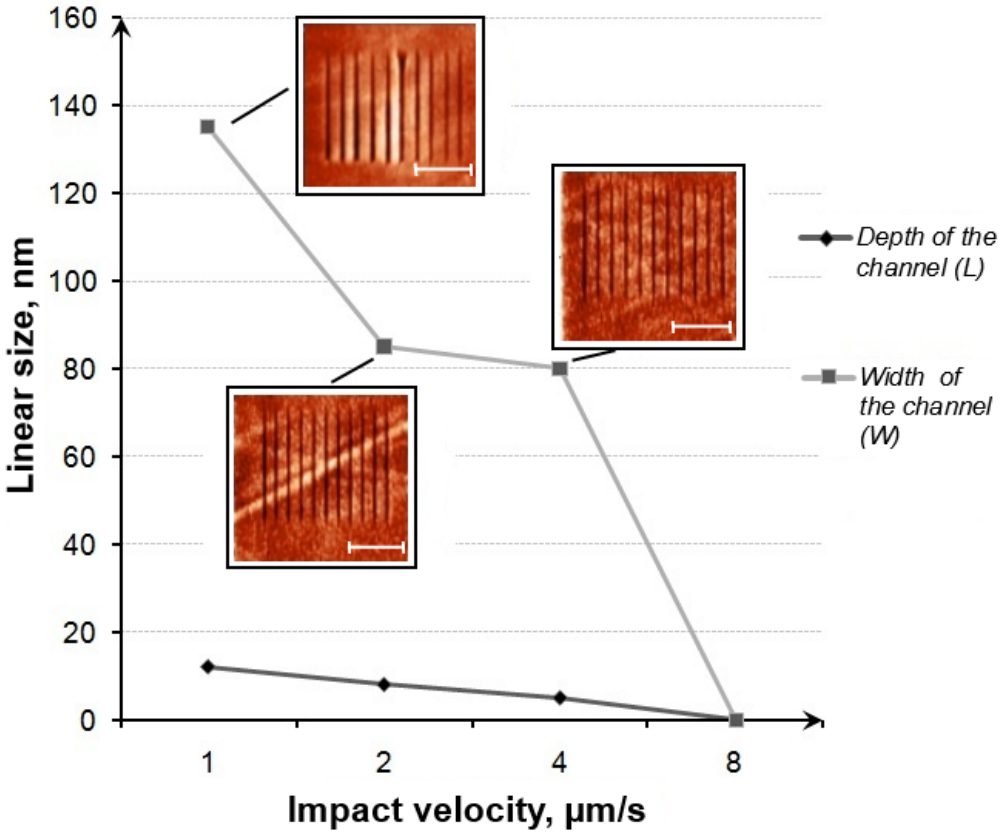


Figure 3. The graph of average features of diffraction gratings (*L, W*) with AFM insertions fabricated by a NS probe at different lithography speeds (the scale bar is about 1 μm).

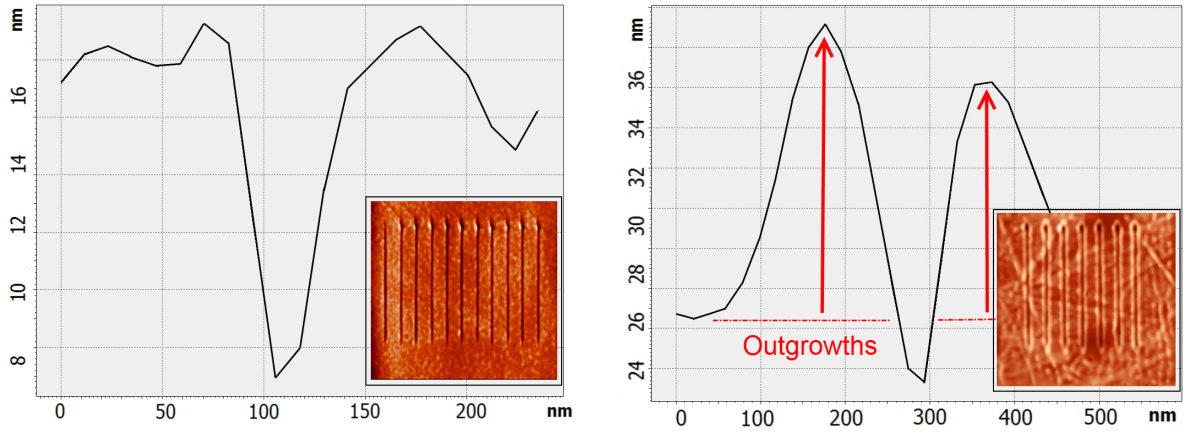
After finding the optimal parameters of lithography impact forces (1–1.5 μN), altering the lithography speed was carried out (figure 3). At a lithography speed of about 1 μm/s, a strong change of the gratings' widths along the lines was found. At a speed of 2 μm/s, the perfect ratio of the depth and width of gratings was found. While increasing the lithography speed up to 4 μm/s, the decreasing of penetration depth without reduction of the line width was revealed. The further increasing of the lithography speed up to 8 μm/s reveals no grating structure which can be explained by the effect of probe slipping over the surface due to a high speed of lithography.

After finding the optimal parameters of the atomic force lithography, the comparison of lithography with standard and NS probes was carried out (figure 4). The standard probes resolution limit was found in the area of 5 μm with periods of grating of about 500 nm, a line length of about 4 μm, a line depth of about 4 nm, and a line width of about 160-180 nm. The optimal area of NS probes was about 3 μm with a grating's period of about 250 nm, a line length of about 2 μm, a line depth of about 11–12 nm, and a line width of about 70-80 nm.

It was found that the depth of grating grooves obtained by the standard probe is 3 times smaller than the one obtained using the NS probe. Furthermore, a noticeable outgrowth of material (17 nm) around grooves was found using the standard probe, which can be associated with the geometry of the standard probe, expanding from the top to the bottom. In other words, the standard probes push the material outward from the groove with increasing the depth of the line while the NS probes have a constant cross-section and don't have this effect.

At the same time, the limit of resolution of the NS probes was in the range of 25–50 nm, which was identified by lithography of pair lines with a gradual reduction of the distance between them. It should be noted that fabricated gratings can potentially be used in tasks of plasmonics, in particular, for

determining the parameters of multipath particle plasmons at the interface of the diffraction grating. The possibility of using NS probes for manipulating particles was discussed previously [10].



(a) (b)

Figure 4. AFM images of the surface of the gold substrate and typical cross-sections of one line of diffraction gratings obtained by lithography with NS probes (a) and standard probes (b). A red mark shows the build-up material around grooves for the standard probe lithography.

It should be noted that the results of lithography are strongly affected by the stiffness and the parameters of the samples. Probes with a low coefficient of stiffness require a large deflection of the probe to achieve the lithography effect, while at a higher coefficient of stiffness, lower deflection values are required. Therefore, it is recommended to use probes with high stiffness for a strong impact and probes with low stiffness for removal of thin layered material.

4. Conclusions

The formation and approbation of the specialized nanoscalpel based probes were carried out. Improvement of lithography results by using the nanoscalpel probes in comparison with the standard probes was shown. The optimal parameters of lithography (a force impact, a speed) were revealed, and the diffraction gratings on gold substrates for plasmonics research with an accuracy of about 25–50 nm were produced.

The studies demonstrated that nanoscalpel probes can significantly improve the results of lithography process that is interesting for the current nanoelectronics and plasmonics research.

Acknowledgments

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References

[1] Ashiri I, Gartsman K, Cohen S R and Tenne R 2003 *AIP Conf. Proc.* **685** 545
[2] Levichev V V, Zhukov M V, Mukhin I S, Denisyuk A I and Golubok A O 2013 *Technical Physics* **58** 1043
[3] Beard J D and Gordeev S N 2011 *Nanotechnology* **22** 175303
[4] Custance O, Perez R and Morita S 2009 *Nature Nanotechnology* **4** 803
[5] Cortes R and Coello V 2009 *NANO: Brief Reports and Reviews* **4** 201
[6] Wu J J 2010 *Progress in Electromagnetics Research* **104** 113
[7] Bonch-Bruevich A M, Libenson M N, Makin V and Trubaev V V 1992 *Optical Engineering* **31** 716

[8] Landau L D and Lifshitz E M 1970 *Theory of Elasticity, A Course of Theoretical Physics* (UK: Pergamon Press)
[9] Zussman E, Chen X, Ding W, Calabri L, Dikin D A, Quintana J P and Ruoff R S 2005 *Carbon* **43** 2175
[10] Mukhin I S, Fadeev I V, Zhukov M V, Dubrovskii V G and Golubok A O 2015 *Ultramicroscopy* **148** 151