Fabrication of probe tips via the FIB method for nanodiagnoses of the surface of solids by atomic force microscopy

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Abstract. In this work, we carried out an investigation of commercial atomic force microscope (AFM) probes for contact and semi-contact modes, which were modified by focused ion beam (FIB). This method was used to modify the original tip shape of silicon AFM probes, by ion-etching and ion-enhance gas deposition. We show a better performance of the FIB-modified probes in contrast with the non-modified commercial probes. These results were obtained after using both probes in semi-contact mode in a calibration grating sample.

1. Introduction

Atomic force microscopy (AFM) is a powerful tool for studying topography and various physical and chemical properties of the surface of solids. The most important factor affecting the quality and accuracy of AFM images is the quality of fabrication and the geometric parameters of the tip of the probe sensor. Standard AFM probes have a limited range of geometric parameters, which often leads to undesirable image distortions [1]. Often, to obtain adequate results of measurements of the relief of nanoscale structures, it is necessary to manufacture probes with a special tip shape, for example, with an increased aspect ratio or a reduced radius of rounding. One of the methods that allows the formation of probe points in a wide range of geometric parameters is the use of a focused ion beam (FIB) for local ion-stimulated etching and deposition of materials from the gas phase [2]. The purpose of this work is to manufacture AFM probes by the FIB method and to study the influence of the geometric parameters of the probes on the accuracy of the study of the relief of nanoscale periodic structures by the AFM method.

2. Modelling

To identify the regularities of the influence of the ion beam parameters on the evolution of the surface relief during ion treatment, numerical modelling was performed using the SRIM software package, which is based on the methods of stochastic Monte Carlo modelling. By simulating the distribution of ion runs in a solid, it is possible to estimate the influence of the ion energy and the direction of their movement on the dynamics of the profile formation on the surface of the solid, and by calculating the sputtering coefficient, it is possible to judge the influence of these parameters on the etching rate or the rate of dissociation of process gases.
For the simulation, a multilayer structure was constructed: the first layer is C10H8 gas with a thickness of 10 nm, the next layer is absorbed C10H8 gas molecules with a thickness of 5 nm, and the last layer is a silicon substrate with a thickness of 200 nm. The choice of the material is determined by the method of the subsequent experiment, which is based on the decomposition of the C10H8 gas, which enters locally through the injector needle, under the action of the Ga+ FIB with an energy of up to 30 keV. Numerical simulations were performed to determine the dependences of the ion penetration depth and the sputtering coefficient (on the angle of incidence of the ion beam (Figure 2, a) and on the value of the Ga+ ion energy (Figure 

Figure 1. Simulation results of the ion-gas-substrate interaction: the dependence of the ion penetration depth and the sputtering coefficient on (a) the energy and (b) the angle of incidence of the ions.

The analysis of the obtained dependences showed that with an increase in the angle of incidence of the ion beam, the depth of ion penetration decreases, and at the same time the sputtering coefficient increases. Such a change in the parameters is interrelated: if the ion energy is equal in the case of a normal fall, it will mainly be spent on the gradual penetration of the ion deep into the substrate, and if the ion hits the surface at an angle to the normal, the energy of the primary ions will be transferred mainly to the near-surface atoms and the probability of sputtering will increase. The change in the accelerating voltage similarly affects both the penetration depth and the sputtering coefficient for the same reasons: as the energy of the incident ion increases, both the number of collisions (and, accordingly, the penetration depth) increases until the ion completely stops, and the probability of sputtering of the substrate atoms increases.

These results were used to determine the parameters of ion-stimulated etching and deposition. The depth of ion penetration into the structure will determine the thickness of the amorphized layer and the dose of embedded gallium ions. When planning and conducting experimental studies, it is necessary to take into account that etching and deposition by an ion beam incident at an angle to the surface can lead to distortion of the geometry of the formed structure; and a decrease in the accelerating voltage can significantly reduce the resolution and efficiency of decomposition of precursor gases. The results of the influence of the beam parameters on the sputtering coefficient allow us to estimate the dynamics of the ion-stimulated deposition or ion-beam etching process.

3. Experimental

Experimental studies were carried out using a scanning electron-ion microscope Nova NanoLab 600 (FEI, the Netherlands), equipped with a FIB system. The tip of the probes was formed on the basis of the beams of standard AFM HA_HR cantilevers (NT-MDT), whose own points were previously removed by ion-beam etching FIB. The new probe points were formed by ion-stimulated carbon deposition, when a volatile compound C10H8 was fed into the zone of impact of the ion beam. To study the influence of the probe shape on the accuracy of measuring the surface topology, cantilevers with two types of points were made: cylindrical with a high aspect ratio and conical stepped. The
technique was also applied, where ion-stimulated etching was used to modify the formed high-aspect probes.

Figure 2. SEM images of (a) high-aspect, (b) stepped, and (c) sharpened probes manufactured using FIB technology.

3.1. High aspect ratio probes
The formation of high-aspect ratio points was carried out by ion-stimulated carbon deposition at FIB current values from 30 to 1 pA, an accelerating voltage of 30 keV, and an ion beam exposure time at a point of 1 μs. The geometric parameters specified in the template were different for each probe. The deposition parameters for each specific tip are shown in Table 1.

| Designation of the cantilever on the beam of which the probe was formed | T1 | T2 | T3 | T4 | T5 |
|---|---|---|---|---|---|
| Ion beam current (nA) | 30 | 30 | 10 | 1 | 1 |
| Height (μm) | 5.62 | 5.91 | 4.92 | 4.58 | 5.15 |
| Radius of curvature (nm) | 521 | 330 | 230 | 171 | 125 |

3.2. Stair-step base probes
The first step probe (S1) was deposited as a structure of five disks with a height of 1.3; 1.2; 0.9; 0.7 and 0.6 microns and diameters of 3; 2.5; 2.0; 1.5 and 0.4 microns, respectively. The ion current during deposition was 30 nA for the first three disks and 1 pA for the last two. The second probe (S2) was deposited as a structure of four disks, the height of which was 1.4; 0.7; 0.7 and 0.7 microns, and the diameter of 2; 1.5 and 0.2 microns, respectively. The ion current during deposition was 300 pA for the first two disks, 100 pA for the third, and 1 pA for the last.

3.3. FIB-sharpened probes
The next probe (M1) was made according to the scheme shown in Figure 3. This method uses a combination of ion-stimulated deposition and etching techniques. At the first stage, a tungsten tip probe with a diameter of 800 nm, was deposited at a beam current of 0.10 nA. Next, ion-stimulated deposition of the formed tip was carried out using the raster templates shown in Figure 3. The final radius of rounding of the probe was 81 nm.

In order to assess the influence of the shape of the obtained probes on the accuracy of measurements using the T1-T5, S2 and M1 cantilevers, the surface of the TGZ3 (NT-MDT) calibration grating was studied in the semi-contact AFM mode. For a comparative evaluation of the results of the study, such a scan was also performed using a standard NSG10 cantilever.
4. Results and discussion

The T2-T5 cantilever probes were broken at the initial stage of scanning due to the base being too thin and consequently brittle. The profilograms of the TGZ3 lattice surface obtained for the NSG10, T1, T2, S2, and M1 cantilevers are shown in Figure 4.

Comparing the obtained profilograms, we can note an increase in the scanning resolution when using modified probes. To numerically evaluate the accuracy of the profilograms, the values of the
calibration grid parameters specified in the passport, namely, the width and height of the steps, were calculated. The data obtained with the corresponding errors are presented in Table 2. The increase in the accuracy of AFM studies using FIB-modified probes was estimated by the value of the deviation from the set values, which was determined by the ratio of the measured structure parameter, taking into account its average deviation and measurement error, and its passport value.

**Table 2. Parameters of the TGZ3 lattice determined by different cantilevers.**

|           | Width     | Height     |
|-----------|-----------|------------|
|           | Measurements (microns) | Deviation | Measurements (microns) | Deviation |
| Technical data | 1.10±0.01 | - | 500±2.5 | - |
| NSG10     | 1.66±0.68 | 113.4 %   | 492.3±3.05 | 2.66 % |
| T1        | 1.79±0.69 | 113.8 %   | 489.1±5.3  | 3.74 % |
| T2        | 1.62±0.44 | 88.6 %    | 495.6±4.0  | 2.18 % |
| S1        | 1.49±0.40 | 72.1 %    | 505.49±2.2 | 2.10 % |
| S2        | 1.36±0.27 | 49.1 %    | 521.9±3.1  | 5.50 % |
| M1        | 1.31±0.26 | 43.6 %    | 496.3±2.7  | 1.77 % |

For a more visual representation of the obtained data, graphs were constructed (Figure 5). These graphs show the parameter values determined by various cantilevers and show the error during scanning. Also, a blue bar was plotted on the graphs, the width of which corresponds to the passport data of the TGZ3 grid, taking into account the confidence interval presented in the passport.

**Figure 5.** Comparison of the obtained values (a) of the width and (b) of the height of the steps of the TGZ3 grid with the passport data, taking into account the deviation during scanning.

The only probe that did not allow for improved scanning quality, compared to the standard NSG10 cantilever, was the T1 cantilever probe. This result is due to the too large radius of the rounding. The remaining manufactured cantilevers showed good results in the study. The obtained parameter values are more accurate than for the NSG10 cantilever, and the scanning errors are also significantly reduced, especially in the lateral direction. For the M1 cantilever, the resolution was the highest, which allowed for a 2.4–fold increase in the accuracy of the width measurement and a 3.55-fold increase in the height measurement.

### 5. Conclusion

The manufactured probes showed less distortion in the image than the standard AFM probe, which allowed to increase the measurement accuracy several times. Tip probes with a baseless less than 330 nm
showed low strength during the scanning process. The stepped probes avoid this problem thanks to a stronger base. Sharpening the probe with a focused ion beam allows to get a smaller radius of rounding (compared to unmodified probes), and this operation is performed simultaneously in one stage. The probes made by this method showed the highest scanning accuracy.

The performed studies have shown that the use of the FIB method makes it possible to form the tips of the probes, the parameters of which differ significantly from the parameters of standard probes, thereby increasing the accuracy of the study of the surface relief. The results of this work can be useful in the study of micro-and nanostructures, and can also be used for further development of the technology for manufacturing and modifying probes by the FIB method.

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