High-precision investigation of nanorod and nanosphere topological structures for nanoelectronic issues by means of atomic-force microscopy

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Abstract. Fabrication and study of specialized single nanowhisker probes are performed for high-precision investigation of elements such as nanospheres and nanorods using the atomic force microscopy. It was found that single nanowhisker probe significantly increases the resolution and contrast of images obtained in the semi-contact mode. Furthermore, the roughness analysis and adhesion forces are investigated in contact mode to comprehensively characterize properties of nanospherical and nanorod electronic structures.

1. Introduction
The deviation in properties of nanoelectronic devices greatly depends on the parameter differences of their structure elements. Therefore, it is necessary to ensure high-precision control of topological features for such elements with significant accuracy. Atomic-force microscopy (AFM) is one of the most advanced methods used to perform high-precision study and modification of nanoelectronic structures \cite{1, 2}.

The principal element in AFM is a probe, or nanoscale needle tip, that interacts with the surfaces during the scanning process. The geometry and material of the probe can greatly affects on the spatial resolution and contrast of the obtained images. To improve the accuracy of AFM investigations, there is some of research works aimed at development new types of probes \cite{3, 4} at the end of standard tip.

Fabrication of nanowhisker structures has significant advantages, such as the possibility to control composition and orientation of growing structures. It is also should be noted that formation of nanowhisker structures goes directly on tip without any additional manipulation. Nevertheless, the most common method of the single or matrix nanowhisker creating is the molecular-beam epitaxy \cite{5}. This method doesn’t allow high-precision orientation of single whisker with controlled geometrical parameters of growth that is very important while scanning complex surface topologies \cite{6}. Such surfaces can be, for instance, structures of nanorods with significant difference in aspect ratio or small nano-objects with smooth walls as nanospheres \cite{7, 8}.

In this work, the new method to creating stable single nanowhisker (SNW) probes by electron beam deposition with precursor gases in vacuum chamber \cite{9} was offered at the top of standard probes with control of SNW geometrical parameters.
The aim of this article is to ensure high precision investigation of nanoelectronic elements with complex surface structure like nanorods and nanospheres by means of atomic force microscopy with specialized single nanowhisker probes.

2. Experimental setup

The SNW structures were grown at the tip of the standard probes under the electron beam in a high vacuum and direct injection system of precursor gases [6]. Fabrication of the SNW structures and control their geometrical parameters were performed by means of scanning electron microscope CrossBeam Neon 40 (Carl Zeiss, Germany) with integrated inlet gas C$_{9}$H$_{16}$Pt.

Light emitting diodes based on GaN nanorods and SiO$_{2}$ nanospheres on their surfaces [10] were selected as nanoelectronic samples with complex topological structure. The investigation of samples was performed by means of scanning probe microscope Ntegra Aura (NT-MDT, Russia) in the semi-contact and contact AFM modes.

3. Results and Discussions

Investigation of SNW probes in the air carried out in tapping mode, which increases the stability of the SNW probes and reducing the damaging effects of the probe to the sample. Figure 1-2 show the distribution of nanospheres and nanorods on the sample surface at the same locations, obtained using standard and SNW probes. It was found that SNW probes allow detect the boundary areas of nanospheres more accurately (diameter ~200-250 nm), whereas applying of standard probes revealed borders thickening of the nanospheres up to diameter ~500 nm (Figure 1). Therefore, the study of nanospheres demonstrated a two-fold decreasing of nanospheres broadening using SNW probes.

![Figure 1. AFM images of nanospheres distribution on the same area of 2x2 mkm, received with standard (a) and SNW probe (b).](image)

Figure 2 shows that the SNW probes area allow visualizing nanorod basis, i.e. penetrating to the basement of the substrate, whereas the standard probes visualize only the upper areas (the tips of nanorods). It was found that SNW probes penetrate to the substrate at the value about 230 nm, whereas the standard probes penetration was only about 150 nm at the same area.

This effect can be explained by the geometry of the SNW probe, which is a cylinder with a radius ~30-40 nm and tip sharpening ~7-12 nm, whereas standard probe is a pyramid, expanding from tip to the basement of cantilever (Figure 3). Thus, the penetration of SNW probes in narrow and deep areas is significantly better because of its high aspect ratio (i.e. the ratio of length to diameter of the whisker). This feature appears most noticeably in the samples having large longitudinal and small transverse dimensions, for instance, nanorods, or areas with sharp height differences and smooth walls, such as nanospheres.
Figure 2. AFM images of nanorods distribution on the same area of 2x2 mkm, received with standard (a) and SNW probe (b).

Figure 3. Schematic visualization of a narrow channel using a cone probe (standard Si probe) (a) and cylinder probe (SNW probe) (b).

Results show an overall improvement of SPM resolution and image contrast using SNW probes. This may be explain due to a better penetration ability of SNW probes (contrast height enhancement) and a clear imaging of sharp height difference of objects borders (spatial resolution), correspondingly.

Figure 4 depicts the visualization of the small area of nanorods with cross-sectional view on purpose to measure their spatial parameters. Thus, the single nanorod has a diameter of basement of about 250-300 nm with an altitude of about 100-150 nm. The radius of the tip curvature changes from 20 to 100 nm.
Figure 4. AFM image of the nanorod distribution (a) and a cross-sectional view of the one nanorod (b), obtained with SNW probe.

Figure 5. Histograms of the height distribution on the surface of nanorods on the area about 2x2 mkm, received with standard (a) and SNW probe (b).

Table. 1. Parameters of the nanorods obtained by different types of probes

| Parameter                      | Standard probe | SNW probe  |
|--------------------------------|----------------|------------|
| Height difference, nm          | 162,917        | 241,514    |
| Average height, nm             | 88,736         | 150,082    |
| Roughness, nm                  | 21,046         | 24,867     |
| Mean square deviation, nm      | 26,454         | 32,879     |

According to distribution histograms (Figure 5) we identify the parameters of the surface area 2x2 µm of nanorods obtained by standard and SNW probes (Table. 1). Based on this data it is possible to conclude that the roughness of the area has increased values with SNW probes, which means greater penetration capability and, therefore, the reliability of the data obtained using this type of probes.
Analysis of adhesion curves was obtained using standard probe in contact AFM mode (Figure 6). The values of the probe position increment $\delta z$ (signal Height) corresponding to cantilever deflection (signal DFL) was measured to find the adhesion of probe to the substrate (label A-B and A'-B'). The value of $\delta z$ calculated as a projection of points A-B (for nanospheres) and A'-B' (for nanorods) on the X axis. According to the formula (1) it is possible to determine adhesion force as [11]:

$$F = \delta z \cdot k,$$

where $\delta z$ - deflection of the probe [nm], $k$ - stiffness of the cantilever [N/m].

We use the standard hydrophobic Si contact probes (SiO2 coating on air) with cantilever stiffness of about 0.01 N/m measured by Sader [12].

![Figure 6. Typical adhesive curves obtained on the surface of nanorods (a) and nanospheres (b) using standard Si probe in contact mode.](image)

The average value of the probe position increment $\delta z$ was about 120±10 nm for nanorods and about 200±10 nm for nanospheres. This fact suggests a greater accumulation of condensate in the area of nanospheres than in the area of nanorods. That indicates advantage of nanorods as a material for photoelements construction in nanoelectronics, as condensation may interfere with light emission from the surface of the light emitting diodes (LED) and other structures.

The investigation shows that the application of specialized SNW probes has better results than standard silicon probes in the study of complex topological elements with thin structures used in nanoelectronic devises.

4. Conclusions
The method of deposition by a focused electron beam was performed to fabricate specialized probes with nanowhiskers and ensure the precise control of the geometry and composition of the grown structures. It was found that these type of probes may significantly increase the resolution and contrast of images obtained in the study of objects of nanoelectronics, such as nanospheres and nanorods.

Roughness analysis displays a greater degree of penetration ability of specialized whisker probes. In addition, the analysis of adhesion surfaces shows considerable adhesion force in the area of nanospheres than in the area of nanorods that indicates accumulation of condensate in this region.
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