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Investigation of the Nanodiagnostics Probe Modes for Semiconductor Resistivity Measurements by Atomic Force Microscopy

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Abstract. The work presents the results of theoretical and experimental investigations of the features and nanodiagnostics probe modes for semiconductors resistivity measurements by current technique of atomic force microscopy and by using test silicon samples with known resistivities (0.01 Ω·cm, 1 Ω·cm, 5 Ω·cm, 10 Ω·cm). It is shown that the measured resistivity data in air and in ultrahigh vacuum (10^{-8} Pa) is 166 Ω·cm and 10 Ω·cm, respectively, for the sample with ρ = 10 Ω·cm of theoretically predicted resistivity. We showed that reducing of the measurements reliability in air, due to the local anodic oxidation of the substrate surface. Experimental studies of the influence of cantilever load forces (0.3 to 6.0 μN) to the samples surface on the current distribution are presented. Based on the experimental results we developed a mathematical model for determining the resistivity of semiconductor materials by current technique of atomic force microscopy. The results are useful to the development of probe methods for nanoelectronic devices analysis by atomic force microscopy.

Introduction

The development of modern electronic components associated with the development of nanoelectronics process of nanoscale structures formation. For testing of such structures is necessary to carry out the investigations of geometrical, electrical and mechanical properties of the substrate materials [1 - 9].

Current technique of atomic force microscopy (C - AFM) is a multifunction method of nanodiagnostics which allows investigating the current distribution on the surface of the sample with simultaneous observation of its topology [6]. This technique is applicable for study of the electrical properties of graphene layers [1, 6], carbon nanotubes [2] and nanowires ZnO [3], and also to determine the electric parameters and the concentration and distribution of dopants in semiconductor structures [4, 5]. However, for research materials by C - AFM, there are several problems associated with: the lack of measurement techniques that can determine the electrical characteristics of the materials resulted from the AFM images; the choice of the optimal modes of interaction between the probe tip to the surface of the substrate; insufficient study of the influence of the environment on the current distribution measurements.

The aims of research, first one, to investigate the influence of the environment and cantilever load forces to the surface of the sample to determine the accuracy of the semiconductor materials resistivity on the example of silicon test samples by C - AFM.

Experimental procedure

Investigations were carried out by using C - AFM on the test silicon wafers with a resistivity of 0.01 Ω·cm, 1 Ω·cm, 5 Ω·cm, 10 Ω·cm (see Table 1), which allowed the comparison of the experimental results with the known values.

The study of environmental influences on the measurement of the resistivity was carried out by C - AFM scanning of the silicon surface BDS-10 in air (probe nanolaboratory Ntegra, NT-MDT, Russia), and in ultra-high vacuum (10^{-8} Pa) (UHV module SPM nanotechnology complex NANOFAB, NT-MDT, Russia). The samples were degreased in a H_2SO_4 (80-90%) and H_2O_2 (10%) solution and pre-treated in a HF (10%) solution. Sequential scanning areas of 5 × 5 μm^2 and a 7 × 7 μm^2 were performed by using cantilevers NSG11, coated with a conductive material W_2C, the tip radius is 35 nm. In the experiments, a DC voltage of 3 V was applied to cantilever with load force of 2.5 μN to sample surface. The Figure 1 shows the resulting AFM images of current distribution.

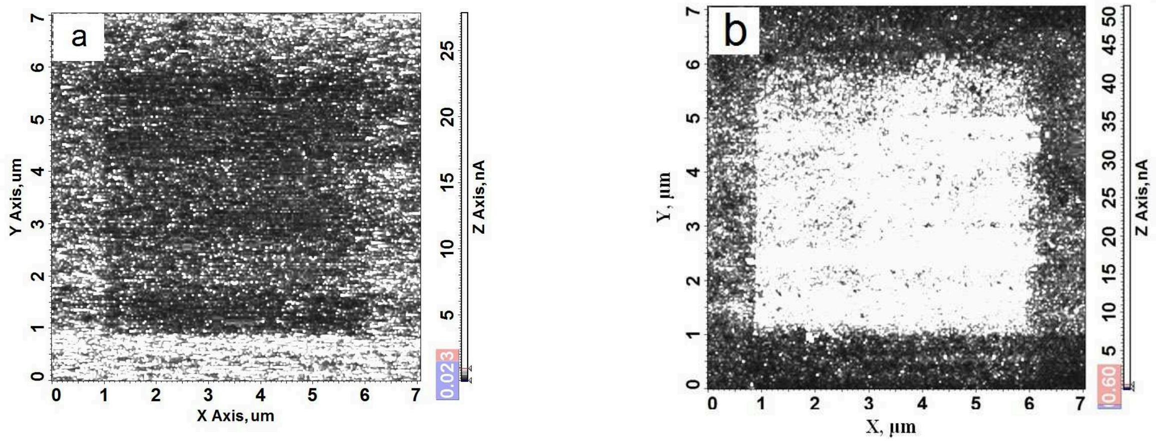

Fig. 1. AFM images of the current distribution on the surface of the silicon substrate, BDS-10 mark, obtained: a) - in air, b) - in an ultrahigh vacuum (10^{-8} Pa)

The influence of the cantilever load forces on silicon substrates surface was carried out in ultra-high vacuum (10^{-8} Pa) by C - AFM. The scans were performed by cantilevers NSG11. A DC voltage of 3 V was applied to cantilever with varied load force in the range of (0.3 ÷ 6.0 μN) to the sample surface. For each value of the load force in C - AFM, current-voltage (I-V) characteristics were obtained.

We present a model to determine the resistivity of semiconductor materials based on the experimental data obtained from C - AFM. The mathematical model described here takes into consideration the several sources of resistance in on ultrahigh vacuum environment. In the system described, the resistance is given by [4, 5].

$$R_{tot} = R_{barrier} + R_s + R_p + R_b, \tag{1}$$

where $R_{barrier}$ – contact resistance between the tip and the surface of the sample; R_s – sample resistance; R_p – resistance of the material coated onto the cantilever; R_b – resistance of solid metal in contact with the surface of the substrate [4, 5].

The resistance of the cantilever R_p and sample R_s are given by [5]:

$$R_p = \rho_p \cdot \frac{L_p}{A_p}, \tag{2}$$

and

$$R_s = \rho_s \cdot \frac{L_s}{A_s}, \tag{3}$$

where ρ_p and ρ_s represent the resistivity of the coating materials of the probe and the sample, respectively; L_p - probe length; L_s-substrate thickness; A_s-area of tip-sample contact.

The resistance of the nano-contact tip with the surface of the substrate is defined by Sharvin's equation [4, 5]:

$$R_{barrier} = \frac{4}{3} \cdot \frac{\rho \cdot \lambda}{\pi a^2}, \quad \rho = \frac{\rho_p \lambda_p + \rho_s \lambda_s}{2}. \tag{4}$$

where λ represents the mean free path of the charge carriers in the material covering the probe and the substrate; ρ – is the resistivity between the tip and the surface of the substrate; a – is the radius of the tip of the probe in contact to the surface of the sample. It depends on the cantilever load forces, according to the Hertz model [10]:

$$a = \sqrt[3]{\frac{FR}{E^*}}, \tag{5}$$

where F – represents cantilever load forces to the substrate surface; R – is the radius of the probe tip; E^* – is the reduced Young's modulus of the probe-substrate materials [10]:

$$\frac{1}{E^*} = \frac{3}{4} \cdot \left(\frac{1-v_p^2}{E_p} + \frac{1-v_s^2}{E_s} \right). \tag{6}$$

where v_p, v_s - Poisson's ratios of the materials of the cantilever tip and the sample; E_p, E_s - Young's modulus of the cantilever tip and the sample, respectively.

From equations (1 - 6) we obtained an equation which allows determining the resistivity of the material of the sample under ultrahigh vacuum conditions:

$$\rho_s = \left[\frac{U}{I} \cdot CF - R_b - \rho_p \left(\frac{2\lambda_p}{3\pi a^2} + \frac{L_p}{A_p} \right) \right] \Big/ \left(\frac{2\lambda_s}{3\pi a^2} + \frac{L_s}{A_s} \right). \tag{7}$$

where term CF represents a correction factor for the various effects occurring in the region of contact between the tip and the sample, such as: generation-recombination currents on the surface states, the presence of near-surface region with a high level of mechanical stress, leading to a shift in the energy levels of the bands of permitted energy; the presence of the semiconductor plastically deformed region with metallic conduction current near the surface, etc. [5] each of which have an impact on the current flow and are not included in the proposed model.

By using the equations (1 - 7), the parameters of test samples (see Table 1) were calculated. Dependences of the current distribution and the silicon resistivity for different cantilever loads (0.3 ÷ 6) µN shown in Figure 2.

Results and discussion

The experimental results on the effect the environment on the accuracy of resistivity measurements showed that when DC voltage is applied to the tip-substrate system in the air, there is a local anodic oxidation of the substrate surface with adsorbed water film [11]. Figure 1 a, shows that the current distribution on the surface BDS -10 in the central region of AFM image is in average 0.1 nA, and in the peripheral region is in average 0.6 nA. In the results, the contrast in the current distribution corresponds to the process of local anodic oxidation of the central area of 5 × 5 µm^2, during the initial scanning by C - AFM in the atmosphere. Scanning the sample surface BDS -10 in ultrahigh vacuum conditions (10^{-8} Pa) showed that in the central area of the AFM image the current distribution is in average 2 nA, and in the peripheral region is in average 0.3 nA. The resulting contrast of the current distribution corresponds to the spreading process of removing the natural oxide layer by scanning the central area of 5 × 5 µm^2 in AFM contact mode.

By applying the experimental data on the model described (1-7), the values of the resistivity of the test sample BDS -10 is: 166 Ω·cm when scanned in scanning in air and 10 Ω·cm when scanned in ultrahigh vacuum. The results show that, in order to obtain reliable data on the electrical parameters of semiconductor materials by C - AFM one should consider the importance of performing the measurements in ultra-high vacuum conditions. In our work, we achieved such conditions in the SPM nanotechnology complex NANOFAB module.

The analysis of results on the effect of cantilever load force on the surface of the sample ADS-0.01 shows that with increasing contact load force, the current distribution increases to almost constant value over the entire surface with load force at 2 µN, which is associated with the formation of a stable tip-sample contact. The same behaviour was observed while we studied the remaining test samples shown on table 1. The results allow establishing of three distinct modes of scanning, according to the cantilever load force: at low loads (for ADS-0.01 less than 1 µN) the probe weakly interacts with the sample and research in these modes of C - AFM is not effective; at medium loads (for ADS-0.01 more than 1 µN and less than 2 µN), the probe interacts with the sample surface. In this mode of C - AFM the research can allow to identify resistance artefacts with distinguishable substrate; at high loads (ADS-0.01 more than 2 µN), the probe interacts well with surface, forming a stable contact with the sample. Furthermore, artefacts as well as natural oxide layers were removed from surface.

These modes can be useful to probe the electrical parameters of the structures at a nanoscale. The analysis of the equations (1-7) shows that the current increase with increasing cantilever load force is primarily due to the influence of load on the contact resistance and also to increase the contact area.

The figure 2 presents the results of experimental and theoretical (equation (1-7)) studies. The analysis shows a good correlation of experimental results and theoretical data, especially at high cantilever load force, which confirms the adequacy of the followed model. From the data presented on Fig. 2 b, the dependency implies the existence of a threshold for cantilever load force, above which the resistivity of the substrate material is constant, thus producing a reliable result. For the samples used in this work, the threshold cantilever load force can be considered as 2 µN (Table 1).

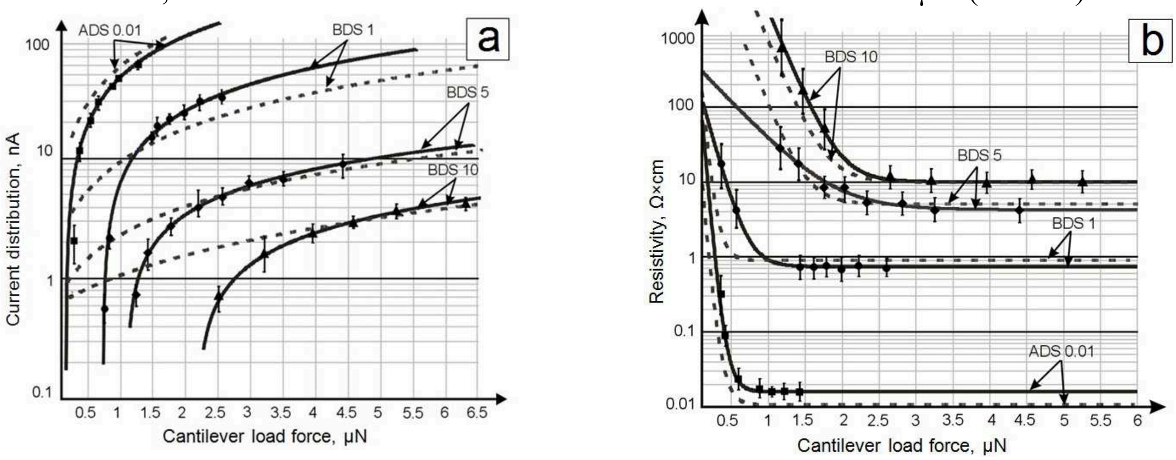

Fig. 2. The dependence of the electrical parameters of the silicon test samples from cantilever load forces between the probe tip to the sample surface: a) - the dependence of the current distribution; b) - the dependence of the resistivity. The dotted line - the theoretical dependence (1-7), the solid line –experimental dependence

Table 1. Silicon substrates resistivity determined by the developed method

Marking the sample	Theoretical resistivity values (Ω×cm)	Conductivity type	Substrate thickness (µm)	Cantilever load (µH)	The measured current (nA)	Experimental Determined Resistivity (Ω×cm)
ADS-0.01	0.01	n	460	2	49.8 ± 1.08	0.04 ± 0.01
BDS-1	1	p			24.89 ± 0.43	0.70 ± 0.10
BDS -5	5	p			8.11 ± 0.24	3.73 ± 0.40
BDS -10	10	p			2.00 ± 0.38	11.24 ±1.20

ADS – antimony-doped silicon, BDS – boron-doped silicon

Conclusion

The paper presents the results on experimental and theoretical investigation of the defining characteristics of the electrical parameters of silicon substrates. We applied a method denominated by current technique of atomic force microscopy. The influence of the environment on the values of the current distribution and resistivity were presented and discussed. It is shown that when the silicon samples are measured in air by C - AFM on the surface of sample is adsorbed a film of H_2O and when a DC voltage is applied to cantilever then between cantilever tip and surface the electrochemical reaction with silicon oxide is appear. In results the resistivity of the silicon samples are increasing. In order to accurately determine the resistivity of silicon test wafers one should measure the current distribution in ultrahigh vacuum. Thus, to obtain of reliable results of investigation of electrical parameters by C - AFM of semiconductor materials is necessary to carry out measurements in ultrahigh vacuum.

Also, in the paper we presented the results on the theoretical and experimental investigations of the influence of the cantilever load force on the surface of silicon test wafers, based on measuring the current distribution in the tip-substrate system. It is shown that, in order to obtain a reproducible measurement of current distribution and to determine the resistivity, it is necessary to form a stable tip-substrate contact. For the case of a silicon test sample, the optimal interaction is achieved with cantilever load force to the surface at 2 μN or more.

We have developed a mathematical model for determining the resistivity of semiconductor materials by C - AFM. The model allows determining the resistivity and the dependence of the resistivity on the cantilever load force to the sample surface. When the cantilever load force to the surface is (2 - 3) μN the resistivity values correlate well with the data here presented, regarding all test substrates.

The results are applicable in the development of probing methods and investigation of the structures of nano- and micro-electronics, nano- and microsystems engineering being investigated under the scope of atomic force microscopy.

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