Kelvin probe force microscopy with high aspect ratio Pt/C nanowhisker probes

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Abstract. Specialized conductive high aspect ratio Pt/C single nanowhisker probes for Kelvin Probe Force Microscopy were fabricated and tested. Improvement the accuracy of contact potential difference and topography measurement was found using nanowhisker probes compared to standard conductive probes. The contact potential difference of Pt, Ti, TiN and ITO materials with respect to Au was studied.

1. Introduction

The application of probes with single carbon nanowhisker (NW) structures allows to improve the quality of atomic force microscopy (AFM) images in the study of complex objects due to their high aspect ratio (length-to-diameter ratio) and small size of the apex [1, 2].

At the same time, the application of NW probes in such methods of scanning probe microscopy as electrostatic force microscopy (EFM), magnetic force microscopy (MFM), and Kelvin probe force microscopy (KPFM) is difficult due to the low conductive properties of amorphous carbon NW. The solution to this problem can be either the deposition of conductive or magnetic materials on a NW [3] or the fabrication of NW from conductive materials [4].

The problem of making nanocontacts with certain properties often arises in the manufacture of a semiconductor devices. In particular, it is important to know the work function of the material when manufacturing ohmic contacts or Schottky contacts for semiconductors having a low density of surface states, such as III-N and II-VI groups of materials [5]. The work function is related to the contact potential difference (CPD) arising when two dissimilar metals come into contact.

Kelvin probe force microscopy (KPFM), also known as surface potential microscopy, is a method for measuring the local surface potential difference between conductive probe and the surface of the sample. Thus, a map of local potential distribution on the sample is obtained.

Thus, the aim of this work was to fabricate conductive high-aspect probes with Pt/C nanowhiskers for Kelvin probe force microscopy and their comparison with standard conductive probes on the nanocontacts of various materials.
2. Experimental setup

The single NWs were fabricated at the tops of standard Si probes with Pt/C composition (~30-40% of platinum), having hydrophilic [6] and conductive properties (figure 1, a). The standard conductive probes (W2C, NT-MDT) and standard probes (NSG01, NT-MDT) with conductive Pt/C whiskers were used. The length of the NW was about ~750±50 nm and diameter was about ~50±10 nm. The electron microscope CrossBeam Neon 40 (Carl Zeiss, Germany) was used with a gas-precursor system C9H16Pt to fabricate single NW [4].

The investigation of nanocontacts was carried out by means of scanning probe microscope Ntegra Aura (NT-MDT, Russia). For the deposition of thin films and contacts the Auto 500 thermal spraying unit (BocEdwards, UK) was used. Gold was selected as the reference material; Pt, Ti, TiN and ITO were selected as the materials to be studied.

3. Results and Discussion

Before measuring the contacts, a thin layer of gold with a sublayer of chromium was deposited using a thermal spraying device on polished silicon substrates with a low roughness. Photolithography of microstructures (array of crosses) was carried out on the substrates (figure 1, b). After the deposition of materials (Pt, Ti, TiN or ITO) the photoresist with the excess deposited material was removed by the method of explosive lithography.

The Pt was chosen as a second support material, and the resulting Au/Pt microstructure was needed to verify the method. The materials Ti, TiN and ITO are of interest in the formation of contacts, in particular, to the GaN material. It is known that Ti and TiN are successfully used in the composition of ohmic contacts to this material [7].

An improvement of topographic lateral resolution of images using the NW probes at the cross-section of Au-Pt edge (~ 110±20 nm) compared to standard probes (~ 800±150 nm) was found when measuring (Si-Au-Pt) structures (figure 2, 3). This can be related to the geometry of the probes (the high aspect ratio of NW probe). At the same time, petite decreasing of maximum CPD value (NW probe ~ 80 mV, standard probe ~ 100 mV) as an increment of Au-Pt contrast was found.

The CPD contrast enhancement (~ 100 mV) and lateral resolution improvement (~ 250±50 nm) at Au-ITO edge was found using NW probes compared to standard probes (CPD contrast ~ 60 mV and lateral resolution ~ 700±100 nm) when measuring (Si-Au-ITO) structures (figure 4, 5). It should be noted that in both cases the quality was worse than in the case of measuring the (Si-Au-Pt) structure.

![Figure 1. SEM image of NW formed at the standard probe apex (a), and optical image of formed structures (array of crosses) and cantilever of the probe (b).](image-url)
Improving the quality of images can be related to the geometry (high aspect ratio and tip shape) and composition of the organometallic NW probes. Developed conductive NW probes can potentially be used to material diagnostics in such areas as nanoelectronics, materials science, engineering, etc.

**Figure 2.** AFM (a) and KPFM (b) image of (Si-Au-Pt) structure; and the typical KPFM cross-section of Au-Pt edge, obtained by the standard probe (c).

**Figure 3.** AFM (a) and KPFM (b) image of (Si-Au-Pt) structure; and the typical KPFM cross-section of Au-Pt edge, obtained by the NW probe (c).

**Figure 4.** AFM (a) and KPFM (b) image of (Si-Au-ITO) structure; and the typical KPFM cross-section of Au-ITO edge, obtained by the standard probe (c).
Figure 5. AFM (a) and KPFM (b) image of (Si-Au-ITO) structure; and the typical KPFM cross-section of Au-ITO edge, obtained by the NW probe (c).

Figure 6. The profiles of CPD with respect to Au layer for different contact materials obtained by NW probe. At the insertion the KPFM image with a cross-sectional line is presented.

Table 1. The average values of CPD with respect to Au for different contact materials.

| Sample coating | Potential difference with respect to Au |
|----------------|----------------------------------------|
| ITO            | +90 mV                                 |
| ITO cured      | +60 mV                                 |
| Pt             | -10 mV                                 |
| Ti             | +10 mV                                 |
| TiN            | -60 mV                                 |

Table 1 and figure 6 shows the potential difference relatively to Au. The obtained results show the existence of the expected regularity of the difference in the work function (the sign of the value of the work function) for Pt, Ti, and ITO materials. Nevertheless, the value of this difference was significantly lower than expected, which in the literature is associated with the presence of contamination on the surface or presence of a thin layer of water on the surface (both reduce the
difference in the work function) [8]. For TiN the result was unexpected, because according to the literature the work function of TiN is about 3-4 eV, which is much larger than the work function of gold (5.1 eV) and should give a positive difference in scanning.

Thus, the contact potential difference of Pt, Ti, TiN and ITO materials with respect to Au was studied. For Pt, Ti and ITO materials the difference in work function is in accordance with expectations. For TiN the work function is likely to be greater than the work function of the reference material (Au), which can be explained by incorrect preparation of the structures or a particularities in the measurement of this material.

4. Conclusions
Thus, the high-aspect ratio conductive probes with Pt/C nanowhiskers were fabricated and tested by Kelvin Probe Force Microscopy. It is shown that nanowhisker probes improve the accuracy of measurement contact potential difference and topography of nanocontacts of different materials compared to standard conductive probes, what can find an application in nanoelectronics. In particular, the contact potential difference of Pt, Ti, TiN and ITO materials with respect to Au was studied.

Acknowledgments
The work was carried out with the support of the Federal Agency for Scientific Organizations (AAAA-A16-11604110123-5), Governm of Russian Federation (grant 08-08).

References
[1] Beard J D and Gordeev S N 2011 Nanotechnology 22(17) 175303 (8 pp)
[2] Beard J D, Burbidge D J, Moskalenko A V, Dudko O, Yarova P L, Smirnov S V, Gordeev S N 2009 Nanotechnology 20 1–10
[3] Zhukov M V, Belousov K I, Mozharov A M, Mukhin I S and Golubok A O 2015 J. Phys.: Conf. Ser. 643 012095 (5pp)
[4] Zhukov M V, Kukhtevich I V, Levichev V V, Mukhin I S and Golubok A O 2014 J. Phys.: Conf. Ser. 541 012042
[5] Blank T V, Goldberg Yu A 2007 Semiconductors 41(11) 1263-1292
[6] Zhukov M V, Mukhin I S, Levichev V V and Golubok A O 2015 Technical Physics Letters 41 149–152
[7] Dimitriadis C A, Karakostas Th, Logothetidis S, Kamarinos G, Brini J, Nouet G 1999 Solid-State Electronics 43(10) 1969–1972
[8] Heras J M, Viscido L 1980 Applications of Surface Science 4(2), 238-241