The measurement of electrical properties of nanostructures with use of conductive diamond tip.

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Abstract. The use of doped full-diamond tips in SPM to measure electrical properties with nanoscale resolution is described. The method enables to measure electrical impedance, capacity and conductivity of semiconductors. The experimental results shows a wide range of benefits in investigation and creation of elements for micro and nano-electronics. According to multiple experiments, the characteristics of diamond tips are very stable during scanning, indenting and electrical measurements.

1. Introduction.
Development of elements in micro and nanoelectronics requires the improvements in methods of controlling the created structures. Nowadays the methods of investigation of electrical and mechanical properties and modification of surface using Scanning Probe Microscopes are well known. However, the traditional silicone probes do not provide enough reliability and durability, therefore, the abilities to use them during measurements of mechanical and electrical characteristics of hard and superhard conductive and semiconductive materials are restricted.

The main object of the current work was to investigate the properties of probes produced from bulk doped single crystal diamond and application them in SPM.

2. Methodology.
A modification of Scanning Probe Microscope (SPM) with the conductive diamond tips has been developed on the base of SPM – nanohardness tester “NanoScan” [1]. The measurements of electrical properties on the sample surface with the nanoscale resolution are implemented in scanning and indentation modes. The mode of electrical measurements allows the recording of DC current as well as impedance in radio-frequency bandwidth at contact point of tip and surface.

The main feature of “NanoScan” is the piezoceramic cantilever working in resonance mode. The cantilever possesses a very high bending stiffness (k ~ 10^5 N/m) that is about three orders higher than conventional SPM cantilevers. These allow to avoid the influence of electrical and ponderomotive forces on the oscillation mode and to combine the measurements of surface profile, elastic and electrical properties in single-pass mode. The use of diamond tips with stiff cantilever allows measuring the properties of superhard materials and coatings. The conductive tips are manufactured in TISNCM from the bulk boron-doped single crystal diamonds grown by the temperature gradient method using the high pressure - high temperature (HPHT) technique. The boron concentration is about 10^{16} - 10^{20} sm⁻³.
During the operation AC and DC voltage is applied between sample and scanning tip. Then, in scanning mode, the DC current and the radio-frequency impedance are recorded in every point simultaneously with surface topography and elasticity map. The lateral resolution was estimated about 10 nm. (Figure 1)

![Diagram](image)

Figure 1.

The scheme of the boron-doped diamond tip indenting the sample and the equal scheme of contact “tip-sample” in DC mode. $R_t$ – tip resistance, $R_c$ – contact area resistance, $R_s$ – sample resistance, $R(U)$ – nonlinear resistance in contact area.

The information obtained with “NanoScan” allows the estimation and the analysis of:
- Surface topography
- Local surface nanohardness value
- Local surface elastic modulus value
- Surface elasticity map
- Surface conductivity map
- Surface capacity map
- Current-voltage characteristic
- Defect observing and current leaks investigation
- Correlation of tip-sample current and contact area or indentation load.

Furthermore, in addition to scanning mode, the local surface modification is possible simultaneously with recording the electrical characteristics, e.g. current-voltage and farad-voltage characteristics during indentation with applied loads up to 20g. Another mode available is the indentation until reaching the given value of the current. We have found the only mention of such experiments in literature [3]. But the silicone tips and low loads are used. The evaporation of thin gold film was carried out with short electrical impulses of voltage up to 150 volts. No damages of tip geometry were found afterwards.

The use of conductive diamond tips with apex curvature about 30 nm opens the wide range of benefits in investigation and creation of elements for nanoelectronics. According to multiple experiments, the characteristics of diamond tip are very stable during scanning, indenting and electrical measurements with working voltage in range of hundreds volts and currents of milliamperes. The versatile investigations allow to claim that doped diamond obtains enough conductivity to create high-energy electrical impulse (with power up to hundreds milliwatts) on the nanoscale and provide well heat spreading from working area due to high thermal conductivity of diamond.
3. Experiment.

3.1. Scanning mode.

The classical application for conductive tips is the measurement of electrical properties during the scanning along sample surface [4]. Figure 2 illustrates measured surface topography, elasticity and conductivity maps for surface of composite MgB$_2$+MgO. Figure 2-(c) represents the alteration of the “tip-sample” current in frame of investigated area: white color corresponds to maximum of current, black – to minimum. Light regions corresponding to conductive inclusions of MgB$_2$ are clearly shown.

![Figure 2](image)

Surface topography, elasticity and conductivity maps for surface of composite MgB$_2$+MgO.

Image size 7 × 7 × 0.4 μm, current range 5 nA.

3.2. Current-voltage characteristics

During the indentation and holding the tip deep in sample surface the current-voltage characteristics were measured for contact points. Figure 3 presents current-voltage characteristics in contact of presented samples and tip from “Blue” boron-doped diamond with concentration $5.5 \times 10^{16}$ sm$^{-3}$. The curves were measured in rigid contact with surface of investigated material with loads from 0.1 to 2 mN. The contact area radius changes from 20 to 200 nm, in dependence from the load applied to the tip.

![Figure 3](image)

a - current-voltage characteristic on gold. Local resistance of contact area 250 kOm.
b - curve 1 - characteristic of contact of tip and SiO$_2$, diode conductivity.
- curve 2 - characteristic of two industrial diodes, connected in counter-parallel.

c - characteristics in different areas of silicone chip. Different types of conductivity:

  curve 1 - quasi-metal conductivity on the highly doped area.
  curve 2 - transition condition,
  curve 3,4 - diode conductivity.

3.3. Approaching curves mode.

By moving the oscillating tip closer to the surface the approaching curves for “current-amplitude-frequency” were measured for range of materials. Figure 4 shows the approaching curves for gold and silicon. The increase of frequency and drop of amplitude correlate to the mechanical processes in the contact area [5]. The contact of tip with surface is supplied by piezo–cantilever, which controls the approaching force and distance to the surface during the measurement of curve due to its own bending. Due to the presence of oxide film on the Si surface, the current increases after penetration of the tip through it. (Fig. 4(b)-C)

![Approaching curves for gold and silicon](image)

Figure 4.

Approaching curves for gold 4(a) and silicon 4(b) doped P – 1.5 x 10^16 with native oxide film.

A – frequency shift; B - fading of amplitude; C – current in tip-sample contact.

1- The moment of contacting of the probe and surface, for gold – beginning of current increasing.

2- The moment of oxide puncture and beginning of current increasing for oxidated silicone.

4. Conclusion

The implementation of electrical measurements on SPM-nanohardness tester NanoScan combining high stiffness of cantilever with ability to measure surface topography, elasticity, conductivity and impedance maps of surface layer with nanoscale resolution opens great potential for measurement and creation of elements for nanoelectronics. The utilization of durable and chemically stable tips produced from doped conductive diamond is very promising. According to multiple experiments, the characteristics of diamond tips are very stable during scanning, indenting and electrical measurements with working voltage in range of volts and currents of microamperes.
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