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Characterization of resistivity of Sb_2S_3 semiconductor nanowires by conductive AFM and in-situ methods

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Abstract. Conductive AFM and *in situ* methods were used to determine contact resistance and resistivity of individual Sb_2S_3 nanowires. Nanowires were deposited on oxidized Si surface for *in situ* measurements and on Si surface with macroelectrodes for conductive AFM (C-AFM) measurements. Contact resistance was determined by measurement of I(V) characteristics at different distances from the nanowire contact with the macroelectrode and resistivity of nanowires was determined. Sb_2S_3 is a soft material with low adhesion force to the surface and therefore special precautions were taken during measurements.

Introduction

Nanowires are perspective materials for application in solar energy [1], nanoelectronics [2,3], in light detection [4], thermoelectrics [5], etc. Investigation of properties of Sb_2S_3 nanowires for energetic and thermoelectric started recently [5]. One of complications in application of nanowires is the large spread of electrical parameters due to impurities, surface structure and size effects, which are difficult to control in production process. For example, Ge nanowires fabricated in one synthesis is exhibiting variations in resistivity in the range 0.001-0.1 Ω.m [6]. Another problem for application is creation of reliable contacts to the interfacing electrodes with well defined properties [7]. Contact resistances depend on contact material, fabrication method and treatment after contact fabrication. To characterize electrical parameters of individual nanowires usually two or more electrodes are used. In a two contact measurements the contact resistance may dominate over resistance of the nanowire. Precise resistivity of nanowires can be determined by 4 contact method however, this method is time consuming and requires electron beam lithography. Simple methods for control of electrical characterization are required for efficient evaluation of large amount of samples. Atomic force microscope with conductive probes (C-AFM) has been shown as a powerful tool for electrical characterization of individual nanowires. C-AFM was used to characterize electrical properties of individual nanowires incorporated inside anodized aluminium oxide membranes and mesoporous silica [8-11]. By this method not only electrical characteristics of individual nanowires, but also information about nanowires extended through the membrane can be obtained. To determine contact resistance in nanowire arrays measurements of resistance along longitudinal axes of nanowire was developed [10]. *In situ* methods also have shown capabilities for characterization of electrical properties of individual nanowires and nanotubes [12,13].

In this work contact mode C-AFM and in situ methods were applied to determine contact resistance and resistivity of soft and weekly adhered material to the surface of individual Sb_2S_3 nanowires.

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Experimental

Nanowires were grown as following. Sb_2Cl_3 (2 mmol) and 3 mmol thiourea were dissolved in 25 mL of ethylene glycol in a three-necked RB flask at room temperature. The mixture was then heated to 195ºC and held for 1 h under stirring in nitrogen atmosphere. After the sample was cooled to room temperature naturally, excess ethanol was added to the reaction vessel, and then the precipitate of Sb_2S_3 was retrieved by centrifugation of the mixture with ethanol several times. The obtained black precipitate was dried at 60ºC overnight in an air oven.

For conductivity measurements nanowires were deposited on oxidized Si surface for *in situ* measurements and on Si surface with macroelectrodes with height of 100 nm for (C-AFM) measurements. Macroelectrodes with height of 100 nm were fabricated on the surface by photolithography. Nanowires were suspended in hexane and spincoated over the surface at 200 RPM. The concentration of nanowires in the solution was optimized to provide optimal density of nanowires on the surface so that individual species are likely to be found in contact with the electrode. Adapted SmarAct GmbH 13D-manipulation probing system inside a Field Emission Scanning Electron Microscope (SEM) Hitachi S-4800 was used to observe directly and measure conductivity of individual nanowires. Etched gold tips were used to contact individual nanowires. An AFM (MFP-3DTM, Asylum Research) was used for topography analysis, mechanical manipulation and conductivity data acquisition.

Results and discussion

SEM image of free standing Sb_2S_3 nanowires after preparation are shown in Fig 1a. Individual nanowires were deposited on the surface and characterized *in situ* inside electron microscope Fig. 1b). I(V) measurement (Fig. 1c) shows nonlinear characteristics, which can be considered as a Schottky barrier of the metal-semiconductor-metal structure and/or insulator layer between nanowire and electrode. Resistivity of nanowires determined by this two-contact method was in around 4 kΩ·m although contact resistance may give impact in resistivity data.

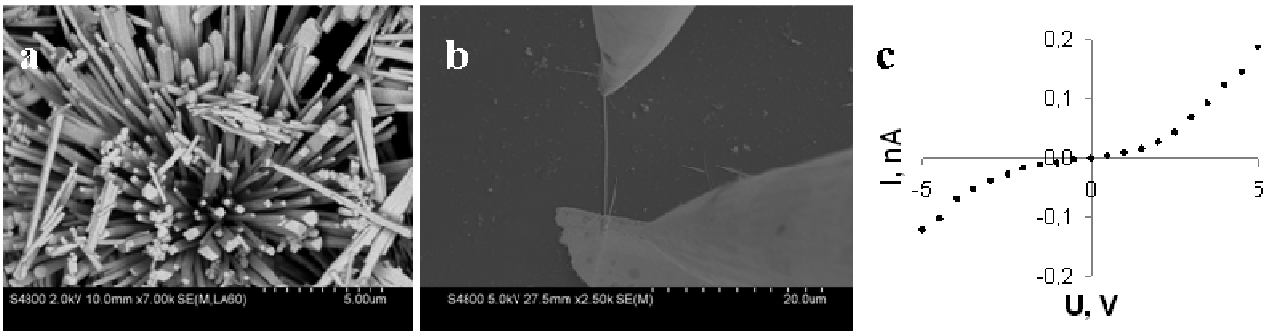

Fig. 1. SEM image of free standing nanowires after fabrication (a); in-situ contact to the nanowire with two gold probes (b); I(V) characteristic of individual nanowire (c).

To determine contact resistance nanowires were deposited on the surface with prefabricated macroelectrodes Fig. 2a. SEM and AFM images of individual nanowires contacting macroelectrode with one end can be seen in Fig. 2 b,c. These nanowires were selected for electrical characterization with conductive AFM.

In comparison to nanowire arrays [8-11] conductivity measurements in contact mode cannot be directly applied for Sb_2S_3 nanowires on the surface because nanowires were moved by the probe even when soft cantilevers with spring constant 0.2 N/m were used. Therefore different approach was used for determination of contact resistance.

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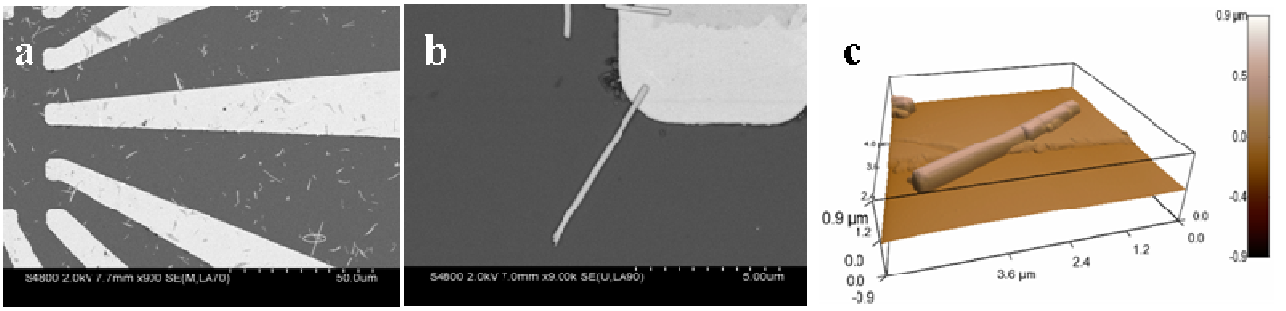

Fig. 2. SEM nanowires after deposition on surface with prefabricated macroelectrodes (a); SEM (b) and AFM (c) images of individual nanowires contacting surface of macroelectrode.

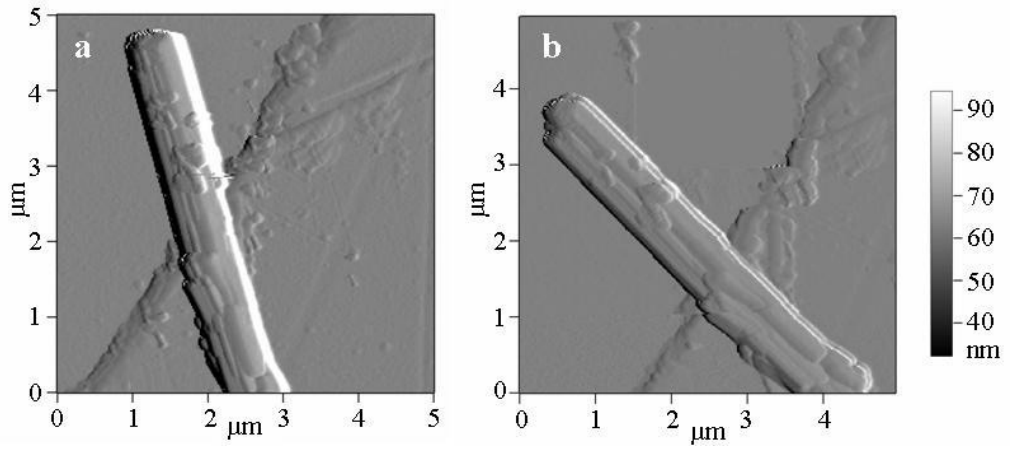

Fig. 3. AFM image of Sb_2S_3 nanowires deposited over the edge of macroelectrode scanned in contact mode: (a) first scan; (b) second scan.

First surface was scanned in tapping mode and on the nanowire surface points with different distances from the macroelectrode were selected. Secondly conductive AFM probe was moved to these positions and I(V) characteristics in each position measured (Fig. 4a). These I(V) characteristics are nonlinear and larger than *in situ* measurements where nonconductive gap around was observed (Fig. 1c and 4a). The contact resistance between interfaces (tip-nanowire and nanowire-macroelectrode) was determined from the summary resistance dependence on the distance from the macroelectrode (Fig. 4b). Contact resistances extrapolated from data was higher than 1 GΩ (in presented Fig.4b it was estimated around 10 GΩ), which may be mostly determined by the nanowire contact with the macroelectrode.

High contact resistance values most probably are related to the poor electrical contact between nanowire and macroelectrode as shown for conductivity measurements of nanowires deposited over gaps between two macroelectrodes [14]. Larger nonconductive gap observed in I(V) characteristics in C-AFM measurements in comparison to the *in situ* measurements with two gold probes shows that most likely the contact resistance to is higher for the position where nanowire is in contact with the macroelectrode, because we expect that properties of contacts between nanowire and probes are similar in both in situ and C-AFM measurements. Despite the high contact resistances between the nanowire and macroelectrode this method is simplest and can serve as express method for determination of resistivity of nanowires. Calculated resistivity of individual Sb_2S_3 nanowires was in the range 1.8÷3.2 kΩ·m which is a few orders of magnitude higher than resistivity of bulk Sb_2S_3 (5000 kΩ·m) [5] which may be result of doping of nanowires during synthesis.

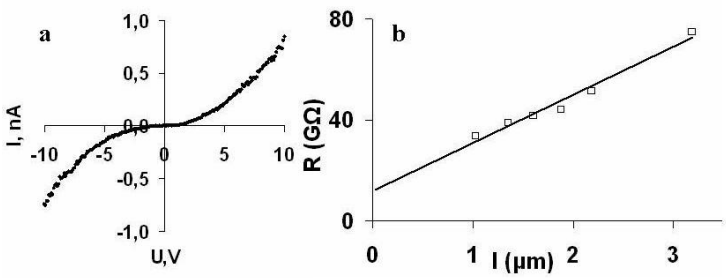

Fig. 4. I(V) characteristics of nanowire (a) and resistance of the nanowires at different distances from the macroelectrode (b).

Conclusions

Conductive AFM and in situ methods are showing to be powerful tool for nanoscale morphology, physical field's distribution measurements and manipulations of nanostructures. Here we developed a method for electrical characterization of fragile and poorly adhesive Sb_2S_3 nanowires. Combined C-AFM and *in-situ* techniques allow more precise characterization of nanowires and interfaces. Deposition of nanowires over macroelectrodes provide a simple express method for measurement of conductive properties of nanowires although it is limited to highly resistive nanostructures only caused by the high contact resistances between deposited nanostructure and the electrode.

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