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Structure, mechanical and electroconductive properties of nanomaterials

Ph.D. thesis in physics

Summary

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The work was done in the Institute of Chemical Physics, University of Latvia, under supervision of Dr. Chem. Donats Erts during 2002-2006 years.

Structure of the work

The promotion work is prefaced with the abstract, list of publications and conferences, where the main results were presented. The work is organized into the following chapters:

- Chapter 1, "Introduction", where the short historical review is given and the importance of chosen theme is argued;
- Chapter 2, "SPM principles", introduces some basic information about used instruments;
- Chapter 3, "Ge nanowire arrays inside AAO", where electroconductive and photoconductive properties of template synthesized nanowires are described;
- Chapter 4, "Nanoparticle catalyst synthesized free Ge and Si nanowires", where electroconductive and mechanical properties of nanoparticle catalyst synthesized free nanowires are revealed and compared with template synthesized nanowire properties;
- Chapter 5, "3D DNA arrays on Au(111)", where structure of this DNA architecture is described and electroconductivity of DNA oligomers probed;
- Chapter 6, "Possible applications", where utilization of Ge nanowires and DNA molecules discussed;
- Chapter 7, "Conclusions and Thesis", summarizes the work;
- Chapter 8, "References", where bibliography is given.

Work is appended with 8 copies of the included papers. The promotion work contains 62 pages, 54 figures, 4 tables and 89 references.

Actuality of the work

In recent times nanoscale materials such as carbon nanotubes, metal and semiconducting nanowires, nanoparticles, fullerenes, nanodots and others were thoroughly investigated in many aspects of their properties. However the technology of integration of many individual nano objects into functional devices is still in its infancy. There are two aspects of this problem. First one is the precise positioning of nano objects on solid surface. The very promising approach of solving this problem is application of self-assembly principles, when nanoobjects spontaneously assemble into ordered 2D (two dimensions) or 3D architectures. The second challenge arises if one would connect nanoobjects into electrical circuit. At this moment the only available technology for the permanent contacting is the electron beam lithography which is expensive, complicated and limited in 2D. Nanotechnology proposes utilization of nanowires for electrical addressing of individual nanoobjects.

In this PhD work structure, mechanical and electroconductive properties of two types of nanomaterials were studied: germanium nanowires and DNA oligomers. These materials were assembled into 3D architectures, what is essential for its future applications.

Single semiconducting nanowire can work as an entire device as already was demonstrated by some researchers [1, 2, 3]. However it should be noted what most previously demonstrated nanowire (and nanotube) based devices have 2D design. It means that in spite of ultra small diameter of nanowires (down to few nanometres) real device dimensions are measured in micrometers and cannot help electronic chip miniaturization. Nanowires ordered into 3D arrays in solid matrix such as AAO is a perspective material for engineering of complex 3D electric circuits and nanodevices. However, there are few data on electrical characterization of semiconducting nanowires [4] and no data on germanium nanowires inside AAO.

Optoelectronics and telecommunications is a perspective field of semiconducting nanowire and nanodot arrays utilization [5]. Investigation of photoconductivity processes in such structures provides information necessary for creation of new device for optical-to-electronic signal conversion. There have been no studies on photoconductivity of elemental nanowire arrays until now.

Although the optical and electronic properties of individual nanotubes and nanowires have been intensely investigated, there are few studies on the force interactions of nanotubes [6] and no studies on the force interactions of nanowires with electrical contacts. Such studies are important for the development of nanoelectromechanical systems (NEMS) and for finding methods of device integration into 3D arrays.

To date, DNA oligonucleotide base pairing appears to be the most thoroughly studied encoding system [7] and DNA is now supposed as an important component of emerging nanomaterials [8]. Learning how to control the formation of 3D DNA assemblies, and consequently combining nano scale building blocks into well-defined meso- and macroscopic structures, is important for nanotechnology and materials chemistry. Investigation of mechanical and electroconductive properties of DNA molecules is of great need for elaboration of DNA assisted electrical addressing of nanocomponents for nanoelectronics.

Aims of the work

The main aims of this work were the following:

- Investigation of structure (architecture), electroconductive and photoconductive properties of 3D germanium nanowire arrays in AAO;
- Comparison of electroconductive properties of free nanoparticle catalyst synthesized nanowires with AAO template synthesized ones;
- To study mechanical properties of free nanoparticle catalyst synthesized nanowires;
- Investigation of structure (architecture), mechanical and electroconductive properties of DNA oligomers in 3D array on Au surface.

Review of main results

High-density, vertically aligned ordered arrays of germanium nanowires of diameter 50 and 100 nm have been synthesized within the pores of anodized aluminum oxide (AAO) matrices using a supercritical fluid inclusion technique (figure 1). Packing density for 50 and 100 nm nanowires is 1.4×10^{10} cm^{-2} and 9×10^{8} cm^{-2} respectively. The conductive properties of germanium nanowires within AAO pores have been characterized by conductive atomic force microscopy (C-AFM) and macro-contact measurements. Nearly all of the semiconductor nanowires contained within the AAO substrates are found to be conducting. Additionally, each individual nanowire within the substrate possessed similar electrical properties demonstrating that the nanowires are continuous and reproducible within each pore.

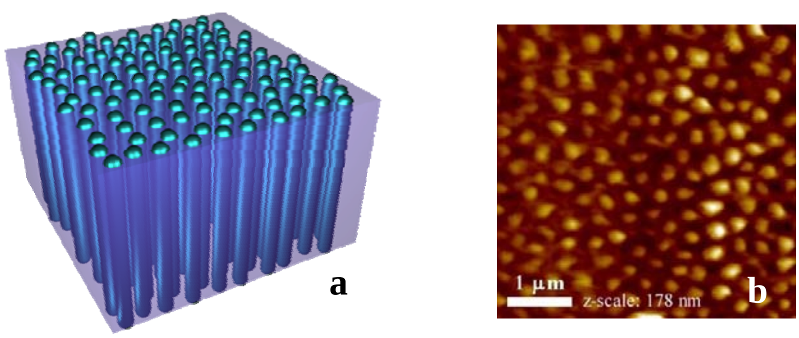

Figure 1. (a) - schematics of nanowire array inside AAO pores; (b) - AFM images of AAO surfaces filled with germanium after mechanical and chemical treatment (top view).

Macro-contacts are used to measure the mean current-voltage characteristics of groups of nanowires. Activation energy was measured at low and high temperatures, and is found to be close (0.58-0.61 eV) to the energy gap of bulk value for germanium (0.66 eV). Contact resistance between the nanowires and metal macro-contacts was minimized by polishing and gradual etching of the AAO surface, to expose the nanowires, prior to deposition of the contacts. Conductivity data from C-AFM and macro-contact measurements are found to be comparable suggesting that both methods are inherently suitable for evaluating the electrical transport properties of encapsulated nanowires within a matrix [10, 11].

Photoconductive properties of same germanium nanowire arrays, incorporated in AAO membrane pores, are also investigated using ITO and thin Au film electrodes. Advantages of different light transparent electrodes are analyzed. Photocurrent grows linearly with increasing voltage up to at least 50 V, and current value is sufficient to detect signal from individual nanowire. This fact let us presume that such nanowire arrays may be adopted for optoelectronics applications. Photodynamics of nanowire arrays is studied. Temperature effects and light redistribution due to photoluminescence in AAO matrix are investigated [12].

Electroconductivity of nanoparticle (Au) catalyst synthesized free germanium and silicon nanowires was studied and nanowire / electrode contact area is simultaneously visualized using TEM-STM. Nanowires are doped with Au and have higher conductivity than corresponding bulk materials. Electroconductivity of free nanowires is compared with electroconductivity of AAO template synthesized nanowires and it is found to be much higher (10^3-10^4 times) due to doping with Au atoms of catalyst nanoparticles. High elasticity (much higher than elasticity of bulk material) of single nanowires is also demonstrated [13, 14, 15].

Spatially distributed 3D DNA oligomer arrays on Au(111) surfaces are created by one-step co-assembly of mixed monolayers of alkanethiol-conjugated DNA and mercaptohexanol (figure 2(a)).

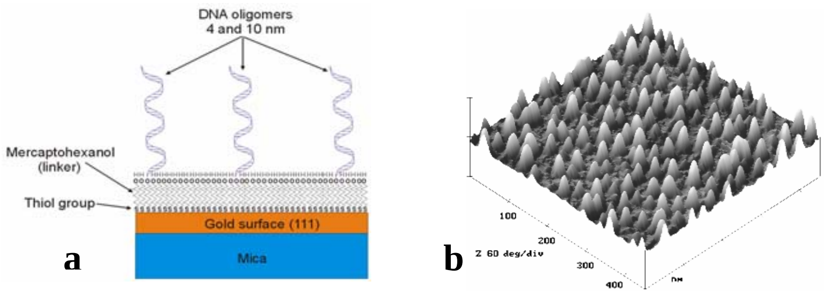

Figure 2. (a) - schematics of DNA array on Au (111) surface; (b) - tapping AFM images of DNA array surface.

Tapping mode and conductive contact Atomic Force Microscopes (AFM) are used to visualize and analyze the DNA distribution on the surface (figure 2(b)), and to study mechanical and conductive properties of individual molecules [16]. The DNA coating density (10^{10}-10^{11} cm^{-2}) is increases nonlinearly with increasing mole fraction of DNA oligomer to MCH in the coating solution. It is found that both duplex and single-stranded DNA molecules extend approximately vertically upward from the surface which confirms adaptability of this architecture for DNA assisted nanoobject assembly [17]. Electroconductivity of individual DNA oligomers was probed directly by conductive AFM tip and it is found that DNA oligomers are poor current conductors.

Novelty of results

In this work both organic and inorganic nanowire architectures created via self-assembly methods are investigated: ordered 3D germanium nanowire arrays and spatially distributed 3D DNA arrays.

To the best of our knowledge this is the first complex study on electrical characterization of germanium nanowire arrays inside AAO. It should be noted that the contact problem of nanowire / electrode is investigated and contact optimization procedure is found. New information on photoconductivity and photodynamics of germanium nanowire array inside AAO is acquired.

Nanoelectromechanical interactions of single semiconducting nanowires are studied for the first time. Information on mechanical properties and acting forces is collected and used to propose and demonstrate some operating principles of nanoelectromechanical device (NEPROM) based on single semiconducting nanowire.

Novel 3D DNA oligomer array on Au (111) surface is created. Viscoelastic behavior and electroconductivity of DNA oligomers attached to solid surface is investigated for the first time.

Practical importance

Ordered arrays of nanowires is a promising type of materials for nano and optoelectronics. This work was focused on problem of nanowire integrity inside surrounding matrix and electrical conductivity of nanowires, as well as making electrical contact with nanowires, which is important for elaboration of any electronic device based on such material. Possibility to utilize of Ge nanowire arrays for photo detection was also studied.

Investigation of electrical and mechanical properties of individual nanowires is important for creation nanoelectromechanical devices, which utilize nanowires. Collected data was used to propose and demonstrate functioning of memory element based on single nanowire. Nanoelectromechanical devices are believed to develop into competitors of traditional electronic switches and memory elements.

Array of DNA oligomers is perspective architecture for both nanotechnology and biotechnology. Similar DNA array preparation procedure is utilized in so called DNA biochip technology (Nanogen) for DNA analysis. DNA was proposed as material for electrical wiring and connection of nanostructures. Our data confirm the necessity to metalize DNA molecule to increase it electrical conductivity.

Thesis and conclusions

1. Electrical properties investigation method for individual nanowires inside AAO matrix using conductive AFM is elaborated and its applicability demonstrated.
2. Method of nanowire/electrode contact improvement for nanowires inside AAO matrix using selective chemical etching is found.
3. Ge nanowires synthesized with supercritical fluid inclusion method in AAO template are proved to be electroconductive and are demonstrated to exhibit photoconductivity.
4. Principles of nanowire based nanoelectromechanical device (NEPROM) operating are proposed and prototype of such device is demonstrated.
5. Method for electrical probing of DNA oligomers in 3D array on solid surface by conductive AFM is elaborated and its applicability demonstrated.

Publications

The main results of this PhD work were published in 8 papers in international journals. The results were also reported at 22 international conferences (25 reports) and 7 local conferences (18 reports), and published in proceeding and abstract books.

Work was presented and discussed at scientific seminars in the Institute of Physical Energetics and the Institute of Solid State Physics.

Papers:

1. B.Polyakov, B.Daly, J.Prikulis, V.Lisauskas, B.Vengalis, J.Holmes, D.Erts. High Density Arrays of Germanium Nanowire Photoresistors. *Advanced Materials,* 18, 14, 1812-1816, 2006.

2. D.Erts, B.Polyakov, B.Daly, M.Morris, S.Ellingboe, J.Boland, J.Holmes. High Density Germanium Nanowire Assemblies: Contact Challenges and Electrical Characterization. *Journal of Physical Chemistry B, 110,* 820-826, 2006.

3. K.Ziegler, B.Polyakov, J.Kulkarni, T.Crowley, K.Ryan, M.Morris, D. Erts and J.Holmes. Conductive films of ordered nanowire arrays, *Journal of Material Chemistry*, 14, 4, 585 - 589, 2004.

4. K.Ziegler, D.Lyons, J.Holmes, D.Erts, B.Polyakov, H.Olin, K.Svensson, E.Olsson. Bistable nanoelectromechanical devices, *Applied Physics Letters,* 84, 20, 3957-4121, 2004. (Also selected for publication in *Virtual Journal of Nanoscale Science & Technology*, 9, 19, 2004)

5. D.Erts, B.Polyakov, E.Saks, H.Olin, K.Ziegler, J.D.Holmes. Semiconducting nanowires: properties and architectures, *Solid State Phenomena,* 99-100, 109-116, 2004.

6. D.Erts, B.Polyakov, A.Lõhmus, R.Lõhmus, H.Olin, M.A.Morris, J.D.Holmes. Metallic and semiconducting nanowires studied by TEM-SPM. *Physics of Low-Dimensional Structures*, 3/4, 65-74, 2003.

7. B.Polyakov, D.Erts, U.Malinovskis, I.Muiznieks, E.Tuite. SPM studies of DNA architectures on Au(111) and mica surfaces. *Physics of Low-Dimensional Structures*, 3/4, 269-277, 2003.

8. D.Erts, B.Polyakov, H.Olin, and E.Tuite. Spatial and mechanical properties of dilute DNA monolayers on gold imaged by AFM. *Journal of Physical Chemistry B,* 107, 3591-3597, 2003.

Conferences:

1. *International Conference on Nanotechnology ICNT2006, Basel, Switzeland, July 30 – August 6, 2006.*
D.Erts, B.Polyakov, P.Birjukovs, B.Daly, J.Xu, J.D.Holmes. Conductive and photoconductive properties of semiconductor nanowire arrays.

2. *European Conference on Organised Films ECOF-10, Riga, Latvia, August 21-24, 2006.*
-B.Polyakov, D.Erts, U.Malinovskis, I.Muiznieks, E.Tuite. Mechanical and electroconductive properties of spatially distributed DNA oligomer arrays on Au(111).
-U.Malinovskis, A.Pastare, B.Polyakov, D.Erts, I.Muiznieks. Self-assembled DNA net-like structures on the mica surface.

3. *2nd Latvian Conference on Nanomaterials and Nanotehnologies,* Riga, Latvia, March 27-28, 2006.
-B.Polyakov, J.Prikulis, L.Grigorjeva, D.Miller, V.Zauls, J.H.Holmes, D.Erts. High density arrays of germanium nanowire photoresistors.
-K.Erta, K.Didriksone, B.Polyakov, J.H.Holmes, D.Erts. Two terminal nanoelectromechanical devices based on individual Ge nanowires and its arrays.
-U.Malinovskis, A.Pastare, B.Polyakov, D.Erts, I.Muizhniekas. Controllable self-assembled DNA net-like structures on the mica surface.

4. International Conference TNT-2005 (Trends in Nanotechnology), Oviedo, Spain, 29 August – 2 September, 2005.
B.Polyakov, B.Daly, J.Prikulis, B.Vengalis, J.Holmes, D.Erts. Photoconductive Properties of Germanium Nanowires Incorporated in Anodic Aluminium Oxide Membranes.

5. 10th INTEL Academic Forum, May 18-20, Gdansk, Poland, 2005.
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6. NSTI Nanotechnology Conference & Trade Show, Anaheim, CA, USA, May, 2005.
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8. 21st Scientific Conference of University of Latvia, Riga, 7-9 February, 2005.

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- B.Redkin, B.Polyakov, J.Holmes, D.Erts. Nanotribology on Ge nanowire architectures.
- U.Malinovskis, A.Pastare, B.Polyakov, I.Muižnieks, D.Erts. Controlled selfassembling of DNA networks on mica surface.
- D.Erts, J.Prikulis, B.Polyakov. New ES structural funds and home-made instruments in the Institute of Chemical Physics.

9. 1st Latvian Conference on Nanomaterials and Nanotechnologies, Riga, Latvia, March 30-31, 2005.
- B.Polyakov, B.Daly, Y.Prikulis, L.Grigorjeva, D.Miller, J.H.Holmes, D.Erts. Conductive and photoconductive properties of Ge nanowires in anodic aluminium oxide membranes.
- B.Redkins, B.Polyakov, J.H.Holmes, D.Erts. Local electrostatic, friction forces and conductivity of Ge nanowire arrays encapsulated within oxidized aluminium.
- D.Erts, B.Polyakov, J.H.Holmes. Nanoelectromechanical devices.
- R.Udris, L.Lauks, J.H.Holmes, B.Polyakov, D.Erts. Light and radiation detection by germanium nanowire arrays.

10. International Conference TNT-2004, (Trends in Nanotechnology), Segovia, Spain, September 13-17, 2004.
B.Polyakov, D.Erts, J.Holmes. Complex studies of Ge nanowire arrays in oxidized alumina membranes.

11. Nordic-Baltic SPM Workshop, Trondheim, Norway, June 15-17, 2004.
- B.Polyakov, D.Erts, J.Holmes. AFM studies of conductive Ge nanowire arrays in oxidized alumina membranes.
- D.Erts, H.Olin, J.Holmes, B.Polyakov, A.Lohmus. Electrical and mechanical properties of nanowires studied by SPM.

12. SPM - 2004 International Workshop, Nizhny Novgorod, Russia, May 2-6, 2004.
U.Malinovskis, A.Pastare, B.Polyakov, I.Muiznieks, D.Erts. AFM studies of DNA networks on mica surface.

13. 20th Scientific Conference of University of Latvia, Riga, February 16-18, 2004.
- B.Polyakov, L.Lauks, R.Udris, J.Holmes, D.Erts. Thin films of conductive ordered Ge nanowire arrays.
- D.Erts, B.Polyakov, E.Saks, A.Patmalnieks, H.Olin, J.Holmes. Metal and semiconducting nanowires and architectures.

14. 8th Conference of Latvian Physical Society, Jelgava, Latvia, July 1, 2003.
D.Erts, B.Poļakovs, E.Saks, U.Maļinoskis. Nanocomponents: properties, architectures, devices.

15. Scanning Probe Microscopy – 2003 International workshop, Nizhny Novgorod, Russia, March 2-5, 2003.
- B.Polyakov, D.Erts, U.Malinovskis, I.Muiznieks, E.Tuite. SPM studies of DNA architectures on Au(111) and mica surfaces.
- D.Erts, B.Polyakov, A.Lõhmus, R.Lõhmus, H.Olin, J.D.Holmes. Properties of metallic and semiconducting nanowires studied by TEM-SPM.

16. E-MRS 2003 Fall Meeting, Warsaw, Poland, September 15-19, 2003.
D.Erts, B.Polyakov, E.Saks, H.Olin, J.D.Holmes. Semiconducting nanowires: properties and architectures.

17. II ESF-Nanotribology Workshop, Antalya, Turkey, October, 2003.
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18. 19th Scientific Conference of University of Latvia, Riga, February 10-12, 2003.
- B.Polyakov, D.Erts, U.Malinovskis, I.Muiznieks, E.Tuite. DNA macromolecular architectures: creation and properties investigation.
- D.Erts, B.Polyakov, A.Truhins, L.Skuja, A.Patmalnieks, K.M.Ryan, J.D.Holmes. Investigation of 3D semiconducting nanowire architectures using TEM, AFM and optical methods.
- U.Maļinovskis, I.Muižnieks, B.Polyakov, D.Erts. DNA macromolecular architectures on solid surfaces.

19. Nordic-Baltic SPM Workshop, Tartu, Estonia, May 29-31, 2002.
- D.Erts, J.D.Holmes, D.Lyons, M.A.Morris, H.Olins, E.Olsson, B.Polyakov, L.Ryen, K.Svensson. Properties of silicon nanowires studied by TEM-STM.
- B.Polyakov, D.Erts, E.Tuite, H.Olin, M.Knite. Conductive and mechanical properties of 2-dimensional DNA-arrays and electroconductive polymer composites.

20. 15th International Congres on Electron Microscopy. Durban, South Africa, 1-6 september, 2002.
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22 International Scientific Conference on Biomedical Engineering and Microtechnologies, RTU, Riga, Latvia, October 10 - 14, 2002.
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23. 18th Scientific Conference of ISSP University of Latvia, February 2002.
D.Erts, B.Polyakov, H.Olin. Mechanical properties and conductivity of single silicon nanowires measured by scanning tunneling microscope compatible with transmission electron microscope.

24. International Scientific Conference on Biomedical Engineering and Microtechnologies, RTU, Riga, Latvia, October 10 - 14, 2002.
D.Erts, B.Polyakov, J.D.Holmes, H.Olin. Properties of silicon nanowires.

25. Adriatico Research Conference on Interaction and Assembly of Biomolecules, Trieste, Italy, 27-31 August, 2001.
B.Polyakov, D.Erts, E.Tuite, H.Olin. Force and conductivity measurements on DNA-arrays and MCH monolayers on Au(111) surface with cAFM.

26. Second International Conference on Scanning Probe Spectroscopy, Hamburg, Germany, July 19 -22, 2000.
D.Erts, B.Polakov, H.Olin, T.Vajakas, E.Tuite. Tunneling and force spectroscopy of self assembled DNA monolayers on Au(111) abstract,

27. Eleventh International Conference on Mechanics of Composite Materials, Riga, Latvia, June 11 -15, 2000.
D.Erts, B.Polakov, and E.Tuite. Spatial, Mechanical, Conductivity Characterization of Self-Assembled DNA Arrays on Au(111) by Atomic Force Microscopy.

28. Nordic-Baltic SPM workshop, Goteborg, Sweden, June 5-7, 2000.
B.Polakov, D.Erts, H.Olin, T.Vajakas, E.Tuite. Conductive and Nanomechanical SPM Characterization of Self Assembled DNA Arrays on Au(111).

29. International Seminar "Medical Engineering and Physics: Science, Practice, Busyness", Riga, 6-9 October, 1999.
D.Erts, E.Tuite, B.Polyakov. Structure and conductive properties of self assembled DNA by scanning probe microscopy.

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