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THERMOELECTRIC PROPERTIES OF NANOSTRUCTURES IN CHRYSOTILE ASBESTOS AND POROUS GLASS

The nanostructures of indium and bismuth antimonides in chrysotile asbestos channels and porous glasses have been studied. In asbestos channels, sufficiently perfect quasi one-dimensional wires are formed, and in porous glasses – three-dimensional nets of semiconductor materials. The electric conductance and thermopower of these nanostructures have been measured in the temperature range of 80 to 400 K. It is shown that transport properties of composites depend heavily on the type of a die and the semiconductor material forming a nanostructure. Many features of transport properties of the investigated nanowires can be, at least qualitatively, described by the Luttinger liquid theory. In the nanowires in chrysotile asbestos channels, the thermoelectric power factor increases quickly with a rise in temperature.

Key words: thermoelectricity, Luttinger liquid, nanowire, chrysotile asbestos, porous glass.

Introduction

In recent years, an active search for materials and structures with dimensionless thermoelectric figure of merit $ZT \gg 1$ has been pursued. The investigations are conducted in various lines, however, it is only in nanostructures that considerable increase in thermoelectric figure of merit as compared to the value of $ZT = 1$ has been experimentally obtained. In 2001 Venkatasubramanian et al. manufactured superlattices based on bismuth telluride with the dimensionless thermoelectric figure of merit 2.4 at room temperature [1]. At about the same time Harman et al. [2] synthesized $PbSnSeTe/PbTe$ superlattices consisting of quantum dots, with $ZT = 2.0$ at $T = 300$ K. In 2005 Harman managed to improve considerably his result [3] and obtained at a temperature of 550 K for $PbSeTe/PbTe$ superlattices the value of $ZT \approx 3.0$. Higher ZT values have not been obtained so far, so a search for novel low-dimensional structures for thermoelectric power conversion continues to be a relevant objective.

According to theoretical estimates, high values of thermoelectric figure of merit can be exhibited by nanostructures consisting of ultrathin nanowires of diameter less than 10 nm [4-7]. However, at the present time in various laboratories worldwide intensive efforts are underway to study sufficiently thick nanowires of diameter ~ 50 nm of such thermoelectric materials as Bi , $Bi_{1-x}Sb_x$, Bi_2Te_3 , $InSb$, Si [8-14], as well as irregular bismuth nanostructures in porous dies [15-17]. All these structures either have large characteristic diameters or differ markedly in their topological structure from quasi one-dimensional wires. So, no sensible increase in thermoelectric figure of merit has been achieved in this line of investigation thus far.

One should note a fundamental distinction of the electronic properties of quasi one-dimensional wires from the respective properties of nanostructures of larger dimensions. In quasi one-dimensional wires the electron-electron interaction results in formation of a specific strongly correlated state of electronic subsystem that came to be known as the Luttinger liquid [18]. The equilibrium and transport properties of the Luttinger liquid are radically different from those of the Fermi gas. For instance, the electric conductance (reverse resistance) of one-dimensional conductor quickly increases with a rise in temperature [19-21], like in semiconductors. The thermopower of the Luttinger liquid increases as a linear function of temperature [22-25], like in metals. Moreover, some calculations predict considerable increase in the thermopower of the Luttinger liquid as compared to that of the Fermi gas [22, 26]. All these properties of the Luttinger liquid hold much promise in terms of thermoelectric applications.

In this paper, some peculiarities of thermoelectric properties of ultrathin wires of indium and bismuth antimonides in chrysotile asbestos and porous glass channels have been studied. The above nanostructures have comparable diameters of the wires they are formed of ($\sim 5 - 7$ nm), but differ in surface topology and roughness degree. It is shown that these features of nanostructures lead to a drastic difference in their transport properties.

Semiconductor nanostructures in porous dies

Estimates show that ultrathin nanowires of indium and, possibly, bismuth antimonide of diameter less than 10 nm must be quasi one-dimensional conductors, since the electrons in them fill only the lower subband of the energy spectrum resulting from dimensional quantization. The manufacture and use of single nanowires of such dimensions is a hard-to-solve problem, so we investigated semiconductor nanostructures obtained by indentation of molten material under high hydrostatic pressure to the pores of dielectric dies [27]. As such dies, chrysotile asbestos and porous glasses were used.

Natural mineral chrysotile asbestos [25, 27] is a tight packing of long nanotubes whose dimensions depend on the geological deposit or synthesis conditions. The internal diameters of asbestos nanotubes are generally 1 to 10 nm, the outer diameters – 10 to 100 nm. In each specific sample the spread of internal diameter values is not large. The samples used in this work had the average channel diameters 5 to 7 nm. These channels were filled with *InSb* or *Bi* melt at a pressure of ~ 15 kbar. The samples cut from asbestos pieces filled in this fashion consisted of 10^5 to 10^7 parallel asbestos nanotubes whose images obtained using electric scanning microscope (SEM) are shown in Fig. 1. Inside each nanotube there is a readily visible channel filled with a semiconductor material.

Analysis of *X*-ray diffraction spectrum of an asbestos die filled with indium antimonide has shown that on entering nanotube channels, the semiconductor does not change its stoichiometric composition. In so doing, high crosswise fillability of channels is observed and nanocrystallites with the average characteristic dimension 4.4 nm are formed [28].

The rigid frame of porous glass [27, 29, 30] consists mainly of *SiO₂*. The concentration of this oxide is approximately 96 %. The pores of this material are interconnected and form a three-dimensional net consisting of short channels. Unlike chrysotile asbestos, the channels in porous glass have an irregular surface shape, differing dramatically from the cylinder one. The specific feature of porous glasses is a narrow maximum in the distribution of pores according to their lateral dimensions. The width of this maximum at half the height is approximately 0.15 of the average diameter of pores in the sample [30]. At the same time, changing glass synthesis conditions, the average diameter of pores can be varied from several angstroms to several microns.

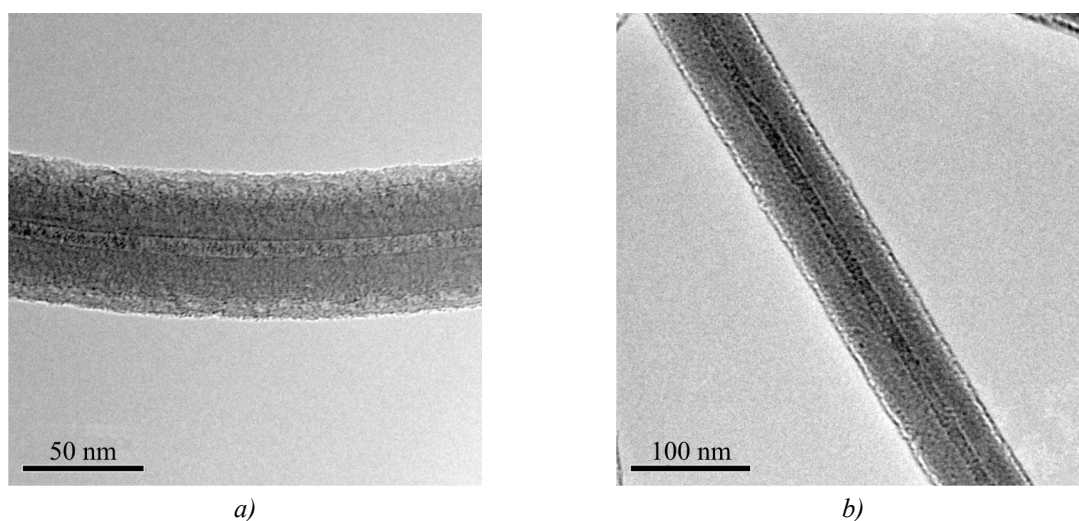


Fig. 1. SEM-images of two chrysotile-asbestos nanotubes filled with InSb.

In this work, porous glasses with the average lateral dimension of pores ~ 7 nm, close to the average diameter of channels in chrysotile asbestos, were selected. In this case possibility emerges to carry out a comparative analysis of thermoelectric properties of long quasi one-dimensional wires and three-dimensional nets consisting of short nanowires with the same characteristic dimensions. Fig. 2 represents SEM-images of the surfaces of two porous glasses with different dimensions of pores. In the pictures, pores are matched by the darker areas. It is seen that in Fig. 2 *a* the average diameter of pores is ~ 1 μm and Fig. 2 *b* shows a glass with characteristic dimensions of pores 6 to 10 nm that are almost coincident with the internal diameters of the above described asbestos nanotubes.

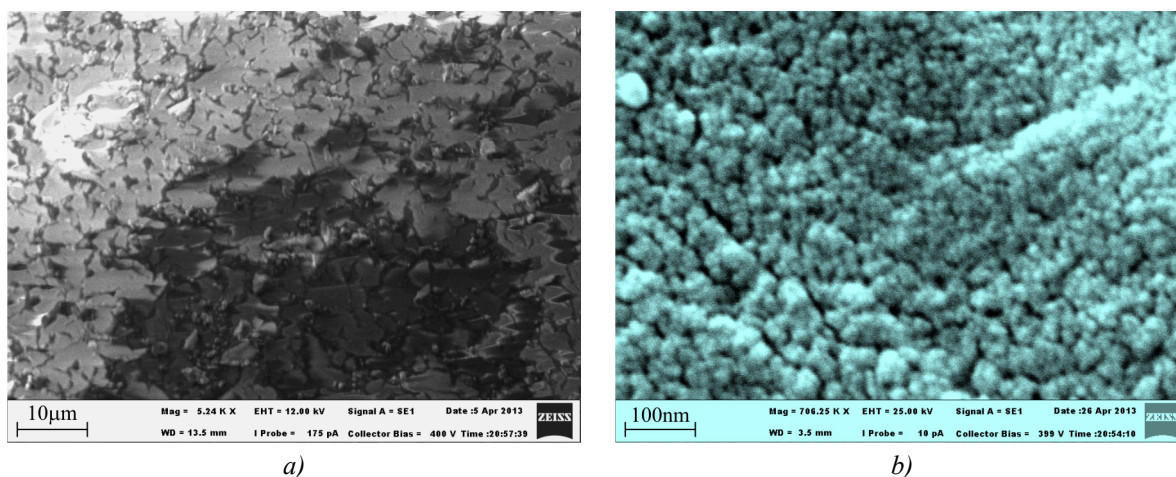


Fig. 2. SEM-images of the structure of two porous glasses with lateral dimensions of pores ~ 1 μm (*a*) and 6 – 10 nm (*b*).

Semiconductor nanostructures in porous glasses were manufactured in the same way as nanowires in asbestos dies. Capsules used for the preparation of composite samples were simultaneously filled with pieces of chrysotile asbestos and the respective porous glass.

To determine the composition of manufactured nanocomposites, the energy-dispersive X-ray analyzer INCA X-Act from Oxford Instruments was used. The X-ray spectra and atomic composition of porous glass filled with InSb are represented in Fig. 3. Within the measurement error the

concentration of indium and antimony in the composite corresponds to stoichiometric composition of the initial semiconductor.

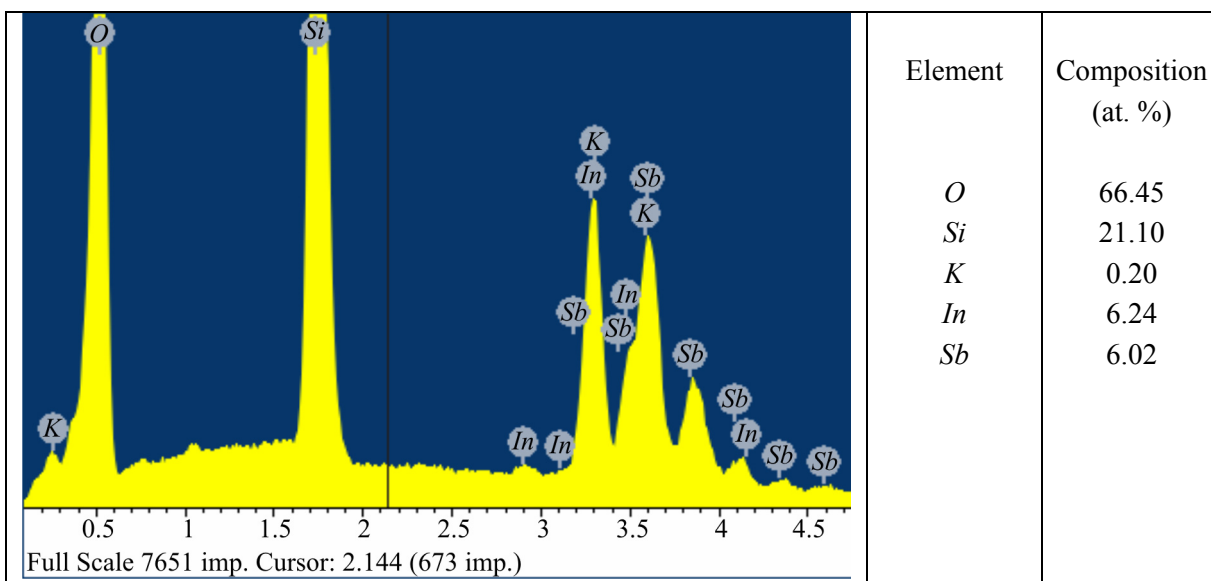


Fig. 3. X-ray spectrum and the results from the compositional analysis of porous glass filled with InSb.

Thus, the above-described procedure for preparation of samples of semiconductor nanostructures allows creating both long quasi one-dimensional wires in chrysotile asbestos channels of diameter 5 to 7 nm and three-dimensional nets of short nanowires of approximately the same diameter. It should be noted that despite the comparable characteristic dimensions, these nanostructures differ in topology. Moreover, the lateral dimensions of a wire in a porous glass change with lengthwise displacement. Therefore, the electron band structure of a semiconductor net in a porous glass to all appearance may be regarded as a totality of connected quantum dots.

Method for measuring the thermopower of nanostructures

The samples of nanostructures obtained by the above-described method can have high electric resistances $\sim 10^6$ to $10^8 \Omega$, since in the process of manufacturing not all the channels in the initial dies are filled with semiconductor materials. The investigation of the thermoelectric properties of such objects requires special methods for measuring kinetic coefficients. The most complicated problem is thermopower measurement at high electric resistance values of samples.

Our experimental setup is intended for measuring the thermopower of nanowire bunches in the temperature range of 80 to 400 K. This setup employs a comparative method of thermopower measurement. On the measured and reference samples an identical temperature difference ΔT is created which is then calculated based on the known thermopower of the reference sample. Such an approach requires no direct measurement of ΔT and increases the accuracy of result due to increased signal-noise ratio.

The samples made of nanowire bunches had a length of 1 to 2 mm and a section of $\sim 0.01 \text{ mm}^2$. Ohmic contact was provided by indium electrodes. With a view to minimize the resistance, the distance between the indium electrodes was set of the order of 0.3 to 0.1 mm. For thermopower measurement high-resistance voltmeters with input resistance at least $10 \text{ G}\Omega$ were used. The basic elements of the experimental setup are represented in Fig. 4.

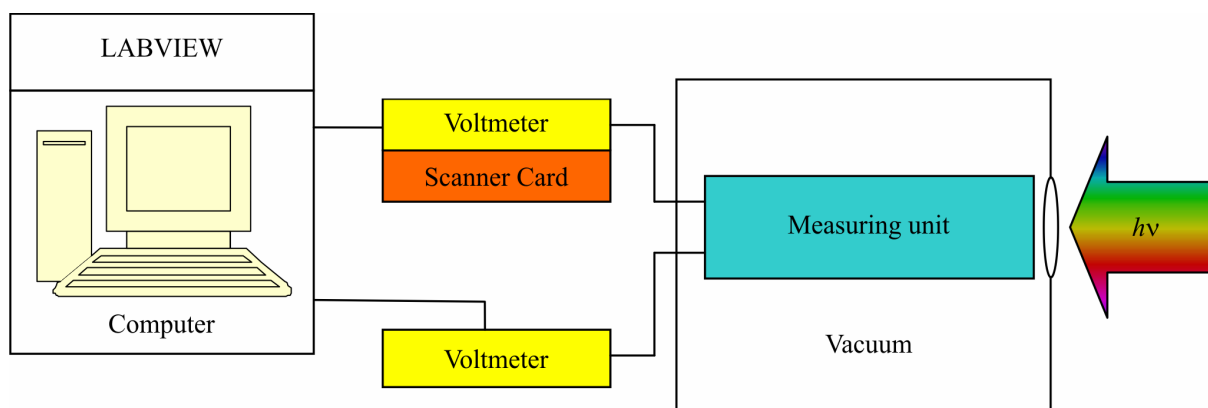


Fig. 4. Simplified circuit of measuring setup

Measuring unit was placed into vacuum chamber. The main element of measuring unit is a holder with two pairs of clamps arranged on it, in one of which the sample under study is clamped, and in the other – the reference. As the reference, calibrated constantan was used. To reduce electric pickup in measuring circuits, the clamps were electrically isolated from the package of measuring unit with retention of the necessary thermal contact. For creation of temperature difference the electric heaters shall not be used because of electric pickup and electric shunting of sample due to leakage currents in the circuits. Therefore, temperature difference on the samples was created by lighting the receiving plate of the hot clamps. In order to minimize parasitic heat fluxes, both clamp pairs were covered with thermal shield.

Fig. 5 shows one of the clamp pairs. On the left there is a cold clamp (8) having a good thermal contact to the holder, so its temperature differs from that of the body at most by 2 K. On the right there is a hot clamp (6). Between this clamp and the copper body (1) there is stainless steel heat spreader (3) having a relatively high thermal resistance and making it possible to obtain temperature difference on the sample. The hot clamp is heated by intensive light beam incident on thermal radiation detector (5) that was blackened with a view to improve light absorption. Such a design assures reliable shading of the object under study.

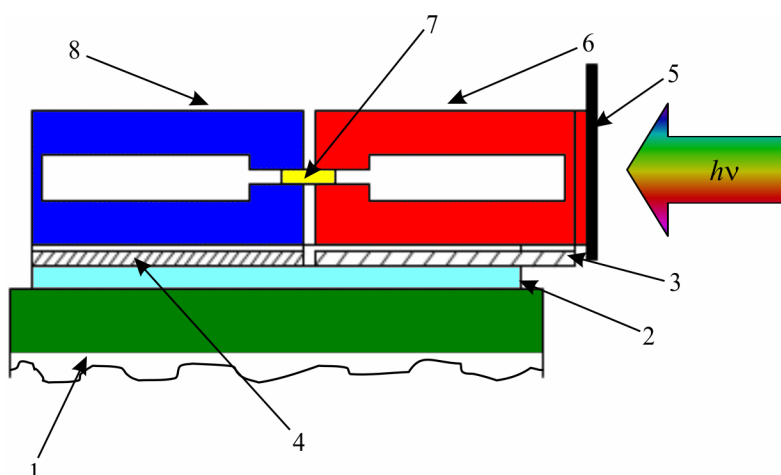


Fig. 5. Schematic of samples holder: 1 – copper body; 2 – dielectric substrate BeO; 3 – stainless steel; 4 – copper plate; 5 – heated element (Cu); 6 – hot clamp; 7 – sample or standard; 8 – cold clamp.

The temperature dependence of the thermopower of the object under study was measured simultaneously with measuring the thermopower of the reference. The measured data was fed into the computer, and the results were used to calculate the Seebeck coefficient of the sample:

$$S_{sample} = S_{ref} \cdot U_{sample} \cdot K(T) / U_{ref}, \quad (1)$$

where S_{ref} is the Seebeck coefficient of the reference, U_{sample} is voltage on the sample, U_{ref} is voltage on the reference, $K(T)$ is correction factor taking into account small diversity of temperature drops on the sample and reference, determined with a parallel measurement of two references.

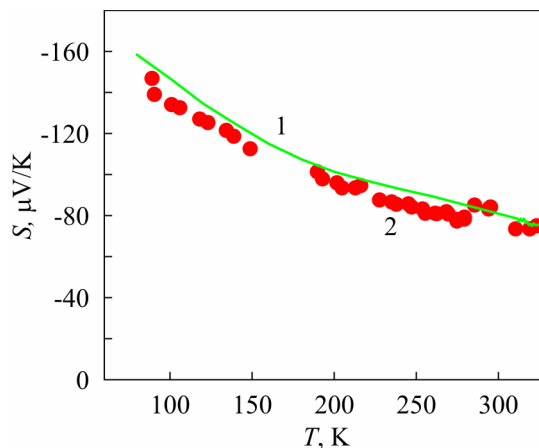


Fig. 6. Temperature dependences of the Seebeck coefficient of $Bi_{0.85}Sb_{0.15}$ single crystal measured by the absolute (1) and relative (2) methods.

Fig. 6 represents the results of testing the above-described experimental setup. The temperature dependence of the Seebeck coefficient S_{11} of $Bi_{0.85}Sb_{0.15}$ single crystal was measured and compared to a similar dependence obtained by traditional absolute method of thermopower measurement. The choice of $Bi_{0.85}Sb_{0.15}$ single crystal as a test material was determined by high homogeneity, stability of properties, high thermopower values and practical isotropy of thermoelectric properties. The difference in the temperature dependences of the Seebeck coefficient of the same $Bi_{0.85}Sb_{0.15}$ crystal measured by the absolute and relative methods does not exceed 10 %, which is a wholly satisfactory result.

Discussion of measurement results

All nanostructures under study consist of ultrathin nanowires having comparable lateral dimensions. Estimates show that strong dimensional quantization of the electron spectrum should cause the emergence in them of a wide forbidden band (~ 500 meV), and the energy gap between the first and upper subbands should be more than 200 meV. Therefore, at temperatures below 300 K only the electrons of the lower spectrum subband must contribute to impurity conduction (all nanostructures under study had n -type conductivity). In the circumstances, one might expect the similarity of temperature dependences of the electron kinetic coefficients in all nanostructures considered. However, measurements have shown that the thermoelectric properties of these nanostructures are qualitatively different. They depend on the type of the die used and the semiconductor material it is filled with. Moreover, they may be drastically different even in the samples cut from the same initial piece of composite. The large variety of properties and their dominant physical processes invite further investigations. Below we turn our attention only to some specific features of nanostructures considered.

All one-type samples (for instance, $InSb$ nanowires in asbestos die) can be conventionally divided into three groups. The first-group samples have high resistance $R > 10^5 \Omega$ at 300 K. This group of samples is characterized by abrupt electric resistance jumps on cooling and heating. As a rule, these jumps are irreversible and after several measurement cycles result in resistance increase to $10^{14} \Omega$ and more. We believe that these samples have only a small amount of defective through wires

(wire clusters in porous glasses). Due to the effect of elastic stresses arising with temperature change and electric migration of defects, the wires are broken, leading to a drastic resistance increase. When a considerable electric voltage is applied, a concurrent break of the entire array of wires is possible [31] which is initiated by the electrostatic field of the first broken wire and leads to practically complete sample failure. In some cases on thermal cycling there are reversible resistance jumps (see Fig. 7). We cannot interpret this effect unambiguously. It is likely that such nanostructure behaviour is caused by the difference in the coefficients of linear thermal expansion of semiconductor and die, leading to the emergence of strains which with a drop in temperature break the wires, and then restore the conductivity on heating.

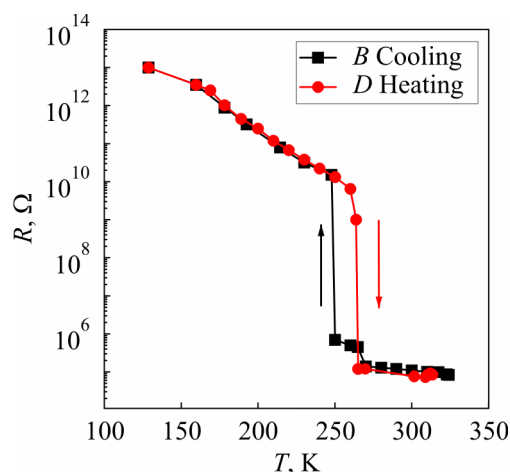


Fig. 7. Temperature dependence of the electrical resistance of InSb sample in the asbestos die belonging to the first group. Temperature change directions are indicated by the arrows.

The second-group samples have low resistances ($< 10^3 \Omega$), smoothly and reversibly changing with temperature variation. The thermopower of these samples often changes its sign with temperature variation, and the temperature dependences of conductance in indium antimonide nanostructures are of activation nature [32], like in the bulk semiconductors. An example of such $S(T)$ dependence for bismuth nanowires in asbestos die is given in Fig. 8 (red curve). Such behaviour of second-group samples is due to the presence in them of a crack filled with semiconductor. In the process, a conducting channel with a characteristic dimension of $\sim 1 \mu\text{m}$ is formed. Electron scattering by the channel surface reduces their contribution to thermopower and results in the Seebeck coefficient's sign reversal (classical dimensional effect).

Of greatest interest from physical and practical standpoints are the third-group samples. They are relatively stable, and at room temperature their resistances are $10^3 < R < 10^7 \Omega$. Their thermopower is proportional to temperature (sometimes there are slight deviations from proportionality), like in metals. Conductance, as a rule, is increased with a rise in temperature, like in semiconductors. However, in contrast to them, $G(T)$ dependence is not of activation nature. The thermoelectric properties of this group of samples are governed by sufficiently perfect quasi one-dimensional semiconductor wires or their three-dimensional nets.

Special emphasis should be placed on the thermoelectric properties of ultrathin InSb wires in the asbestos die (third-group samples). As mentioned above, in quasi one-dimensional conductor due to increased effect of electron-electron interaction, a strongly correlated state of electron subsystem is formed which was called the Luttinger liquid [18]. Unlike the Fermi liquid, the elementary excitations in the Luttinger liquid are collective excitations similar to phonons, rather than quasi-particles. According

to theory, the temperature dependence of a linear conductance of the Luttinger liquid containing a defect (or several independent defects) should be described by power function $G \propto T^\alpha$, where power exponent α is determined by the value of electron-electron interaction [19, 20]. The thermopower should be proportional to temperature [22, 23]. These dependences for one of the samples with quasi one-dimensional *InSb* wires are represented in Fig. 9, and, for the sake of illustration, the plot of $G(T)$ dependence is built in log-log scale. It is seen that the plot in Fig. 9 a at $T < 250$ K is practically linear and corresponds to $G \propto T^{5.2}$ dependence. The thermopower of the same sample represented in Fig. 9 b in the range of $T < 300$ K is really proportional to temperature. Slight deviations of the represented curves from the theoretical dependences at $T > 250$ K seem to be related to the violation of electron subsystem strong degeneracy condition which governs the applicability of the Luttinger liquid theory.

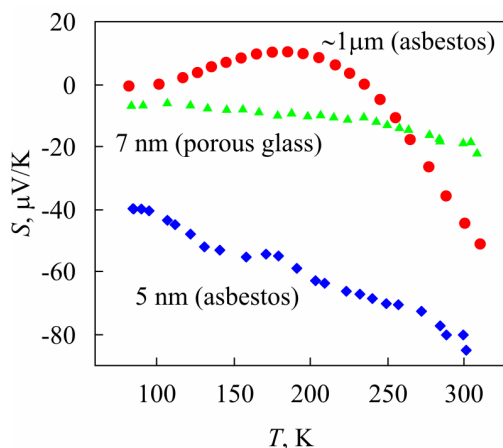


Fig. 8. Temperature dependences of thermopower of bismuth in chrysotile asbestos nanotubes (blue diamonds), in porous glass nanopores (green triangles) and in asbestos die crack (red circles). The approximate characteristic dimensions of structures are indicated.

It should be noted that the sample whose data is represented in Fig. 9 was manufactured with the use of tellurium-doped indium antimonide. By our estimates, the concentration of electrons in it was $\sim 4 \cdot 10^{18} \text{ cm}^{-3}$. In so doing, the Fermi energy in the bulk *InSb* should be ~ 270 meV, and in the wires of diameter 7 nm – ~ 60 meV. Therefore, the thermopower values in Fig. 9 b are not high. In similar samples, but not doped with tellurium, the absolute thermopower value at room temperature can reach 200 $\mu\text{V/K}$ [24].

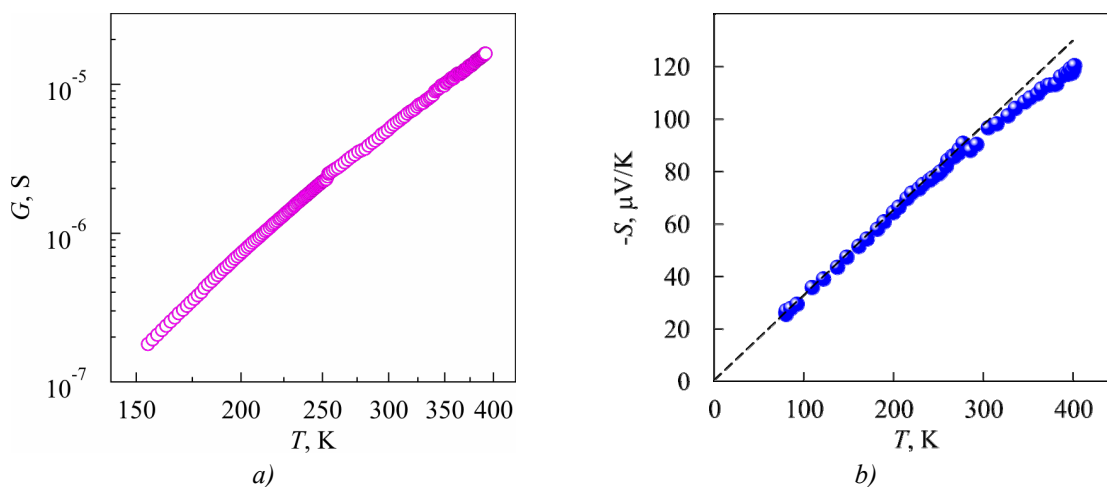


Fig. 9. Temperature dependences of conductance (a) and thermopower (b) of a sample cut from chrysotile asbestos filled with *InSb*. The dashed line shows the $S \propto T$ dependence.

Unlike quasi one-dimensional *InSb* wires in asbestos die, similar samples consisting of bismuth wires of the same diameter have practically temperature-independent conductance. The temperature dependence of thermopower for one of such samples is shown in Fig. 8 (blue curve). It is little different from the dependence represented in Fig. 9 *b*. However, in this case strict proportionality of the Seebeck coefficient to temperature, if any, exists only in the low-temperature region. Such differences in the properties of one-type nanostructures cannot be explained so far. It may be related to the surface states [33] that determine the electronic properties of bismuth thin films and wires.

The thermoelectric properties of nanostructures based on porous glass differ from the considered properties of nanowires in chrysotile asbestos. The conductance of three-dimensional nets formed by short wires of both indium antimonide and bismuth slowly grows with a rise in temperature [21, 34]. The temperature dependence of thermopower of bismuth in porous glass is shown in Fig. 8 (green curve). The distinctive feature of this dependence is the abnormally low thermopower value. It is, for instance, 4 to 5 times lower than the thermopower of quasi one-dimensional bismuth wires in asbestos. There is no unambiguous explanation of such a drastic difference in the properties of nanostructures with equal characteristic dimensions. At first glance, it may seem that strong shape irregularity of bismuth wires in porous glass should result in strong Anderson localization of electrons. However, in [34] it is shown that in such nanostructures at low temperatures only small localization of charge carriers is possible.

The most probable reason for a strong difference between thermopower values of the two considered nanostructure types lies in the different nature of their electron states. In quasi one-dimensional wires in asbestos dies the Luttinger liquid is formed, whereas in nanostructures in porous glass due to their three-dimensionality a conventional Fermi-liquid is formed. Moreover, in theoretical works [22, 23, 26] it is asserted that the thermopower of the Luttinger liquid should exceed manifold that of the Fermi gas. However, in our opinion, the study of this problem is far short of completion. Electron spectrum nonlinearity does not contribute to the thermopower of the Luttinger liquid [35]. The contribution to transport properties of electron-phonon interaction should not be considerable either, since in one-dimensional degenerate semiconductors there are few phonons capable of inverting electron pulse. At the same time, the contribution to the Luttinger liquid transport properties of electron scattering on defects (potential barriers) is governing due to formation of a quasi-gap in the tunneling density of states [18, 19]. In [26] it is shown that the thermopower of the Luttinger liquid comprising two closely-spaced barriers (quantum well) should be increased by electron-electron interaction. However, depending on quantum well parameters this contribution can be of any sign. Therefore, with averaging over the defective structure of a real nanowire this process should not be a major contribution to thermopower. Of great significance is a model considering the Luttinger liquid with one potential barrier (or several independent barriers). It is stated [23] that here again the thermopower should be increased manifold as compared to that of the Fermi gas. However, another calculation [36] of a similar module yielded the opposite result, namely the contribution to thermopower of electron scattering on the barrier proved to be considerably less than the respective contribution to thermopower of the Fermi gas. Thus, further investigations are necessary to clarify the peculiarities of nanostructures considered.

Conclusion

Sufficiently perfect semiconductor nanostructures have been manufactured in chrysotile asbestos and porous glass channels with characteristic dimensions of 5 to 7 nm. The semiconductor

material retains its stoichiometric composition on indentation into nanoporous dies. Very long quasi one-dimensional wires are formed in asbestos channels. The nanostructures in porous glasses are a three-dimensional net consisting of arbitrary-shaped short wires. The thermoelectric properties of nanostructures depend heavily on the type of a die and the semiconductor it is filled with. Moreover, certain property variations are observed even in the samples cut from the same composite. Of greatest interest, both in terms of basic physics, and for practical application, are quasi one-dimensional indium antimonide wires in the channels of asbestos nanotubes. The temperature dependences of thermopower and electrical conductance of these wires are adequately described by the Luttinger liquid theory. For practical purposes it may be beneficial to use the high thermopower of these wires that far exceeds the thermopower of the bulk filling material and that of the respective nanostructure in a porous glass. Moreover, in quasi one-dimensional indium antimonide wires the power factor increases quickly with a rise in temperature. This feature may be of advantage in the development of nanostructured materials for thermoelectric power conversion.

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