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Detection of internal cracks and ultrasound characterization of nanostructured Bi_2Te_3-based thermoelectrics via acoustic microscopy

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ABSTRACT

The search for thermoelectric (TE) materials for highly efficient devices aims at improving the TE efficiency and broadening their areas of applications. We created nanostructured thermoelectric Bi–Sb–Te-family materials by high energy (ball milling) pre-treatment of the parent materials followed by high-pressure/high-temperature treatment. $Bi_{0.5}Sb_{1.5}Te_3$ compositions with the superfluous maintenance of tellurium was used for the synthesis of the samples with p-type electrical conductivity. Acoustic microscopy was used to study elastic properties and bulk irregularities and to detection of internal cracks both in the parent materials and in the created nanostructured samples. The data has been used for optimization of parameters of synthesis of nanostructured thermoelectrics.

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1. Introduction

The best heat-to-power conversion efficiency of conventional TE materials is just about 4% [1]. The efficiency is assessed with a figure of merit Z defined as $S^2\sigma/\kappa$, where S is the Seebeck coefficient (measured in μV/K), σ the electric conductivity, and κ the thermal conductivity. For practical applications, the nondimensional parameter defined as ZT is used (where T is the temperature). It takes a maximum value of 0.8 in bulk TE materials, while it could be greater than 2.5 in nanostructures. Z can be improved for nanostructured materials by (1) reduction of lattice thermal conductivity by means of enhanced phonon scattering on the interfaces between nanoparticles and (2) increase of S by electrons tunneling through interfaces between nanoparticles, with both goals helping to increase ZT [2,3]. Nanostructured TE materials offer a promising approach for the preparation of bulk specimens with nanostructured constituents.

The tasks for producing nanostructured TE materials are the following: improve mechanical properties (reduce brittleness and improve machining) and improve TE properties (figure of merit).

Much efforts have been applied to improve properties of thermoelectrics by doping and alloying, but little progress was made until the reports for $Bi_{2-x}Sb_xTe_3$ alloys [4]. Appropriately doped Bi_2Te_3-based alloys are the most competitive TE materials.

Among other extensive applications of TE materials one of important and intensively studied is biomedicine. Thus, worldwide efforts are undertaken to expand the technology of TE devices into the field of micro-systems technologies. TE devices are attractive as potential power sources in biomedical applications because they directly convert thermal gradients into electrical power [5,6]. Bi_2Te_3-based alloys are a proven TE system, and provide an excellent prototype devices. Bi_2Te_3 may be a material system to implement in hyper-dermal designs, but tellurium toxicity will probably prevent sub-dermal usage [7].

Among methods of creation of bulk samples of the nanostructured TE materials the considerable part is powder technologies based on high pressure/high temperature technique. It is one of the most common for creation of bulk nanostructured materials. High energy mechanical pre-treatment (ball milling) allows mixing and crushing initial components down to nanosize. Ball milling followed by high-pressure/high-temperature treatment was found to be “good technological chain” to produce nanomaterials with unique properties [8]. This technology allows producing compact nanocomposites with zero porosity. Among the advantages of such technology are rather low price and readiness for mass production.

In the present work the employed method of the production of bulk materials with the nano-crystalline structure assumes the use of methods of powder metallurgy, namely, sequential realization of obtaining the nano-powder of the initial synthesized material and the transformation of it by means of cold or hot pressing into the compact material with the assigned geometric dimensions and structure. The pressed TE materials can

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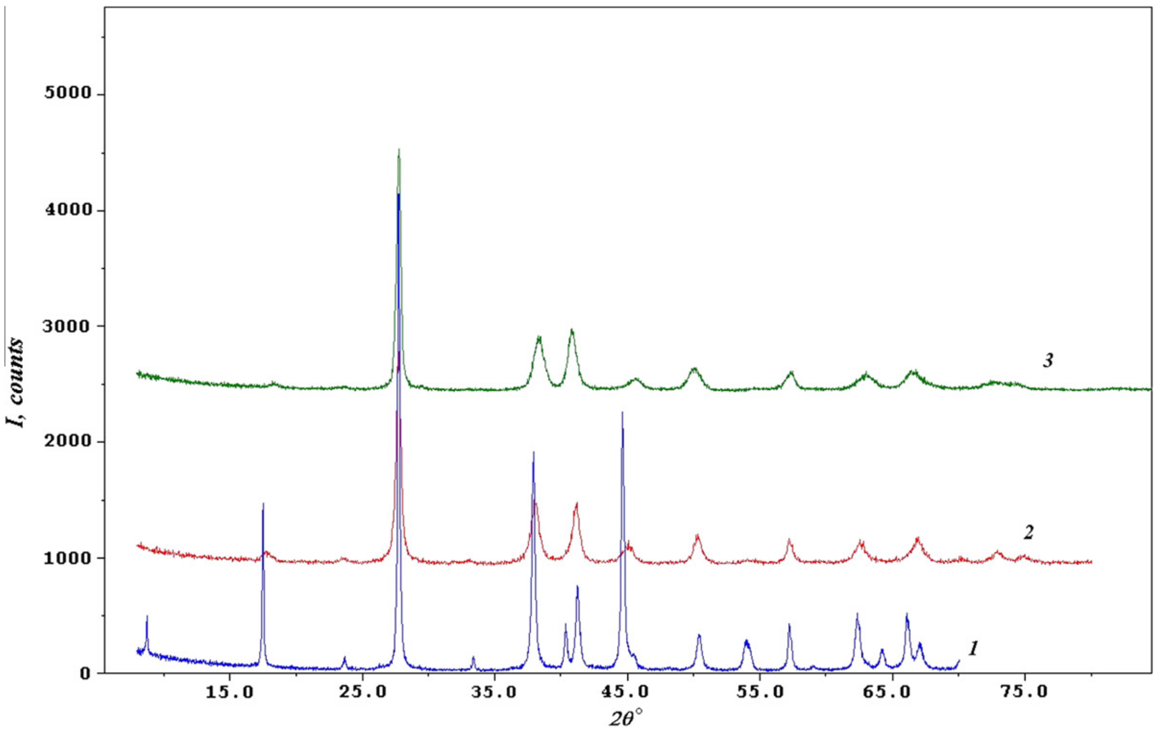

Fig. 1. X-ray diffraction patterns of $Bi_{0,5}Sb_{1,5}Te_3$ nanopowder. Diffraction peaks corresponding to triple solid solution of $Bi_{0,5}Sb_{1,5}Te_3$ only (a = 4.284 Å and c = 30.440 Å).

be obtained both from the TE alloy powder of stoichiometric or close to it composition, preliminarily synthesized by the metallurgical method and by the method of mechanochemical synthesis from the mixture of the powders of the initial components, taken in the stoichiometric ratio.

2. Experimental details

We used the following technological process to fabricate bulk specimens of nanostructured Bi_2Te_3-based thermoelectrics:

a. Fine crushing of a ingot material. $Bi_{0.5}Sb_{1.5}Te_3$ polycrystalline ingots was used for the nanostructured samples synthesis with p-type electrical conductivity.
b. Weigh, sizing (fraction less then 0.8 mm was extracted) and high energy ball milling. Refinement and homogenization was carried out in a high-power and high-speed planetary mill which provides the effective crushing and mixing of powders at impact of working bodies with acceleration up to 20 g.
c. Extraction of the activated mixed powders from mill drums.
d. Assembly of high pressure containers.
e. Consolidation under high pressures/high temperatures (thermobaric treatment) of the activated powder mixes.

At last stage there are three versions of obtaining compact specimens from the highly active ultra-dispersed powders were tested: sintering of the compacted powder at high temperature and ambient pressure (A-series); sintering at high pressure and high temperature in graphite molds (B-series); sintering in steel molds at high temperature and higher pressures compared to the previous method (C-series).

To exclude pollution all operations with powders were carried out in the argon atmosphere.

The wide-field pulse scanning acoustic microscope was applied in a reflection mode at the driving frequencies f = 25 MHz to measure local values of ultrasonic velocities (microacoustic technique) and to visualize bulk microstructure and internal cracks of a specimen (scanning acoustic microscopy). Ultrashort probing ultrasonic 30–40 ns pulses were used for measurements. Experimental procedure was described in details previously [9]. Elastic moduli were calculated on the basis of the measured sound velocities and densities of the samples. The data on sound velocities of longitudinal V_L and transverse V_T waves were obtained with an accuracy of ∼1%; elastic moduli ∼2–3%.

Structural investigations were also performed by high resolution electron microscopy (HRTEM) and X-ray diffraction studies.

3. Samples and results

3.1. Powders after high energy ball milling

X-rays diffraction (Figs. 1 and 2) and HRTEM (Fig. 3) observation of samples (alloys) show grains of 10–20 nm size without preferentially orientations.

HRTEM and X-ray investigations revealed that $Bi_{0,5}Sb_{1,5}Te_3$ was nanopowder after high energy ball milling.

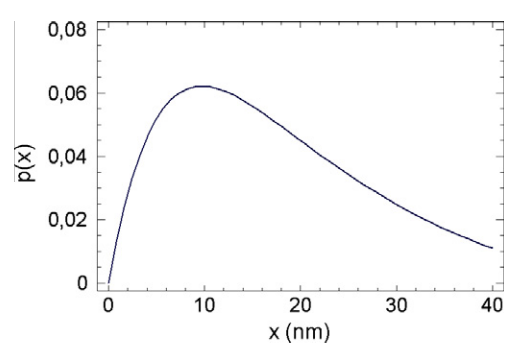

Fig. 2. Coherent scattering length (CSL) distribution in $Bi_{0,5}Sb_{1,5}Te_3$ nanopowder. Maximum value CSL is 40 nm. The mean size of nanoparticles ∼10 nm.

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