

INDIUM IMPURITY EFFECT ON GROWTH AND STRUCTURAL PERFECTION OF LEAD-TIN TELLURIDE WIRE CRYSTALS

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(Received 13 January 2010)

Abstract

In this work, we present the results of studying the indium impurity effect on the structural perfection of wire crystals (WCs) of the $\text{Pb}_{0.8}\text{Sn}_{0.2}\text{Te}$ solid solution, which were obtained by sublimation in quartz capillaries.

The microscopic and X-ray investigations revealed that indium doped lead-tin telluride single crystals with uniform distribution of indium impurity can be grown by the zone sublimation method.

The relationship between the method of treatment of the surface of indium doped lead-tin telluride WCs of the $\text{Pb}_{0.8}\text{Sn}_{0.2}\text{Te}$ solid solution and the density of structural defects, microhardness, and electric parameters was found.

Introduction

Solid solutions of lead-tin telluride and its alloys have been used for many years for designing emitters and detectors of infrared radiation in a spectral range of 3-14 μm that operate within the range from room temperature to 600-650°C. In addition, these materials are used in thermoelectricity and tensometry [1-3].

However, specific features of the crystallochemical properties of $\text{A}^{\text{IV}}\text{B}^{\text{VI}}$ materials lead to the formation of a high concentration of intrinsic defects, dislocations, and other disruptions of the crystal lattice, which renders difficult to obtain devices with high physical parameters. In order to design effective semiconductor converters, it is necessary to obtain crystals of solid solutions of high structural quality with the given composition and the required electrophysical and mechanical characteristics. Therefore, the main task of this work was to grow homogeneous lead-tin telluride wire crystals and to study some physical characteristics depending on the technology of their fabricating.

Experimental results

In this work, we present the results of studying the structural defects and some mechanical properties of wire crystals of the $\text{Pb}_{0.8}\text{Sn}_{0.2}\text{Te}$ solid solution doped with indium, which were obtained by sublimation in quartz capillaries with an inner diameter of 10-120 μm and a length of about 20 cm of p- and n-type conductivity. Indium impurity in the amount of 0.2-0.6 at % was introduced into the charge in the course of synthesis of the material.

The electron microscope investigation of samples of resultant WCs showed that they had a smooth cylindrical surface with characteristic metallic luster; the size of step-shaped

defects on their surface was much smaller than that of the melt-grown WCs of the same composition. At the end face of the samples, there was the (100) growth plane; its surface was smooth and shiny (Fig. 1a, 1b).

Measurements of the electrical parameters of the WCs obtained from a charge containing 0.2-0.3 at % indium showed that they were of the p-type conductivity. As the indium content increased to 0.4 at %, we obtained WCs of both p- and n-type; from a charge containing 0.5 at % indium, WCs of the n-type conductivity were grown. WCs with the most uniform electrical characteristics were obtained from a charge with 0.6 at % indium.

The X-ray examination showed that the WCs were single crystals; they grew mainly along crystallographic direction $<100>$; a uniform distribution of indium impurity along their length was observed; their structure was more perfect than that of bulk crystals grown by a similar method. The composition of the samples grown by sublimation, which was determined by the lattice parameter, was the same along the entire length of the WCs and nearly coincided with the composition of the growth charge, whereas for the melt-grown WCs of PbSnTe it differed from the initial one by 10-15% [4].

The results of microhardness measurements and dislocation density investigations along longitudinal and transverse sections testify to the high homogeneity of the indium doped lead-tin telluride wire crystals. A special technique of preparation of longitudinal and transverse section for studying microhardness and dislocations structures was elaborated. This method is based on chemical-mechanical polishing of WCs not involving the grinding process for the minimal damage of the surface layer up to 2 μm , whereas mechanical grinding results in the damaged layer depth up to 20 μm .

After chemical-mechanical polishing, the samples were exposed to etching in a solution containing 10 parts of (95% HBr + 5% Br) and one part of toluene [5]. The use of this etchant for chemical polishing of lead-tin telluride WCs with indium impurity ensures obtaining of mirror smooth surfaces without oxide film. This is due to the fact that the composition of the proposed solution, in addition to the active element HBr, contains toluene that governs the intensity of dissolving. For revealing the dislocation etch pits, we used a solution of the following composition: 10g KOH + 10ml H₂O + 1ml C₃H₈O₃ + 0.5ml 30% H₂O₂.

The revealing of dislocation pits was carried out during 0.5–1 min. It should be noted that dislocation pits dimensions depend on the duration of etching. It was found that dislocation pits of the WCs under study appear normally to their surface and have a strict rectangular shape both in the longitudinal and transverse sections. The diagonals of the etching pits always coincided with crystallographic direction $<100>$. However, such dislocation pits on nondoped lead telluride and lead-tin telluride WCs have not been observed earlier. The relatively low dislocation density ($7 \cdot 10 - 3 \cdot 10^3$) cm^{-2} of the indium doped lead-tin telluride WCs under study also are indicative of the high homogeneity of the grown WCs.

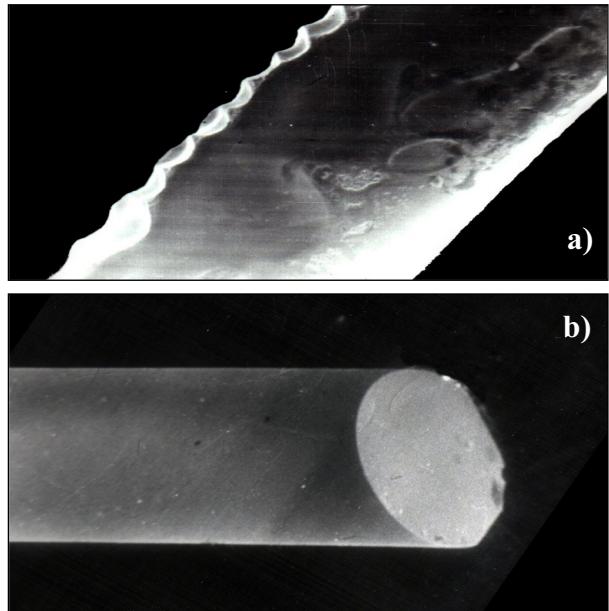


Fig. 1. Microphotograph of longitudinal surfaces of indium doped lead-tin telluride wire crystals (x 1500); a) grown from the melt, b) obtained by zone sublimation in quartz capillaries.

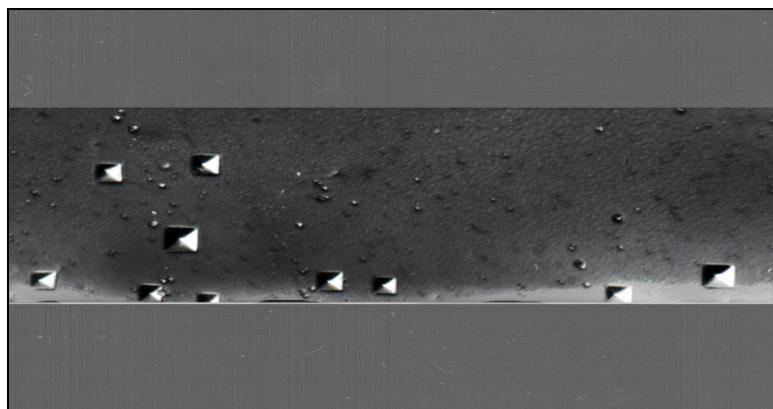


Fig. 2. Dislocation etch pits on the $\text{Pb}_{0.8}\text{Sn}_{0.2}\text{Te}$ WCs doped with indium at a depth of 7 μm after chemical-mechanical polishing (x 1000).

The sample microhardness was measured by indentation on the transverse and longitudinal surfaces of WCs polished by the above mentioned method. The local indenter loading of the crystals was carried out by a PMT-3 device at a working load of (2–10)g. The indentation microhardness was calculated by the generally accepted correlation $H = 1854 P/d$, where P is the indenter load in kg and d is the indentation diagonal in μm .

It was found that the microhardness of indium-doped WCs was higher than that of nondoped samples; it was 56–72 kg/mm² for p-type samples and 50–63 kg/mm² for n-type conductivity in the range of WC diameters of 10–80 μm . The increase in microhardness by a factor of 2–3, depending on indium impurity concentration in the range of 0.1–0.4 at %, was observed for samples of both p- and n-type conductivity. As the indium concentration increased (above 0.6 at %), a decrease in microhardness took place. It should be noted that the value of microhardness along the longitudinal and transverse sections of WCs samples was constant, which counts in favor of the structural homogeneity of the WCs.

Conclusions

It was established that it is possible to obtain indium doped lead-tin telluride WCs of high quality by the method of zone sublimation in quartz capillaries.

The results of the study showed that the quality and the physical parameters of lead-tin telluride WCs can be controlled by indium impurity doping in a considerably wide concentration range up to 0.6 at.%.

The relationship between the method of treatment of the surface of indium-doped PbSnTe WCs and the density of structural defects and electrical parameters was found.

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