

Electrophysical and Thermoelectric Properties of Nano-scaled $\text{In}_2\text{O}_3\text{:Sn}$, Zn, Ga-Based Thin Films: Achievements and Limitations for Thermoelectric Applications

G. Korotcenkov, V. Brinzari, B. K. Cho

Abstract—The thermoelectric properties of nano-scaled $\text{In}_2\text{O}_3\text{:Sn}$ films deposited by spray pyrolysis are considered in the present report. It is shown that multicomponent $\text{In}_2\text{O}_3\text{:Sn}$ -based films are promising material for the application in thermoelectric devices. It is established that the increase in the efficiency of thermoelectric conversion at $C_{\text{Sn}} \sim 5\%$ occurred due to nano-scaled structure of the films studied and the effect of the grain boundary filtering of the low energy electrons. There are also analyzed the limitations that may appear during such material using in devices developed for the market of thermoelectric generators and refrigerators. Studies showed that the stability of nano-scaled film's parameters is the main problem which can limit the application of these materials in high temperature thermoelectric converters.

Keywords—Energy conversion technologies, thermoelectricity, In_2O_3 -based films, power factor, nanocomposites, stability.

I. INTRODUCTION

CURRENTLY, the search for alternative energy sources is one of the most demanded and promising areas of research. In this regard, thermoelectric energy conversion is of particular interest, because home heating, automotive exhaust, and industrial processes all generate an enormous amount of unused waste heat that could be converted to electricity by using thermoelectrics [1]. This means that a huge amount of the waste heat, including human kind activities, can be converted into useful energy, helping to increase the production efficiency and improve living conditions. Thermoelectric generators, which can be used for these purposes, are solid-state devices with no moving parts; they are silent, reliable and scalable, making them ideal for small, distributed power generation [2]. However, conventional thermoelectric converters have low efficiency, and therefore their application is limited to niche markets where high reliability, less maintenance, and small size are more

important than magnitude of energy output. This means that extension of the area of thermoelectric generators applications is possible only via increasing the efficiency of thermoelectric conversion.

At present, there are three main directions in studies aimed at improving the efficiency of thermoelectric converters [3]-[7]. They include the followings: (1) optimization of bulk electrophysical properties of materials aimed at increasing the concentration and mobility of the charge carriers; (2) search for materials capable to increase the operating temperature; and (3) elaboration of the technology of the material nanostructuring, providing an increase in the efficiency of thermoelectric conversion due to a significant reduction in thermal conductivity of material caused by a phonon scattering at the physical boundaries of the nanoscale structure. Theoretical calculations have shown that the effect of the grain boundary filtering of the low energy electrons is another opportunity to increase thermoelectric efficiency in nanostructured materials. It has been found that by filtering effect the thermoelectric efficiency can be increased in several times [5], [8]. Thus, research conducted in material engineering for thermoelectric applications should focus on the search for new materials, not only with the required electrophysical properties, i.e. high electroconductivity, but also with certain structural and surface properties, which can guarantee the formation of the grain boundaries with interface properties optimal for phonon scattering and filtering effect. The experiment showed that the semiconductor metal oxides are the best materials for solving these problems [4], [6]. It could be explained by the fact that the semiconductor metal oxides can have a high conductivity at extraordinary high thermal stability, which allows devices operating in temperatures up to 1000 °C, and the metal oxides have a simple technology of nanostructuring. The analysis showed that In_2O_3 is one of the most promising metal oxides for such applications [9], [10].

Indium (III) oxide is a transparent semiconducting metal oxide of n -type of conductivity with a wide band gap. In_2O_3 possesses a large variety of unique properties like high electrical conductivity, high optical transparency in the visible region and high reflectance in the infrared spectral regions, high thermal stability, good adhesion to the substrate and ease of patterning to form transparent circuitry [11], [12]. To date

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In_2O_3 is heavily exploited in different practical applications from optoelectronics [12] to gas sensor design [13].

The aim of our study was (i) to evaluate the real prospects of nanostructured In_2O_3 films for thermoelectric applications, (ii) to find out the factors that control the thermoelectric properties of these films, (iii) to determine the influence of different dopants on the electrical and thermoelectric properties, and (iv) to evaluate limitations that may occur when using nano-scaled In_2O_3 films in high temperature thermoelectric converters.

II. EXPERIMENTAL DETAILS

$\text{In}_2\text{O}_3:\text{Sn}$ (ITO) films ($C_{\text{Sn}} = 0\text{--}50$ at.%) were the main object for our research. Doping with tin, which is a donor to In_2O_3 , provided necessary increase in the conductivity of the In_2O_3 films. More complicated multicomponent nanocomposites tested in these studies, such as $\text{In}_2\text{O}_3:\text{Zn}$ ($C_{\text{Zn}} = 0\text{--}75$ at.%), $\text{In}_2\text{O}_3:(\text{Sn} + \text{Zn})$ ($C_{(\text{Sn}+\text{Zn})} = 0\text{--}50$ at.%), $\text{ITO}(10\%\text{Sn}):\text{Ga}$ ($C_{\text{Ga}} = 0\text{--}30$ at.%), and $\text{Zn}_{0.5}\text{InSn}_{0.5}\text{O}_{1.5}:\text{Ga}$ ($C_{\text{Ga}} = 0\text{--}20$ at.%), belonged to $\text{In}_2\text{O}_3\text{--SnO}_2\text{--ZnO}$ and $\text{In}_2\text{O}_3\text{--SnO}_2\text{--ZnO--Ga}_2\text{O}_3$ systems. We proceeded from the fact that the introduction of new elements in metal oxide provided additional opportunities to affect the surface properties and to control the state of the grain boundary interface. This effect may be achieved by incorporation of dopants into the lattice of the basic metal oxide, and their segregation at the grain surface.

Studied In_2O_3 -based films were deposited on dielectric substrate by using spray pyrolysis at $350\text{--}520$ °C [14]. 0.2 M water solutions of InCl_3 with addition of SnCl_4 , GaCl_3 , and ZnCl_2 were used as precursors. Usually films had thickness varied in the range of $50\text{--}200$ nm.

Structural characterization of the films was performed by using X-ray diffraction (XRD), scanning electron microscopy (SEM) and atomic force microscopy (AFM). Thermoelectric properties, i.e. conductivity and the Seebeck coefficient, were studied over a temperature range of $20\text{--}450$ °C. Power factor ($\text{PF} = \alpha^2 \cdot \sigma$, where α is the Seebeck coefficient and σ is the conductivity), was used as a parameter characterizing the thermoelectric conversion efficiency [15].

III. RESULTS AND DISCUSSIONS

As a result of studies, it was shown that electroconductivity, Seebeck coefficient as well as nanostructure of films studied were strongly dependent on the composition of nanocomposite and deposition temperature. Typical AFM images of In_2O_3 films are shown in Figs. 1 (a) and (b). In particular, it was observed a sharp decrease of grain size and changes in grain shape in the range of $0\text{--}10$ at.% of Sn content. In addition, at this composition ITO films demonstrated grain size growth on film's thickness. The power factor (PF) as a function of the composition of In_2O_3 -based multicomponent films also demonstrated non-monotonous behavior with the maxima at a certain concentration of doping additives. Namely, near 5 at.%, there was found maximal thermoelectric PF for our films deposited at $T_{\text{pyr}} = 350$ °C. XRD studies have shown that films with the given optimal composition are textured with

preferential surface orientation in direction (100). It is also found that the main changes in the properties of multicomponent metal oxide films took place at concentrations of dopants corresponded to their limit solubility in In_2O_3 . Previous studies of gas sensing characteristics of In_2O_3 films deposited by spray pyrolysis showed also that these films are porous [16], [17]. Earlier it has been noted [7] that large dispersion of grain size, which was observed in our nanostructured films (see Figs. 1 (c) and (d)) [18], and the presence of porosity are favorable factors for enhanced phonon scattering and drop in thermal conductivity. As it is known, a low thermal conductivity of thermoelectric materials is necessary to achieve high values of the figure of merit ZT ($ZT = \text{PF} \cdot T/k$, where T is the temperature and k is the thermal conductivity [14]). In accordance with estimations, thermoelectric material suitable for real applications should have $ZT > 1$ [18].

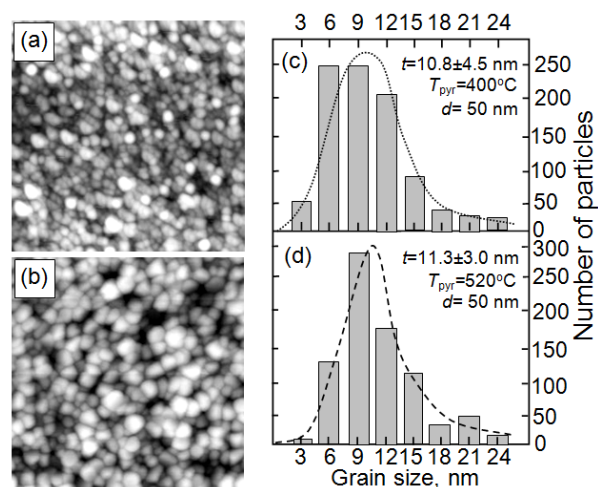


Fig. 1 Influence of pyrolysis temperature and film thickness on (a, b) AFM images and (c, d) grain size dispersion in In_2O_3 films deposited by spray pyrolysis: (a)- $T_{\text{pyr}} = 400$ °C, $d \sim 50$ nm; (b) - $T_{\text{pyr}} = 520$ °C, $d \sim 200$ nm

It was also established that nano-structured $\text{In}_2\text{O}_3:\text{Sn}$ films ($d \sim 90\text{--}100$ nm, $C_{\text{Sn}} = 5\%$) with a grain size of $25\text{--}30$ nm had much higher efficiency of thermoelectric conversion in comparison with ceramic In_2O_3 prepared using conventional approach. If the best sintered $\text{In}_2\text{O}_3:\text{Sn}$ samples with a crystallite size of tens of micrometers had a power factor of $0.4\text{--}0.5$ mW/m·K² [19]–[22], then our nanoscaled $\text{In}_2\text{O}_3:\text{Sn}$ films had PF reaching $4\text{--}5$ mW/m·K² [23]. At the moment this is the best result obtained for the n -type metal oxides designed for thermoelectric applications. This demonstrates that nanoscaled $\text{In}_2\text{O}_3:\text{Sn}$ metal oxides are promising materials for thermoelectric converters, because observed increase in the efficiency of converting heat into electricity makes the use of thermoelectric generators cost-effective. Oxides, forming the $\text{In}_2\text{O}_3\text{--SnO}_2$ binary system, have n -type conductivity and can be used in tandem with already well-established thermoelectric materials of p -type, for example such as cobaltites Na_2CoO_2 [24]. It was also found that all studied

samples from $\text{In}_2\text{O}_3\text{-SnO}_2\text{-ZnO}$ and $\text{In}_2\text{O}_3\text{-SnO}_2\text{-ZnO-Ga}_2\text{O}_3$ systems had power factor smaller than the power factor of the $\text{In}_2\text{O}_3\text{:Sn}$ films with optimal doping concentration (see Fig. 2). Only for a few compositions of ITO:Ga-based films at a certain Ga concentration, close to the limiting solubility in In_2O_3 , the power factor had values acceptable for practical applications [10].

A more detailed examination of the reasons of such behavior of thermoelectric properties of the $\text{In}_2\text{O}_3\text{:Sn}$ films led to the conclusion that the increase in the efficiency of thermoelectric conversion at $C_{\text{Sn}} \sim 5\%$ occurred due to nano-scaled structure of the films studied and filtering effect.

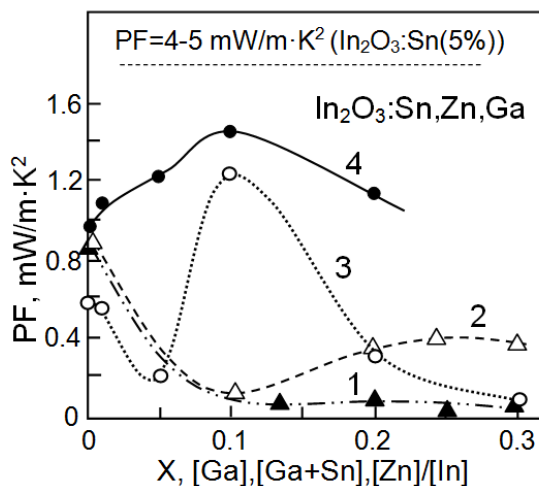


Fig. 2 Influence of the composition of (1) $\text{In}_2\text{O}_3\text{:Zn}$, (2) $\text{In}_2\text{O}_3\text{:}(\text{Sn}+\text{Zn})$, (3) $\text{ITO}(10\%)\text{:Ga}$, and (4) $\text{Zn}_{0.5}\text{InSn}_{0.5}\text{O}_{1.5}\text{:Ga}$ films on their power factor: $d \sim 100 \text{ nm}$

This conclusion was based on the results of simulation carried out for $\text{In}_2\text{O}_3\text{:Sn}(10\%)$. In the framework of the electron filtering model (EFM), the basic thermoelectric parameters (i.e., electrical conductivity, mobility, Seebeck coefficient, and power factor) were calculated for ITO in the temperature range of $100\text{--}500^\circ\text{C}$ as a function of potential barrier height at inter-grain boundaries. Ionized impurity scattering (IIS) and polar optical phonon (POP) scattering were considered as the main bulk electron scattering mechanisms, which had the greatest contribution to total relaxation time and mobility in nanoscale indium tin oxide films. Calculations showed that the maximum growth in the power factor due to the filtering effect can be achieved in semiconductors of n -type conductivity if the band bending upwards and the height of potential barrier U_b exceeds only for a few kT the position of the Fermi level in the conduction band. According to our estimations, filtering effect in the $\text{In}_2\text{O}_3\text{:Sn}$ films could give the rise in power factor to more than five times [25]. Results of simulations are shown in Fig. 3.

We assume that in the $\text{In}_2\text{O}_3\text{:Sn}(5\%)$ film it was possible to realize situation, which contributed to the maximum impact of filtering effect on the efficiency of thermoelectric conversion. We believe that the formation of the optimal band bending at

the interface between crystallites in the $\text{In}_2\text{O}_3\text{:Sn}$ film is a result of combined action of two factors. The first one is the segregation of the tin atoms in the surface layer, which is accompanied by the accumulation of charged defects in the structurally distorted surface region, and the second one is oxygen chemisorption on the surface of the $\text{In}_2\text{O}_3\text{:Sn}$ grains, which is accompanied by the capture of electrons from the conduction band.

Our studies also showed that the real use of nanostructured films in thermoelectric generators and refrigerators designed for application at high temperatures might be accompanied by difficulties that could lead to limitations in their use. First of all, it concerns the stability of nano-scaled film's parameters.

As it was mentioned before, the increase in operating temperature up to 1000°C is the easiest method of increasing the efficiency of the thermoelectric conversion. However, our study showed that nano-scaled $\text{In}_2\text{O}_3\text{:Sn}$ films did not possess high stability at such high temperatures. It was found that long-term exposure of the $\text{In}_2\text{O}_3\text{:Sn}$ films already at 700°C led to a 30% decrease in PF [10]. This means that for successful commercial application of $\text{In}_2\text{O}_3\text{:Sn}$ films in high-temperature thermoelectric converters, it is necessary to solve the problems associated with improving high temperature stability of this nano-scaled material's parameters.

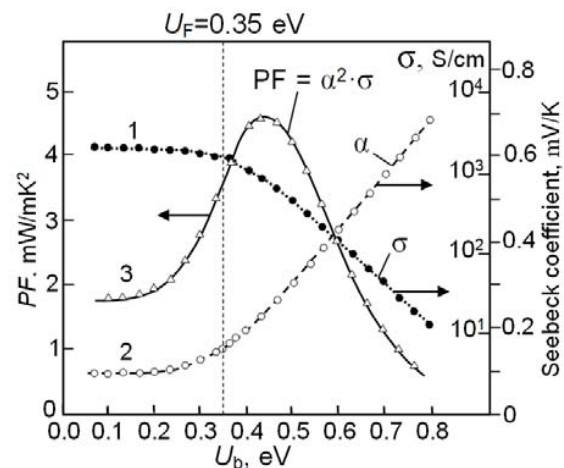


Fig. 3 Influence of filtering effect on (1) conductivity, (2) Seebeck coefficient and (3) power factor of $\text{In}_2\text{O}_3\text{:Sn}(10\%)$ in dependence on the height of the potential barrier at the inter-grain boundaries: $T=500^\circ\text{C}$

Generally, an improvement in the structural stability of the metal oxide is being solved by introducing a second component in the metal oxide, which due to segregation at the surface of the crystallites prevents interaction between crystallites and their coalescence [26]. This means that if one can find a suitable impurity, it would be possible to stabilize the structure and properties of nano-scaled materials. However, the experiment showed that the introduction of the second component cannot completely prevent the growth of crystallites during high temperature annealing. Doping only reduces the possible structural changes in metal oxides [26]. In

addition, there is no certainty that the doping with additive, which contributes to the stabilization of the film structure, will not be accompanied by deterioration of the initial thermoelectric characteristics. For example, doping can be accompanied by the growth of the concentration of structural defects and the decrease in the mobility of charge carriers. Significant change in the potential barrier height at the inter-grain interface is also possible [27]. Therefore, we think that more promising direction is to find conditions under which the maximum influence of the filtering effect on the efficiency of thermoelectric conversion may be realized in the range of intermediate temperatures. In this case, the metal oxides may be at a temperature at which the structure of the nano-scaled films will be stable. This means that nanoscale films in addition to high efficiency of thermoelectric conversion will be also able to provide stable operation of thermoelectric generators.

Studies showed that in the range of intermediate temperatures these conditions can be created through a changing either the temperature or the surrounding atmosphere. It is known that the chemisorption mechanism controls the formation of a potential barrier on the surface of metal oxides in the temperature range of 200–600 °C [28], [29]. This means that the height of potential barrier at the surface of crystallites depends on the concentration of chemisorbed oxygen, which captures electrons from the conduction band of the metal oxide. Thereby, through the change in concentration of oxygen chemisorbed on the surface of metal oxide, it is possible to influence the height of the inter-grain potential barrier and thus control the efficiency of thermoelectric conversion. Our experiments confirm this assumption.

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