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CRYSTALLIZATION BEHAVIOR AND ELECTRICAL PROPERTIES OF InSe AND In_2Se_3 THIN FILMS PREPARED BY THERMAL EVAPORATION

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ABSTRACT
In recent years, semiconductors based on indium selenide have attracted significant attention in advanced research due to their unique electronic properties. Characteristics such as low effective electron mass, high thermal conductivity, and high photosensitivity are particularly pronounced in two-dimensional (2D) InSe nanocrystals. However, challenges associated with their large-scale fabrication limit their application in industrial technologies. In this work, the crystallization of amorphous In_xSe_y thin films deposited by thermal evaporation is investigated through annealing in an inert gas atmosphere to obtain large-area InSe and In_2Se_3 thin films. It is shown that annealing films with an In/Se stoichiometry of 50/50 at 350 °C leads to the formation of the β-InSe phase, whereas films with a stoichiometry of 40/60 form a two-phase system consisting of β-In_2Se_3 and γ-In_2Se_3 under the same conditions. The kinetics of electrical resistance during film crystallization is analyzed. Measurements of the temperature dependence of electrical resistance reveal a sharp decrease in the resistance of the InSe film near 170-180 °C, while the In_2Se_3 film exhibits a two-step decrease at approximately 150-190 °C and 270 °C. The results demonstrate the potential of these thin-film phase change materials for application in memory devices.

INTRODUCTION

The indium-selenium phase diagram contains many thermodynamically stable crystalline phases with different stoichiometries, among which InSe and In_2Se_3, are of particular interest due to their unique electronic properties (Bergeron et al., 2020; Günkel et al., 2025; J. Li et al., 2021; Wojnar et al., 2025). High-temperature In_2Se_3 crystal is known for its ferroelectric properties (W. Zheng et al., 2025; X. Zheng et al., 2022), whereas InSe is considered a promising material for optoelectronic applications due to its high charge carrier mobility (Zhao et al., 2020). The band gap of single-layer InSe crystal is approximately ~1.3 eV (Mudd et al., 2013), while for In_2Se_3 crystal this value can vary from 1.3 to 1.9 eV depending on the polymorphic phase. (Chanchal et al., 2022). In general, In_2Se_3 can exist in stable layered α-, β-, and δ- phases, as well as in a non-layered, wurtzite-like γ-phase (W. Li et al., 2018; W. Zheng et al., 2025). In contrast, crystalline InSe has only a layered structure and can exist in α, β, ε, and δ polytypes depending on the stacking sequence of the layers. Within each layer, In and Se atoms are covalently bonded, while adjacent layers are held together by weak van der Waals interactions.

Atomically thin layers of two-dimensional (2D) In_xSe_y nanocrystals can be obtained by mechanical exfoliation. Due to quantum confinement effects, the electronic properties of these 2D crystals differ significantly from those of their bulk counterparts. For instance, it has been demonstrated that the band gap of monolayer InSe crystal increases to ~2.6 eV (Bandurin et al., 2017). Consequently, it paves the way to obtain InSe crystals with the desired electronic structure by controlling the number of layers.

In_xSe_y crystals have attracted attention in the field of optoelectronics, and in recent years their application as functional elements of electrical and optical memory devices has been extensively investigated. The heating-induced transformation of the low-temperature α-In_2Se_3 crystal into the β-In_2Se_3 or γ-In_2Se_3 phases leads to a dramatic change in the material's electrical properties. Based on this phenomenon, prototypes of electrical memory devices have been developed (Choi et al., 2017; W. Zheng et al., 2025).

In the context of development of electrical and optical memory cells, thin In_xSe_y films produced by physical vapor deposition (PVD) are of particular interest (Bergeron et al., 2020; Chanchal et al., 2022; J. Hossain, M. Julkarnain, 2013; Jeengar et al., 2023). This method enables the production of amorphous (a-In_xSe_y) thin film over a large area, which structural and electrical properties can be tuned through post-deposition thermal treatment. Depending on the annealing condition the various crystalline phases can form, leading to significant changes in the electrical properties of the film. Therefore, a comparative analysis of the films with different stoichiometry In_xSe_y under identical annealing and measurement conditions can provide a better understanding of the differences in their functionality, including the evolution of electrical resistance associated with the structural transformation under heat treatment.

In this study, amorphous a-InSe and a-In_2Se_3 thin films were obtained by thermal evaporation of pre-synthesized bulk InSe and In_2Se_3 polycrystals in a vacuum. A comparative study of their crystallization behavior during annealing in an inert atmosphere was also carried out together with an analysis of the temperature dependence of electrical resistance during the heating.

MATERIALS AND METHODS

InSe and In_2Se_3 crystals were synthesized by melting metallic In and elemental Se in a vacuum at a temperature of 860 °C. First, In and Se with a purity of 99.9% were placed in a quartz ampoule at the required mass ratio and hermetically sealed under high vacuum (10^{-3} Pa). The ampoule was then placed in the active zone of the heating furnace described in our previous paper (Oman et al., 2025) and slowly heated. To ensure proper mixing of the molten elements, a vibration motor was connected to the quartz tube in which the ampoule was placed. The formation of high-quality crystals was ensured by a controlled decrease in the temperature of the ampoule with the melt down to 500 °C at a rate of 1–2 °C per minute, followed by further natural cooling to room temperature.

In the next stage, thin InSe and In_2Se_3 films were obtained by thermal evaporation under high vacuum (~10^{-4} Pa) using a resistive heating technique. As a heating stage we used tungsten plates coated with an alumina (Kurt J. Lesker). Such heater minimizes direct contact between the InSe polycrystals and heating material, i.e. metallic tungsten. As a substrate, a high-transparency KU-1 quartz glass (2×2 cm) and a monocrystalline silicon wafer (Si (100), 5-10 Ω×cm, 1×1 cm, Zhejiang Lijing Optoelectronics Technology Co., Ltd.) were used. Before the deposition, the substrates were cleaned in isopropanol using ultrasonic treatment. The deposition was carried out without heating a substrate holder. The distance between evaporation source and the substrate holder was fixed at 10 cm.

The structure of the obtained materials was studied by Raman spectroscopy (Solver Spectrum, NT-MDT) using 633 nm excitation laser source and X-ray structural analysis with the

CuKα monochromator (Rigaku MiniFlex 600), while their elemental composition was analyzed using energy dispersive spectroscopy (EDS) based on SEM with EDAX device (Quanta 3D 200i, FEI company). To determine the film thickness, a scratch was made on the surface of films, then the step edge between film and substrate was scanned using atomic force microscopy (AFM).

The annealing of the as-deposited amorphous thin films was carried out in an ultra-high purity argon (Ar, Ihsantechnogaz, 99.999%) gas atmosphere at 350 °C for a 20 min with the heating rate of 2°C /min. The temperature dependence of the electrical resistance of thin films was studied in situ during the annealing. The measurements were performed in a sealed chamber equipped with two stainless steel needle type electrodes, a heating stage and a gas flow system. The electrodes were brought into direct contact with the film surface, while the distance between the electrodes was fixed at 2 mm. The measurements were carried out using a Keithley 6485 picoammeter connected to computer for automated recording of the electrical current as function of the temperature.

RESULTS AND DISCUSSION

To obtain thin-film samples of InSe and In_2Se_3, bulk polycrystalline InSe and In_2Se_3 were first synthesized, and then the resulting crystals were thermally evaporated onto the selected substrates in high vacuum.

Characterization of bulk InSe and In_2Se_3 crystals

InSe and In_2Se_3 crystals were initially synthesized by melting In and Se elements in a sealed ampoule at a temperature of 860 °C. The Raman spectrum of the obtained InSe crystal is characterized by three peaks at 114, 175 and 224 cm^{-1}. These peaks correspond to the A_1, E'' and A'_1 phonon vibrational modes characteristic of the β-polytype of the InSe crystal (Fig. 1a).

The Raman spectrum of the stoichiometric In_2Se_3 sample is characterized by several distinct peaks at 87, 104 (A_1 (LO+TO)) and ~187 (A_1 (LO)) cm^{-1} (Fig. 1b). It is evident that the broad peak at ~187 cm^{-1} consists of several components. In general, this set of peaks corresponds to the vibrational modes of the α-In_2Se_3 phase. A low-intense peak at ~250 cm^{-1} in the spectrum indicates the presence of a small amount of free selenium, while the peak at 150 cm^{-1} is associated with the γ-phase of the In_2Se_3 crystal.

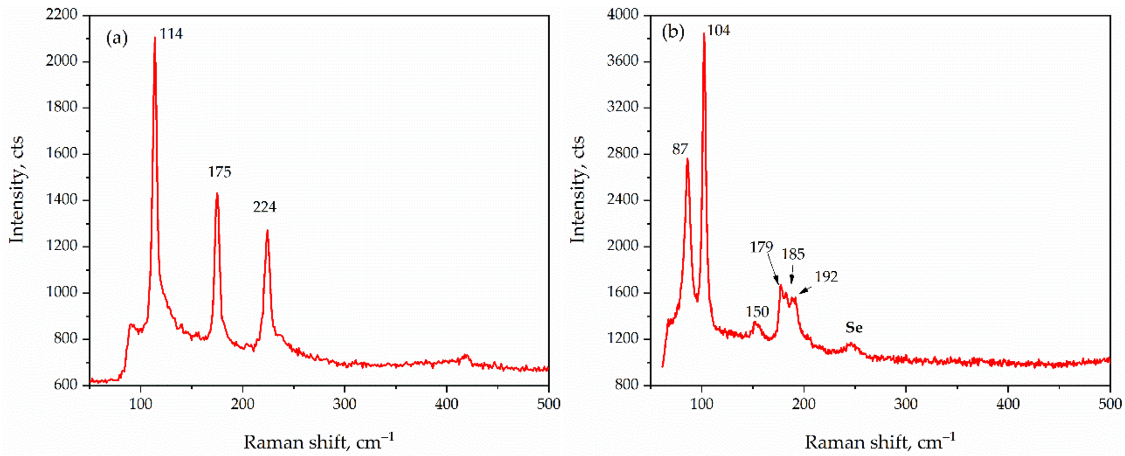

Figure 1. Raman spectra of InSe (a) and In_2Se_3 (b) crystals

The results of EDS analysis along with SEM images of the obtained crystals are presented in Table 1 and Figures 2(a) and 2(b). It can be seen from the SEM images that both InSe and In_2Se_3 crystals possess a layered structure.

Table 1. Elemental composition (EDS analysis) of InSe and In_2Se_3 samples

Element	InSe crystal	InSe thin film	In_2Se_3 crystal	In_2Se_3 thin film
In, at. %	48.81	49.68	40.54	40.49
Se, at. %	51.19	50.32	59.46	59.51

The structure of the synthesized crystals was characterized by X-ray diffraction (XRD) analysis using a Rigaku X-ray analytical system with a CuKα monochromator. The XRD pattern of the InSe crystal (Figure 3(a)) confirms a hexagonal crystal structure belonging to the $P6_3/mmc$ space group with lattice parameters: a = b = 4.0 Å, c = 16.6 Å. The X-ray diffraction pattern of the In_2Se_3 crystal also indicated a formation of hexagonal structure (space group $P6_3/mmc$) with lattice parameters a = b = 4.0 Å, c = 19.2 Å. Additional reflections corresponding to the (100) and (101) planes of elemental selenium were also observed. Phase identification was performed using the PDXL software package (Rigaku) by comparison of experimental diffraction patterns with reference ICDD PDF database card. The card number is shown at the inset of the figure.

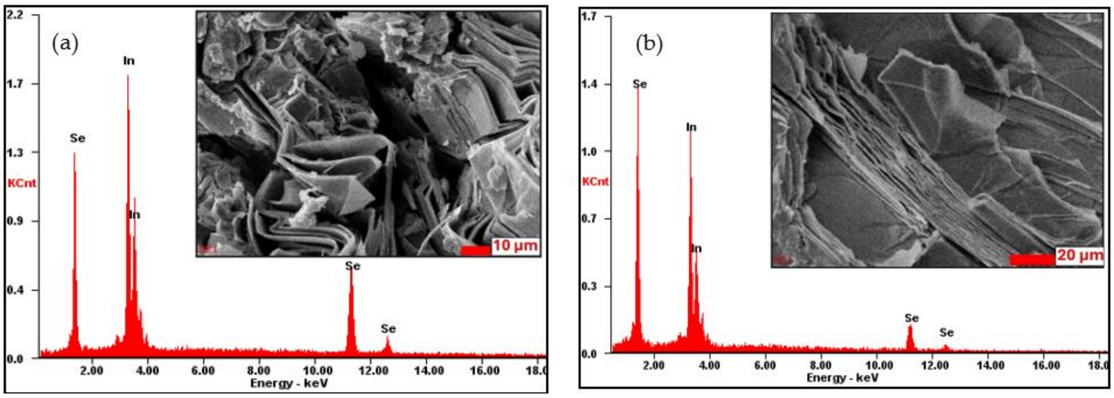

Figure 2. EDS spectra and SEM images of InSe (a) and In_2Se_3 (b) crystals

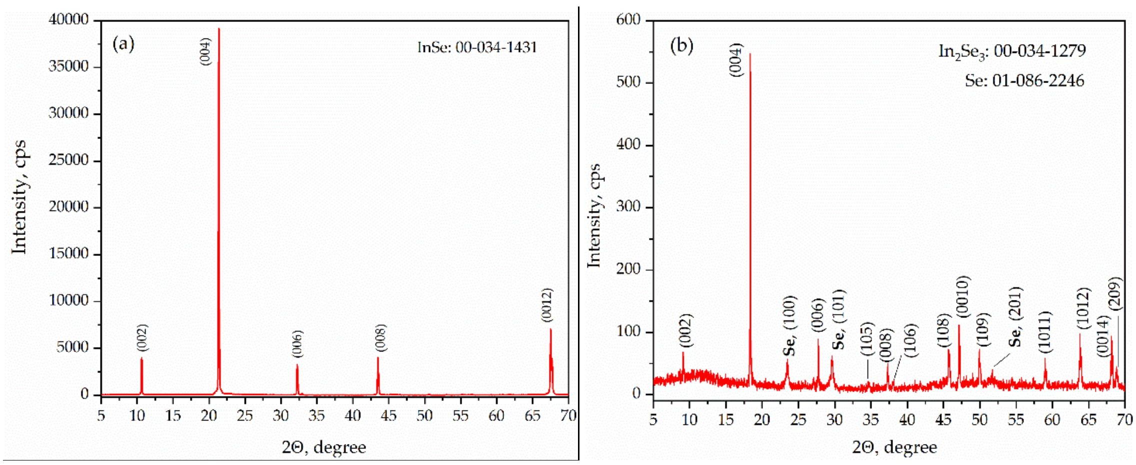

Figure 3. XRD patterns of InSe (a) and In_2Se_3 (b) crystals

Characterization of InSe and In_2Se_3 thin films

Pre-synthesized bulk InSe and In_2Se_3 crystals were thermally evaporated on highly transparent quartz substrates in high vacuum (~10^{-4} Pa) to obtain thin films. The evaporation was carried out using a tungsten plate coated with an aluminum oxide layer. The thicknesses of the deposited films were 215 nm for InSe and 450 nm for In_2Se_3. The halo-type of XRD patterns and the absence of distinct peaks in the Raman spectra indicate an amorphous structure of the obtained films (Fig. 4-5).

Thermal treatment of as-deposited amorphous thin films in inert atmosphere leads to their crystallization. The peaks at 114 cm^{-1}, 175 cm^{-1} and 225 cm^{-1} appearing in the Raman spectrum of the InSe film (c-InSe), annealed at 350 °C for 20 min in Ar, are characteristic of the β-InSe polytype (Fig. 4a). This result was also confirmed by the XRD analysis data - the diffraction pattern shown in Fig. 5(b) corresponds to a hexagonal structure with lattice parameters a=3.99 Å and c=16.62 Å (Space group: 194:P63/mmc, No. 00-034-1431). The EDS analysis showed that the stoichiometry of the films was preserved similar to that of the initial crystals (Table 1). Thus, proposed technique for production of crystalline InSe films over a large area is considered feasible and effective. It is important to note that annealing the compounds based on In-Se at temperatures other than 350 °C leads to the formation of various phases, In_4Se_3 or In_3Se_4, in the film. Experiments have shown that amorphous a-InSe film annealed at the temperature interval of 300-350°C transforms into biphasic InSe/In_4Se_3 system.

As-deposited amorphous In_2Se_3 thin film was annealed at the same conditions. The appearance of a single peak at 148 cm^{-1} in the Raman spectrum of the annealed crystalline (c-In_2Se_3) thin film shown in Fig. 4(b) indicates the formation of a γ-phase In_2Se_3 crystal. However, XRD pattern shown in Fig. 5d reveals reflections from the planes of the γ-In_2Se_3 and β-In_2Se_3 phases. These findings can be explained by the fact that the temperature of 350 °C is not sufficient for the formation of a uniform single-phase γ-In_2Se_3 crystalline film. It is known that In_2Se_3 crystals typically transform from the α-phase to the β-phase at 200 °C, while further transformation from the β-phase to the γ-state may start at a temperature of 350°C (Han et al., 2014).

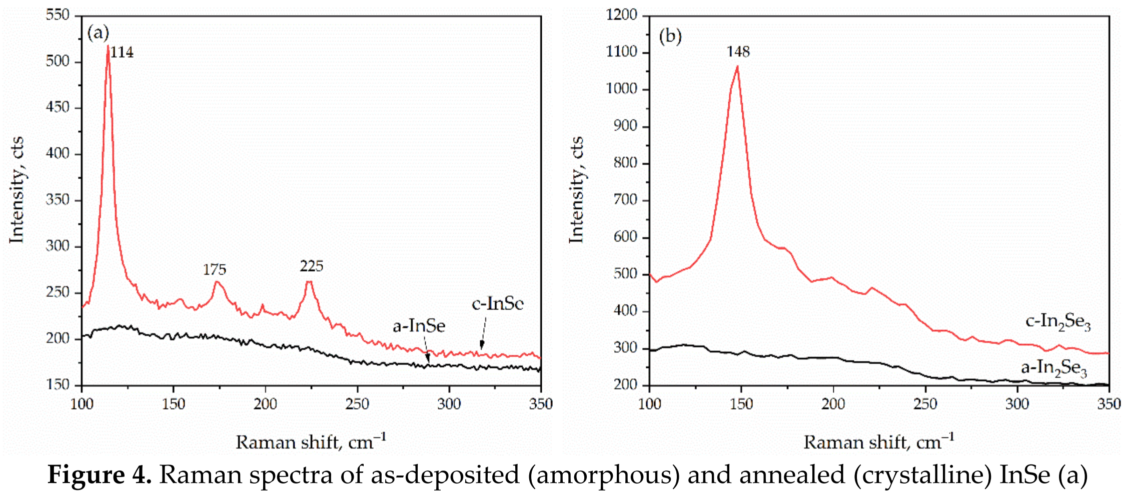

Figure 4. Raman spectra of as-deposited (amorphous) and annealed (crystalline) InSe (a) and In_2Se_3 (b) thin films

Structural transformations are typically accompanied by a change in the physical properties and thermodynamic state of the material. In our case, the crystallization of the films induces a sharp change in their electrical resistance. Therefore, one of the key tasks of this work is to study the temperature dependence of the electrical resistance of thin InSe and In_2Se_3 films. Plots of the obtained dependencies are presented in Figure 6. As can be seen from the figure, the resistance of InSe film demonstrates a sharp decrease by 5 orders of magnitude at around 170-180 °C from ~10^9 Ω to ~10^4 Ω. This can be explained by the formation of conductive channels in the film because of the structural transformation, i.e. amorphous-to-crystalline phase transition.

Several measurements of resistance under heating showed the transition in the temperature indicated interval. These observations correlate with the many reports of other authors. Numerous studies have shown that this transition in InSe films typically occurs in the range of 120-200 °C, depending on the film thickness (Fedyanina et al., 2025; J. Hossain, M. Julkarnain, 2013). The presented experimental curve is typical for films with thickness of 100-300 nm. Raman spectroscopy and XRD analysis of annealed InSe thin film revealed the formation of crystalline β-InSe, suggesting that the resistance drops correlate with the crystallization of the film.

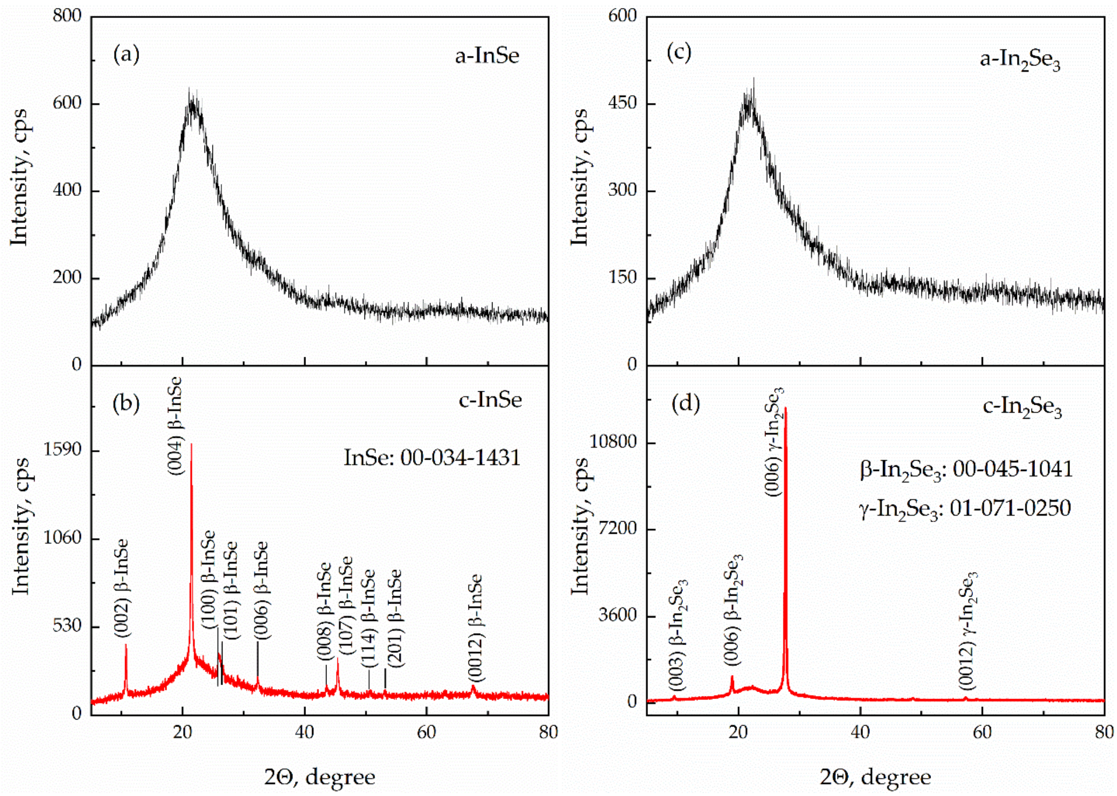

Figure 5. XRD patterns of as-deposited (amorphous) (a,c) and annealed (crystalline) (b,d) InSe and In_2Se_3 thin films

The same analysis for In_2Se_3 thin film revealed a two-step transition from a high-resistance state to a low-resistance state. Initially, the resistance decreases from ~10^{12} Ω to ~10^7 Ω near 150-190 °C, followed by a second transition near 270 °C, in which the resistance further decreased to ~10^5 Ω. The observed behavior may indicate a more complex crystallization process involving several structural transformations. The first transition step is possibly associated with the crystallization of the material (formation of low temperature phases α and β In_2Se_3), and the second step can be explained by the transition of already crystalline film from the β -In_2Se_3 phase to the γ -In_2Se_3 phase. Overall, a two-step drop in the electrical resistance of thin In_2Se_3 films was previously demonstrated by Xu et al. in 2023 and multi-level electrical memory cells were designed based on this system. According to the ex-situ Raman and XRD results, the annealed film contains γ-In_2Se_3 and β-In_2Se_3 phases, which suggests that the electrical resistance evolution is related to phase transformation.

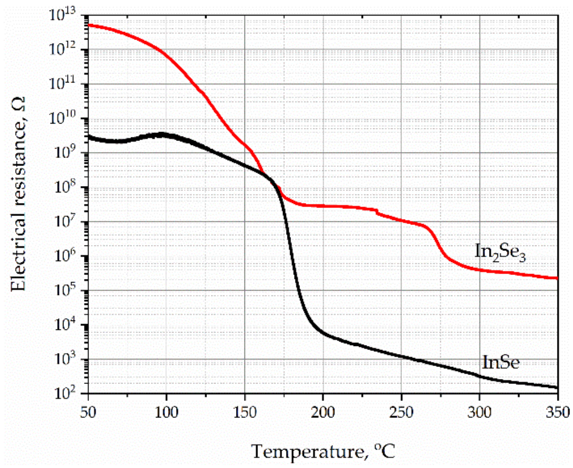

Figure 6. Temperature dependence of the electrical resistance of InSe (black) and In_2Se_3 (red) thin films

CONCLUSIONS

This study presents a comparative analysis of the crystallization conditions of InSe and In_2Se_3 thin films deposited by thermal evaporation. Annealing of the as-deposited thin films in an inert gas atmosphere at 350 °C for 20 min results in the transformation of amorphous InSe into crystalline β-InSe, while amorphous In_2Se_3 crystallizes into two-phase system consisting of β- and γ- In_2Se_3. These results demonstrate that precise control of annealing conditions is essential for obtaining crystalline indium selenide thin films with the desired phase states.

Since the key feature of these materials is a pronounced change in physical properties during crystallization, an analysis of the temperature dependence of the electrical resistance of the films was carried out. The R(T) measurements reveal that the resistance of InSe film decreases sharply by several orders of magnitude at ~170°C, whereas In_2Se_3 film exhibits a two-step decrease in the range from 170°C and 270°C. These findings further highlight the potential of In-Se thin films not only for optoelectronics but also for the development of electrical memory cells.

CONFLICT OF INTEREST The authors declare no conflict of interest.

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