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Structural Characterisation of Grown InSe:Mn Semiconductors and Effect of Doped Manganese

Büyütülen InSe:Mn Yarıiletkenlerin Yapısal Karakterizasyonu ve Katkılanan Manganezin Etkisi

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Abstract

Scientific studies on binary semiconductors have been going on for half a century. The growth and research of semiconductors have contributed greatly to the advancement of semiconductor technology. Indium and selenium were synthesized from elements in stoichiometric ratios and doped with manganese. InSe and InSe:Mn single crystals were successfully grown by the Bridgman/Stockbarger crystal growth method. Since the surfaces of the grown samples do not contain contamination, chemical contamination was not caused by chemical treatment. Since the grown semiconductors have a layered structure, the samples were easily prepared for characteristic analyses along the 001 planes. The structure of the InSe and InSe:Mn semiconductors was analysed using x-ray diffractometers and energy dispersive X-ray techniques. An important study has been carried out on the structural properties of the grown InSe and InSe:Mn semiconductors. The hexagonal structure of the InSe semiconductor with lattice parameters a = b = 4.025 Å and c = 16.732 Å was confirmed with the help of X-ray diffraction. III-VI semiconductors are used in visible and infrared light-emitting diodes, infrared detectors, converters, amplifiers, optical parameter oscillators, and far infrared generator.

Keywords: Crystal Growth, InSe, InSe:Mn, Bridgman/Stockbarger Method, Structural Characterisation.

Öz

İkili yarıiletkenler üzerine yapılan bilimsel çalışmalar yarım asırdır devam etmektedir. Yarıiletkenlerin, büyütülmesi ve araştırılması yarıiletken teknolojisinin ilerlemesine büyük katkı sağlamıştır. İndiyum ve selenyum elementlerden stokiyometrik oranlarda sentezlenmiş ve manganez katkılanmıştır. InSe ve InSe:Mn tek kristalleri, Bridgman/Stockbarger kristal büyütme yöntemiyle başarıyla büyütülmüştür. Büyütülen numunelerin yüzeyleri kontaminasyon içermediğinden kimyasal işleme tabi tutulmayarak kimyasal kirlenmeye sebep olunmamıştır. Büyütülen yarıiletkenler tabakalı bir yapıya sahip olduğundan, numuneler kolayca (001) düzlemleri boyunca karakteristik analizler için hazırlanmıştır. InSe ve InSe:Mn yarı iletkenlerinin yapısı X-ışını difraktometresi ve enerji dağılımlı X-ışını teknikleri kullanılarak analiz edilmiştir. Büyütülen InSe ve InSe:Mn yarıiletkenlerin yapısal özellikleri üzerine önemli bir çalışma yapılmıştır. InSe yarıiletkeninin a = b= 4.025 Å ve c = 16.732 Å kafes parametrelerine sahip olan altıgen yapısı, X-ışını kırınımı yardımıyla doğrulanmıştır. III-VI yarıiletkenler, görünür ve kızılötesi ışık yayan diyotlarda, kızılötesi detektörlerde, dönüştürücülerde, yükselteçlerde, optik parametreli osilatörlerde ve uzak kızılötesi jeneratörlerde kullanılmaktadır.

Anahtar Kelimeler: Kristal Büyütme, InSe, InSe:Mn, Bridgman/Stockbarger Yöntemi, Yapısal Karakterizasyon

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Introduction

In recent years, solid and thin films grown by various growth techniques have attracted great interest in producing technological devices and solar cells. Various deposition techniques are employed for preparing thin films, including vacuum evaporation (El-Sayed, 2003), flash evaporation (Julien et al., 1990), chemical vapor deposition (Park et. al., 2003), and van der Waals epitaxy (Lang et. al., 1996). Among these methods, the electrodeposition technique stands out as an inexpensive, simple, and low-temperature option capable of producing high-quality films for device applications such as heterojunction devices and switching devices.

InSe single crystals have been grown by the Bridgman-Stockbarger method, starting from stoichiometric and non-stoichiometric melts (De Blasi et. al., 1982). Semiconductors grown by the Bridgman/Stockbarger technique usually belong to the b and g modifications. Their unit cells crystallize in a hexagonal and rhombohedral structure (Camassel et. al., 1978; Gürbulak 2004). InSe belongs to a large class of layered semiconductors in which a prototype of covalent or ionic forces extends in two dimensions instead of being three-dimensionally bonded as in group III-VI or III-V semiconductors (El-Moiz et al., 1993).

There are few studies available on the impurity levels in p-type InSe, with only group II elements Zn and Cd being used as dopants. Investigations into the impurity levels in Zn and Cd-doped InSe have employed deep-level transient spectroscopy (DLTS), photoluminescence (PL), and Hall-effect measurements. The 1.17 eV emission band observed in the Zn-doped sample can be explained by a self-activated luminescence process, which is described by a configurational coordinate model (Ikari et. al., 1981). III-VI dilute magnetic semiconductors are formed by bonding a group III element to a group VI element and replacing some group III atoms with transition metal atoms. These transition metal atoms impart magnetic moments to the sample. III-VI dilute magnetic semiconductors are of great interest for electro-optical applications due to their high nonlinear optical properties. In InSe with 1.25 at % Mn, two magnetic subsystems containing Mn ions inside ionic-covalent layers and inside the interlayer van-der-Waals gap were discovered (Suhre et al., 1997; Errandonea et al., 1999; Watanabe et al., 2003; Slyn’ko et al., 2005a; Slyn’ko et al., 2005b).

For different studies, single crystals were grown by the Bridgman method from a non-stoichiometric melt with the composition $In_{1.03}Se_{0.97}$. Indium monoselenide single crystals were combined with magnesium from the vapor phase. Electron concentration, electron Hall mobility, anisotropic electrical conductivity, and thermoelectric power of the InSe semiconductor were measured as a function of temperature (Zaslonkin et al., 2006). The crystals have a g-polytype structure and n-type conductivity. In indium monoselenide, the interatomic bond between the layers is ionic-covalent, while the layers are connected by weak van der Waals forces (Kovalyuk et al., 2009). Different studies have been carried out with doped and undoped InSe semiconductors (Kaminskii, 2009; Boledzyuk et al., 2014; Ertap et al., 2019; Sang et al., 2019; Emir et al., 2024).

The aim of this research is to grow pure and doped InSe semiconductors by a modified Bridgman-Stockbarger method. On the other hand, to determine the structural changes induced by the doped manganese (Mn) element in the InSe compound.

Growth and Structural Characterization

The Bridgman-Stockbarger method is a growth technique widely used in the growth of semiconductors. The Bridgman-Stockbarger method is an improved version of the Bridgman method. The Bridgman-Stockbarger method involves cooling a molten alloy very slowly to form a single crystal. As the alloy solidifies, the temperature gradient within the furnace is used to promote controlled crystallization. The sample to be grown is placed in a crucible, usually made of graphite, alumina, or quartz materials that can withstand high temperatures. The vertical temperature gradient of the furnace is determined by the semiconductor to be grown. Growth furnaces usually have two zones. The upper zone is hotter and keeps the material melting, while the lower zone is cooler. The growth furnace heats the elements above the melting point. The temperature of the furnace is gradually reduced. During the formation of the compound, it is very important to control the sensitivity of the temperature change and the cooling rate. It provides a controlled solidification front that moves slowly from the bottom of the to the top. This causes the melting material to solidify from bottom to top. The slow cooling process helps to reduce impurities and defects in the crystal.

Considering the studies (Shih et. al., 1986; Gürbulak, 2024), it was decided to grow InSe and InSe:Mn crystals using a single ampoule. Indium and selenium are prepared in ratios obtained by stoichiometric calculations. The lower end of the growth ampoule is shaped to be oval according to the layered structure, and the elements (In, Se, and Mn) are placed inside the bulb. The air in the growth ampoule is reduced to 10^{-6} Torr, and the end of the ampoule is sealed under vacuum. Movement up and down in the horizontal growth furnace is required to ensure homogeneity of the ampoule, InSe, and InSe:Mn-doped (0.003) mixture. InSe and InSe:Mn semiconductors were grown by a modified Bridgman-Stockbarger method under specified conditions.

It is very important that binary compounds are grown and the grown semiconductor is of a quality that can be used in scientific studies. In general, the Bridgman-Stockbarger method is a fundamental technique in materials science and engineering. It plays a critical role in the development of high-quality semiconductors for various scientific applications.

The crystal growth process was started after the pre-reaction process of the crystal growth process was completed in approximately 52 hours. It was thought that the In, Se, and Mn mixture would increase the vapor pressure of selenium by chemical reaction as a result of thermal conductivity. The lower and upper zone temperatures of the growth furnace were increased to 640 °C within 30 hours. It was kept at 640 °C for 15 hours. Since the exothermic chemical reaction between the elements In, Se, and Mn continues at 640–650 °C, a long period of time is needed to eliminate the risks such as ampoule explosion and cracking. Again, the alloy was heated to 1050 °C in 25 hours and kept for 12 hours. To ensure homogeneous distribution of In, Se, and Mn, the mixture was agitated by moving the growth furnace up and down at an angle of about 50° for 10 hours. The growth furnace was fixed at an angle of 65-70° to the horizontal.

The temperature in both zones of the crystal growth furnace was first kept constant at 1050 °C for 20 hours. Then the temperature was reduced to 750 °C for 80 hours. The upper zone temperature of the growth furnace was kept constant at 750 °C for 48 hours. It was reduced to 600 °C for 70 hours, 250 °C for 50 hours and 30 °C for 15 hours. The oven bottom zone temperature was reduced to 600 °C in 48 hours, 450 °C in 70 hours, 250 °C in 50 hours, 30 °C in 15 hours, and the growth process was terminated. Thus, using the same environment and the same growth temperature program, the InSe and InSe:Mn semiconductor pre-reaction processes and growth processes were completed in approximately 17 days and 19 hours, including the reaction.

At the end of the growth process, the compounds were removed from the growth furnace. In order to prevent any strain or deformation of the compound in the ampoule, it was removed from the ampoules with the help of a suitable cutter. The compounds should be kept in a very clean environment to prevent external contamination of the samples (Gürbulak, 1997; Gürbulak, 2024). This process is very important for the use of semiconductors in structural, optical, electrical, and magnetic applications.

Experimental results

In the crystal growth laboratory, the modified Bridgman-Stockbarger method was used to grow InSe and InSe:Mn solid compounds. The first basic step in obtaining high-quality binary crystals is to ensure that the basic elements in the structure are of 6N purity. Indium, selenium, manganese doped (0.03%) elements were precisely weighed on a five-digit balance to grow InSe single crystals.

Basic indium and selenium elements were obtained with a purity of about 99.999%, and the growth of InSe and InSe:Mn semiconductors was attempted. The stoichiometric ratios of the basic InSe compound were determined to be 52% indium and 48% selenium. The total mass of the chemical elements was determined to be about 50 grams. InSe crystals were grown in our crystal laboratories using a modified horizontal Bridgman-Stockbarger method. Since the sample ingots have a layered structure, they were easily separated from the layers with plastic tweezers in thicknesses of 50 – 60 mm for use in the study. Photographic views of the InSe:Mn solid crystals grown in Figure 1 are given.

As can be seen from the photographic appearance of the InSe:Mn crystals grown in Figure 1, they have a high quality and shiny surface that is very suitable for structural analysis. The effect of Mn doping on the InSe compound is seen.

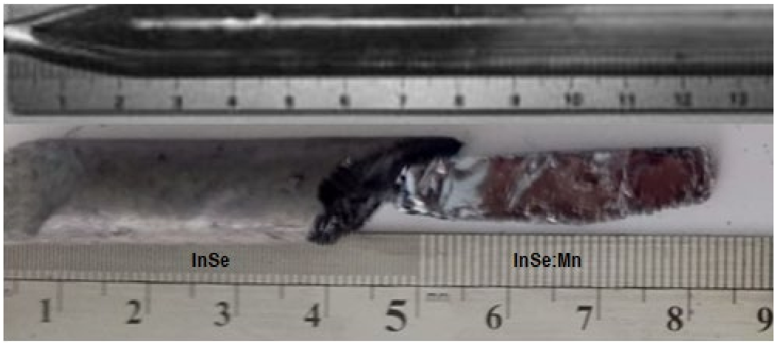


Figure 1. The photographic appearance of InSe:Mn bulk crystals

Experimental results

An important study has been conducted on the structural properties of InSe and InSe:Mn semiconductors grown with the modified Bridgman/Stockbarger technique. It belongs to the family of layered binary semiconductors known as InSe and InSe:Mn compounds. While the bonding in InSe is predominantly covalent between Se atoms and metal cations, weaker van der Waals forces also exist between adjacent layers. Covalent bonding between layers provides stability to the crystal structure, while weaker interlayer interactions allow easy separation along planes. The InSe semiconductor, characterized by anisotropic properties, was examined using X-ray diffraction with a wavelength of 0.154049 nm to determine its crystalline structure. The resulting single crystals were not subjected to any additional annealing. Samples were prepared from InSe and InSe:Mn binary semiconductors with shiny and smooth surfaces. The crystal structure of these samples has been characterized. As a result of the analysis, it was found that the InSe semiconductor has a hexagonal structure with lattice parameters a = b = 4.025 Å and c = 16.732 Å. The crystal structure was found to be hexagonal, which coincides with the study by Viswanathan et. al. (2005). Many scientists have grown InSe single crystals using the Bridgman method and the Bridgman-Stockbarger method and carried out structural analyzes of semiconductors. It is concluded that the grown crystal has a hexagonal structure (Chevy et. al., 1977; Imai et. al., 1981; De Blasi et. al., 1982). XRD analysis of the sample phase composition showed that the hexagonal phase with reduced lattice constants a = 4.0026 Å and c = 16.634 Å is the main phase of InSe (P63/mmc) (Lashkarev et. al., 2007).

X-ray diffraction (XRD) is an important technique used to analyze crystal structures and crystal structural changes. It is used to determine the atomic arrangement of crystal structures. Bragg's Law is expressed by the formula $2d \sin\theta = n\lambda$. X-rays are sent to a sample of crystal. The diffraction pattern resulting from the refraction of these rays by the crystal is analyzed. Diffraction patterns usually appear as peaks. The positions of these peaks provide information about the crystal structure in the sample. The crystallization of microstructures formed at different growth stages was analysed using XRD. The XRD spectra of the InSe and InSe:Mn semiconductors are given in Figure 2. Interplane distance was obtained from d_{hkl} XRD data. The θ-2θ scan data corresponding to (002), (004), (006), and (008) (0012) and (0014) diffraction planes of the InSe:Mn semiconductor were 11.20°, 21.92°, 32.95°, 44.08°, 68.07°, and 81.42°, respectively. Although the intensities of the XRD spectra of the InSe:Mn binary compounds increased compared to the spectra of the InSe compound, no noticeable new peaks were formed due to impurities.

As shown in Figure 2, the XRD peaks in the Mn-doped samples are sharper than those in the undoped sample. The increase in sharpness may be due to the reduction of impurities in the pure material through Mn doping. This can be expected due to the contribution of Mn atoms in InSe. It shows that n-InSe single crystals contain many impurities, which are eliminated by doping with Mn. The doping process can remove impurities between layers and is thought to neutralize existing impurities by forming complexes.

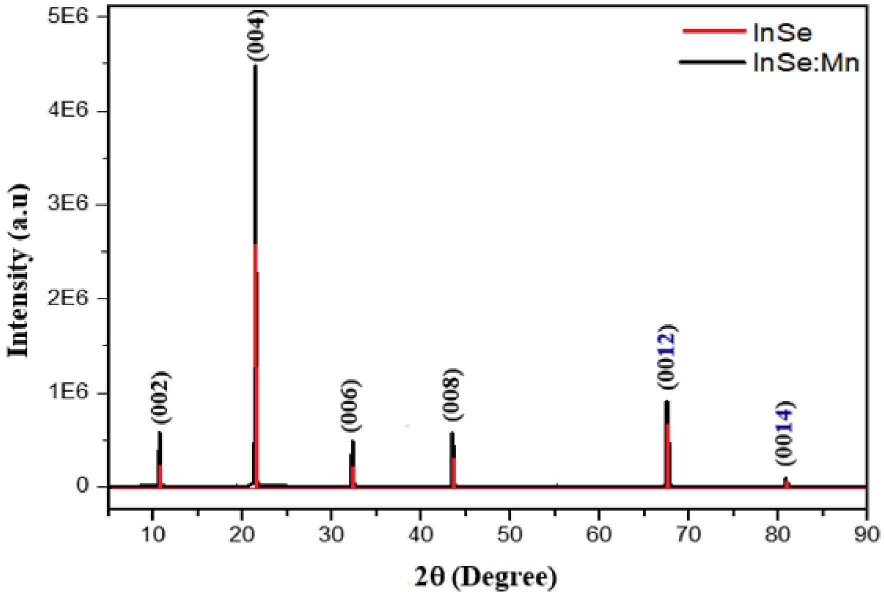


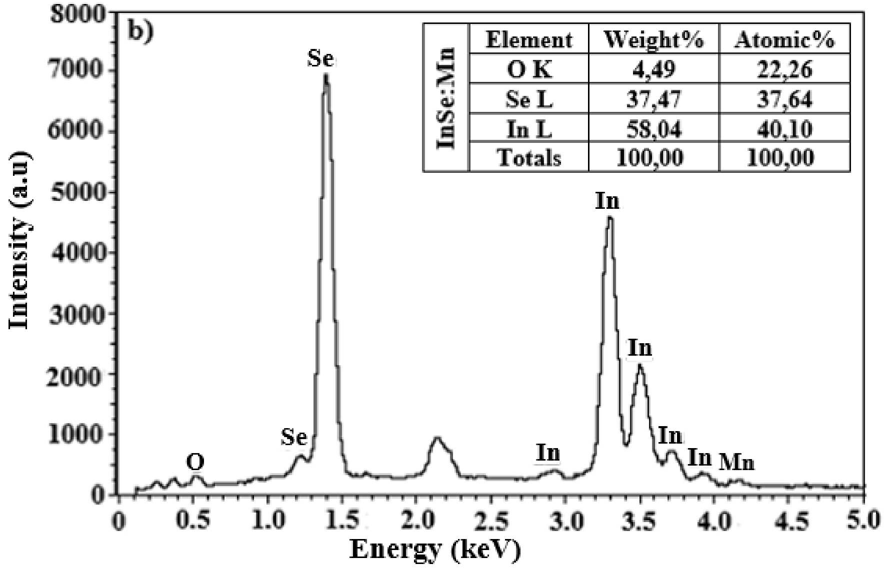
Figure 2. The effect of doping the Mn element on the InSe semiconductor on the peak intensity

If the percentage of Mn dopant ratio is appropriate, the XRD spectra of InSe single crystals increase and become sharper. For doping levels above 0.03 at. %, Mn atoms are predominantly localized between layered packings and can form localized states. In this way, charge carriers recombine intensively. This situation was observed in the study.

Some properties of the grown InSe:Mn compound were realized by interpreting the data taken and calculated with the help of XRD technique. Considering the (004) diffraction plane of the InSe:Mn semiconductor and using the given equations (Williamson, 1956; Cullity, 1972); crystal size (D_{exp}) = 895.75 (Å), dislocation density (ε) = $4.35\text{x}10^{-4}$ ($\text{lin}^{-2}\text{m}^{4}$), residual strain ($\delta$)=$1.27\text{x}10^{14}$ (lin/m^{2}) values were found. It was carried out with the help of high-resolution X-ray diffraction (XRD) data to detect the perfection of the as-grown single crystal.

Energy dispersive X-ray spectrometry (EDX) is an analytical device that can be integrated with an electron microscope. EDX is widely used in many research fields. By measuring the characteristic x-rays of InSe and InSe:Mn semiconductors with the help of EDX, the percentage ratios of the elements in the sample were determined. Energy dispersive x-ray spectrometry data of the Mn-doped InSe semiconductor is given in Figure 3. According to EDX results, InSe and InSe:Mn, In = 55.89%, Se = 38.86% and O = 5.25%, respectively, and InSe:Mn, In = 58.04%, Se = 37.47% and O = Contains 4.49%. The EDX spectrum of the InSe crystal was found to be close to that of the InSe:Mn crystals. The compositions of these samples were characterized by EDX. It has been determined that the Mn effect creates a more homogeneous and high-quality structure in the InSe crystal. It has been observed that the grown InSe and InSe:Mn semiconductors have high quality stoichiometric ratios and homogeneous surfaces. These values are very close to the expected values, and in addition to the formation of some In or Se bonds with O, the effect of added Mn was also observed. Results similar to those obtained from EDX analyzes have been observed in many studies.

This study presents some results of systematic studies. InSe and InSe:Mn single crystals were grown under high vacuum. Single crystals of InSe and InSe:Mn were grown under a high vacuum condition. The structural characterization of these crystals was attempted using X-ray diffraction (XRD) measurements and X energy-dispersive X-ray spectroscopy (EDX). Single crystals of InSe and InSe:Mn were grown using the modified Bridgman-Stockbarger method. The structure and melting points of these InSe and InSe:Mn binary compounds have been determined. The grown samples are shiny and layered and crystallize in hexagonal structures. Interest in the growth and characterization of such derived layered compounds has increased in recent years. Hot probe techniques revealed that both undoped and Mn doped InSe samples are n-type semiconductors. Doping with Mn can change the crystal structure and lead to a new appearance.



InSe:Mn	Element	Weight%	Atomic%
	O K	4,49	22,26
	Se L	37,47	37,64
	In L	58,04	40,10
	Totals	100,00	100,00

Figure 3. EDX data of InSe:Mn crystal grown at room temperature

Conclusion

An important study has been carried out on the structural properties of the grown InSe and InSe:Mn single crystals.The hexagonal structure of the InSe semiconductor with lattice parameters a = b = 4.025 Å and c = 16.732 Å was confirmed with the help of X-ray diffraction. Doped InSe single crystals were grown by the Bridgman method using Mn-containing In-Se alloys. Samples were taken from the ingot and annealed at 595 K for 70 hours. Mn content was determined by X-ray fluorescence analysis. It has been shown that the impurity is distributed unevenly throughout the nugget and the Mn concentration increases at the tip.

XRD investigation of the sample phase composition was conducted, and it was found that the hexagonal phase InSe (P63/mmc) with lattice constants a = b = 4.0026 Å and c = 16.634 Å was the main phase (Lashkarev et. al., 2007). As is known, b-InSe crystallizes in the hexagonal lattice defined as the D^4_{6h} space symmetry group. The lattice constants are a = b = 4.048 Å, and c = 16.930 Å (Man et. al., 1979). Eight atoms in the unit cell of b-InSe belong to two layers with the Se-In-In-Se structure. This study's results are consistent with those of numerous other studies.

InSe are layered III-VI semiconductors. Each of the structures consists of regularly stacked layers consisting of four atomic planes tightly connected according to the Se-In-Se structure. These structures exhibit strong bonding within the layers and very weak bonding between them. Due to this configuration, crystals of these compounds have high polarizability and optical uniformity, with a naturally mirror-smooth surface. The doping of the layered InSe binary compound with manganese may have led to the formation of an ordered atomic structure. Moreover, the complexation of manganese with impurities in the compound may improve the quality of the InSe semiconductor. Carrying out the procedure of a long-term growth program may have led to the arrangement of layers consisting of indium and selenium atomic planes and may have contributed to a more homogeneous formation of the layers.

When added to semiconductors, manganese alters their optical, magnetic, and electronic properties, enabling controlled optimization of these materials. Manganese-doped semiconductors exhibit magnetic and electronic properties. Ideal for use in magnetic memory and spin-based electronic devices. Manganese can alter semiconductors' band structure and carrier density, leading to phenomena such as magnetic phase transitions at low temperatures. These properties significantly affect critical parameters of the material, such as magnetic resistance, and offer great potential for advanced technology applications (Boledzyuk et al., 2014; Sang et al., 2019; Emir et al., 2024). This study and subsequent studies aim to reveal some of these properties.

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Financial Disclosure: This work ''ID 8833, FBA-2021-8833'' and ''FYL-2023-10176, Project ID: 10176 Structural Characterization of $X^{II}In_2Se_4$ (X^{II}: Cu, Mn Fe, Ni, etc.) Ternary Semiconductors Grown by Bridgman/Stockbarger Technique,'' was supported by Atatürk University BAP (Scientific Research Projects)..

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