Annealing and plasma treatment effect on structural, morphological and topographical properties of evaporated $\beta$-In$_2$S$_3$ films

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Keywords: In$_2$S$_3$ films, annealing, plasma treatment, structure, surface topography

Abstract

Indium sulfide (In$_2$S$_3$) is a wide bandgap semiconductor, which is widely used as a window/buffer layer in thin film solar cell applications. In$_2$S$_3$ thin films were deposited using thermal evaporation technique and were annealed in sulfur ambient at 200 °C and 250 °C. Further, these films were treated in inductively coupled argon plasma sputtering with an average argon ion energy of 75 eV for 30 s. The paper presents the effect of Ar-plasma treatment on structure, elemental composition, morphology and topography of In$_2$S$_3$ films and the results were reported. Further, the optimized In$_2$S$_3$ layers were continued for plasma etching process with an average argon ion energy of 25 eV to study the effect of plasma etching duration on the growth of metallic indium nanoparticles over the film surface and the results were discussed in detail.

1. Introduction

Indium sulfide (In$_2$S$_3$) is an n-type semiconductor, which is widely used as a window/buffer layer in thin film solar cell applications due to its wide band gap energy ($\sim$2.2 eV), photoconducting nature and tunable electrical properties [1–5]. It is a promising material in thin film solar cells, an alternative to toxic CdS. It exhibits polymorphic nature, such as tetragonal $\beta$-In$_2$S$_3$ (stable up to 420 °C), cubic α-In$_2$S$_3$ and cubic $\beta$-In$_2$S$_3$ (>$\sim$420 °C) and γ-In$_2$S$_3$ (>750 °C) with trigonal structure depending upon the growth conditions [6]. Among these phases, $\beta$-In$_2$S$_3$ is the stable phase at room temperature. Recently, Spiering et al [7] reported, a record cell efficiency of 18.2% using evaporated In$_2$S$_3$ thin film as a buffer layer in Cu(In, Ga)Se$_2$ thin film solar cells.

Several methods have been used to modify the physical properties of In$_2$S$_3$ thin films, such as adding impurity elements in the material [8, 9], post-deposition annealing [10–13], treating with gas plasma [14] and swift heavy ion or gamma irradiations [15, 16]. It is known that plasma treatment of binary metal chalcogenides and complex compounds based on them in argon plasma leads to new phenomena associated with nanostructuring of the surface [17]. This is due to the presence of volatile chalcogen and of low melting point metals that enhance the effect of nanostructure formation. Similar processing in pure argon plasma for creation of nano- and microstructured surface of chalcogenide materials such as Cu(In, Ga)Se$_2$, SnS, PbSnTe and PbSe were reported in literature [18–21]. As per literature survey, there is no report available on physical characteristics of In$_2$S$_3$ thin films treated with Ar-plasma. Special interest in indium sulfide films is caused by the presence of highly volatile chalcogen, sulfur and very low melting point (156.7 °C) metal, indium. Therefore in the present work, the effect of argon plasma treatment on the structure and surface properties of $\beta$-In$_2$S$_3$ thin films deposited by thermal evaporation technique was investigated and the obtained results were compared with the untreated films.
2. Experimental details

2.1. Deposition and annealing conditions of In$_2$S$_3$ films

In$_2$S$_3$ thin films were deposited using vacuum thermal evaporation technique (HHV BC 300 model) at a substrate temperature of 300 °C on soda lime glass substrates. In$_2$S$_3$ powder (Sigma Aldrich 99.9999% purity) was used as source material with a source to substrate distance of about 14 cm. The thickness of the deposited layers was measured using a quartz crystal thickness monitor as ~500 nm. Further, these deposited layers were annealed under sulfur ambient at 200 °C and 250 °C for an hour using two zone tubular furnace at 2 × 10$^{-3}$ mbar.

In our studies, according to the characterization analysis of as-deposited and annealed films, there was an improvement in the physical properties of the films annealed upto 250 °C. Above this temperature, the properties of the layers started to deteriorate, particularly the structure and optical properties. Therefore in the present study, the annealing temperature of the films was optimized to 250 °C. (See reference [22] for details). Further, annealing at lower temperatures is more economical to scale up the technology.

2.2. Plasma treatment conditions

The as-deposited and annealed β-In$_2$S$_3$ films were plasma treated with radio frequency (RF) high-density low-pressure inductively coupled argon plasma of radio frequency 13.56 MHz using RF ICP reactor [17]. The experiments were carried out with the following parameters: the inductive RF power $P$ — 800 W; the argon gas flow — 10 sccm; the operating argon gas pressure — 0.07 Pa; the RF bias power to the substrate holder $P_{bias}$ — 100 W. The ion flux is determined by the RF power $P$ applied on the inductor. Ion energy is controlled by the RF bias power $P_{bias}$ applied on the substrate [17]. In this case, the function of the ion energy distribution (IED) on the substrate surface for RF ICP has a double energy maxima [23, 24]. Measurements of the IED and self-bias potential $U_{ds}$ in the RF ICP reactor showed that the $U_{ds}$ is positioned symmetrically between the two maxima of the IED curve. At a RF bias power of 100 W, the average ion energy was 75 eV. In this case, on the basis of theoretical consideration [25] and experimental data [26], it can be assumed that at a high frequency of RF bias power $P_{bias}$ (13.56 MHz), the maximum peaks of the bimodal IED will be approximately ±20 eV in relation to average ion energy. However, the bimodal nature of the IED should not significantly affect the process of nanostructuring of the In$_2$S$_3$ film surface. If RF bias power is not supplied to the electrode, then there is no double peak in the IED function and the ion energy will be more monoenergetic.

The plasma treatment was carried out in vacuum at room temperature with an average argon ion energy of 75 eV for a duration of 30 s. From the physical point of view, the treatment of the sample involved bombarding the surface with argon ions that led to surface heating (~230 °C) and UV glow of argon plasma. Further, the optimized sample was treated with 25 eV of argon ion energy (where the RF bias power was kept as 0 W) by steps with duration of 15 s to study the effect of plasma treatment time on surface morphology of In$_2$S$_3$ layers.

2.3. Characterization techniques

The as-deposited as well as plasma treated In$_2$S$_3$ films were characterized using various techniques. The structural details of the films before and after plasma treatment was investigated using Ultima IV X-ray diffractometer (Rigaku) with Cu Kα radiation source ($\lambda$ = 1.5406 Å) in grazing incidence X-ray diffraction (GIXD) geometry at 1 degree of grazing angle. The surface composition of the untreated and treated In$_2$S$_3$ films was studied by energy-dispersive spectroscopy (EDS) analysis with the aid of an INCA Energy attachment (Oxford Instruments). Hitachi S–4800 with Supra 40 scanning electron microscope (SEM) was used to analyse the surface morphological properties. The topographical properties of the films were analysed using Solver Nano, NT–MDT atomic force microscope (AFM) instrument at a resonance frequency of 227 kHz in semi-contact mode with a scanning probe of 10 nm tip radius.

3. Results and discussion

3.1. Structural analysis

Figures 1(a) and (b) shows the respective GIXD patterns of In$_2$S$_3$ films before and after argon ion (Ar$^+$) plasma treatment. From figure 1(a), it is observed that the as-deposited and annealed films exhibit polycrystalline nature with the crystallinity being improved with increase of annealing temperature. The diffraction patterns of as-deposited In$_2$S$_3$ films were well matched with the cubic β-In$_2$S$_3$ phase (JCPDS 32-0456) with (300) plane as the preferred orientation and the peaks appeared at 2θ = 17.02° and 18.67° are related to the (112) and (105) orientations respectively that belongs to the tetragonal β-In$_2$S$_3$ (JCPDS 73-1366). Both cubic and tetragonal phases were identified with a change in the preferred orientation from (300) to (111) for films annealed at 200 °C. The existence of both tetragonal and cubic phases of In$_2$S$_3$ is most commonly observed in the literature for In$_2$S$_3$ thin films, irrespective of the deposition techniques used [22, 27–29]. For films annealed at 250 °C, the
secondary tetragonal phase was completely suppressed and only cubic phase with an intense (111) plane was observed as the preferred orientation with better crystallinity. From figure 1(b), it can be seen that the plasma treated In2S3 films initially had mixed phases of cubic and tetragonal β-In2S3 with a shift in crystallographic orientation from the (300) to (400) plane with improved crystallinity than the untreated films. Further, the annealed films treated with argon plasma exhibited only cubic phase of β-In2S3 (JCPDS 65-0459) with different preferred orientations. Moreover, an interesting feature observed in plasma treated films is the presence of peaks related to elemental indium (In) in all plasma treated In2S3 films (JCPDS 05-0642).

During the plasma treatment process, Ar+ ions interact with the film surface and hence affect the structural properties of In2S3 films. The various structural parameters of the layers before and after plasma irradiation were evaluated using the following relations (1–5) and the obtained values were tabulated in table 1. It is observed from the determined parameters that there is an improvement in the crystallite size with reduction in structural defects for plasma treated In2S3 films than the untreated layers.

The interplanar spacing (d) was calculated using the Bragg’s diffraction law:

\[ d = \frac{n\lambda}{2 \sin \theta}. \]

The lattice constant (a) can be calculated using the following equation:

\[ a = d \sqrt{h^2 + k^2 + l^2}. \]

### Table 1: Structural parameters of In2S3 films.

| Condition | Temperature (°C) | 2θ (°) (hkl) | FWHM (radians) | L (nm) | d (Å) | a (Å) | δ (lines m⁻²) | ε × 10⁻² |
|-----------|------------------|--------------|----------------|-------|-------|-------|--------------|---------|
| Before plasma treatment | As-grown | 25.10 (300) | 0.046141 | 3.0 | 3.54 | 10.62 | 1.1 × 10¹⁷ | 5.5 |
| | 200 | 14.25 (111) | 0.008671 | 15.8 | 6.21 | 10.75 | 4.0 × 10¹⁵ | 1.9 |
| | 250 | 14.25 (111) | 0.007626 | 18.0 | 6.21 | 10.75 | 3.1 × 10¹⁵ | 1.6 |
| After plasma treatment | As-grown | 33.05 (400) | 0.008054 | 17.6 | 2.70 | 10.82 | 3.2 × 10¹⁵ | 0.7 |
| | 200 | 33.05 (400) | 0.006227 | 22.8 | 2.70 | 10.82 | 1.9 × 10¹⁵ | 0.5 |
| | 250 | 14.35 (111) | 0.006202 | 22.1 | 6.16 | 10.67 | 2.0 × 10¹⁵ | 1.3 |

Figure 1. GIXD patterns of In2S3 films (a) before and (b) after plasma treatment [in figures 1(a) and (b), T represents tetragonal structure; *In represents elemental indium].
The size of coherent scattering region was estimated by the Debye–Scherrer formula [30]:

$$L = \frac{0.94 \lambda}{\beta \cos \theta},$$

(3)

where $\beta$ is an integral breadth of the prevailing peak in radians. Gaussian approximation of the peaks was used, when $\beta = (\pi / 4 \ln 2)^{1/2} \times \text{FWHM}$ (where, the FWHM is full width at maximum of the prominent peak).

The dislocation density was calculated using the Williamson-Smallman relation [31]:

$$\delta = \frac{1}{L^2}.$$

(4)

The lattice deformation was calculated using the following relation [32]:

$$\varepsilon = \frac{\beta}{4 \tan \theta}.$$

(5)

The analysis of table 1 shows that the as-deposited films have the lowest size of coherent scattering, high dislocation density and the highest value of mechanical stresses. In the annealing process, the quality of the film improves, and further plasma treatment continues this trend. The results are in good agreement with the earlier reported work of Revathi et al [33] and Nehra et al [12] for annealed In$_2$S$_3$ films.

3.2. Morphological studies and EDS analysis

Figure 2 shows the SEM micrographs of In$_2$S$_3$ films (a) before and (b) after plasma treatment. It can be observed that the surface of the films was covered with uniformly distributed grains that had different shapes without any cracks. In the case of untreated films, the grain size was increased for films annealed at 200 °C and 250 °C due to the coalescence of smaller grains [33, 34]. In contrary, the appearance of nanoparticle ensembles isolated from each other on the surface became typical after plasma treatment. The details about the nanostructures over the surface can be studied in more detail through SEM pictures taken at higher magnification.

Figure 3 shows the SEM images of plasma treated In$_2$S$_3$ films taken at higher magnification (100 K×). Here, an array of nanoparticles with quasi-spherical or spread droplet shapes are observed. Nanostructures of this shape are typical for metal droplets. It can be assumed that these droplets correspond to metallic indium that had an average size of 30–100 nm with a surface density of $10^{10}$ cm$^{-2}$. Before plasma treatment, the thickness of the films was $\sim$500 nm (varied from 503 nm to 522 nm) for annealed films. Further, the plasma treatment of the films was done for constant duration of 30 s with an average Ar ion energy of 75 eV, which causes surface etching of the layers by $\sim$200 nm. Because of this, the thickness of the films was finally decreased to $\sim$300 nm after plasma treatment.

The study of the chemical composition of films by EDS showed that indium to sulfur (In/S) ratio increased after plasma treatment. This ratio was in the range, 0.95–0.65 before treatment, that was increased to 1.20–1.38 after plasma treatment. Given that the depth of EDS analysis is smaller than the thickness of the film, the results confirm the appearance of indium nanoparticles after evaporation of sulfur on the surface of the film. A similar trend in compositional variation was reported in literature for polycrystalline In$_2$S$_3$ films prepared by chemical bath deposition method and irradiated using gamma rays with different doses [16].

3.3. Topographical studies

Figure 4 shows the 3D AFM images (5 μm × 5 μm area) of In$_2$S$_3$ films before and after plasma treatment. The average grain size and surface roughness of the films were evaluated from typical AFM data. The grain size was increased for annealed films (260 nm and 360 nm) compared to as-deposited layers (250 nm). At higher annealing temperatures, the ad-atoms gain sufficient energy to combine with the neighbouring atoms on the surface and form larger grains. Also, the surface roughness of the annealed layers was found to be higher than the as-deposited layers. After plasma treatment, the surface roughness of the films was further increased with a decrease of grain size from 163 nm to 130 nm. This is because of etching of the film surface due to irradiation of argon plasma. Further, the grain size of In$_2$S$_3$ films is fairly in agreement with that estimated values using SEM analysis. The skewness for the plasma treated films was low (0.28–0.56) compared to the untreated films (0.41–0.89) and all films had positive skewness except for plasma treated films annealed at 250 °C (−0.49). The positive skewness indicates that the films had more number of peaks than valleys on the surface, whereas the films annealed at 250 °C followed by plasma treatment showed negative skewness, which reveals that valleys are more predominant than peaks on the surface with respect to the mean line. Thus, both positive and negative skewness values represent the asymmetrical distribution of heights on the film surface [35]. All the films before and after plasma treatment showed leptokurtic nature (kurtosis value is $>3$), which indicates that the film surface profile distribution consists of a few extremely high peaks [36].
3.4. The dynamics of nucleation and growth of indium drops during plasma treatment

In order to study the effect of plasma treatment on the nucleation and growth of indium drops over the surface of In$_2$S$_3$ layers in more detail, the treatment was performed for different time durations of 30, 45 and 60 s with an interval of 15 s for each treatment in order to cool the sample. The experimental studies were carried out for In$_2$S$_3$ films annealed at 250 °C with Ar-ion energy of 25 eV. Figures 5(a)–(d) shows the SEM images of In$_2$S$_3$ films for different plasma etching durations taken at a tilt (stage) of 70°. The picture for initial state (figure 5(a)) clearly

![SEM images of In$_2$S$_3$ films (a) before and (b) after plasma treatment.](image1)

![SEM images of In$_2$S$_3$ films after plasma treatment at high magnifications. The SEM images were captured at a tilt of 70°.](image2)
showed that the film surface was rough with uniformly distributed grains. After the first step for 30 s (figure 5(b)) irradiation of the film, the larger grains of untreated film started to disintegrate into smaller grains and finally became an ensemble of nanodroplets formed on the surface. This can be clearly seen from figure 5(b) where small droplets of indium appear to be distributed in the background of larger grains. Therefore for 30 s of plasma etching in the first step, the particle size of indium droplets was 10–33 nm with a surface density of \(9 \times 10^{11} \text{ cm}^{-2}\). Each subsequent step of 15 s (figures 5(c)–(d)) led to a gradual increase in the size of indium nanoparticles reached up to 50 nm, but their surface density remained constant and was in the range \((6–8) \times 10^{11} \text{ cm}^{-2}\). The study of the chemical composition of films by EDS showed that the ratio of In/S increases with the increase of plasma treatment time. If for the initial state the ratio was 0.61, then after 30 s treatment, it was 0.76, and after 45 s plasma treatment it increased to 0.91 and for 60 s, to 0.96. This confirms the formation of indium nanoparticles on the surface of In\(_2\)S\(_3\) films after evaporation of sulfur from the film surface.

The following physical model can be proposed to explain the formation of indium droplets on the surface of In\(_2\)S\(_3\) films when processed in argon plasma. Experimentally determined sputtering rates are 2.7 nm s\(^{-1}\) and 7.1 nm s\(^{-1}\) for ion energies of 25 eV and 75 eV respectively. The bombardment of the surface by argon ions

Figure 4. AFM images of In\(_2\)S\(_3\) films (a) before and (b) after plasma treatment.
involves the physical sputtering of surface material, so that the sputtering products are above the surface of In$_2$S$_3$ film. It is reported in literature [37] that the ion sputtering process of In$_2$S$_3$ films result in the appearance of S$_2$ molecules over In$_2$S surface. Further, two additional factors need to be taken into account. The first is due to the fact that the surface temperature during plasma treatment increases. Under close experimental conditions, it can reach approximately 230 °C in 30 s [20], which significantly exceeds the melting point of indium. The second factor is associated with the presence of an intense UV glow of argon plasma. This leads to the fact that both S$_2$ molecules and In$_2$S surface are being subjected to continuous bombardment of argon ions at elevated temperatures and UV glow that dissociates S$_2$ into S and In$_2$S into indium and sulfur. Rodriguez et al [14] also reported the process of dissociation of In$_2$S$_3$ layer into indium and sulfur when irradiated with nitrogen plasma. In this case, the volatile sulfur leaves the reaction zone and the relatively heavy metal settles on the surface of the plasma treated film. The result of this process is the formation of liquid indium nanoparticles, which at the end form ensembles of quasi-spherical droplets. The size, shape and density of the droplets can be controlled by changing the plasma treatment conditions. Moreover, the annealing temperatures (200 °C, 250 °C) and the surface temperature during plasma treatment (230 °C) of the films are nearly same. The effect of both temperatures annealing and plasma treatment led to a change in the film composition. In the former case, the S/In ratio reached to stoichiometry upon sulfur annealing, whereas in latter case, the films are indium rich due to re-evaporation of sulfur from the film surface.

4. Conclusions

The effect of Ar-plasma treatment on structural, morphological and topographical properties of thermally evaporated In$_2$S$_3$ films has been investigated. The GIXD analysis confirmed that the untreated films had mixed phases of tetragonal and cubic $\beta$-In$_2$S$_3$ structure. But, the plasma treated films showed mixed phases initially and then cubic $\beta$-In$_2$S$_3$ phase only for annealed films. An important feature observed was the formation of ensemble of indium nanoparticles on the surface of In$_2$S$_3$ films during plasma treatment that was evident from the GIXD, EDS and SEM analysis. The nanostructured array of indium particles formed on the surface of the layers reduces the surface roughness of the plasma treated films than the untreated films. Thus, the Ar-plasma strongly influenced the morphological and topographical properties of $\beta$-In$_2$S$_3$ layers. The cleaning of the surface of In$_2$S$_3$ films during plasma treatment and additional appearance of ensembles of indium nanodrops on the surface can be used also for the subsequent realization of the growth of nanorods, nanowires, nanohillocks of various

![Figure 5. Formation of ensemble of nanoparticles during plasma treatment for (a) initial state (b) 30 s (c) 45 s (d) 60 s. The SEM images were captured at sample tilt of 70°.](image-url)
semiconductors on In$_2$S$_3$ surface by the vapor-liquid-crystal (VLS) mechanism with catalyzing liquid indium. Therefore, plasma treatment can serve as an approach for the self-formation of indium nanostructure arrays on the surface of In$_2$S$_3$ layers, which can be useful for multilayered device fabrication such as solar cells. The formation of metal particles on the surface can radically change the processes of reflection and transmission of light, which will be considered in our future work.

**Acknowledgments**

One of the authors, S Rasool thanks the University Grants Commission (UGC), New Delhi for the financial assistance via the ‘UGC-BSR fellowship’.

The authors, Prof K T Ramakrishna Reddy and Prof M S Tivanov wish to acknowledge the Dept. of Science and Technology, Govt. of India (Grant no: DST/INT/BLR/P-30/2019) and the State Committee on Science and Technology of the Republic of Belarus (Grant no: F19INDG-008).

SEM investigations were carried out at the Facilities Sharing Center ‘Diagnostics of Micro- and Nanostructures’ with the support of the Ministry of Science and Higher Education of Russian Federation.

This work was performed in the framework of R&D initiative of Yaroslavl State University, project AAAAA16-116070610023-3.

The investigation was partially supported by Program no. 0066-2019-0002 of the Ministry of Science and Higher Education of Russian Federation for Valiev Institute of Physics and Technology of RAS.

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