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INFLUENCE OF QUANTITY OF SPRAY SOLUTION ON THE PHYSICAL PROPERTIES OF SPRAY DEPOSITED NANOCRYSTALLINE MgSe THIN FILMS

Nanocrystalline MgSe thin films have been prepared by varying the quantity of spray solution using the spray pyrolysis technique. The effect of the varying quantity of spray solution on the structural, electrical, morphological and optical properties of MgSe thin films was investigated. The X-ray diffraction studies revealed that the deposited MgSe has a cubic lattice with (111) as the preferred orientation. The optical studies showed that the deposited MgSe exhibited direct optical band gap which varied from 2.45 to 2.75 eV depending on the quantity of spray solution. The electrical resistivity of MgSe decreased with an increase in temperature indicating its semiconducting nature. The electrical resistivity, activation energy and optical band gap energy were found to depend upon the quantity of spray solution. The measurement of thermo-emf with applied temperature gradient across the film confirmed its *p*-type conductivity.

THIN FILMS, X-RAY DIFFRACTION, OPTICAL PROPERTIES.

1. Introduction

The alkaline earth chalcogenides such as MgSe, CaSe, SrSe, etc., have attracted the scientific community due to their potential applications in various optoelectronic devices, especially luminescent ones. M.W. Wang, et al. [1] have studied the growth of MgSe films of zinc-blende structure on ZnTe substrates by the molecular beam epitaxy (MBE) method. The surface reconstructions of MgSe under different flux ratios and growth temperatures were studied. A.L. Rouff, et al. [2] have studied MgSe using the energy dispersive X-ray diffraction to 202 GPa and local density approximation to ultra-soft pseudo-potentials to 500 GPa. It was reported that MgSe underwent continuous phase transformation from rocksalt to FeSi. P. Prete, et al. [3] have reported growth and characterization of ZnMgSe and MgSe on (100) GaAs by low-pressure metallorganic vapour phase epitaxy (MOVPE). The crystallographic phases of as grown MgSe and ZnMgSe were investigated. F.A. Sahraoui, et al. [4] have reported the structural and electric properties of MgSe at high hydrostatic pressure by the pseudo-potential plane wave method. R. Pendey, et al. [5] have studied structural phase transition on MgSe from wurtzite zinc-blende to rocksalt phase by the periodic Hartree-Fock method. As compared to low pressure, the MOVPE and MBE methods used for the deposition of MgSe thin films, the spray pyrolysis method has many advantages, such as high deposition rate, control of various deposition parameters and low cost [6 – 12]. The spray method has been used for several decades in glass industry and

in solar cell production to deposit electrically conducting electrodes. Thin film formation using this technique involves spraying a metal salt solution onto a heated substrate. The sprayed droplet reaching the hot substrate surface undergoes spyrolytic decomposition and forms the desired product. The other volatile by-products escape in the vapour phase. The quality and properties of the films depend largely on substrate temperature, precursor solution concentration, spray rate and substrate, etc. Previous studies have shown that very few reports are available on growth of MgSe films by solution growth [13, 14] by the metalorganic vapour chemical deposition (MOVCD) [5] and MBE [1] methods. The effect of triethanolamine (TEA) on the physical properties of MgSe thin films grown by the chemical bath deposition method has been reported in our earliear report [15]. The purpose of this work is to investigate the effect of the quantity of spray solution on the physical properties of MgSe thin films. This paper reports structural, electrical and optical properties of MgSe thin films prepared by varying the quantity of the spray solution from 5 to 30 ml.

2. Deposition of MgSe thin films

MgSe thin solid films were prepared using the versatile spray pyrolysis technique, which is simple and economic as compared to the MBE and MOCVD methods. The properties of the film can be controlled by adjusting the preparative parameters such as substrate temperature, spray rate, quantity of spray solution, pH of spray solution, etc. Aqueous solutions of magnesium chloride ($MgCl_2$), selenium oxide (SeO_2) and triethanolamine NH $(CH_2\text{-}CH_2)_3$ (TEA) were prepared using double distilled water. The glass substrates were ultrasonically cleaned and purged with acetone prior to the deposition. The spray solution was prepared by mixing 10 ml of 0.12 M $MgCl_2$, 10 ml of 0.10 M TEA and 10 ml of 0.13 M SeO_2. The MgSe thin films of various thicknesses were deposited by spraying 5, 10, 15, 20, 25 and 30 ml of spray solution onto preheated amorphous glass substrates, maintained at a temperature of 523 K. The spray rate was kept at 4 ml/min. The distance of 25 cm was kept between the substrate and the nozzle. The MgSe film thickness was measured using weight difference method (by sensitive microbalance for thin films) and considering bulk density of MgSe.

The X-ray diffractogram was recorded using PANalytical X'Pert PRO MRD diffractometer with CuK_α line in 2θ range from 20 to 90°. The compositional analysis of the deposited film was carried out by Energy Dispersive X-ray Spectroscopy and microstructure aspects of the films were studied with a JOEL'S JSM-7600F Scanning Electron Microscope with the resolution of 1 nm and an Atomic Force Microscope (AFM) from Park Scientific Instruments. The optical absorption spectrum was recorded using the JASCO V-530 UV/ Visible Spectrophotometer at normal incidence in the wavelength range from 300 to 900 nm. The electrical resistivity of the film was measured in the temperature range from 300 to 550 K by employing the two point probe method; quick drying silver paste was used as a contact material. A chromel-alumel thermocouple was used to measure the temperature of the sample. The type of electrical conductivity of the films was determined from the sign of the thermo-emf developed across the hot and cold junction.

3. Results and discussion

Film formation mechanism. The aqueous solution containing Mg^{2+} ions complexed with TEA, i.e. $[Mg\ (TEA)]^{2+}$ along with SeO_2, was sprayed onto hot substrates kept at a temperature of 523 K. The sprayed droplets underwent thermal decomposition and well-adherent milk-white colored MgSe thin films were deposited. The film formation takes place according to the following reaction:

$$SeO_2 + H_2O \rightarrow H_2SeO_3; \qquad (1)$$

$$[Mg(TEA)]^{2+} + H_2SeO_3 + 4\ Cl^- \rightarrow (523\ K) \rightarrow MgSe\downarrow + TEA + HCl\uparrow + 2O_2\uparrow. \qquad (2)$$

The deposited films were uniform, pinhole-free and adherent to the glass substrate. Fig. 1 shows the variation of film thickness as a function of the quantity of spray solution. The film thickness is 145 nm for a 5 ml spray solution and it increases to 188 nm as the spray

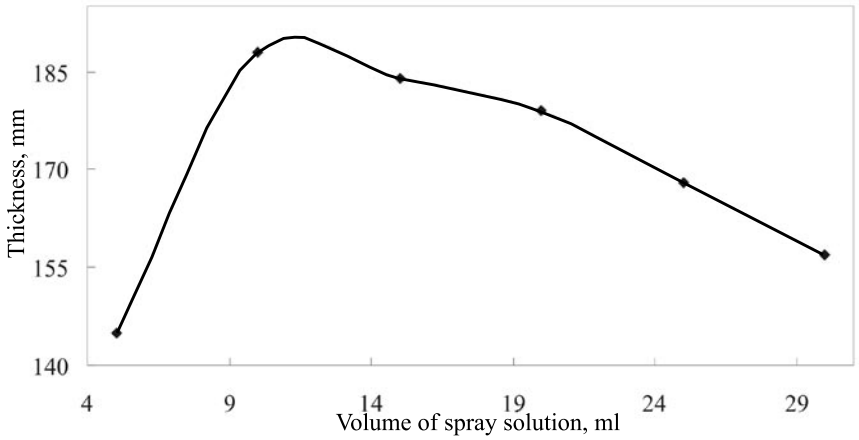


Fig. 1.The plots of MgSe film thickness versus the volume of the spray solution

solution becomes 10 ml. However, a slight decrease in the film thickness was observed with a further increase in the quantity of the spray solution. This may be due to incomplete thermal decomposition of the spray solution. Here, the optimum quantity of the spray solution was found to be 10 ml and above it the film surface started becoming somewhat powdery, giving lower thickness [16].

Structural studies. The grown films were characterized by X-ray diffraction (XRD) using CuK_α radiation, for structural identification and changes in the crystallinity of MgSe. Fig. 2 shows XRD patterns of MgSe films deposited by varying the quantity of the spray solution.

The films are nanocrystalline in nature with cubic lattice (see Table). The XRD pattern exhibits the major (111) orientation which becomes prominent with an increase in the thickness. Beside this, other diffraction peaks

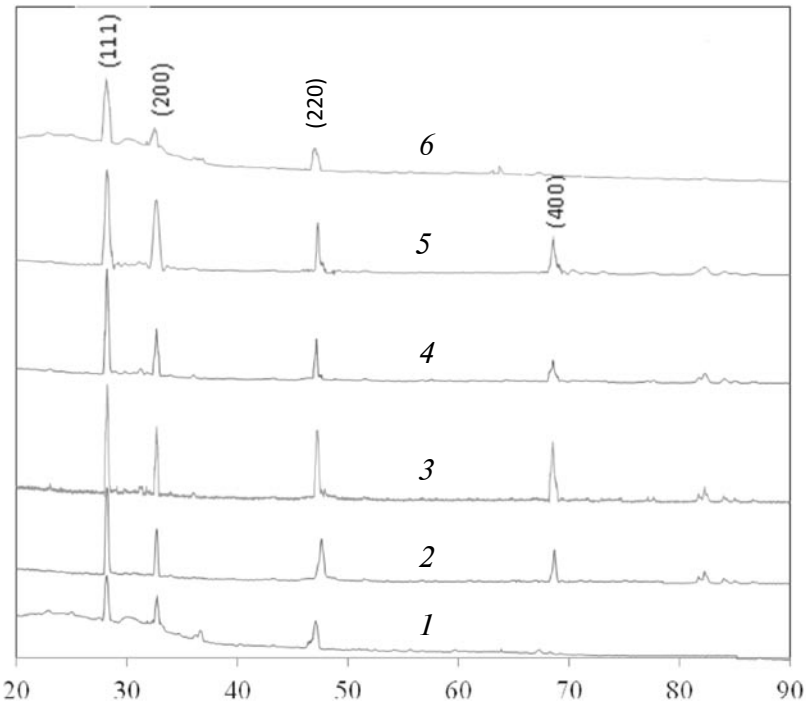


Fig. 2. X-ray diffraction patterns of MgSe thin films deposited changing the spray solution volume, ml: 5 (*1*), 10 (*2*), 15 (*3*), 20 (*4*), 25 (*5*), 30 (*6*)

Table

The obtained physical properties of MgSe thin films depending on the spray solution volume

Spray solution volume, ml	Film thickness, nm	Band gap energy, eV	Activation energy, eV	Grain size, nm	$\rho \times 10^2$, Ω-cm, at 373 K
05	145	2.75	0.97	11	23
10	188	2.45	0.86	24	11
15	184	2.50	0.87	21	13
20	179	2.60	0.88	17	14
25	168	2.65	0.90	13	07
30	157	2.69	0.92	10	20

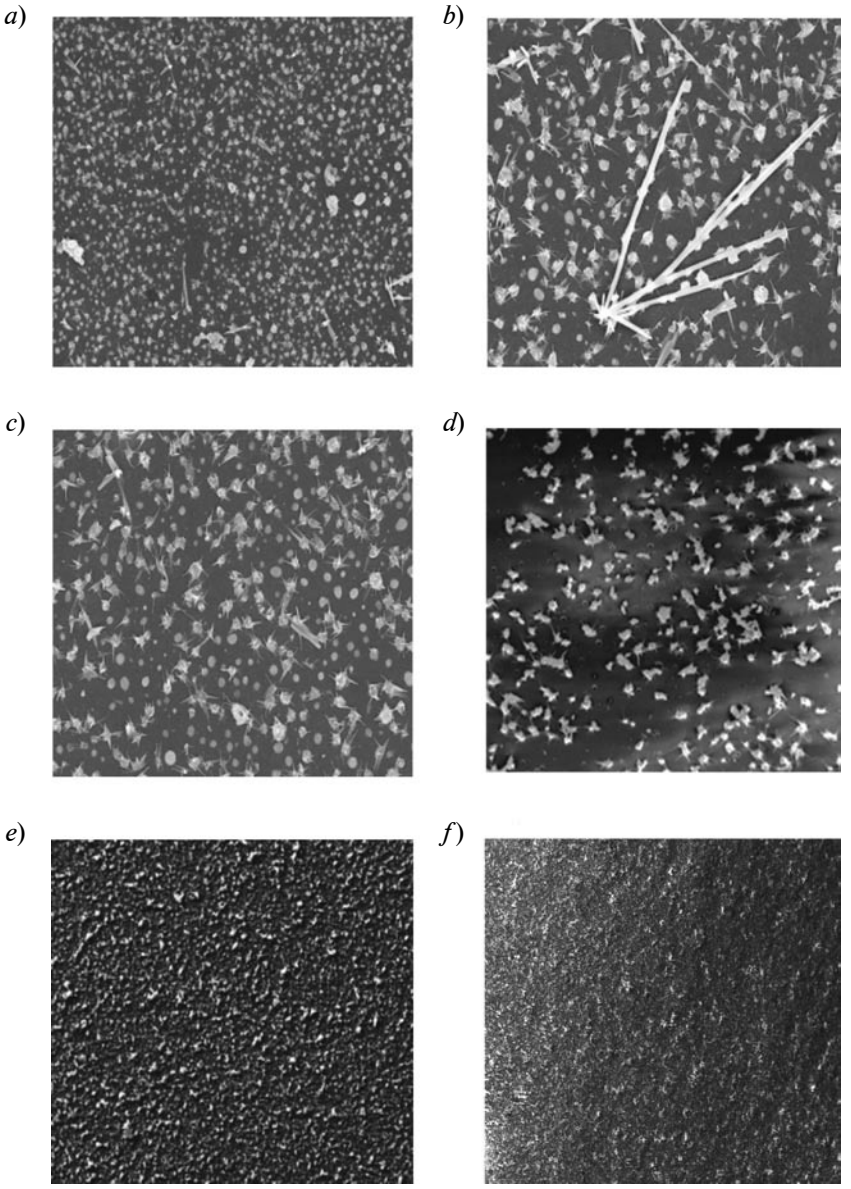


Fig. 3. Scanning electron microscopy images of MgSe thin films deposited by changing the spray solution volume, ml: 05 (*a*), 10 (*b*), 15 (*c*), 20 (*d*), 25 (*e*), 30 (*f*)

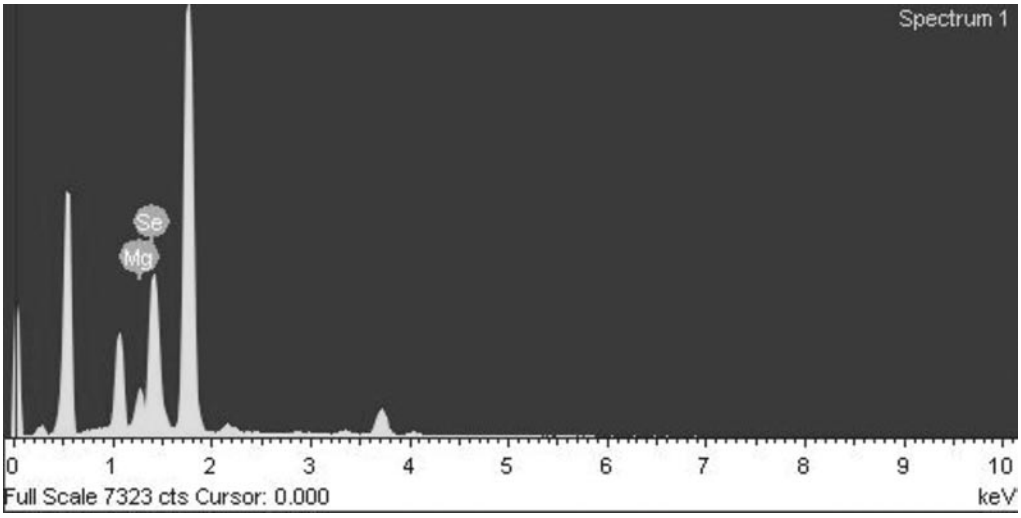
of (200), (220) and (400) are observed. These patterns clearly demonstrate that the film thickness of 188 nm deposited by spraying 10 ml of the solution is more oriented than others. The variation in the peak intensity with respect to the quantity of the spray solution may also be attributed to the variation in the film thickness and the grain structure. The average crystallite size of the deposited material was determined using Debye-Scherer's formula:

$$d = 0.9\lambda/(\beta\cos\theta), \qquad (3)$$

where β is the peak width at half-height (in radians), λ is the wavelength of CuK_α radiation (λ = 1.5418 Å), θ is Bragg's diffraction angle at peak position (in degrees).

The average crystallite size was found to vary between 10 to 24 nm as the film thickness was changed from 145 to 188 nm (see Table). Above 10 ml of the spray solution, the film thickness and the grain size gradually decrease due to the formation of powdery film. Consequently, the thicker films consist of larger grains as compared to the thinner ones.

Surface morphology. Fig. 3 shows scanning electron microscopy (SEM) micrographs of spray-deposited MgSe thin films. The SEM micrograph reveals that the substrates were



Energy, keV

Fig. 4. Typical energy dispersive X-ray analysis spectrum of MgSe thin film of thickness 188 nm (the volume of the spray solution is 10 ml)

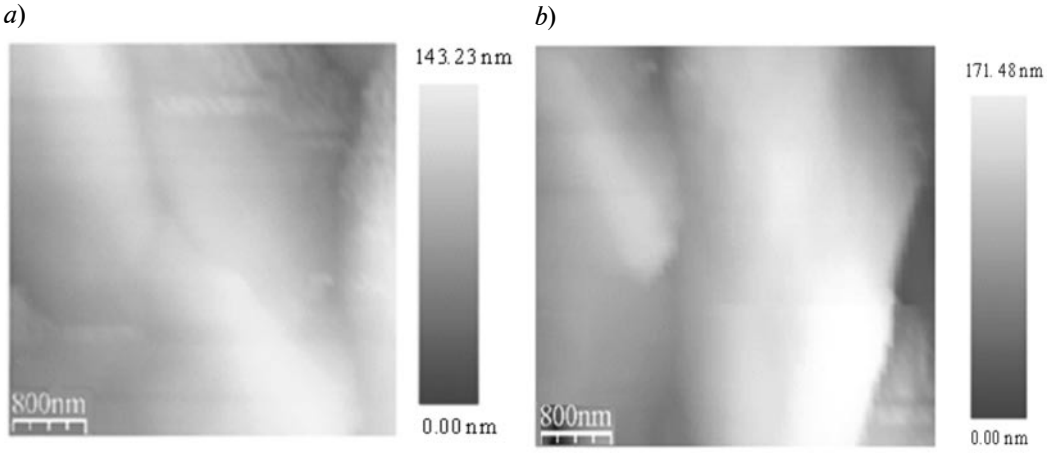


Fig. 5. AFM images of MgSe thin films deposited by changing the volume of the spray solution: 5 ml (*a*) and10 ml (*b*)

well-covered with a uniform distribution of a large number of fine grains. As the quantity of spray solution increased from 5 to 10 ml, a remarkable grain growth is observed. The film deposited using 10 ml spray solution has well-defined MgSe nano-rods along with spherical grains showing spikes on the surface. However, this type of morphology is suppressed as the quantity of the spray solution is increased above 10 ml. This fact confirms that films deposited with the optimum quantity of the spray solution, i.e., 10 ml, are more useful due to their porous nature.

A typical energy dispersive X-ray analysis (EDAX) spectrum of MgSe thin film deposited using the spray solution of the volume ranging from 5 to 30 ml is shown in Fig. 4. The formation of MgSe is confirmed from the elemental analysis. Other peaks appearing due to glass substrate can also be observed.

Fig. 5 shows an atomic force microscopy (AFM) image of MgSe films deposited with 05 and 10 ml of the spray solution. It is evident that the film surface is uniform and homogenous.

Optical properties. The optical absorption spectra of the films of different thicknesses were recorded and analyzed to estimate band gap energies of MgSe. The variation in absorption density αt with a wavelength λ for all the samples is shown in Fig. 6. It is evident that the absorption coefficient of the MgSe film is of the order of 10^4 cm^{-1}.

To confirm the nature of the optical transition in these samples, the optical data was analyzed using the classical relationship [17]:

$$\alpha h\nu = A(E_g - h\nu)^n, \qquad (4)$$

where α is the absorption coefficient, A is the constant, h is the Planck constant, ν is the photon frequency, E_g is the optical band-gap energy, and n is 1/2 and 2 for the direct and the indirect transition, respectively.

Fig. 7 shows the variation of $(\alpha h\nu)^2$ values versus $h\nu$ values for MgSe thin films of various thicknesses. The extrapolation of the straight-line portion of the plot to zero absorption coefficient gives the value of the band gap energy E_g.

Fig. 8 shows the changing of the optical band-gap energy of the MgSe thin film with the volume of the spray solution. The energy gap increases from 2.45 to 2.75 eV as the film thickness decreases from 188 to 145 nm [18, 19]. The red shift observed in the absorption edge of MgSe thin film results from the smaller grain size of the nanostructured material as the

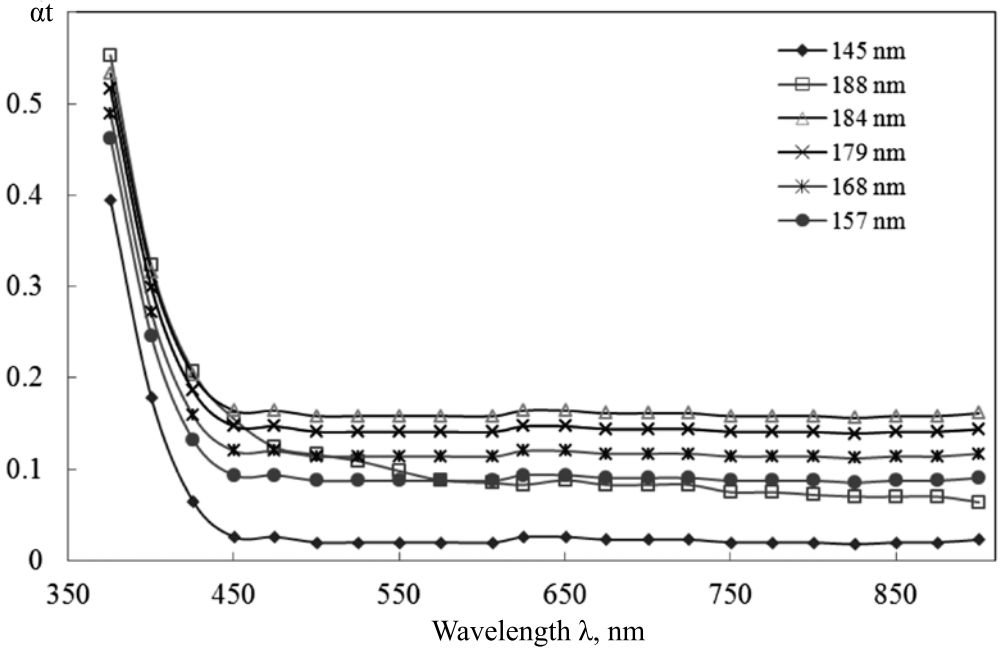


Fig. 6. Plots of the optical absorption αt versus wavelength λ for MgSe thin films deposited by changing the quantity of the spray solution (see Table)

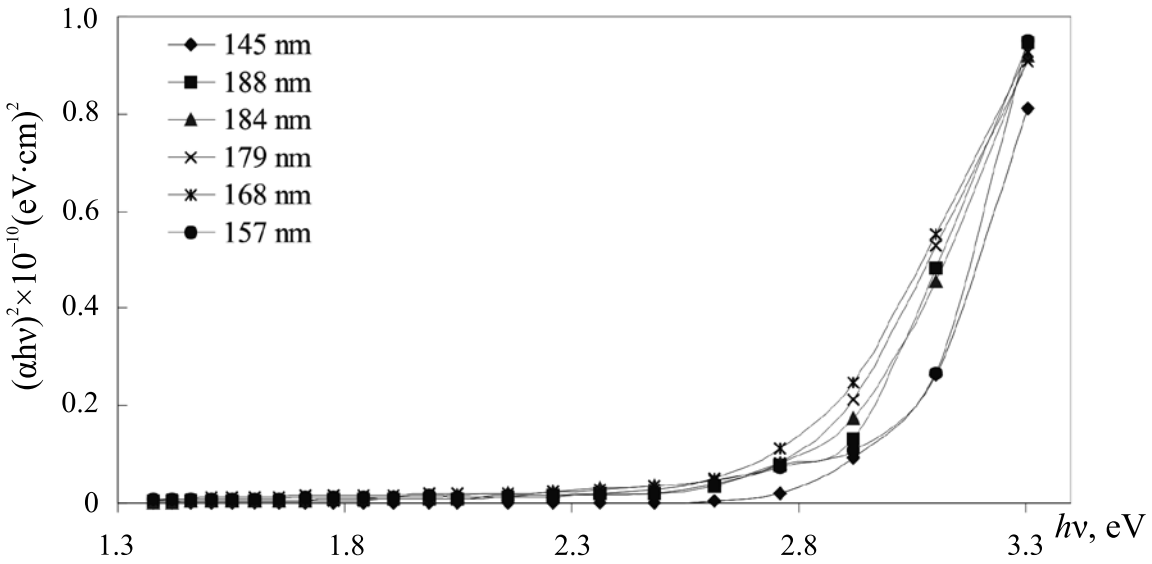


Fig. 7. Plots of $(\alpha h\nu)^2$ values versus $h\nu$ values for MgSe films. Volumes of spray solution are given in Table

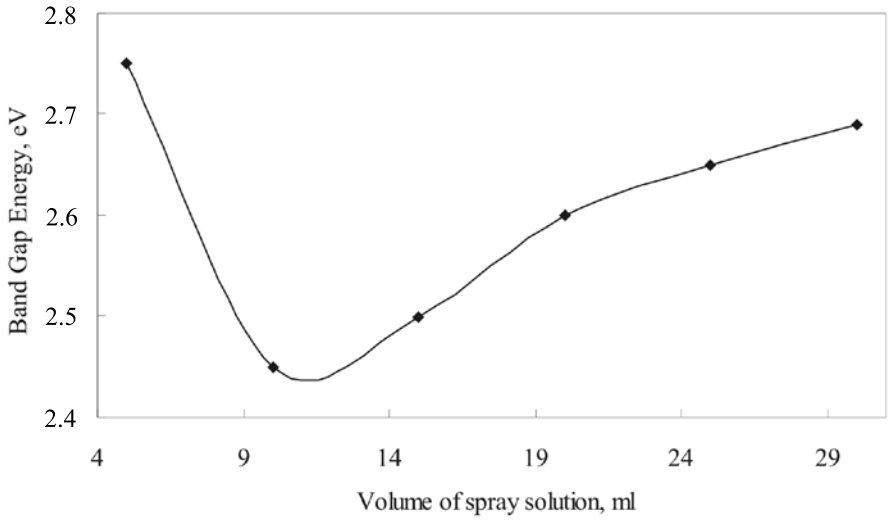


Fig. 8. The dependence of the optical band gap energy of the MgSe film on the volume of the spray solution

quantum size effect becomes prominent [20].

Electrical properties. The d.c. electrical resistivity of all the deposited samples was studied in the temperature range from 300 to 500 K using the two-probe method. The variation of the log ρ value with the reciprocal of temperature ($1000/T$) is shown in Fig. 9. The electrical resistivity follows the relation

$$\rho = \rho_0 \exp(-E_a/(kT)), \qquad (5)$$

where ρ is the resistivity at temperature T, ρ_0 is the constant, k is the Boltzmann constant, E_a is the activation energy required for conduction.

The electrical resistivity of thinner film was found to be $2.3\cdot10^3$ Ω·cm and it decreases to $1.1\cdot10^3$ Ω·cm as film thickness becomes 188 nm. The improvement in crystallinity and the grain size with thickness induces conductivity. Fig. 10 shows the dependence of electrical resistivity on the volume of the spray solution for the MgSe thin film. For all the samples it was obtained that the electrical resistivity decreases

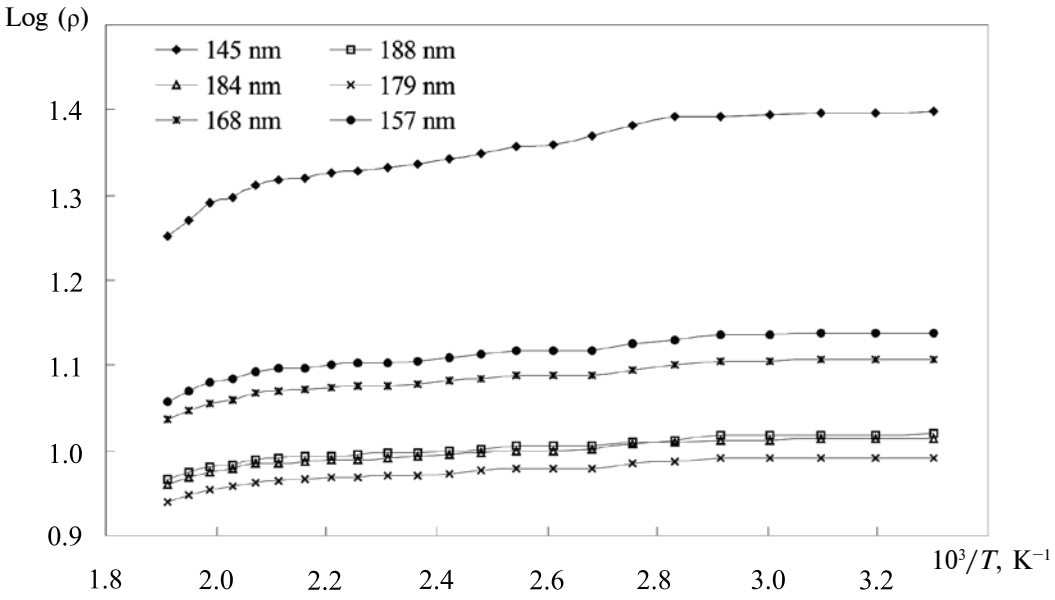


Fig. 9. The plots of Log (ρ) value versus ($10^3/T$) for MgSe thin films deposited varying the quantity of the spray solution (see Table)

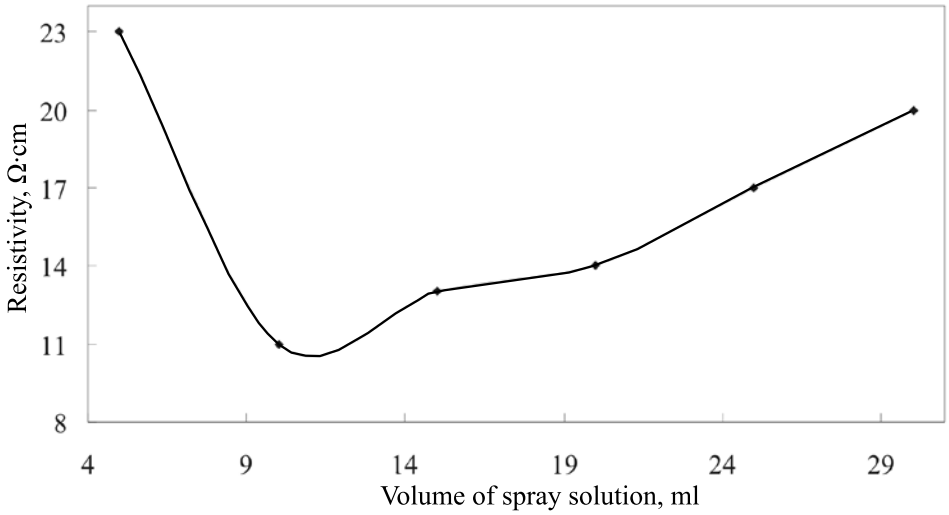


Fig. 10. The dependence of the resistivity of MgSe thin films obtained at 373 K on the volume of the spray solution

with an increase in temperature, confirming the semiconducting nature of MgSe. The thermal activation energies were calculated and were found to vary from 0.97 to 0.87 eV depending on the film thickness (Fig. 11).This variation in the activation energy with the film thickness is due to the changes in the crystalline structure and the density of dislocations in the film.

Thermo-emf measurement. The temperature difference applied across the film causes the transport of carriers from the hot end to the cold one thus creating an electric field. It was concluded from the polarity of the thermoelectric-emf that MgSe film exhibited *p*-type conductivity. Fig. 12 shows the variation of thermo-emf with applied temperature difference.

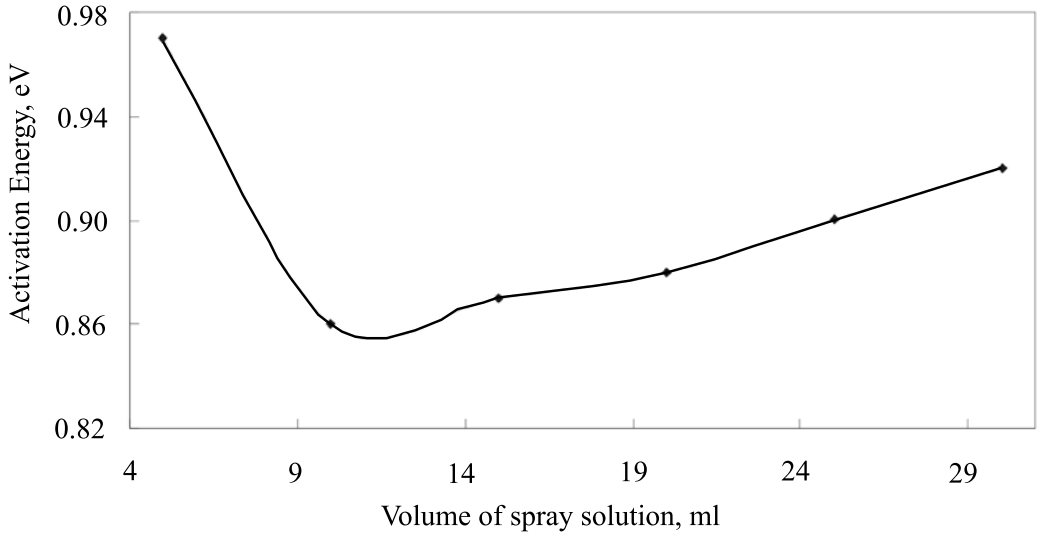


Fig. 11. The dependence of the activation energy of MgSe thin films on the volume of the spray solution

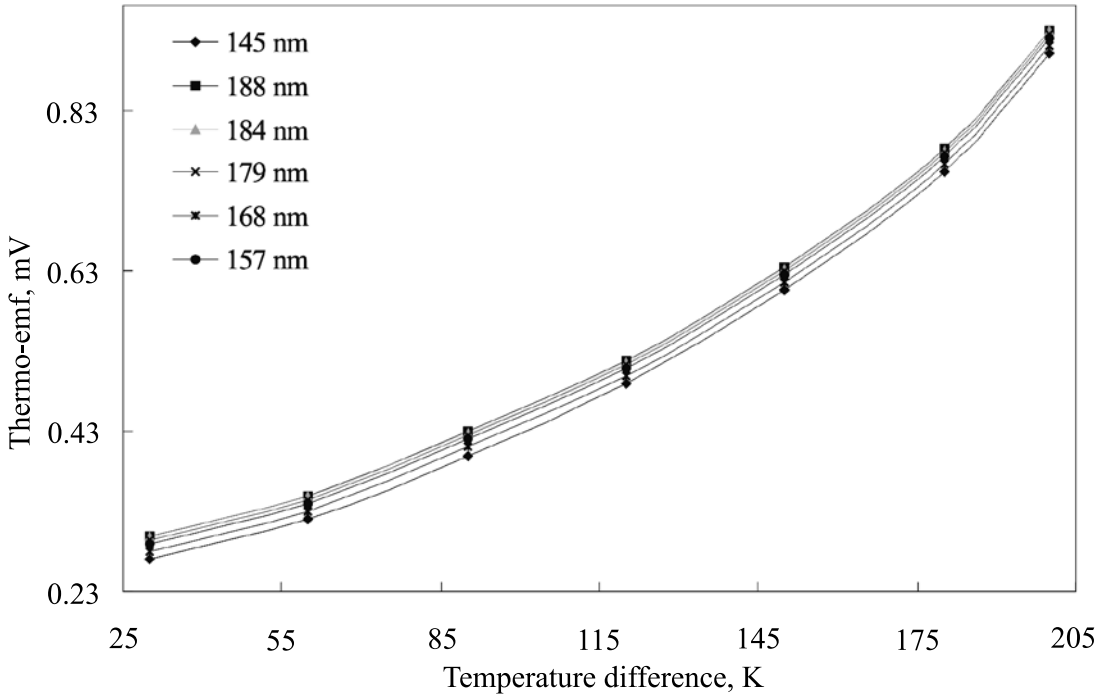


Fig. 12. The plots of thermo e.m.f. versus temperature differences applied across the MgSe thin films

It is evident that the thermo-emf increases linearly with the applied temperature difference. The thermo-emf developed across the film increases with thickness, which may be due to the improvement in the crystalline structure of the film [21]. At lower thickness the grain boundary scattering is prominent which may decrease with thickness due to the grain growth.

4. Conclusions

Magnesium selenide thin films of thickness from 145 to 188 nm were deposited onto glass

substrates at 523 K using the spray pyrolysis technique with varying the quantity of the spray solution. The grown films are nanocrystalline in nature with cubic structure. The crystallinity and the grain size of MgSe is improved with thickness. The films deposited by spraying 10 ml of the solution are porous, showing growth of nano-rods. The optical band-gap energy decreases with an increase in film thickness. It is, therefore, concluded that the physical properties of the magnesium selenide films can be customized by merely controlling the film thickness, which in turn becomes suitable for a particular application.

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Сахаре Ё.С., Такаре Н.Р., Убале А.У. ВЛИЯНИЕ КОЛИЧЕСТВА РАСПЫЛЯЕМОГО РАСТВОРА НА ФИЗИЧЕСКИЕ СВОЙСТВА ТОНКИХ НАНОКРИСТАЛЛИЧЕСКИХ ПЛЕНОК СЕЛЕНИДА МАГНИЯ, ОСАЖДЕННЫХ МЕТОДОМ СПРЕЙ-ПИРОЛИЗА.

Тонкие нанокристаллические пленки селенида магния MgSe были приготовлены методом спрей-пиролиза с использованием различного количества распыляемого раствора. Рассмотрено влияние изменения количества этого раствора на структурные, электрические, морфологические и оптические свойства пленок MgSe. Методом рентгеновской дифракции установлено, что осажденный MgSe имеет кубическую кристаллическую решетку с преимущественной ориентацией (111). Оптические исследования показали, что для полученного объекта характерна запрещенная зона с прямыми оптическими переходами, ширина которой меняется от 2,45 до 2,75 эВ, в зависимости от количества распыляемого раствора. Электрическое сопротивление пленки MgSe снижается с повышением температуры, что указывает на ее полупроводниковую природу. Установлено, что электросопротивление, энергия активации и ширина запрещенной зоны зависят от количества распыляемого раствора. Измерение термоэдс путем создания температурного градиента через пленку подтвердило, что она проявляет проводимость дырочного типа.

ТОНКИЕ ПЛЕНКИ, ДИФРАКЦИЯ ОПТИЧЕСКИХ ЛУЧЕЙ, ОПТИЧЕСКИЕ СВОЙСТВА.

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