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Structural, Raman and Electrical Characterization of Nanocrystalline CdSe Thin Films Deposited by Pulse Laser Deposition Technique

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Abstract- Cadmium Selenide (CdSe) thin films were deposited by Pulse Laser Deposition(PLD) technique onto Indium Tin Oxide (ITO) coated glass substrates at different substrate temperatures. The structure of CdSe confirmed by using the X-ray diffraction pattern. The surface element analysis and morphology of thin films were done by X-ray photoelectron spectroscopy and Atomic force microscopy, respectively. Raman spectroscopy is used for atomic bond behavior at room temperature and lower than room temperature. The band gap of the thin films was estimated (1.75 eV to 2.3 eV) using the UV-Visible absorption spectra. Electrical behavior I-V characteristic) of thin films also studied at different temperatures. These films have possible applications in thin films based on solar cells, and sensors.

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I. Introduction

Cadmium selenide is one of the II-VI materials which have been extensively considered by researchers due to their various applications such as photovoltaic applications, solar cells, thin-film transistors, gamma-ray detectors, lasers, sensors [1-3], and photo electrochemical cells [4] because of having high efficiency of radiative recombination, high absorption coefficient, high photosensitivity, direct band gap matching to a wide spectrum of wavelengths from ultraviolet to infrared regions[5]. Semiconducting nanocrystalline thin films such as CdSe, CdS, and CdTehave attracted considerable attention owing to their remarkable optical and electrical properties, which depend on crystallite size[6-9]. The optical properties, including broadband absorption and high luminescence, make these materials attractive in photovoltaic applications[10, 11]. CdSe has shown to be the most promising candidate owing to its direct band gap, high absorption coefficient, n-type conductivity, and crystallite size- tunable band gap that can vary its optical response over the entire visible range of the electromagnetic spectrum[12-14].Although there are many reports available on the optical and transport properties of CdSe thin films, the optical and sub-band gap absorption parameters are much less explored [15-19]. The performance of the devices based on CdSe thin films depends on the structural and electronic properties of the layers obtained under various experimental conditions [20]. The reported thin film deposition methods for CdSe are mainly based on vacuum and non- vacuum film deposition, spray pyrolysis[21], electrochemical deposition [22], vacuum evaporation, co evaporation[23-25], chemical deposition[26], laser ablation[27], hot wall deposition technique[28], and molecular beam epitaxy[29].In recent years, many efforts have been devoted to the research of doped metal chalcogenide nanostructured materials [30 - 34]. The electronic and optical properties of semiconductors are strongly influenced by the methods of preparation and doping process, which provides the basis for tailoring the desired carrier concentration and conscquently the absorption, emission, and transport properties. Nanocrystalline thin films with varying concentrations of dopants are a matter of interest from the view point of basic physics and applications. The Raman spectroscopy has been widely used to characterize the phonon properties of thin films.Due to the dependence of Raman shift, intensity, and linewidth on temperature, recently, Raman spectroscopy has also been actively used for thermometry.

In the present work, we present a systematic study on the investigation of structural, Raman, and electrical properties CdSe thin films deposited by PLD with varying different substrates temperaturewhich performance a significant impact on the CdSe layer. The substrate temperatures were fixed at 150^{0}C (S1), 200^{0}C (S2), 250^{0}C (S3), and 300^{0}C (S4), respectively, during the growth of samples.

II. Experimental

CdSe Powder(99.999%, Sigma Aldrich Chem. Co.) was used to make the target of the PLD technique.

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The target was sintered at 850^{0}C in the Ar gas atmosphere in vacuum condition for 5 hours. The laser beam was focused onto the target material surface using a lens of focal length 5 cm. The target was maintained in continuous horizontal and vertical displacement to refresh the ablated zone. The incident laser pulse energy and repetition rate for the film deposited on ITO substrates was obtained as 90 mJ, and 10 Hz, respectively. The target and the glass substrate holder were rotating at 5 rpm and -5 rpm, respectively. The distance between the substrate holder and the target inside the vacuum chamber was ~5 cm. A resistive type heater was used to heat the substrate temperature to reach the set value. The substrates were clean up sequentially, using isopropyl alcohol, detergent solution, methanol followed by deionized water in the ultrasonic bath cleaner and subsequently,CdSe thin films were deposited by PLD on the highly cleaned Indium Tin Oxide (ITO) coated glass substrates. To obtain free-risking pinhole thin film each parameter of the PLD was optimized (25 milli torr, 250 milli J) at high vacuum condition using a KrF Laser(248nm, 10number of shots per sec,30Hz) to ablate a sintered CdSe target in nitrogen atmosphere. Laser ablation was carried out by scanning the sample inside the vacuum chamber which is kept at 10^{-6} Torr, by using a molecular pump together with a mechanical pump [26]. Thin film of thickness around 200nm were prepared using pulsed laser deposition technique. No reactive gas was introduced to the chamber only N_2 gas pass in the chamber for deposition with the 25ml torr pressure.

Phase purity of synthesized CdSe thin films were characterized using X-ray diffraction method (Rigaku, Smartlab) using Cu(K_α) 1.54Åoperated in $\theta/2\theta$ configuration. Long scanning times were carried out to obtain good quality of diffraction patterns. A PHI 5000 Versaprobe system was used to examine the surface chemistry with X-ray photoelectron spectroscopy (XPS) analysis using Al K_α X-rays of energy 1486.6 eV. Raman spectroscopic measurements were carried out using a Horiba–Yobin (T-64000) *micro*-Raman system using a laser excitation line 514.5 nm from an argon ion laser.The surface morphology of the films were investigated employing atomic force microscopy (AFM) (Digital Instruments, Veeco Metrology Group) in the contact mode.The transmittance of thin films was performed using a Lambda 950 UV–vis spectrophotometer from PerkinElmer equipped with a spectralon integrating sphere. The I-V measurements were carried out by using aprecision impedance Analyzer (4294A) in the frequency 40 Hz –110 MHz in the temperature 273 – 373K in the interval of 10 ^{0}C.

III. Results and Discussion

a) Structural characterization

Fig. 1 shows the typical XRD pattern of CdS thin films. Diffraction patterns were recorded in the 2θ range of 10^0 to 80^0 for each layer. These patterns were used to identify the crystal structures, phases and lattice planes for the observed diffraction peaks from atomic planes of deposited thin films.The XRD pattern shows the highest intensity peak at $2\theta = 24.3^0$ along (111) with another small intensity peak at $2\theta = 44.6^0$ along (311).These peak are indexed using standard results JCPDS card no.00-019-0191 having cubic structure with lattice constant a = b = c = 6.077.Some distortion also obtained due to ITO substrate and most distortion are marked as (*). As the substrate temperature increased from 150°C to 300°C the crystallinity of thin films also changed. The effect of grain size, peak broadening may be due to other reasons, such as inhomogeneous strains, lattice bending, twinned structure or other point defect that may be present in the CdSe nanomaterials; hence Scherrer's method may produce results which are different from the actual size[35].The crystallite size varied from 50 nm to 120 nm at different substrate temperature. The cubic structure is obtained corresponding to the peak (111) with a=b=c =5.984.

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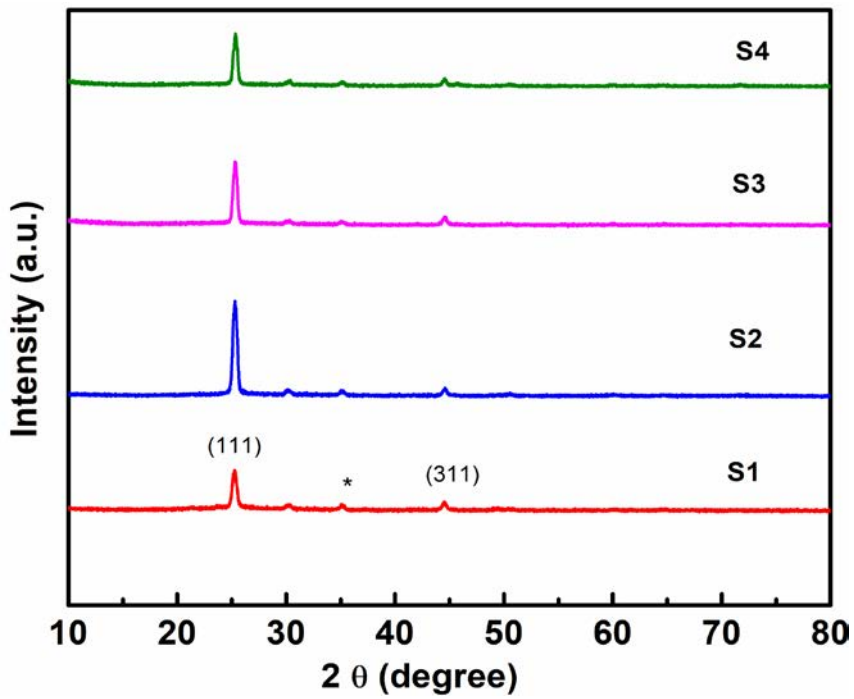

Fig.1: XRD diffraction Pattern CdSe thin films

b) X-ray Photoelectron spectroscopy (XPS)

The XPS spectra of the CdSe thin films were analyzed to confirm their compositions as shown in Fig.2. The different survey spectra obtained using Al K_α X-rays with energy 1486.6 eV. The survey diagram (Fig. 2(a)) of CdSe thin film indicates the presence of cadmium (Cd), selenium (Se), carbon (C), and oxygen (O) elements. The detected carbon is related to the carbon adsorbed on the surface during the exposure of the sample to the ambient atmosphere. All binding energies were corrected for the charge shift using the C 1s peak of graphitic carbon (284.6 eV) as a reference [36].High resolution core level spectrum for Cd 3d is shown in Fig.2 (b). The two peaks in Cd 3d core level spectrum arise with Cd3d5/2 peak position at 405.03 eV and Cd3d3/2 at 412.18 eV binding energies respectively. The binding energy of Cd $3d_{5/2}$ indicates the Cd^{2+} states; this is in good agreement with the literature [37, 38]. Both sample S1 and S3 shows the same peak position for Cd 3d. High-resolution core level spectrum for Se 3d depictedin Fig.2(c). Core level spectrum for Se 3d was fitted using singlepeak for binding energy at 54.10 eV [39, 40].The area of the peak remain same but a little shift obtained in Se 3d peak that may be due to lattice mismatching as some distortion obtained in the XRD results. The values of binding energies for Cd and Se are at their respective positions for Cd^{2+} and Se^{2-} states. It means that Cd^{2+} and Se^{2-} exist in as-deposited CdSe thin films with stoichiometric formula CdSe, confirm the presence of the Cd and Se materials.

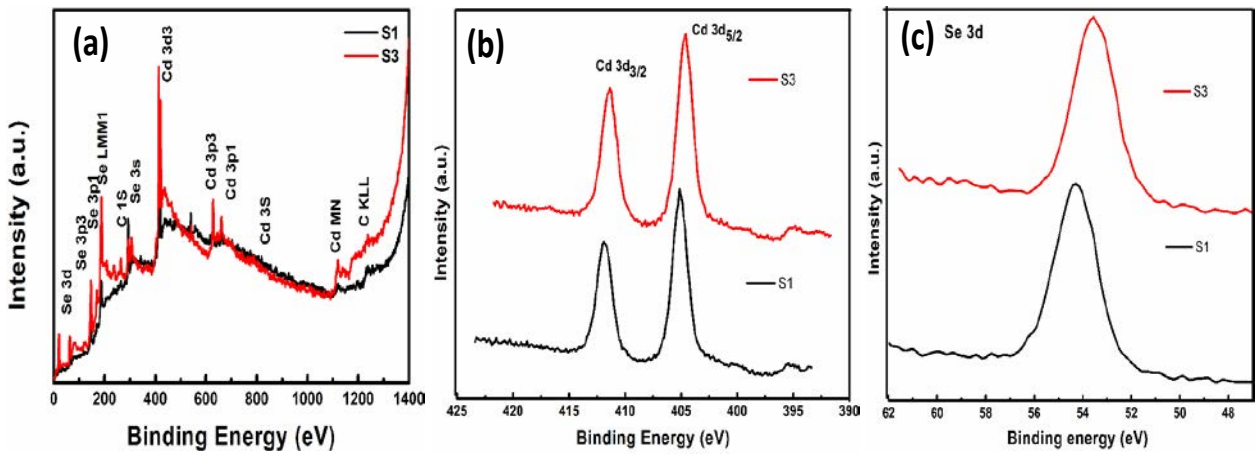

Fig. 2: XPS studies of CdSe thin films

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c) Optical absorption and transmission

Trasmission studies are carried out in the wavelength range280-800nmto investigate the optical absorption properties of CdSe thin films. Fig.3 shows the variation of transmittance T with wavelength (λ). The transmission spectra exhibit interference fringes for photon energies below the absorption edge, which shifts towards longer wavelengths with increase in temperature. The shift in absorption edge can be assigned to thermally induced defects, quantum confinement, or the formation of additional phases and change in the structural properties [41]. Moreover, a lower grain size, resulting in an increase in scattering of photons at grain boundaries, could contribute to the decrease in transmittance.

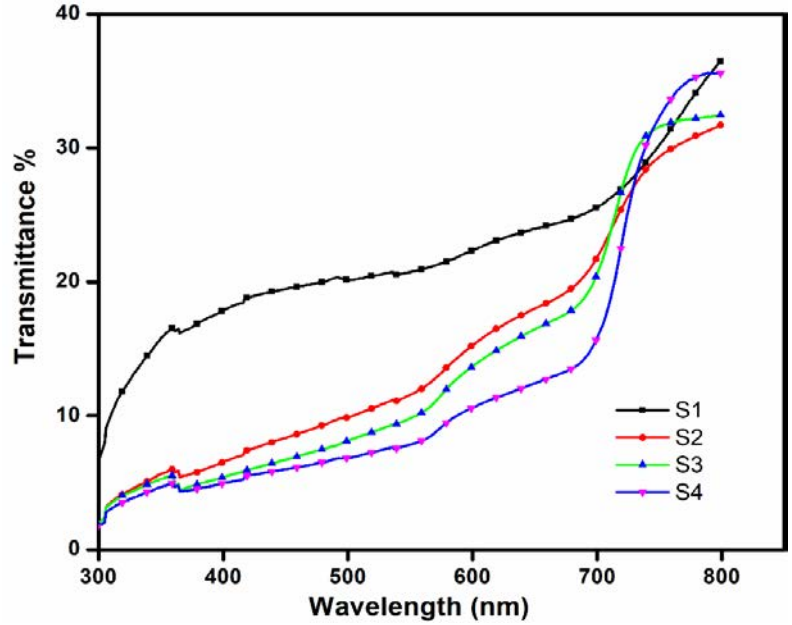

Fig. 3: Transmittance spectra of CdSe thin films

The absorbance spectra obtained from transmittance data shown in Fig. 4 (a). The optical band gap of the thin films has been determined from the absorption coefficient using the equation [42];

$$(\alpha h\upsilon)^n = B(h\upsilon - E_g)$$

Where α is the absorption coefficient, hυ is the energy of incident photon, B is a constant which is characteristic of the material, $E_g$is the optical band gap and n = 2 for direct band gap transitions or n =1/2 for indirect band gap transitions. The intercept on the abscissa of the plot of $(\alpha h\upsilon)^2$versus hυ·given in fig.4 (b), provides the direct band gap energy. The value of Eg varies from 2.3 eV to 1.75 eV for CdSe thin films owing to size quantization effect as reported earlier [43].

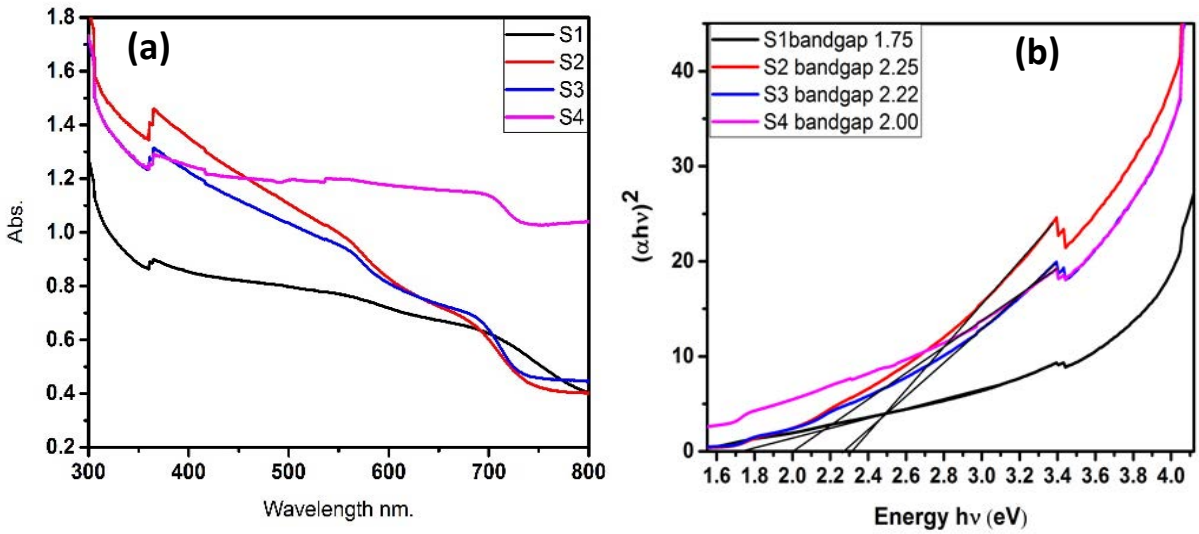

Fig. 4: Absorbance spectra and bandgap diagram of CdSe thin films

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d) Low temperature Raman Spectroscopy

Raman spectroscopy is used to identify the various vibrational modes of the molecules in the material by shining the laser beam of light onto the CdSe thin films. Raman spectroscopy is an alternative method for quality control of materials grown in aproduction line. Raman spectra are generally applied as fingerprints for identifying material phases. The optimal conditions (power and wavelength of excitation, accumulation time) for Raman scattering measurements chosen in such a way to provide the good quality spectra. In present work micro-Raman scattering was performed in the backscattering configuration using the 514.5 nm line of an argon ion laser to a spot size of ~2 μm, and with an incident power of ~3 mW.Fig. 5 shows the Raman spectra of the samples: S3 (CdSe thin films at substrate temperature 250^{0}C) at low to room temperature 83K to 300K. In the Raman Spectra it has been found that the peak at 208 cm^{-1} corresponds to the basic vibrational (longitudinal) LO mode of CdSe structure vibrations [44]. The peak in ~ 415 cm^{-1} region corresponds to the second order 2LO mode which is observed at low temperature. Some distortion also obtained marked as (*) in the spectra. The Raman peak at ~517 cm^{-1}originates from substrate property and get wider with decreasing temperature. It should be noted that scanning of the surface of CdSe thin films by micro-Raman did not reveal any changes in values of Raman shift for the related modes across the surface. It means that surface distribution of compound elements in samples is uniform.

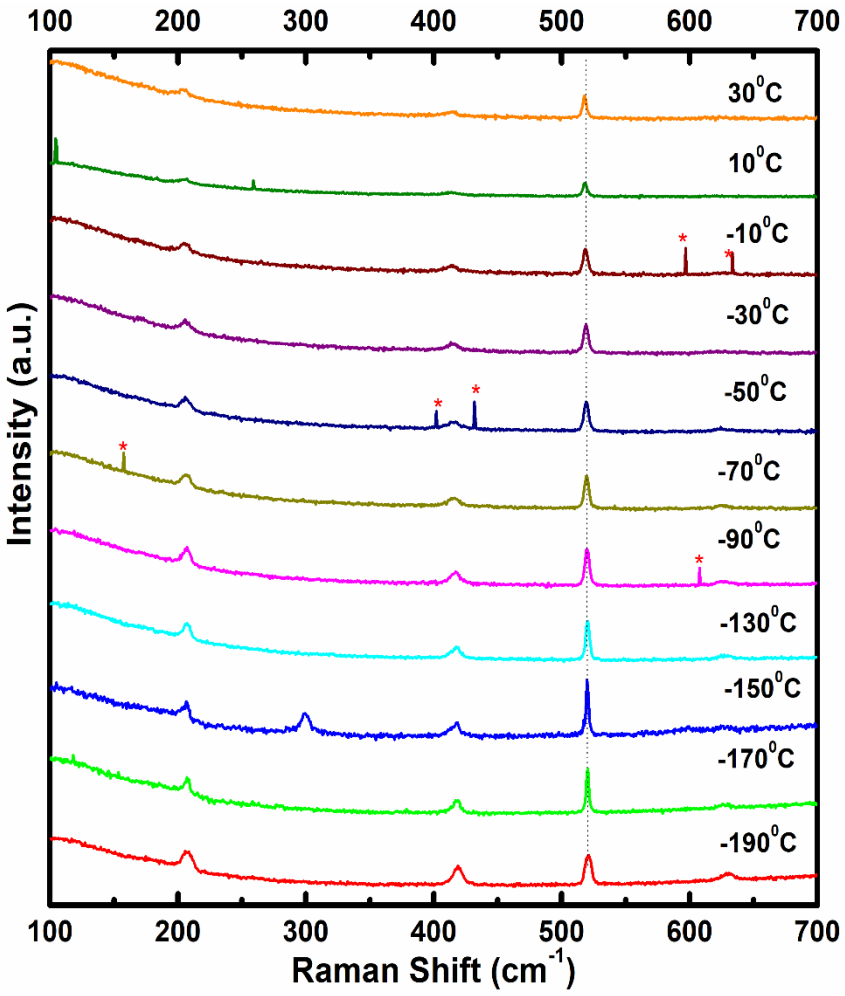

Fig. 5: Raman spectra of CdSe thin films at different temperature

e) Surface Analysis

Figure 6 shows the surface morphology of the grown films analyzed by atomic force microscopy. It is observed from the surface image that the particles are uniformly distributed on the surface of the thin film and the grains of CdSe particles were found to exist in spindle shape. Highly elliptical shape of the grains has a narrow size distribution on the surface of the film. The roughness of the film is found to be relatively low with a RMS value of 20 nm.

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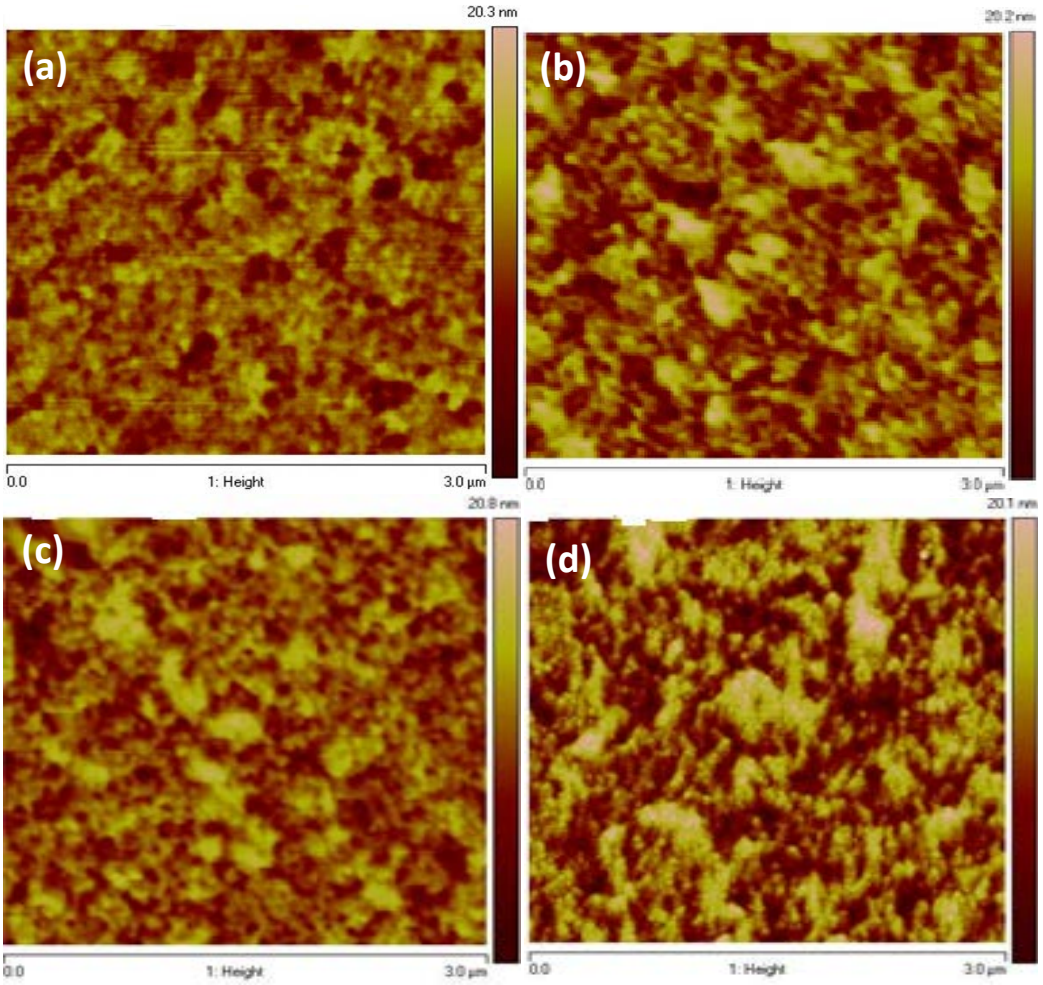

Fig. 6: AFM image of CdSe thin films at different substrate temperature

f) Electrical Measurements

The electrical property of the materials is an important factor which reveals the important and reliable information about the transport phenomenon and other physical properties of the material. The electrical properties are dependent on various film or growth parameters such as composition, thickness, and substrates temperature and deposition rate.The study of I-V characterization is done by varying the temperature from 273K to 373K of CdSe thin films. Measurement of electrical conductivity of CdSe/ITO (S1 and S3) was made by using d.c. two probe method at different temperatures and the corresponding characteristic curves are shown in Fig-7. At room temperature the current flow for 1 volt potential difference is 0.005µA while at low temperature 273K the current increases to 0.024µA which is approximately 5 times to the room temperature value. Similar work is done on the CdSe thin films (S3) and obtained that when the substrate temperature increased the conductivity also increased.S. Mahato et al. [45] also observed the variation of the electrical conductivity with temperature during heating and cooling cycles was found to be different. The low value of conductivity may be attributed to the nanocrystalline nature of thin film, discontinuities at crystallite boundary, presence of surface states and small thickness of the film. It is observed that the electrical conductivity is increased with the increase of temperature and the curves show typical semiconducting nature of thin films. [46, 47].

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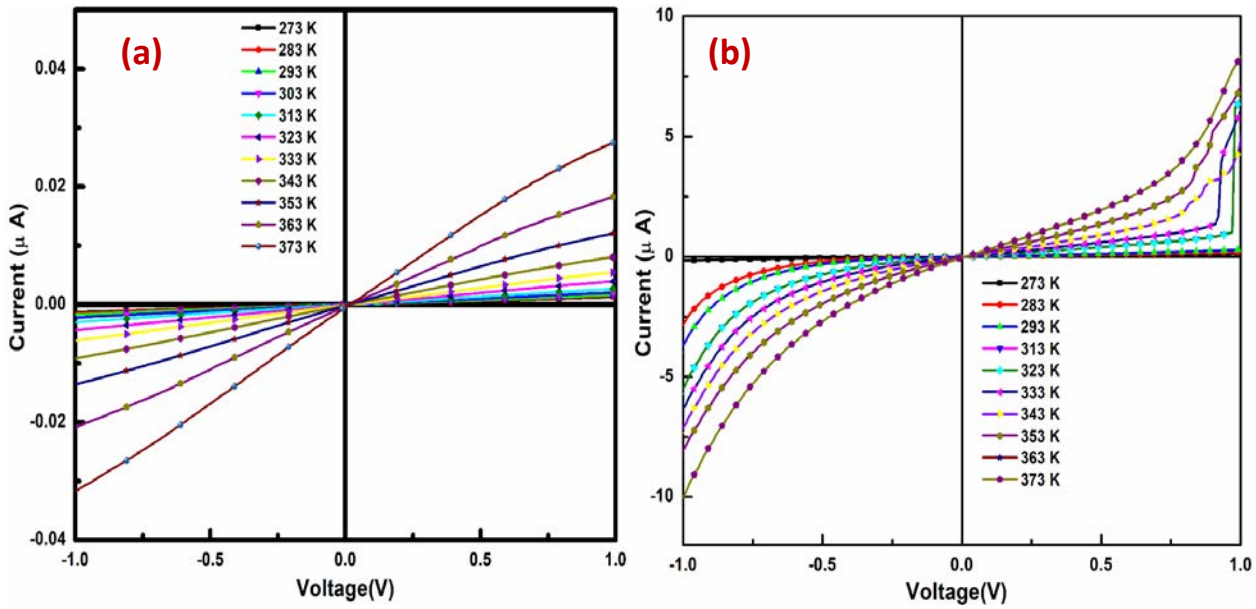

Fig. 7: I-V measurement of CdSe thin films at different substrate temperature

IV. Conclusion

The CdSe thin films successfully synthesized by pulse laser deposition method at different substrate temperature. The recorded diffraction pattern matched with the standard data and have cubic structure. The XPS results of CdSe thin film indicates the presence of cadmium (Cd), selenium (Se), carbon (C), and oxygen (O) elements. The Cd 3d (Cd^{2+}) and Se 3d (Se^{2-}) exist in as-deposited CdSe thin films. The bandgap calculated by the absorption coefficient varies from 2.3 eV to 1.75 eV for CdSe thin films. Raman Spectra shows that basic vibrational LO mode observed at 208 cm^{-1} and it's double corresponds to at 415 cm^{-1}. The AFM image shows spindle shape morphology with lest roughness. The I-V results shows that at room temperature, the current flow for 1-volt potential difference is 0.005μA while at low temperature 273K the current increases to 0.024μA which is approximately five times to the room temperature value.

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