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EVALUATION OF SURFACE MORPHOLOGICAL STUDIES OF MnS, CdS, AND Cdi_xMn_xS THIN FILMS

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ABSTRACT:

The scanning electron microscope (SEM) is a powerful tool for studying surfaces at the nanoscale level that is utilised in a wide range of fields. SEM probes only a section of the complete image at a time, and the image is built up serially by scanning the probe. It has the ability to create high-resolution photographs of a sample surface. SEM is a device that focussed a beam of high energy electrons to generate a range of signals at the surface of solid specimens. It gives a visual display of the surface layer with a high depth of focus larger than that attainable with an ordinary electron microscope. The signals produced by electron sample interaction disclose information about the sample's exterior shape, chemical content, crystalline structure, and orientation of the materials that make it up. The incorporation of Mn into the host matrix was confirmed by a drop in (002) peak intensity and a minor change in peak location as Mn content increased. The presence of peaks in the XPS survey scan spectrum corresponding to Cd, Mn, and S validated the compositional purity of the deposited films. In this research study**,** the decrease in particle size with increasing Mn content was confirmed by scanning electron microscopy .

Keywords: Scanning Electron Microscope, Particle, Size, Films.

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INTRODUCTION:

SEM scans an object's surface with an electron beam. Depending on the topography of the object, electrons strike the surface and cause more or less electrons to be released. A detector collects the difference in electron density and generates a two-dimensional image with black and white pixels. In scanning mode, areas ranging from 1 cm to 5 microns in width can be studied using traditional SEM techniques. A system of ion optics focuses electrons produced by thermionic emission from an electron cannon down to a location on the object (i.e. electromagnetic coils). The spot is scanned (or rastered) over the surface of the sample with a set of scan coils, and reflected electrons are collected, amplified, and transformed into a video signal. As a result, a micrograph of the specimen in the form of a 2-D plot of reflected electron intensity is obtained. SEM was used to examine the surface morphology of deposited films (Tescon Vega 3) [1,2].

EXPERIMENTAL METHODS:

The microstructure of MnS films was examined using a scanning electron microscope. SEM images of NSP produced MnS films on glass substrate at different molar ratios (1:1 and 1:3) and substrate temperatures (300 and 400 °C) are shown in Fig. shows SEM images of MnS films formed on glass substrate with the addition of EDTA at the specified conditions. The surface morphology of MnS films is dependent on the molar ratio and substrate temperature, as seen in Figs.. The micrographs show that the films are homogeneous, without cracks, dense, and almost completely cover the substrate surface.

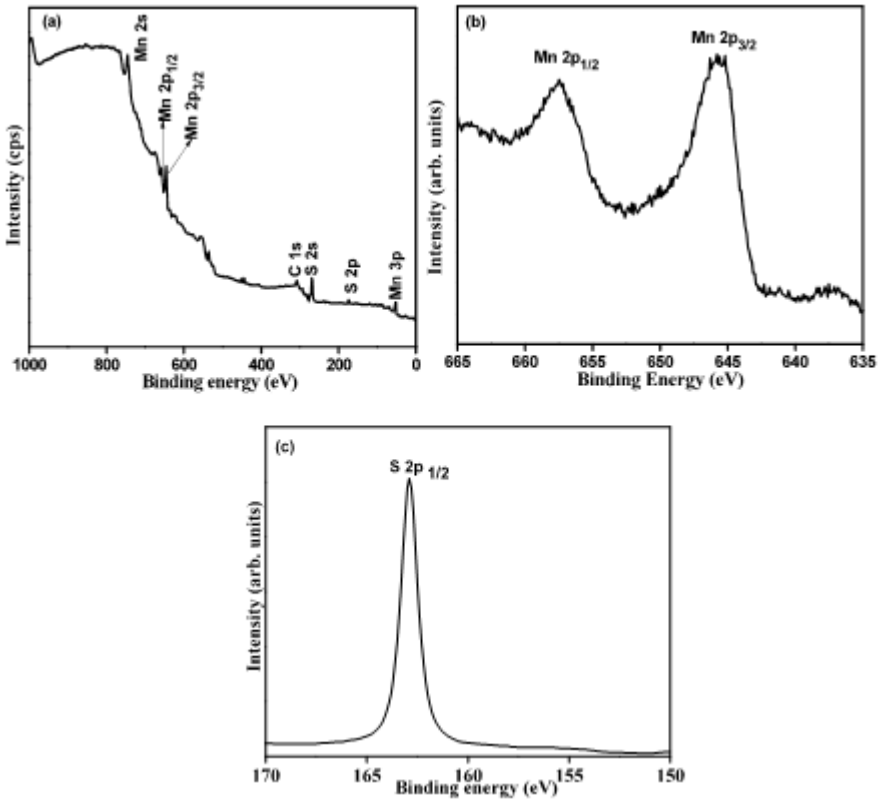

Fig. 1: XPS spectrum of MnS film deposited on glass substrate at 1:1 molar ratio: (a) survey scan spectrum, (b) Mn 2p core level spectrum, and (c) S 2p core level spectrum.

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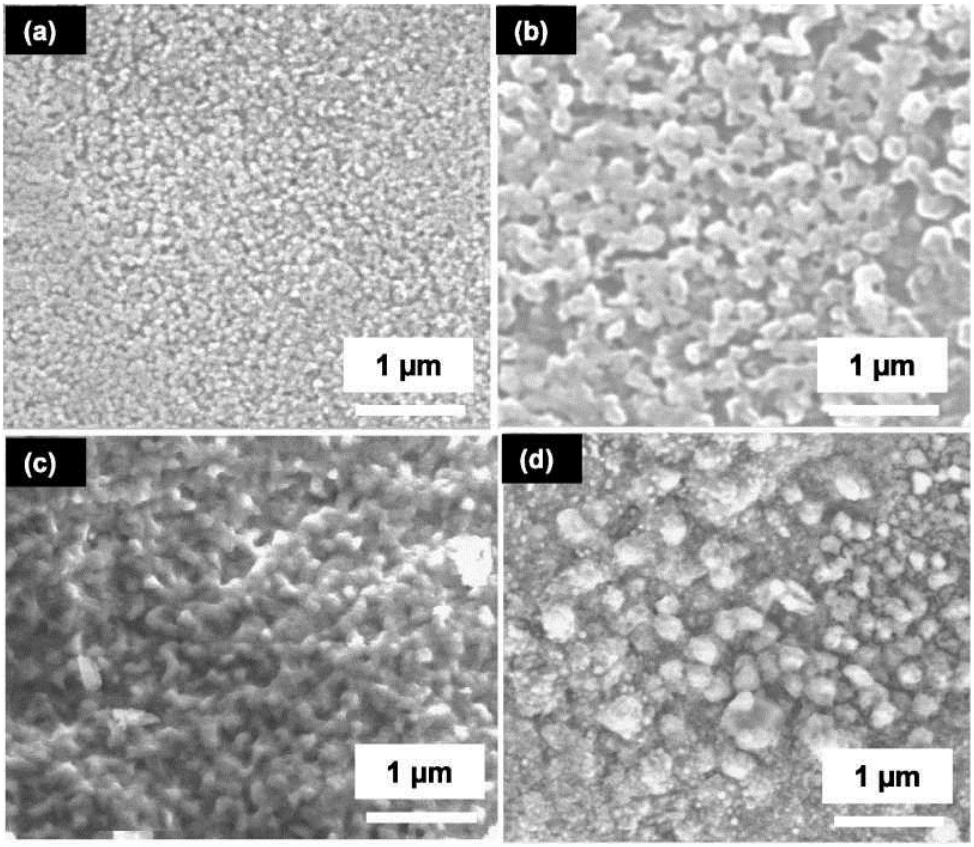

Fig. 2 : Scanning electron microscopic images of MnS films deposited on glass substrates at various molar ratios and substrate temperatures: (a) 1:1 at 300 °C, (b) 1:1 at 400 °C, (c) 1:3 at 300 °C, and (d) 1:3 at 400 °C.

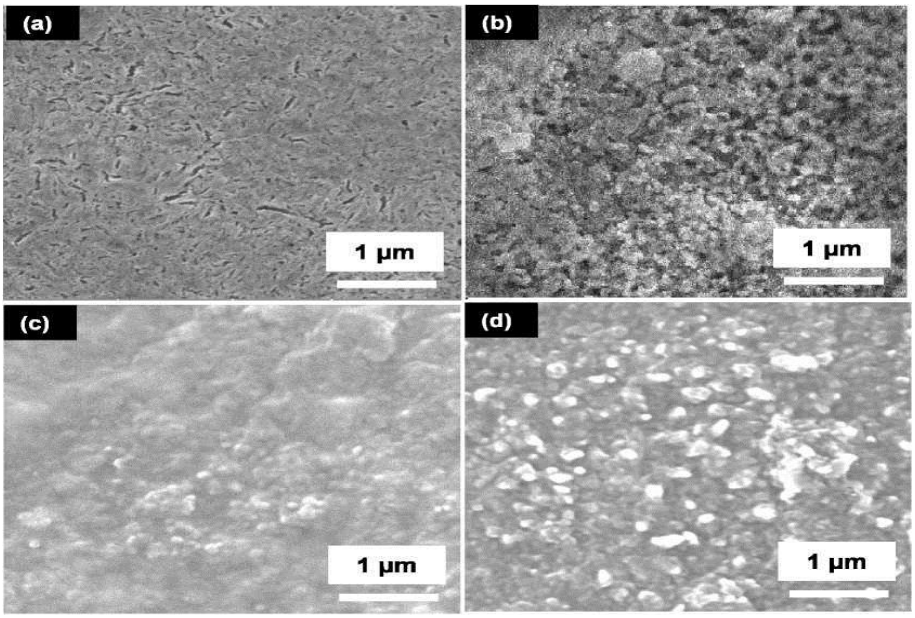

Fig. 3 : Scanning electron microscopic images of MnS films deposited on glass substrate at various molar ratios and substrate temperatures with the addition of EDTA: (a) 1:1 at 300 °C, 1:1 at 400°C, (c) 1:3 at 300 °C, and (d) 1:3 at 400 °C.

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SEM pictures of CdS thin film formed on glass substrate at 300°C and 400°C for 0.04 and 0.20 molar concentrations are shown in Fig.. As the substrate temperature rises from 300 to 400 degrees Celsius, the surface morphology of the films changes from less crystalline and porous to more crystallised and dense films. In most situations, the resulting coatings are uniform, free of cracks, dense, and cover the substrate surface almost completely. The SEM image of CdS film deposited at 300 °C with 0.04 M is shown in Fig.. The samples have a hierarchical profile of three-dimensional leaf-like structures, which can be seen clearly in the SEM image. The sample is made up of well-defined CdS dendrites, as illustrated. An solitary dendrite has several trunks in its intricate arrangement. Each trunk is made up of several branches, each of which is made up of many smaller sub branches, giving the CdS a hierarchical structure. This morphology resembles those described in the literature [3]. The image in Fig. shows a well-defined uniform distribution of agglomerated particles. In the SEM picture for film deposited with 0.20 M at 300 °C, as shown in Fig., the massive crystal grains are clearly apparent (c). The grain size of CdS grows as the molar concentration increases from 0.04 M to 0.20 M.

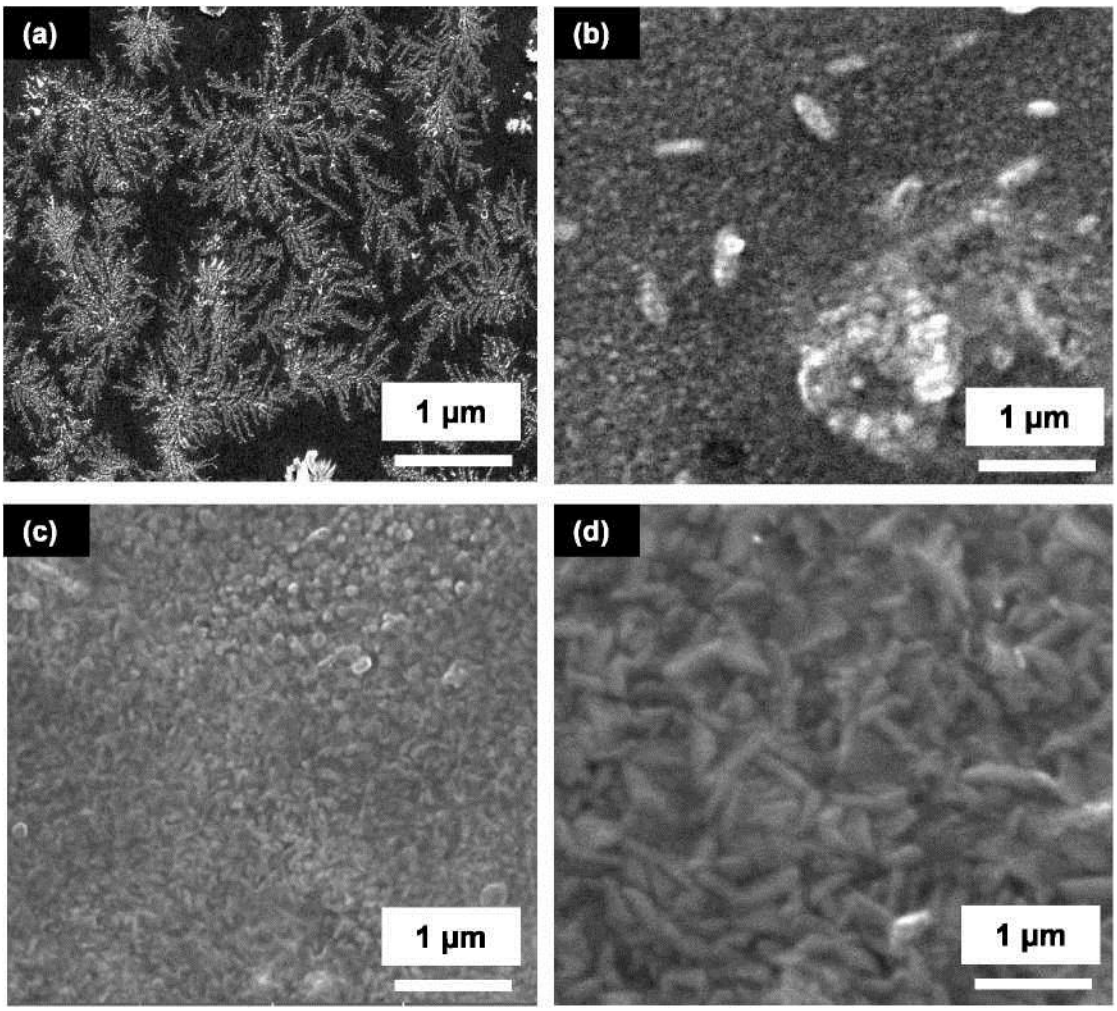

Fig. 4 : Scanning electron microscopic images of CdS films deposited on glass substrate at various molar concentrations and substrate temperatires : (a) 0.04 M at 300°C (b) 0.04 M at 400°C (c) 0.20 M at 300°C and (d) 0.20 M at 400°C

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The surface topology of Cdi-xMn_xS film was studied by scanning electron microscope. Fig. shows the SEM micrographs of Cdi-xMnxS (x = 0.04, 0.10, and 0.20) films deposited at 300 °C and 400 °C. The surfaces of all the films are smooth with well covered grains on the substrate. It is observed from the SEM images that the films are having the surface with distribution of particles and clear grain boundaries with some pores.

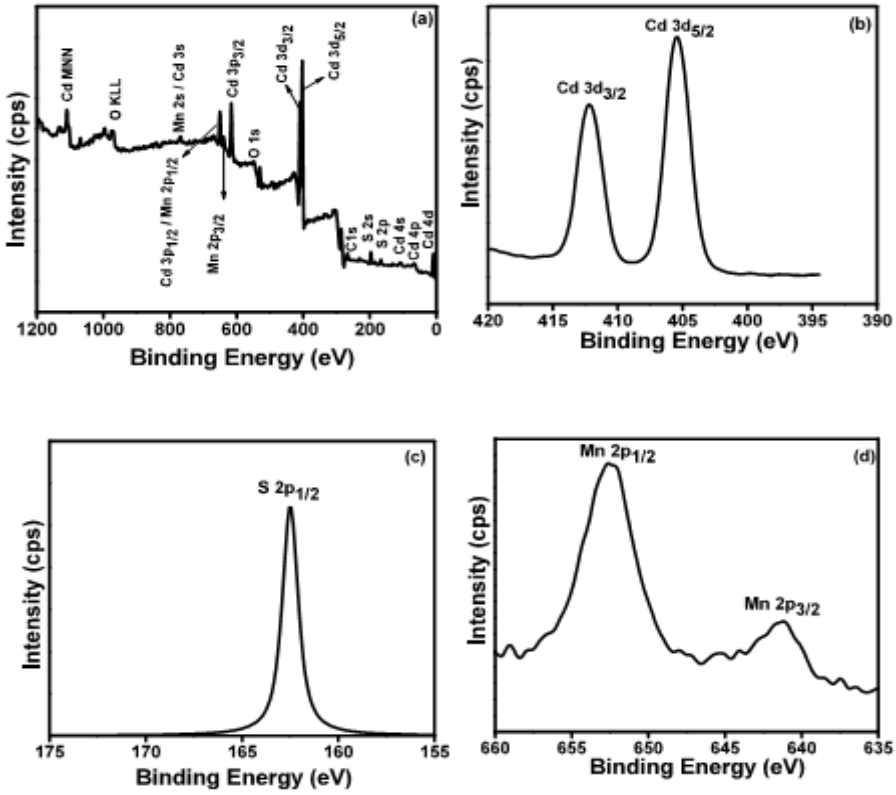
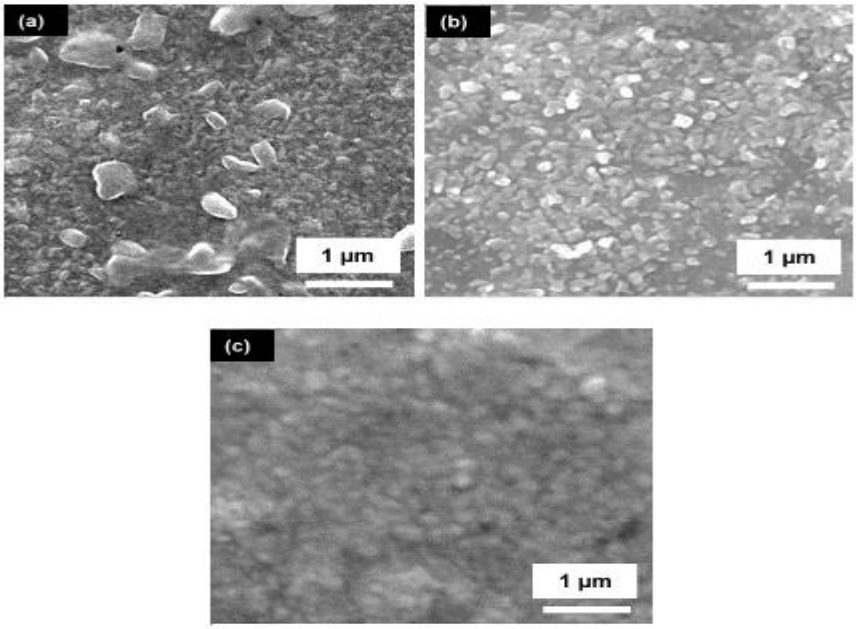


Fig. 5: XPS spectrum of Cdi-$_x$$Mn_x$S film deposited at 400 °C with x=0.20: (a) survey scan spectrum, (b) Cd 3d core level spectrum, (c) S 2p core level spectrum and (d) Mn 2p core level spectrum



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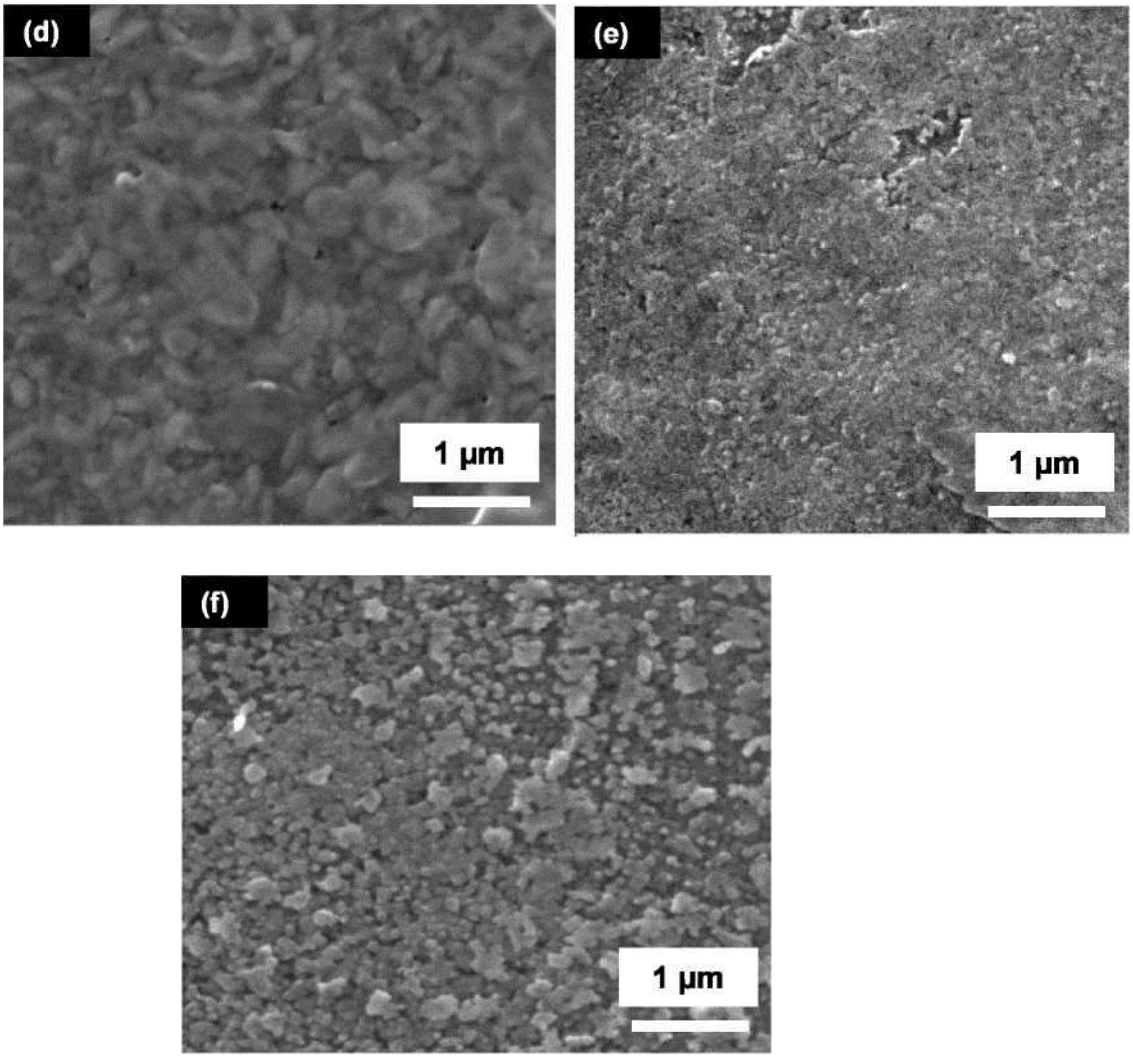

Fig. 6 : SEM images of $Cd_{1-x}Mn_xS$ thin films deposited at various 'x' and sustrate temperatures : (a) x = 0.04 at 300°C, (b) 0.04 at 400°C, (c) x = 0.10 at 300°C, (d) x = 0.10 at 400°C, (e) x = 0.20 at 300°C, and (f) x = 0.20 at 400°C

RESULT AND DISCUSSION:

The grains on the MnS film surface at lower substrate temperatures and molar ratios are obviously smaller in size than those at higher substrate temperatures and molar ratios. For the film deposited at lower molar ratio and substrate temperature, homogeneously distributed spherical particles with almost uniform size were found. The creation of a netted surface by the agglomeration of crystal grains occurs when the precursor solution concentration ratio and substrate temperature increase (Fig. b-d). This is because more particles are accessible to coalesce at greater solution concentration ratios, resulting in increased particle size and film uniformity. The content of S2" ions produced by thiourea is low to form highly crystalline MnS thin films when the S/Mn molar ratio is 1.0. With a higher S/Mn molar ratio (2.0 and 3.0), the high concentration of thiourea causes more S2' to be released, resulting in faster deposition on the substrate, faster nucleation with Mn2+, and highly crystallised MnS thin films. The granularity, on the other hand, is barely visible in the film deposited with EDTA at

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1:1 and 300 °C. The grains begin to agglomerate to beginning crystal formation as the molar ratio and substrate temperature increase. The SEM pictures reveal increased grain formation and a compact nature made up of single type densely packed grains. The particles agglomerate and grow dense as the substrate temperature rises, forming a homogeneous pattern. The addition of EDTA caused the grain size to decrease, as shown in Figs.. The gradual release of Mn2+ ion from the combination is responsible for the reduction in particle size. The S/Mn molar ratio appears to have a significant impact on the crystallisation and shape of MnS thin films. Ulutus et al. [4] reported similar morphological findings. The agreement with XRD results is strengthened by a significant improvement in topographical features and the substrate temperature and molar ratio driven enhancement in crystallisation of the films. Based on the SEM findings, it is determined that nebulized spray pyrolysis is a viable approach for depositing device-quality MnS films.

The grain size of CdS grows as the molar concentration increases from 0.04 M to 0.20 M. The morphology of the sample is rod-like. The morphology of the grains changed from interconnecting regularly formed grains to well grown rod-like morphology with better surface area for light scattering and absorption as the substrate temperature increased. The evolution of such surface morphology can be explained by the fact that as molar concentration rises, linked grains form first, giving enough time for well-ordered and well-grown rod-like morphology to emerge. The creation of a densely packed and homogeneous surface morphology as the reaction progresses is related to the minimising of surface free energy and the accumulation of CdS particles. Such a homogeneous, self-organized structured morphology is advantageous for increasing photon energy absorption surface area. Because of the substrate temperature, grains are numerously grown throughout the entire substrate surface with improved crystallinity and laterally grown sharp edge rod-like morphology (length-500 nm) with reduced grain boundaries. The deposition temperature aided in the creation of homogeneous surface morphology and improved crystallinity [5].

The particles size decreases with the increase in Mn concentration in the morphology of Cdi-xMnxS films. This may be due to the fact that the incorporated Mn^{2+} acts as a grain growth inhibitor [4]. In addition, the decrease in grain size may be due to pinning effect induced by Cdi-xMn_xS particles on grain boundaries and also due to the dragging effect between the doped Mn and grain boundaries [5]. On seeing the morphology of the films, it is found that the compactness slightly increases with increase in substrate temperature. There are hard white regions on the surfaces of Cdi-xMnxS films and the size of these regions increases depending on the Mn concentration (x = 0.2). These undesired formations may be a result of the high stress in the films or thermal difference between the films and the substrates [5]. Grain boundaries are well known to be carrier-scattering centers, so carrier scattering at grain boundaries declines when the grains grow. Thus it is clear that the films deposited at lower substrate temperature have more grain boundaries than the one deposited at higher substrate temperature. The decrease in grain size leads to the increase in grain boundaries which produce more strained films. This is consistent with our XRD results. The substrate temperature and Mn concentration play a vital role for the change in the morphology of Cdi-xMnxS films. The granular surface morphology got diminished at higher value of ‘x’ which clearly explains the effect of Mn incorporation in CdS.

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CONCLUSION:

SEM analyses reveal the production of homogeneous, crack-free coatings with greater grain formation and well-defined edges. The sluggish release of Mn2+ ion from the combination caused the grain size to decrease when EDTA was added.

The CdS films are homogeneous, without any cracks, dense, and almost completely cover the substrate with diverse nanostructured morphological features, according to surface morphological examinations using a scanning electron microscope. The variance in surface energy of the structure could explain the variation in morphological properties of CdS films with growing circumstances.

The decrease in particle size with increasing Mn content in Cdi-xMnxS films was confirmed by scanning electron microscopy and atomic force microscope surface morphology examinations. This could be owing to the grain growth inhibitory effect of incorporated Mn2+ ions.

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