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PREPARATION OF COLLOIDAL TIN SULFIDE NANOPARTICLES BY PULSE LASER ABLATION IN LIQUID (PLAL)

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ABSTRACT

Nanoparticles (NP_S) of Tin sulfide has been prepared by laser ablation of a solid target (SnS) in water solution. SnS colloidal nanoparticles NPs have been synthesized by laser ablation Nd:YAG (1064nm ,100 pulses, pulse energy= 400mJ) when the solid target Tin sulfide SnS was immersed in water H_2O. Structure, topography,morphology Optical studies of the SnS NP_S have been studied using X-ray diffraction (XRD) , atomic force microscope (AFM), scanning electron microscope,tunneling electron microscopy (TEM) and UV-Vis spectrophotometer respectively.

KEYWORDS: ***Tin sulfide, Thin film, AFM , SEM, Energy gap, Laser ablation***

1. INTRODUCTION

Tin Sulfide thin films (SnS) have attracted much attention, due to its high absorption (>10^4 cm^{-1}) (Mariappan R.,et al., 2011), (Yue G.H.,et al., 2009), (Guneri E.,et al., 2010), (Kawther A., et al., 2017) nontoxic and friendly environment, low cost material, can be n or p type depending on kind of application. Its crystal structure was found as zinc blend (Gao C., et al., 2011) and as orthorhombic (Sohila S., et al., 2011), (Reddy K.T.R., et al., 2006), (Loferski J.J., 1956). Its conductivity can be controlled by adding different

dopants (Guneri E., et al., 2010). Sn and S are abundant in nature (Calixto-Rodriguez M., et al., 2009).The optical properties of SnS can be tailored by variation of fabrication process, but nearly all researchers agree with tunable direct (1.2 - 1.5) eV and tunable indirect (1.0 - 1.2) eV band gap values. These properties make SnS thin films as a good candidate for optoelectric and photovoltaic applications(Koteeswara Reddy N., et al., 2016), (Ran Fan-Yong, et al., 2016), (Avellaneda D., et al., 2015), (Patel M., Ray` A, 2014), (Vasudeva R. M., et al., 2015), (Hegde S.S. , et al., 2018), (Zaki Sh. A., et al., 2018). Tin Sulfide thin films can be prepared utilizing different techniques such as electrochemical deposition(Ichimura M., et al., 2000), Successive ionic layer adsorption and reaction (SILR) (Ghosh B., et al., 2008) plasma-enhanced chemical vapor deposition (Sa´nchez-Jua´rez A., et al., 2005), chemical spray pyrolysis (Sajeesh T.H., et al., 2010) chemical bath deposition(Joshi L. P., et al., 2015), (Loranca-Ramos F.E., et al., 2018) and dc magnetron sputtering (Ramakrishna Reddy K.T., et al., 2002) and Liquid phase deposition(Shahara Banu, et al., 2017), In this work, SnS NPs is obtained by laser ablation in liquid (water) at 400 mJ in order to study their structural and morphological properties.

2. EXPERIMENTAL WORK

Nd: YAG laser (type HUAFEI) operating at 1064 nm and 7 ns pulse duration was used together with 11 cm positive lens to ablate 99.99 % purity, 2.0 cm diameter SnS pellet. The pellet was placed at the bottom of a glass vessel; containing 5 mL of water without the addition of any surface active substances. The ablation process was performed at normal pressure and in open air with laser energy 400 mJ per pulse and 20 min ablation time.Laser pulse energy was measured using well calibrated joulmeter. The diameter of the laser beam was 2.12 mm measured by an optical microscope. XRD (XRD-6000, supplied from Shimadzu Japan) was used to estimate crystallinity of nanoparticles. Tunneling electron microscope (TEM) (type CM10 pw6020 Philips) and Scanning electron microscope SEM (T-scan Vega III Czech) was used to study the morphology of nanoparticles (In case of SEM , the material was deposited on glass substrate with film thickness not exceed 200 nm, while for TEM we use a solution of 100 ml. Atomic force microscope AFM (AA 3000 scanning probe microscope) was employed to study the topography of nanoparticles. The absorbance of colloidal nanoparticles was measured by

using UV–Vis double beam spectrophotometer (CECIL, C. 7200, France). Hall measurements were carried out using Ecoia, HMS-3000 system (van der pauw configuration) to estimate the ohmic .

3. RESULTS AND DISSECTION

The XRD diffraction patterns of synthesized SnS films which deposited on glass at 50 ^{o}C annealing temperatures for one hour is shown in figure 1. The spectra reveals that the presence of traces of other phases along with predominant SnS phase.In the lower angle side it seen that the material exhbit amorphous nature , which might be attributed to the transform the material to nanoparticle , which led to disappear the Bragg surface and decrease the disorder of crustallanity. Degree of crystallinty was also found to increase with substrate annealing temperature . The films deposited on glass showed peaks mainly of SnS phase, corresponding to the (111) plane of orthorhombic crystal structure , which were well matched with standard peaks (JCPDS card 33-1375) for herzenbergite SnS, and this result good agreement with Parenteaum and Carlone (Parenteaum M. , Carlone C., ,1990). While the peak at ≈25.3^{o} indexed with (021) plane, which agrees with Zainal et al. (Zainal Z., et al., 1997).

The crystalitte size D for a knowing X-ray wavelength λ at the diffraction angle θ of SnS nanoparticles was calculated by using Scherrer formula (Subramanian B., et al., 1999)

$$D = \frac{0.9\lambda}{\beta \cos(\theta)} \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

where the β is the full width at the half maximum of the preferred peak in units of radians and K is the shape factor ,their values are in the range of (1-0.89). The crystallite size is found to be around 28 nm.

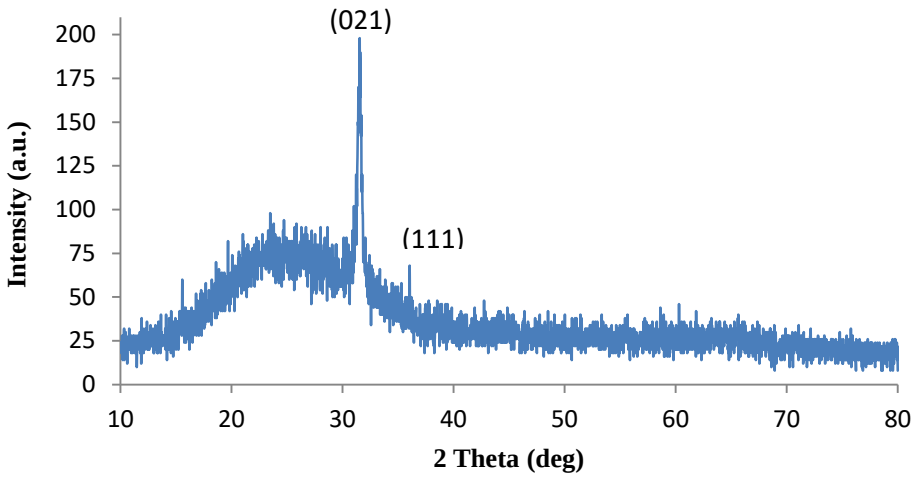

Fig. 1: XRD spectra of SnS thin films deposited on glass substrate.

The composition investigation of the SnS films shows that the tin and sulfur content in the films gradually varied with the increase of annealing temperature.

Scanning electron microscopy (SEM) is a convenient technique to study nanostructure of SnS films which deposited on glass substrates .

Figure 2 shows SEM image of multi-arm nanostructured SnS prepared at 400 mJ laser energy. It is clearly seen that a mixture of, monopod rods, bipod rods, and tripod rods were observed.For SnS nanostructure synthesized with 400 mJ, the average particle size was less than 50 nm and agglomerated particles were noticed, the nanoparticles were converted into multiarm micro and nanostructures with arms .

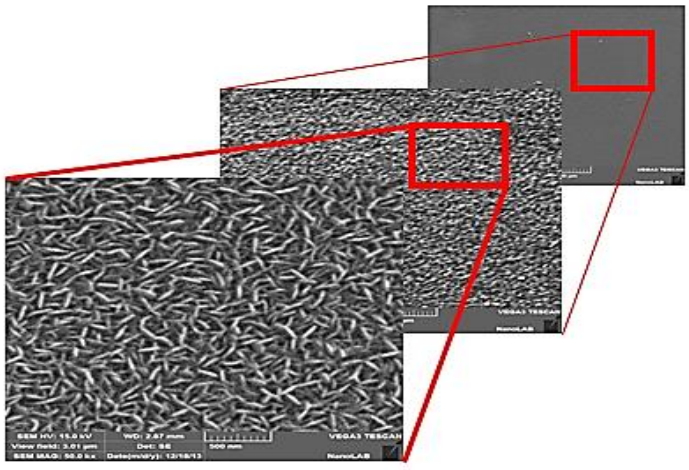

Fig. 2 SEM images of SnS nanoparticles prepared with laser energy 400m J .(magnified images)

Figure 3 shows 3D and 2D AFM image of nanostructured SnS prepared at laser energy 400m J . The surface of the substrate is well covered with SnS nanoparticle distributed uniformly on the surface. It is obvious from this figure that the nanoparticles prepared at 400 mJ laser have small ordered particles with pencil shape with the existence of some monopod rods.The average particle size estimated with the aid of software was about 50 nm. The value of particle size is higher than that calculated from XRD analysis . This is because XRD depends on the size-defect free volume, while AFM directly visualizes the grain without taking into account the degree of crystal defects (Albers W.,et al.,1961).

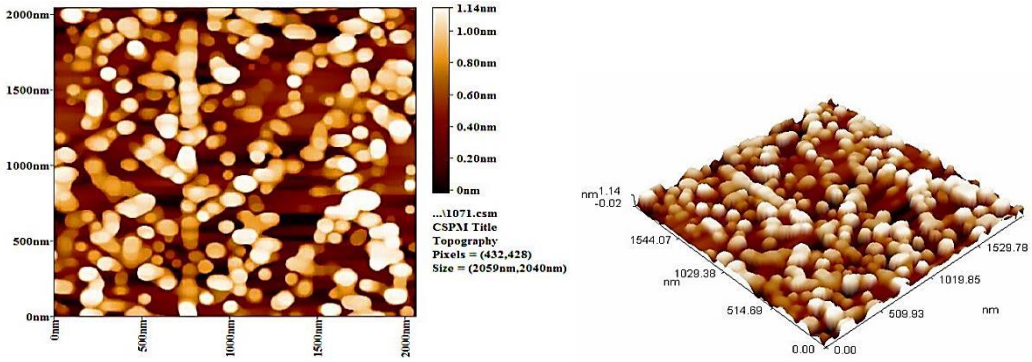

Fig. 3:AFM images and granularity accumulation distribution chart of SnS thin .

Table (1) illustrate the grain size of nanoparticle and roughness.

Table 1: Average grain size, Roughness average and RMS .

Thin film	Average grain size (nm)	Roughness average (nm)	RMS (nm)
SnS thin film	54	2.14	2.5

The TEM images for SnS nanoparticles ablation in methanol is shown in Figure 4. The micrographs confirm the formation of well-defined spherical nanoparticles also, shows that the SnS NPs prepared with 400 mJ laser fluence have different size, it varies from 10 to 45 nm. These NPs have qusi-sperical shapes.

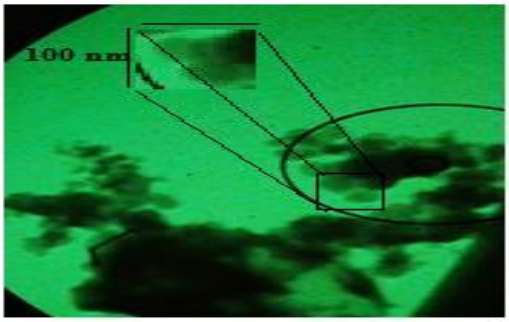

Fig.4 *TEM of SnS colloidal nanoparticles dissolved in water and prepared with 400 mJ laser fluence.*

The optical band gap of the SnS films was calculated from the absorption spectra.

Figure (5 inset) displays the absorption as a function of wavelength. It is obvious that the film gives good absorbance characteristics at the spectral range (400- 900) nm. The films show an increase in absorbance after annealing the film at 50 ^{o}C, respectively. This is perhaps due to the growth in grain size and the diminution in the number of faults.

Figure 5 shows the band gap of SnS thin films estimated from $(\alpha h\upsilon)^2$ versus $h\upsilon$,where α is the absorption coefficient ,by extrapolating the linear portion of the curve with the photon energy axis . The value of the optical energy gap was found to be 2.55eV. The increasing of energy gap which was attributed to fluctuation of absorption edge, could be due to the energy band structure and the variation of density of state to the energy level. The poor crystallinity of the films may also lead to higher optical band gaps (Ismail R. A., 2011).

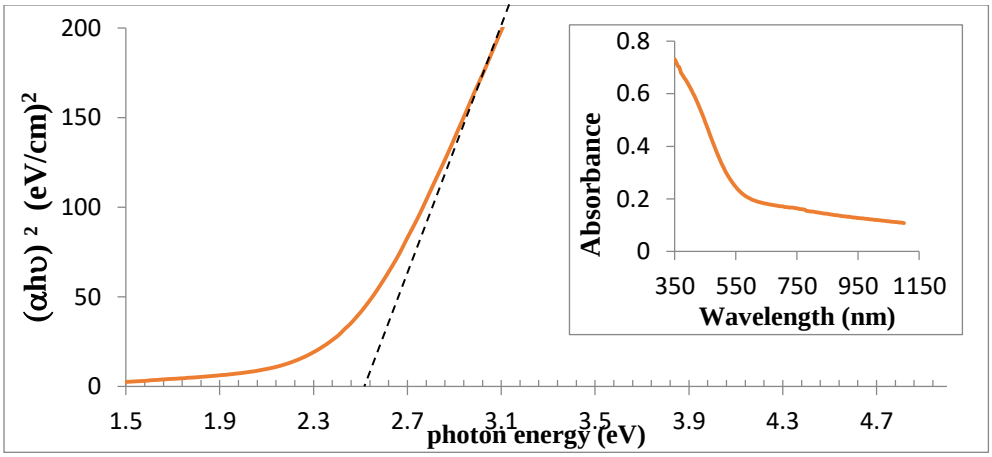

Fig.5 *Optical Absorbance of SnS thin films and* $(\alpha hv)^2$ *versus photon energy gap plot*

The FTIR spectra of SnS thin films prepared at of thin film, which deposited on glass substrate is presented in Fig.5.

Figure 5 shows SnS characteristic vibration bands at 2350 cm^{-1} and 930 cm^{-1} attributed to the hydroxyl groups and SnS groups. The decrease intransmittance is observed at the vibration bands centered at 2350 cm^{-1} and 830 cm^{-1} with increasing bath temperature. All the absorption peaks are found to appear at the wave number (930 cm^{-1}) in the spectra of SnS (Fig. 6). These peaks are ascribed to the stretching vibration of Sn–S bonds, indicating the formation of SnS film.

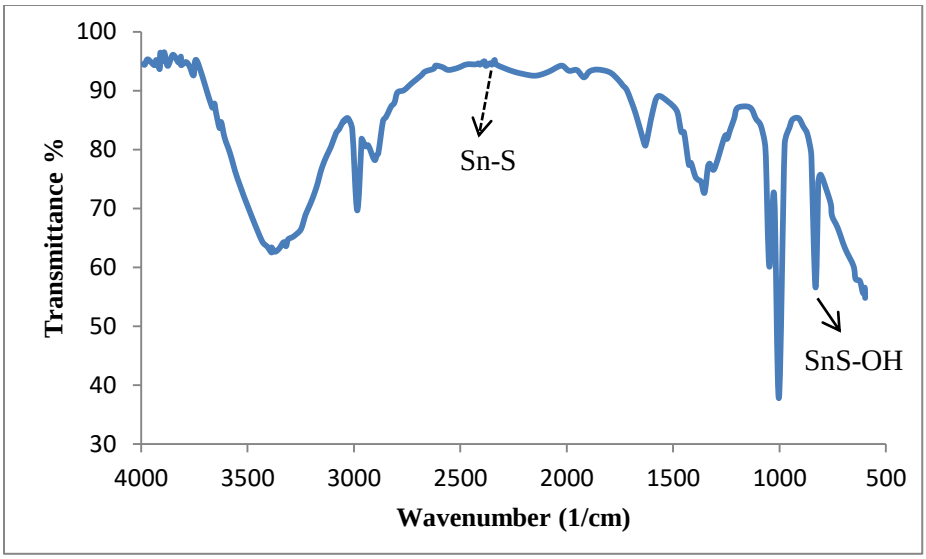

Fig.6: FTIR spectra of SnS thin film

The room temperature dark Current–Voltage characteristics of Al/SnS/AL NPs layer contact prepared with 400mJ laser energy without any post annealing is given in Fig. 7. the figure shows an ohmic contact over the whole applied voltage range. Hall effect measurements which were conducted for SnS at room temperature,these measurements confirmed the n-type conductivity of the synthesised SnS nanoparticles.

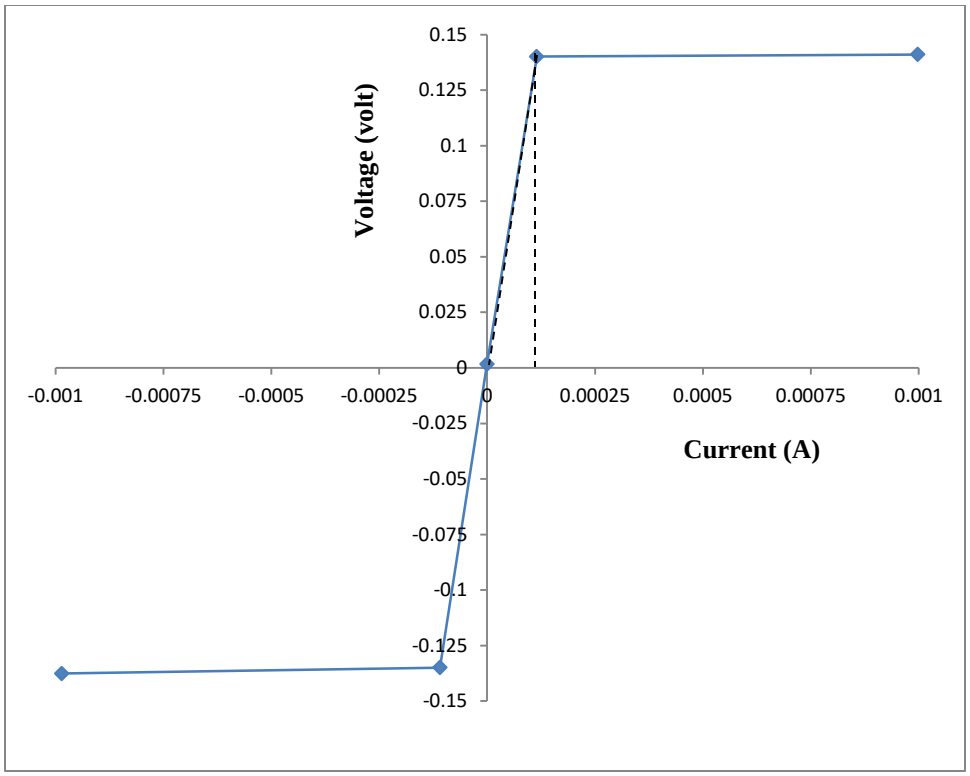

Fig. 7 Room temperature I–V characteristics of Al/SnS/Al nanoparticles contact

4. CONCLUSION

The reported work demonstrates the preparation of SnS NPs by laser ablation in water and study the structural,optical and morphological properties of SnS thin films . XRD results indicated that the thin films had a preferred (021) and (111) orientation

One step synthesis nanoparticles SnS was demonstrated and analysed by using nanosecond laser ablation of SnS target in methanol without using any surfactant. XRD and SEM investigations reveal that the nanoparticles are single-phase tetragonal structure of SnS with morphology and size controlled by laser fluence. at 400 Mj laser fluence, SEM observation confirmed the existence of highly agglomerated 20-40 nm SnS particles. Our forecoming paper will focused on the the increase in carrier concentration for optoelectronic applications.

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