STRUCTURAL AND OPTICAL PROPERTIES OF Cu_2Se/Yb/Cu_2Se THIN FILMS

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Abstract - In this study, two stacked layers of Cu_2Se are nanosandwiched with 50 nm thick Yb slab. The films which are prepared by the thermal evaporation technique under vacuum pressure of 10^{-5} mbar are examined by the X-ray diffraction, scanning electron microscopy, energy dispersion spectroscopy and optical spectrophotometry techniques. The cubic phase of Cu_2Se exhibits lattice contraction which results in strained structure and reduction in grain size upon insertion of Yb between two layers of Cu_2Se. The nanosandwiching of Yb layer does not influence the optical energy band gap but significantly altered the optical conductivity parameters. The optical conductivity parameters are determined by the help of Drude-Lorentz approach for the dielectric dispersion. Particularly, while the scattering time at femtosecond levels and the drift mobility values increases, the free hole density decreases via the nanosandwiching procedure. The drift mobilityand plasmon frequency of Cu_2Se reached values of 35.2 cm^2/Vs and 9.85 GHz, respectively, bythe Ybnanosandwiching. The enhancements in mobility and free carrier density values make the Cu_2Se thin films more appropriate for optoelectronic applications.

Keywords - Cu_2Se; X-ray; Optical Conductivity; Dielectric; Mobility

I. INTRODUCTION

Copper (II) selenide is regarded as a promising material suitable for thermoelectric [1] and optoelectronic [2] applications. By doping with iodine, this compound is reported to exhibit a thermoelectric figure of merit up to 1.1 at 773 K. The iodine doping reduced the transition temperature to 380 K. These features of Cu_2Se make it highly performing during thermoelectric applications [1]. In another work, the seebeck coefficients and electrical resistivity of the Cu_2Se are observed to increase by the Pb doping [3]. It is mentioned that the defects and reduced carrier concentration cause low thermal conductivity and shifts the figure of merit to a value of 1.52 at 973 K. In addition, studies on the Cu_2Se nano-discs grown by the imidazoline-2-selenone technique has shown that these discs are promising for use in optoelectronics and could represent a good alternative material for improving the performance of pn junctions [2]. Works which take into account the effects of doping on the properties of Cu_2Se [4, 5, 6] have shown that an increase in the optical transmittance and widening in the energy band gap with increasing bismuth content could be achieved [4]. The Bi doped Cu_2Se displayed strong band gap emission at 629 nm. Such properties make the copper selenide preferable optical media. In addition, the chemical bath deposition of Sn^{+4}, Cu^{+}- and Zr^{4+}-doped CuS/reduced graphene oxide (rGO) hybrid films have shown that the S/Cu molar ratio and appropriate doping can control the conductivity type increases the optical transmittance and lowers the electrical resistivity significantly [5, 6]. The similar properties of CuS with CuSe make the application of this doping procedure attractive for altering the properties of CuSe. On the other hand, another technique of doping materials which is known as nanosandwiching is reported to have important technological applications [7, 8, 9]. The significance of this technique appears in the uniform distribution of the doping agent in the body of the sample. In the case of metal slabs it can behave as a plasmonic surface which support quantum confinements and enhances the optical performance of materials. In other cases it can also behave as an entire metallic layer that forces charge accumulation due to the Schottky nature of interfacing. In this case, remarkable decrease in the free carrier density could be achieved. Itis important for applications which concern cancelation of semiconductor degeneracy or reducing the charge carrier tunneling property. In one of recent works [7], the nanosandwiching of a ytterbium layer of thickness of 70 nm between two layers of CdS was sufficient to enhance the crystalline nature without changing the unit cell parameters of CdS. It also succeeds in improving the light absorbability by ~8 times in the IR region. In addition, a jump in the value of drift mobility from 281 to 996 cm^2/Vs is achieved by the nanosandwiching of Yb. Similar type of nanosandwiching were carried out for CdSe thin films. The ytterbium nanosandwiching effect on the structural and optoelectronic properties of CdSe have also shown that a systematic redshifts in the interference patterns of the optical transmittance and reflectance associated with redshift in the energy band gap could be achieved The Ybnanosandwiching enhanced the responsivity to blue light by three orders of magnitude [8, 9].Such remarkable enhancements in the structural and optoelectronic properties motivated us to repeat the same

nanosandwiching for coppers selenide films. For this reason, here in this work, we will insert a 50 nm thick layer of Ybbetween two 500 nm thick Cu_2Se layers. The effect of the Ybnanosandwiching on the structural parameters, optical transmittance, reflectance and absorption coefficients as well as energy band gap and dielectric constants will be explored. In addition, the effects of the Yb participation on the optical conductivity parameters which are computed from the imaginary part of the dielectric constant will be reported.

II. EXPERIMENTAL DETAILS

Thin films of Cu_2Se were prepared by the thermal evaporation technique using VCM600 physical deposition evaporator equipped with an in situ thickness monitor (Inficon STM-2). The Cu_2Se film whose thickness is 500 nm were grown onto ultrasonically cleaned glass slides. The films were then coated with ytterbium layers of thickness of 50 nm. The resulting Cu_2Se/Yb films were re-coated with another 500 nm thick Cu_2Se to form the $Cu_2Se/Yb/Cu_2Se$ stacked structure. Another un-sandwiched films that are composed of two layers Cu_2Se each of thickness of 500 nm are also prepared to allow exploring the effects of Yb. The films werecharacterized by the X-ray diffraction (XRD) technique using Miniflex 600 XRD unit, scanning electron microscopy using COXEM 200 SEM unit, energy dispersive X-ray analyzer (EDS) using EDAX unit and by the ultraviolet-visible light spectrophotometry technique using Thermo scientific Evolution 300 spectrophotometer.

III. RESULTS AND DISCUSSION

The optical images of the two stacked layers of copper selenide thin films before and after the nanosandwiching of Yb thin metal of thickness of 50 nm are illustrated in inset-1 of Fig. 1. It is clear from the image that the first layers whose thickness is 500 nm is brown. Coating the first layer with 50 nm Yb did not alter the color but when the second copper selenide layer (500 nm) was coated onto the surface of Yb to form a nanosandwiched structure of copper selenide, the sample became dark brown colored. In this work, we will consider the effect of Yb on the properties of copper selenide. The X-ray diffraction patterns for two stacked layers of copper selenide before and after the nanosandwiching process are shown in Fig. 1. Four and three diffraction peaks before and after the insertion of Yb respectively, are observable in the figure. The respective maximum peaks which are also enlarged in inset-2 of the same figure appear at diffraction angles of $2\theta = 27.30^{o}$and$2\theta = 27.35^{o}$. The analysis of the observed diffraction patterns with the help of "TREOR 92" software packages have shown that the samples under investigation correspond to a cubic crystal structure being best oriented in the (111) direction. The calculated lattice parameters for the cubic unit cell before and after the nanosandwiching are found to be 5.6533 and 5.6431 Å, respectively. These values of lattice constants which are consistent with literature data and crystallography open database cards (COD: 9015293) indicate that the obtained films are Cu_2Se. It is classified in the cubic system with space group $O_h^5 - Fm3m$. The formation of this type of unit cells comprises four selenium ions occupying the(4)a sites and seven Cu ions are distributed over the $32(f)_I$ sites of the space group and the remaining Cu ion is distributed over the $32(f)_{II}$ sites [10]. The diffraction patterns that are shown in Fig. 1 displayed a diffraction peak at $2\theta = 24.70^{o}$which cannot be index in accordance with the cubic system. Various trails of solution were tested. The most probable solution that reveals this diffraction peak corresponds to the hexagonal CuSe [11] (COD: 9000063, JCPDS Data File No.-00-020-1020). The weight of this peak compared to the maximum peak of the cubic structure is 29.5%. It is also observable from the figure, that the broaden peak which is oriented in the (200) direction has disappeared when the material was nanosandwiched with Yb. In addition, as inset-2 of Fig. 1 shows, there is a shift toward larger 2θ values upon Yb nanosandwiching. This shift indicates a contraction in the lattice that usually results in strained structure. The crystallite size (D), micro strain(ε) and defect density (δ) that are calculated in accordance with the published equations [12]are found to exhibit values of 36 nm, $4.24\times 10^{-3}$and$3.08\times 10^{11}cm^{-2}$ for the Cu_2Se/Cu_2Se layers. The Ybnanosandwiching decreased the crystallite size to 32 nm and increased the strain and defect density to 4.77×10^{-3} and $3.92\times 10^{11}cm^{-2}$, respectively. These attenuations in the structural parameters that are associated with the decrease in the lattice constant values from 5.6533 to 5.6431 Å by insertion of Yb layer between two layers of Cu_2Se can be ascribed to the bonding mechanism between Cu_2Se and Yb. The bond lengths of Cu-Se, Yb-Se, Cu-Cu and Yb-Yb are 2.404 Å [13],2.809 Å [14], 2.638 Å [15] and 3.588 Å [16], respectively. Since the bond length between Yb-Se is much shorter than that of Yb-Yb, the ytterbium atoms at the interface boundaries prefer forming the bond with selenium. In another study, lattice contraction in boron doped SiGe epitaxial layers was also assigned to the favorability of the Si-B bond than Si-Ge bond [17]. It is mentioned that, boron atoms bonds with lattice more strongly than that of silicon resulting in an increase in the lattice contraction coefficient in SiGe to minimize the total energy of the system [17]. In order to get information about the atomic contents and the grain distribution in the Cu_2Se thin films, the scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS) measurements were carried out. The results of the measurements before and after the nanosandwichingare shown in

Fig. 2 (a) and (b), respectively. In accordance with the presented spectra of Fig. 2 (a) and its inset, the material display atomic contents of glass, and copper selenide only. The Au peak appeared due to the coating of the samples with 5 nm thick gold layer to prevent electron accumulation. The atomic content for theun-sandwiched films is found to be 66.30 at. % Cu and 33.70 at. % Se. The formed material is $Cu_{1.97}Se$. When the samples are nanosandwiched with 50 nm thick Yb, the produced films are composed of 64.37 at.% Cu, 32.79 at. % Se and 3.80% Yb. The chemical compound becomes $Cu_{1.96}Yb_{0.12}Se$. Recalling that the experimental error is ~3% of the measured value, the formula suggests that no atomic substitutions has occurred. In other words, the nanosandwiching of the Yb between two layers of Cu_2Se is of physical nature. In addition, the differences in the measured values of the EDS data that vary in 3% from region to region on the same sample could be assigned to the higher partial pressure of Se relative to Cu. The same behavior was observed for CdS thin films in which the higher pressure of S relative to Cd causes deviation from standard stoichiometry by ~10% [18]. On the other hand, the SEM images for an enlargement of 120,000 times which are collected from the samples without Yb which appears in the inset of Fig. 2 (a), display well-ordered homogenous distribution of grains with average grain size of 73 nm. The image which collected for the $Cu_2Se/Yb/Cu_2Se$ and is shown in inset-1 of Fig. 2 (b), display dark and bright grains of average grain size of 60 and 100 nm, respectively. The EDS test on the bright colored grains which are rarely observed revealed atomic composition of$Cu_{1.65}Yb_{0.13}Se$. In addition to the high pressure of Se relative to copper, we also believe that the deviation from stoichiometry in some region could have happened due to the cation exchange reactions [19]. In a study which target the doping of Pb into Cu_2Se, it is observed that Pb^{+2} ions selectively react with different planes of cubic and hexagonal stoichiometric Cu_2Se nanoparticles in cation exchange reactions [20]. The on-stoichiometry in $Cu_{2\pm x}Se$ alters the electronic properties and crystalline nature as well as cation exchange properties of the material under study [19]. Fig. 3 (a), (b) and (c) display the transmission (T), reflection (R) and absorption (α) coefficients spectra, respectively, for Cu_2Se films before and after the nanosandwiching process. The transmission coefficient of the un-sandwiched films sharply increases with increasing incident light wavelength reaching a maxima of 29.7%at 698 nm. It then sharply decreases with increasing light wavelength. The same trend of variation are observed for the Yb sandwiched Cu_2Se films. The nanosandwiched films exhibit maxima of 33.9% at 708 nm. T% spectra exhibit higher values for the Yb-sandwiched films in the incident wavelength range of 630-1100 nm. The increase in the transmittance values upon metal dopinginto materials was also observed for, Cu^{+}- and Zr^{4+}-doped CuS/reduced graphene oxide films [5, 6]. The enhancement in the transitivity was assigned to the formation of highly efficient interface between graphene and CuS. It is also possible to think that, the Ybnanosandwiching which forms new Yb-Se bonds in vacant sites of Cu leads to a significant reduction in the defect density. The reduced defect density decreases the traps and as a result band to band transitions become more dominant [21]. In contrast to these observations, the reflection coefficient spectra which are illustrated in Fig. 4 (b) did not show any significant difference between the Yb sandwiched and un-sandwiched films. On the other hand, the absorption coefficient spectra for the un-sandwiched and nanosandwiched films which is calculated from the relations [22],

$$T \approx (1-R_g)(1-R_{g/Cu_2Se})(1-R_{g/Cu_2Se/Cu_2Se})\exp(-\alpha d) \quad (1)$$

and

$$T \approx (1-R_g)(1-R_{g/Cu_2Se})(1-R_{g/Cu_2Se/Yb})(1-R_{g/Cu_2Se/Yb/Cu_2Se})\exp(-\alpha d), \quad (2)$$

respectively, and shown in Fig. 3 (c) reveal lower values of absorption coefficient for the sandwiched films. In these two equations R_g is the reflection coefficient of glass and d is the films thickness. It is noticeable from the absorption coefficient spectra, that Yb participation did not alter the trend of variation, but alter the values only. It is also worth of considering that the absorption coefficient increases with decreasing incident photon energy in the region of 1.72-1.14 eV. The increase in α values that is associated with decreasing transmittance coefficient values with decreasing energy is ascribed to the free carrier absorption in Cu_2Se. The free carrier absorption near the IR region is believed to be due to degeneracy [23] of the samples that raised from the Cu content compared to Se and due to the lattice distortion effect [24]. The lattice distortion causes lattice absorption owing to the changes in the electric dipole moments that is changed by the dislocations, impurities and inclusions which displaces the positions of the atoms. In addition, the ability of the IR radiation to transfer energy to the lattice causes charge carrier movement via charge carrier-phonon interactions [24]. At particular energy value, the decrease in the absorption coefficient values and increase in the transmissivity upon insertion of Yb can be ascribed to the flowing of free electrons from the semiconductor to the metal (Yb) to reach the equilibrium in Fermi levels [25]. Furthermore, in accordance with the optical absorption spectra, the Tauc's equation ($(\alpha E)^2 \propto (E - E_g)$ [7, 26]) plots for the samples under study which are illustrated in Fig. 4 (a) reveal no significant effect on the direct allowed transitions energy band gap value. The direct energy band gap values for the sandwiched and not-sandwiched Cu_2Se films is 2.33 eV. The value is consistent with literature data [27]. The effect of the Ybnanosandwiching on the dielectric properties of Cu_2Se films is also evident from Fig. 4 (b) and (c). The real and imaginary parts of the dielectric constant

which are calculated by a previously described method [7] exhibits the same trend of variation before and after the nanosandwiching process. In accordance with the shown figures, the nanosandwichingdoes not significantly affect the values of the dielectric constants. Particularly, while, the real part of the dielectric constant decreases by 2-3% (near 2.0 eV), the imaginary part decreases by 20-28% when the double layer of copper selenide is nanosandwiched with ytterbium. In addition, in contrast to the $\varepsilon_r$spectra of the studied samples which decreases with increasing incident photon energy indicating less dielectric performance in the ultraviolet region of light, ε_{im} values sharply increases as the incident light energy approaches the infrared region indicating an enhancement in the optical conductivity performance ($\sigma(w) = \varepsilon_{im} w/(4\pi)$). The modeling of the imaginary part of the dielectric spectra reveal information about the optical conductivity parameters which are practically the most significant in optoelectronic technology. Following the Drude-Lorentz approaches for optical conduction [7, 26] which represents the imaginary part by the equation,

$$\varepsilon_{im} = \sum_{i=1}^{k} \frac{w_{pei}^2 w}{\left((w_{ei}^2 - w^2)^2 + w^2 \tau_i^{-2}\right)}. \quad (3)$$

where, τ_i is the average scattering time of free charge carrier (holes in our case as tested by the hot probe technique), $w_{pei}=\sqrt{\frac{4\pi p e^2}{m^*}}$ is the plasmon frequency,w_{ei} is the hole-plasmon reduced resonant frequency and p is the free hole concentration, k is the number of possible linear oscillators, m^* is the effective mass of holes. The fittings of the experimental data are actualized assuming holes effective masses of value of $m^* = 0.5m_0$ [10] for Cu_2Se and electrons effective mass value of $m^* = 1.0m_o$ for Yb metal [7]. The effective mass for the Cu_2Se/Yb/Cu_2Se ($m^* = (2/m^*_{Cu2Se} + 1/m_{CuSe})^{-1}$) is $0.2m_o$. As the figure shows a good correlation between the theoretical and experimental data can be observed. The fitting procedure in accordance with Eqn. (3) reveal the fitting parameters that are shown in Table-1. The obtained data of Table-1 was determined with a mathematical computational method that allow altering the values of the fitting parameters in such a way that, the series is run up to k=4 and the oscillators are linearly added. i.e the resuls for $k = 1$ is added to $k = 2$ and so on. The consistency of the computed data is obtained by the least square method. The computational high-convergence minimization program makes use of regression and residual sums of squares (R^2), coefficient of determination and residual mean squares statistical analysis. The errors in the data were evaluated to be 2–10%. The consistency between the computed and experimental data are illustrated in Fig. 4 (c). As the table illustrates, the free hole concentration being in the range of 4.8×10^{18}-$100.0\times 10^{18}\ cm^{-3}$is very high compared to the semiconductors standards (~$10^{17}cm^{-3}$ [11]) for optoelectronic applications. Even though this value is lower than that reported as $1.9\times 10^{20}cm^{-3}$[10]. The highest drift mobility being 8.8 cm^2/Vscorresponds to the first linear oscillator of hole-plasmon coupled frequency of $1.75\times 10^{15}Hz$ is still higher than that reported as 2.83×10^{-3}cm^2/Vs for Cu_2Se prepared by the chemical bath deposition technique [28]. The doping of these films with Na slightly increased the mobility of the films. One of the reported as the highest hole mobility and lowest hole concertation values are 10 cm^2/Vsand$10^{18}cm^{-3}$, respectively, are registered for $Cu_{1.90}Se$ thin films prepared in a vacuum co-evaporation of elemental Cu and Se onto heated glass substrates at temperatures from 160 to 250 ^oC [29]. The insertion of the Yb layers between two layers of copper selenide increased the mobility value to 35.2 and to 10.5 cm^2/Vsfor the first and second linear oscillators, respectively. It also lowers the free hole concertation to $1.15\times 10^{18}cm^{-3}$. In general, the range of variation of the carrier concertation shifts to$1.15\times 10^{18} - 5.5 \times 10^{19}cm^{-3}$. In addition, it is also observed from the table that the Yb nanosandwiching increased the free hole scattering time at femtosecond level. The increase in the scattering time indicates less electronic friction coefficient ($\gamma = \tau^{-1}$) which we believe that it happens due to band bending as a result of flowing of holes from valence of Cu_2Se to the Yb. The electron affinity of Cu_2Se is 3.0 eV [30]. As it is p-type material with band gap of 2.33 eV, the work function is larger than 4.17 eV. This value is much larger than the work function of Yb (2.51 eV [14]) which make the interface of Schottky type. Another interesting feature which is achieved by the nanosandwiching process is the extension of the plasmon frequency range. The plasmon frequency ranges before the nanosandwiching process is 1.84-8.40 GHz. It becomes 1.42-9.85 GHz after the insertion of Yb. This feature is preferable as it indicates the usability of Cu_2Se/Yb/Cu_2Se as suitable material for fabrication of plasmonic devices [31]. In the light of the estimated values of the energy band gap for p-Cu_2Se and the literature data for CdSe based solar cells [32], it is possible to suggest that the p-Cu_2Se could be used as optical window for n-CdSe solar cells. For the p-CdSe cells an n-CdS layer with band gap of 2.42 eV was used as optical window [32]. The studies which take into account the doping of Cu into CdSe has shown that the current density of the quantum dot synthesized solar cells increased dramatically from 12mA/cm^2 for pure CdSe QDs to 19.915mA/cm^2 for Cu^{2+} ions doped on pure CdSe. The copper ion raised the conduction band of pure CdSe and reduced recombinationand enhanced the efficiency of the cells [33].Such properties make the suggestion of replacing the CdS by Cu_2Se possible and promising. It is worth noting that as a future work, the studied Cu_2Se/Yb/Cu_2Se films could be subjected to an annealing process to motivate the

proper diffusion of Yb into the Cu_2Se layer. This type of compositional and structural modifications by annealing process could enhance the optical properties of the films under study further. It was observed that for $MgCl_2$ treated CdSe thin films the annealing at 470 ^oC enhanced the crystallinity by increasing the grain size, reducing the strain and decreasing the defect density. Such improved properties in the MgCl2 treated CdSe thin films increased the optical absorbance by 70% and redshifted the energy band gap from 2.34 to 1.74 eV [32].

IV. CONCLUSION

In this work, we have shown the ability of improving the optical properties of Cu_2Se by the nanosandwiching technique. Aytterbium thin layer of thickness of 50 nm which results in lattice contraction is sufficient to enhance the optical conductivity parameters and make the material suitable for plasmon and optoelectronic related applications. Another important feature which is detected through this study is that the presence of this metal between two layers of Cu_2Se does not alter the energy band gap value but reduces the free carrier density to make the material more acceptable in electronic device technology. The optical investigations have shown that it is possible to use the Cu2Se thin films as optical window in solar cells to improve the efficiency of the cells. In addition, an annealing process that target the diffusion of Yb into the structure of Cu_2Se is advised so that the performance of this optical cavity is improved further.

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List of Figure Captions

Fig.1 (a)The X-ray diffraction patterns for theCu_2Se/Cu_2Se double layer before and after Ybnanosandwiching. The optical image for the sample is shown in inset-1of the figure. Inset-2 show the deformation due to thenanosandwiching.

Fig. 2 The EDS spectra for Cu_2Se/Cu_2Se films (a) before and (b) after the Ybnanosandwiching. Insets of (a) and (b) show the SEM images for the copper selenide samples before and after the Ybnanosandwiching, respectively.

Fig. 3 (a) thetransmission, (b) the reflection and (c) the absorption coefficients spectra for Cu_2Se/Cu_2Seand $Cu_2Se/Yb/Cu_2Se$.

Fig. 4 (a) The $(\alpha E)^2 - E$ plottings, (b) the realpart of the dielectric spectra and (c) the imaginary part of the dielectric spectra for the Cu_2Se/Cu_2Seand $Cu_2Se/Yb/Cu_2Se$ films.. The blue and red colored points in (c) show the Drude-Lorentz fittings obtained by eqn. 3.

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