Eco-friendly method to synthesize and characterize 2D nanostructured (1,2-bis(diphenyl-phosphino)ethyl) tungsten tetracarbonyl methyl red/copper oxide di-layer thin films

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Abstract. Three-layer thicknesses ($T_1 = 50$, $T_2 = 75$ and $T_3 = 100$ nm) of 1,2-bis(diphenylphosphino)ethyl tungsten tetracarbonyl methyl red (DPE-W-MR) were deposited onto the CuO thin film (50 nm) to produce DPE-W-MR/CuO di-layer thin films by sol-gel spin-coating technique. The composition and the chemical structure of the as-prepared thin films were characterized using various techniques including elemental analysis, Fourier transform infrared spectroscopy, ¹H-NMR and X-ray diffraction (XRD). Scanning electron microscopy was used to investigate the size and shape of the CuO nanoparticles and the fabricated thin films. The films are crystalline as evidenced by the XRD pattern and DPE-W-MR has an orthorhombic crystal system. The crystallite size was calculated from an analysis of the line broadening features using the Scherrer formula; the average crystallite sizes of DPE-W-MR/CuO di-layer thin films are 52.92, 56.24 and 72.26 nm for T_1 , T_2 and T_3 , respectively. Thermogravimetric analysis and the thermal curve of DPE-W-MR complex were studied. Optical properties of DPE-W-MR/CuO di-layer thin films are discussed. The optical band gap energies of DPE-W-MR di-layer thin films/CuO decreased (2.25, 2.1 and 1.88 eV) as the film thickness increased (from T_1 to T_3). Based on the optical results and the quantum confinement effects, the DPE-W-MR/CuO di-layer thin films may be candidates as semiconductor materials for optoelectronic devices.

Keywords. Phosphine; copper oxide; thin film; XRD; SEM; optical properties.

1. Introduction

Two-dimensional (2D) organic-inorganic hybrid structures have attracted attention due to their interesting structural and optical properties [1–5]. Copper is one of the most technologically important metals to form as 1D nanowires at the nanoscale [6] and/or 2D thin films [7]. Copper element remains the material of choice for consumer electronics due to its favourable conductivity/cost ratio. Metallic copper thin films have various applications, especially in electrical contacts in printed circuit boards [8,9]. Based on the high conductivity of copper films, they were utilized as highly uniform and reproducible substrates for plasmon-enhanced fluorescence [10]. In addition, the copper thin films are used in photocatalysis and solar photovoltaics technologies. Nanocrystalline semiconductor particles have drawn considerable interest because of their useful properties, such as electronic properties and unique optical properties, as compared to those of the bulk materials. The oxides of transition metals are an important class of semiconductors, which

have applications in magnetic storage media, solar energy transformation, electronics and catalysis. Therefore, preparation of metallic/semiconductor thin films with fine-tuned properties for specific applications is considered a challenge for chemists. There are different preparations, which were considered, including wet and dry methods were used for the deposition of metal/semiconductor thin films. These methods, such as atomic layer deposition (ALD), photochemical deposition [11], physical vapour deposition (PVD) [12], in addition to molecular precursor [13], ion beam-assisted deposition (IBAD) [14] and hydrothermal methods [15] were applied. When the same thin film is deposited by different techniques, these techniques give the film with different phase structures, degree of crystallinity and variable optoelectronic properties.

At the same time, much great progress was devoted to the applications of phosphine compounds in various catalysed reactions because these compounds are easy to prepare and modify. Particularly, triphenyl phosphine is the most commonly used as it was prepared with high yields and high

selectivity in a wide range of catalysed reactions. Recently, nano-sized diphosphine complexes containing aromatic substituents were attracted much attention [16]. Diphosphine complexes have low oxidation states with transition element and could be used to fine-tune electronic and their steric properties in corresponding coordinated compounds. The connected substituents with phosphorus atom can alter electron donating or electron withdrawing property of the ligand. Bulky phosphines can be used as effective compounds with specific transition metal based on the reaction type between the phosphine compound and transition metal [17]. Phosphine ligands with bulky aromatic substituents showed a good catalytic property in the alcohol oxidation and hydrosilylation reactions [18,19] with a proper transition metal. Complexes with metal hexacarbonyl (group VI) with diphenyl phosphine ligands were used to synthesize phosphide-metals that were used to synthesize nanocrystalline spintronic materials [20]. Complexes of tetra-carbonyl metals were reported as therapeutic CO-releasing molecules [21,22].

Herein, the preparation and characterization of DPE-W-MR/CuO nanocomposite di-thin films were fabricated based on the facial synthetic route to produce 2D thin films by sol-gel spin-coating technique. The composition and the chemical structure of the fabricated di-thin films were characterized using elemental analysis (EA), Fourier transform infrared spectroscopy (FTIR), ^1H -NMR, thermogravimetric analysis (TGA) techniques, X-ray powder diffraction (XRD) and optical parameters. The surface morphology of the di-thin film was investigated using scanning electron microscope (SEM).

2. Experimental

2.1 Materials

1,2-bis(diphenylphosphino)ethyl (DPE), methyl red (MR), ethanol, 2-methoxyethanol, monoethanol amine and copper(II) acetate monohydrate ($\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$) were purchased from Sigma-Aldrich and used as received without further purification.

2.2 Methods

2.2a Preparation of (DPE-W-MR)·3H₂O complex: 1,2-bis(diphenylphosphino)ethyl tungsten tetracarbonyl methyl red (DPE-W-MR) complex was prepared by dissolving MR in ethanol (0.269 mmol in 5 ml; w/v). Subsequently, MR solution was added dropwise to DPE-W solution (0.692 mmol in 6 ml; w/v dichloromethane) under magnetic stirrer (500 rpm) at room temperature [23–26]. The resulting mixture was completely stirred for ~15 min at 110°C, then, the volume of the solution was concentrated to $\cong$ 2 ml under reduced pressure. A red precipitate DPE-W-MR complex was washed several times by appropriate volume of n-hexane and dried under vacuum. Yield: 86% of a red powder. Chemical formula

(CF): $\text{C}_{43}\text{H}_{42}\text{N}_3\text{O}_7\text{P}_2\text{W}$, molecular weight: 958.61, EA: C, 53.88 (53.83); H, 4.42 (4.28); N, 4.38 (4.30).

2.3 Fabrication of DPE-W-MR/CuO di-layer thin films

DPE-W-MR/copper oxide (DPE-W-MR/CuO) di-layer thin films were fabricated by sol-gel spin-coating technique using TC100 spin coater on quartz substrates. $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ used as a precursor to produce CuO, while 2-methoxyethanol used as a solvent. Monoethanol amine was used as stabilizer during the fabrication process. The ratio between the stabilizer (mono-ethanol amine) and precursor/s either $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ or DPE-W-MR was 1:1. The quartz substrate was cleaned in methanol and acetone baths for 10 min by an ultrasonic cleaner, then the substrate was rinsed with deionized water and dried. Subsequently, $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$ precursor was dropped onto the quartz substrate at 1500 rpm per 150 s. After the spin coating, the film was dried at 150°C for 10 min on a hot plate to evaporate the solvent and remove the organic residuals. The resulting CuO film was calcined at 500°C for 2 h in air furnace and left to cool to room temperature kept in an evacuated desiccator for testing to avoid the moisture effect. The thickness of the resulting film was found to be 50 nm, which was measured by a digital micrometer with an accuracy of ± 0.001 mm. To fabricate DPE-W-MR/CuO di-layer thin films, DPE-W-MR solution was deposited onto the resulting CuO (50 nm) film with 50, 75 and 100 nm. The deposition was applied according to the previous step under the same previous conditions. These three different thicknesses were labelled as T₁, T₂ and T₃, respectively.

2.4 Characterization

EA of the fabricated thin films were performed with 2400 Perkin-Elmer, USA elemental analyzer. Perkin-Elmer FTIR type 1650 spectrophotometer was used to FTIR analysis of the wavenumber range of 4000–400 cm^{-1} . ^1H -NMR spectra were recorded by a Bruker (400 MHz) spectrometer. TMS was used as an internal standard reference. Dimethyl sulfoxide-d₆ (CD_3)₂SO₄ was used as a deuterated solvent and the spectra extended from 0 to 14 ppm. TGA of DPE-W-MR complex was studied in the range of 45–1000°C under nitrogen atmosphere (20 ml min⁻¹) with a heating rate of 20°C min⁻¹ by DTG-60H Shimadzu simultaneous TG/DTG apparatus. XRD of DPE-W-MR/CuO di-layer thin films was studied by Philips X'Pert Pro diffractometer equipped with $\text{CuK}\alpha$ radiations. Optical parameters of the DPE-W-MR/CuO di-layer thin films were calculated at room temperature by a Shimadzu UV-3101UV-vis-NIR pc spectrophotometer in the wavelength range of 250–2500 nm. The morphology of the DPE-W-MR/CuO di-layer thin films was investigated using SEM by JEOL-JSM-5400 LV electron microscope.

Figure 1. ^1H -NMR spectrum of the synthesized DPE-W-MR complex.

3. Results and discussion

3.1 FTIR of (DPE-W-MR)·3H₂O complex

IR (KBr, cm^{-1}): the band around 3625 cm^{-1} can be attributed to the molecules as a moisture in the complex during the synthesis process [27,28]. The medium intensity band observed at 3143 cm^{-1} is attributable to $\nu(\text{C-H})_{\text{aromatic}}$ vibration. The weak intensity band appeared at 2985 cm^{-1} is referred to $\nu(\text{C-H})_{\text{aliphatic}}$ vibrational mode. The strong intensity band at 1750 cm^{-1} is due to the overlapping of $\nu(\text{C=O})$ of both carboxylic acid and ester. The band medium intensity at 1555 cm^{-1} is referred to $\nu(\text{C=C})$ mode. Finally, the bands located at (strong 2045 ; medium 1990 ; medium 1985) and (weak 1970 ; weak 725 ; strong 680 cm^{-1}) may be mainly due to $\nu_{\text{sym}}(\text{C}\equiv\text{O})$ and $\nu(\text{P-C})$, respectively.

3.2 ^1H -NMR of the DPE-W-MR complex

Figure 1 shows ^1H -NMR spectrum of the DPE-W-MR complex. The spectrum shows multiple types of resonances at $\delta \cong 3.11$ and 7.29 ppm for six protons in dimethyl groups and 16 protons in four phenyl groups of the complex, respectively. The two strongest peaks located at $\delta \cong 2.45$ and 3.4 ppm can be attributed to DMSO protons and water, respectively, in DMSO. A doublet of doublet singles of relative intensity 2H at a range $\delta \cong 5.45\text{ ppm}$ were observed and assigned to $(-\text{CH}=\text{CH}-)$ protons coupling together and in the presence of two phosphorous atoms, $^2J(\text{CH}=\text{CH})\ 46\text{ Hz}$. The two weak peaks located at $\delta \cong 6.65$ and 7.03 ppm can be attributed

to the two protons in benzene ring containing $(\text{CH}_3)_2\text{-N-}$ group. The weak peaks observed at $\delta \cong 7.52$, 8.15 , 8.55 and 9.29 ppm have a relative low intensity CH proton in benzene ring containing a carboxylic group.

3.3 TGA of DPE-W-MR complex

Thermal decomposition profile of DPE-W-MR complex was studied by the TGA in the range of $45\text{--}1000^\circ\text{C}$. TG/DTG data of the complex $[(\text{DPE-W-MR})(\text{H}_2\text{O})_2]\text{H}_2\text{O}$ is tabulated in table 1. The decomposition of $[(\text{DPE-W-MR})(\text{H}_2\text{O})_2]\text{H}_2\text{O}$ complex was carried out through four stages (figure 2). The first one, within the temperature range of $45\text{--}104.4^\circ\text{C}$ (DTG peak at 100°C), is related to weight loss (obs.: 1.85% ; calc.: 1.87%). This weight loss can be attributed to the removal of lattice water molecules. The second one, within the temperature range of $140\text{--}193^\circ\text{C}$, is related to the removal of two coordinated water molecules and one mole of carbon monoxide [29,32]. This stage of weight loss was found to be 6.67% (theoretical value of 6.68%). The third stage of weight loss was found to be 27.78% within the temperature range of $255\text{--}383^\circ\text{C}$ (with a DTG peak at 383.89°C). This can be attributed to the removal of MR part. The fourth one, within the temperature range of $424\text{--}470^\circ\text{C}$, is related to the removal of DPE ligand. The weight loss within the fourth stage was found to be 22.52% . The total weight loss through the four steps by TGA was found to be 77.45% . Thus, these results are in a good agreement with the theoretical value of 77.48% . The remaining weight found to be 22.52% is related to tungsten dioxide.

Figure 4. X-ray diffraction pattern of the sol-gel synthesized CuO nanoparticles.

On contrary, the intensity of characteristic peaks of DPE-W-MR gradually increases. By comparing appropriate regions of figures 4 and 5, it is found that the illustrated peaks in the range of $2\theta = 5\text{--}35^\circ$ are associated to DPE-W-MR, which are not detected in figure 3.

The average particle size of DPE-W-MR/CuO di-layer thin films were calculated by the use of Debye–Scherrer's equation. The average crystallite sizes of DPE-W-MR/CuO di-thin films are 52.92, 56.24 and 72.26 nm for T_1 , T_2 and T_3 , respectively.

3.5 SEM of the CuO thin film and DPE-W-MR/CuO di-layer thin film

Figure 6a and b showed SEM image of CuO thin film with two different magnifications. The average particle size of copper

Figure 5. XRD diffraction pattern of the as-deposited of DPE-W-MR/CuO di-layer thin films of T_1 , T_2 and T_3 . The intensity of the characteristic peaks of CuO gradually decreases from T_1 to T_2 , whereas the intensity of characteristic peaks of DPE-W-MR gradually increases.

Table 2. The XRD data of copper oxide nanoparticles and DPE-W-MR/CuO di-layer thin films; miller indices (hkl), intensity, d -spacing, β -values and crystallite size, D (nm) as a function of 2θ position.

2θ	hkl	Intensity CuO	d -spacing ($\text{\AA}$) CuO	β (nm)				D (nm)			
				CuO	50/50 ^a	50/75 ^b	50/100 ^c	CuO	(50/50) ^a	(50/75) ^b	(50/100) ^c
32.40	(110)	2344	2.760	0.13	0.12	0.12	0.11	63.69	69.00	69.00	75.27
35.36	(002)	14082	2.535	0.28	0.26	0.24	0.23	29.80	32.10	34.77	36.28
38.04	(111)	21968	2.363	0.33	0.31	0.28	0.26	25.48	27.13	30.04	32.35
48.12	(202)	10430	1.889	0.36	0.33	0.31	0.25	24.19	26.39	28.09	34.83
53.28	(020)	5215	1.717	0.15	0.14	0.12	0.09	59.30	63.54	74.13	98.84
57.72	(202)	4254	1.595	0.16	0.15	0.13	0.11	56.74	60.52	69.83	82.53
61.8	(113)	10430	1.499	0.22	0.2	0.19	0.15	42.12	46.33	48.77	61.77
65.76	(022)	7598	1.418	0.25	0.23	0.21	0.17	37.87	41.16	45.08	55.69
67.32	(113)	8822	1.389	0.28	0.24	0.22	0.18	34.12	39.80	43.42	53.07
72.24	(311)	1754	1.306	0.10	0.09	0.10	0.06	98.43	109.36	98.43	164.05
74.92	(004)	2981	1.266	0.19	0.15	0.13	0.10	52.72	66.78	77.05	100.16

^aDPE-W-MR (50 nm)/(50 nm) CuO.

^bDPE-W-MR (75 nm)/(50 nm) CuO.

^cDPE-W-MR (100 nm)/(50 nm) CuO di-layer.

Figure 6. SEM of CuO nanostructure thin film (50 nm). SEM of CuO nanoparticles at scale bar of (a) 500 and (b) 400 nm.

Figure 7. Selected SEM images of (a) DPE-W-MR di-layer thin film/CuO (T_3) (scale bar = 5 μ m) and (b) cross-section of (T_3) (scale bar = 1 μ m).

oxide thin film (D_{av}) was found to be $\cong 53.12$ nm with ball-like shapes. The average particle size of copper oxide is in a good agreement with previous XRD data (table 2). Similar results were reported for ball-like CuO nanostructures synthesized by a hydrothermal method via controlling pH values of the solution [33].

Figure 7a shows the SEM micrographs of the surface as-deposited DPE-W-MR di-layer thin film/CuO (T_3). The SEM image shows that the thin film is flower-like and luminous with a homogeneous particle-size distribution. Cross-sectional di-layer thin film of T_3 was imaged by SEM (figure 7b) at 1 μ m scale bar. It can be observed that CuO layer is located below the red line, while DPE-W-MR layer is located on the upper surface. There are two different types of particle sizes, which can be ascribed to MR group and other diphenyl phosphine (DPE) group around tungsten atoms. In figure 7a and b, the smoothness of the surface of the upper layer (DPE-W-MR) and its homogenous structure are clearly visible. Finally, the average nanoparticle size of DPE-W-MR di-layer thin film/CuO (T_3) determined by SEM images was

found to be 96.45 nm in comparison with 72.26 nm (XRD data).

3.6 Optical absorption of DPE-W-MR/CuO di-layer nanostructure thin films

The absorbance and transmittance of the DPE-W-MR/CuO di-layer nanostructure films were recorded in the wavelength range of 250–2500 nm at room temperature (K). The absorption spectra of the as-deposited DPE-W-MR/CuO di-layer films are depicted in figure 8. UV-visible spectra consist of two distinct regions: in the near ultraviolet (Soret bands) and visible regions (Q bands). The multi-peaks of DPE-W-MR/CuO di-layer films in the absorption spectra can be attributed to the vibrational excitation form of a ground state to an excited one. The absorption bands in DPE-W-MR/CuO di-layer films arise from transitions between two HOMOs and two LUMOs and it is the identities of the metal centre and the substituents on the ring that might affect the relative energies of these transitions. It is observed that the wavelength of

Figure 8. Optical absorption spectra of DPE-W-MR/CuO di-layer nanostructure thin films (T_1 , T_2 and T_3). Inset is UV wavelength region at a wavelength of 250–500 nm for DPE-W-MR/CuO di-layer nanostructure films.

Figure 9. Optical transmission spectra of as-deposited DPE-W-MR/CuO di-layer thin film; T_1 : black, T_2 : red and T_3 : blue. Inset is the dependence of the complex refractive index (n) on the wavelength (λ , nm).

the as-deposited DPE-W-MR/CuO di-layer films varies from 250 to 500 nm from T_1 to T_3 (figure 8 inset). The absorption peaks (two weak peaks) appear in the UV wavelength region at wavelength of 250–500 nm for DPE-W-MR/CuO di-layer nanostructure films.

From figure 9, it can be noticed that the absorption peaks shift towards longer wavelength as the DPE-W-MR layer thickness increases from T_1 to T_3 . The particle size and the quantum confinement effects of DPE-W-MR layers have the major effect to the red shift in the absorption edge of the semiconductors. Therefore, one-dimensional confinement in the layers was also responsible for enhanced quantum confinement effects with the increase in DPE-W-MR layer thickness (except for the formation of nanostructure film).

Figure 10. Plots of absorption coefficient, α , as a function of photon energy, $h\nu$, of as-deposited di-layer thin film (T_1 , T_2 and T_3). Inset: the band gap of DPE-W-MR/CuO di-layer thin films were 2.25, 2.1 and 1.88 eV for T_1 , T_2 and T_3 , respectively.

Experimentally, the absorption coefficient (α) (m^{-1}) was calculated by the following relationship [34]

$$\alpha = \frac{1}{d} \ln \left(\frac{1}{T} \right), \quad (1)$$

where d is the thickness of the film (in cm) and T is the transmitted intensity of the film layer. Figure 10 shows the plot of absorption coefficient (α) (lies in between 0 and 5 ($\times 10^7 \text{ m}^{-1}$)) with photon energy ($h\nu$). Hence, the band gap of DPE-W-MR/CuO di-layer thin films was 2.25, 2.1 and 1.88 eV for T_1 , T_2 and T_3 , respectively (figure 10 inset).

The optical properties of T_1 film were evaluated from transmittance data using Myburg [35] method. Swanepoel's [36] method is employed for spectroscopic determination of optical properties of DPE-W-MR/CuO di-layer thin films using transmittance data do not have 'Swanepoel' author name. The application of the above method is used to calculate the maximum and minimum transmittance envelope curves by parabolic interpolation to the experimentally determined positions of peaks and valleys. Figure 9 shows that the values of refractive index (n) decrease with an increase in wavelength, λ (nm). The real values of refractive index (n_s) depended on maximum and minimum transmittance points are calculated using the expression proposed by Swanepoel's [35] as given below:

$$n = \left[N + \sqrt{N^2 - n_s^2} \right]^{1/2}. \quad (2)$$

In the weak and medium absorption regions, the value of N is given by the following equation:

$$N = 2n_s \frac{T_{\max} - T_{\min}}{T_{\max} T_{\min}} + \frac{n_s^2 + 1}{2}. \quad (3)$$

Table 3. Values of wavelength, λ (nm), $T_{\max}$ and $T_{\min}$ for the transmittance spectrum of T_1 of as-deposited multi-layer DPE-W-MR/CuO di-layer films and refractive index (n) to the equivalent wavelength, λ (nm).

λ (nm)	$T_{\max}$	$T_{\min}$	N
720	0.81133	0.780401	2.076383
854	0.83803	0.80374	1.976471
960	0.84133	0.81506	1.932849
1102	0.85209	0.82447	1.893732
1280	0.86833	0.83815	1.836922
1508	0.87822	0.84913	1.792917
1698	0.89437	0.85689	1.756996
1963	0.901034	0.86674	1.718866
2234	0.91229	0.87821	1.672258

The n_s value being the refractive index of the quartz substrate. Generally, the n_s value is calculated by the maximum of the transmission in the transparent region $T_{\max}$ using the equation [37]:

$$n_s = \frac{1}{T_{\max}} + \sqrt{\left(\frac{1}{T_{\max}^2} - 1\right)}. \quad (4)$$

The refractive index (n) values at the extremes of the spectra of wavelength (λ , nm), $T_{\max}$ and $T_{\min}$ are obtained from the plot of figure 9. The applied optical method, the dependence of the complex refractive index (n) on the wavelength (λ) is shown in figure 9 (inset). A sharp fall in the refractive index (n) for T_1 is observed at the lower wavelength region and a gradual destruction is observed for the refractive index corresponding to the higher wavelength region. The values of refractive index (n) calculated from equation (2) are resented in table 3. This variation in refractive index indicates a normal dispersive behaviour of T_1 thick as-deposited multi-layer DPE-W-MR/CuO di-layer nanostructure films [38–41].

4. Conclusions

Nanostructured DPEs-W-MR/CuO di-layer thin films were prepared by sol–gel spin-coating technique. Three sets of DPE-W-MR/CuO di-layer films were deposited on quartz substrates by spin-coating process with different DPE-W-MR layer thicknesses (50, 75 and 100 nm) and fixed CuO layer thickness (50 nm). The composition and the chemical structure of the fabricated thin films were characterized using a combination of spectroscopic and diffraction techniques. The particle sizes of CuO nanoparticles and thin films were calculated by applying Debye–Scherrer's formula. The optical band gap values for thin films varied from 1.88 to 2.25 eV depending upon the layer's thickness of thin films.

The variations of the refractive index (n) with the wavelengths was determined. SEM observations of DPE-W-MR/CuO di-layer thin films revealed that agglomeration of the W-phosphine/CuO clusters to form spherical CuO nanoparticles. The thermal characteristics (TGA) of DPE-W-MR complexes were discussed. The XRD data and optical characteristics of DPE-W-MR thin films show strong dependence on the film thickness. The structural and optical characteristics of DPE-W-MR thin films are of high relevance, especially for potential applications in semiconductor optoelectronic device development

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