Materials Research Express

PAPER

Surface morphology and electrical transport properties of Aberchrome 670 (\(\text{E-3(admantan-2-ylidene)} -4-[1-(2,5-dimethyl-3-furyl) ethyldene] dihydro-2,5-furandione\)) nano thickness films

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Keywords: AFM, organic compounds, Thin films, photochromic, seebeck coefficient, dielectric characterizations

Abstract

In this paper, surface morphology and electrical properties of Aberchrome 670, [(E-3(admantan-2-ylidene)-4-[1-(2,5-dimethyl-3-furyl) ethyldene] dihydro -2,5-furandione)], thin films with nano thickness, have been studied, for the first time, to provide one with knowledge concerning microscopic features, for electrical applications of organic photochromic compounds. The atomic force microscope (AFM) has been used to convene a general spectacle about the surface. The cyclic-voltammetric measurement has been inserted to determine the electrochemical gap. The thermal effect on the electrical resistivity, the thermoelectric power and Seebeck coefficient have been measured from 298 to 373 K. The influence of the frequency on some dielectric characteristics; such as the capacitance, the temperature capacitance coefficient (T.C.C), the real part of the complex dielectric permittivity \(\varepsilon\), the dissipation factor \((\tan(\delta))\) has been studied in the frequency zone \((50 - 5\text{ MHz})\) and temperature range \((\sim 298–373\text{ K})\) at different steady temperatures.

1. Introduction

The organic compounds are interesting and attractive in the field of commercial and industrial applications. Therefore, recognizing the behaviour and estimating their properties have become essential [1–6]. The organic photochromic materials are exciting compound because of their prospect applications, especially, optical memories and switches. Fulgides are inspiring examples of these materials [7–13]. Aberchrome (670) compound is belonging to a class of fulgides, which is \(\text{E-3(admantan-2-ylidene)} -4-[1-(2,5-dimethyl-3-furyl) ethyldene] dihydro-2,5-furandione\). It has a chemical formula of \(\text{C}_{23}\text{H}_{24}\text{O}_{4}\). The conveying in thin films provided one with knowledge concerning microscopic characteristics of semiconductor material and, its determination is crucial for utilizing it in abundant electronic and optoelectronic enforcements [2, 14]. In the immediate past, a variety of practical and industrial enforcements of thin films is considered the importance of technological developments of them [14]. In the previous work [10], we studied the structural, and the photochromic properties of thermal evaporated thin films from the tested compound, for the premier time to enrich the field of organic photochromic compounds. They have deduced the polycrystalline nature for the powder with monoclinic structure, where \(a = 18.646\text{ Å}, b = 2.55\text{ Å}, c = 4.425\text{ Å}, \beta = 94.53^\circ\) and \(V = 468\text{ Å}^3\). FTIR indicates the consistency of both the powder and the as-deposited thermal evaporated thin films. The UV - irradiation causes in the activation of the photo-coloration through the thermal films which reached to saturated colour after 6 min of exposure time, while the photo-bleaching time was \(\approx 13.5\text{ min}\). The photochromic effect has been explained according to the conversion of the open cycle E-isomer to closed stable C-isomer (see figure 1(a)). Using Smakula’s equation, the concentration of the coloring centres is \(1.18 \times 10^{10} \text{ cm}^{-3}\). The analysis of the absorption coefficient \(\alpha\) exposed a direct optical gap with 3.1 eV, for the precipitated films, which have been reduced to 2.48 eV by photo-coloration. Also, the author [7] studied the photovoltaic characteristics of E-admantan fulgide thin film/P-Si single crystal solar cell to feed the field of renewing source of energy. In the sitting work, the author has intended to continue the investigation of more physical properties.
of that Fulgide, for industrial applications. It has been planned to study the surface morphology, Cyclo-
voltammetric characterization, electrical resistivity, thermoelectric Power, thermal capacitance coefficient (T.C. C) and dielectric characterizations of Aberchrome (670) nano thickness films. These structural and electrical properties have investigations of which to the best of our knowledge, have not been published so, the aim is to feed the field of photochromic organic compounds for more industrial and commercial applications based on clean, low cost and safe organic photochromic compounds.

The present research has been formulated as follows; part 1, the introduction; part 2, the experimental details, part 3; the results and discusses. The last section reports the conclusions, and then the research ends with the references.

2. Experimental details

The source of Aberchrome 670 was obtained from Tokyo chemical Ind. Co., LTD [(M.F/M.W = C22H24O4/352.4 g mole⁻¹, CAS No. 94 856-25-4), purity (HPLC); 98%, melting point: 179 to 183 °C min⁻¹]. Thin films with various nano thickness, constructions and dimensions have been evaporated by conventional thermal evaporation technique using high vacuum coating unit (Edwards type E 306 A, UK) [7, 10]. Before the evaporation process, the substrates were initially immersed in chromic acid for a bout 24 h. After that they were washed with distilled water and rinsed by isopropyl alcohol and then again with distilled water. The pre-cleaned substrates were fixed on a rotating holder at ~25 cm far from a molybdenum boat heated in a vacuum of 10⁻⁴ Pa. The substrates, temperature was kept at room temperature. The deposition rate was ~1.3 mA s⁻¹. The thickness of the films were determined depending on both the quartz crystal thickness

monitor (Model FTM4, Edwards Uk) and the SEM microscope. The surface morphology of the films has been studied by contact mode atomic force microscope (AFM; Model: Wet- SPM9600; Shimadzu; Japan). AFM is a technique works by scanning a very sharp probe along the surface of the sample. The probe usually, is formed by silicon or silicon nitride cantilever with a sharp integrated and the vertical bending of the cantilever due to forces acting on the tip is detected by a laser focused on the back of the cantilever. The laser is reflected onto a distance photodetector. The movement of the laser spot on the photodetector gives a measurement of the probe. The optical lever and the highly precise movements by the scanner, combined with the careful control of the probe – sample forces allow the high resolution of AFM technique [15]. The morphological data of the surface has been estimated over the whole film with cited numbers resulting from image 5 x 5 μm² and for thickness ~ 539 nm. Also, cyclo-voltammetric measurement has been operated using a potentiometer (CAMARY interface 1000/04001) with three- electrode mode [the counter electrode (CE), the reference electrode (RE) and the working electrode (WE), (figure 1(b))] [16]. The maximum current = ± 100 mA, the maximum current resolution =0.3 pA, the maximum applied potential = ±5 V, the minimum time base = 1 m sec, and the frequency range (0.1—10 KHz) . The co-planer dark electrical resistivity (ρ) of Aberchrome (670) thin films (with different nano-thickness(423–850 nm)) has been measured in the thermal zone (~293–373 K). The initial qualitative check of the type of the majority carriers through the prepared films has been achieved using the hot probe method. The differential Seebeck coefficient (S₁₂) of the films has been measured by depositing masked rectangular films of it onto clean glass substrates. It has been calculated using the following equation [17–21]:

\[
\frac{d\Delta E}{dT} = S_2(T_{12}) - S_1(T_{12})
\]

For studying some dielectric characterizations, the capacitance (C) and the loss tangent (tan δ) have been measured by a computer controlled Hoki 3532–HI-tester LCR meter in the thermal extent from 298 to 373 K and frequency scope (50–5 MHz).

3. Results and discussion

3.1. AFM characterization

Figures 2(a) and (b) represent a planner and 3D images of Aberchrome 670 thin film with 539 nm thickness, respectively, while, figure 2(c) exhibits the distribution of surface particles versus the mean radius (nm) through the same film, using AFM technique. The high sensitivity of that technique give one the ability to got a reasonable information about the topology of the surface by scanning a very sharp probe along the surface of the sample and also, through z-axis. Depending on the degradation of the color, and either continuity of surface particles or not. In 2D image, nearly all the surface particles have the same color relates to a homogenous and continuous distribution of them through the surface. The inset figure is a zooming of part of the surface, where most of the particles have a spherical shape. The dissection of the gained data, result in a mean radius of the surface particle \( \geq 83 \) nm and the standard deviation \( \geq 21 \) nm. The slight degradation in the color in 3D image reflects the minor change in the thickness from point to point through the whole film through Z-axis. The deep black color relates to the deepest thickness while the white relates to minimum thickness contact to the substrate on which the film has been participated. Above the substrate and moving in z-axis, nearly all film have the same deep brown color. Both 2D and 3D images of AFM reflect homogenous and continues distribution of the particles through the surface of Aberchrome 670 thin film.

3.2. Cyclic-voltammetric characterization

Figure 3 represents the cyclic voltammetry (CV) curve of Aberchrome (670) compound. Since the figure exhibits a one electron reversible reduction and oxidation, the cyclovoltametric technique was recognized as an important technique for measuring the energy of the higher occupied molecular orbital (HOMO) and the lower unoccupied molecular orbital (LUMO) [2, 15, 16]. The oxidation process relates to removal of electron from the (HOMO) energy level. The reduction cycle corresponds to addition of electron to the (LUMO) level. HOMO and LUMO energy levels have been elaborated according to the following equations [2, 15, 16]

\[
E_{\text{HOMO}} = -(E_{\text{ox}} + 4.71 \text{ eV})
\]

\[
E_{\text{LUMO}} = -(E_{\text{red}} + 4.71 \text{ eV})
\]

Where; \( E_{\text{ox}} \) is the onset of oxidation and \( E_{\text{red}} \) is the reduction potential versus Ag/NaCl, which has an oxidation potential of 4.71 eV versus vacuum. Depending on the previous equations ((2a) and (2b)), The electrochemical gap could be calculated according to the following formula;

\[
E_{\text{HOMO-LUMO}} = E_{\text{red}} - E_{\text{ox}}
\]
It has been found to be 2.98 eV compared to 3.1 eV for the thermal evaporated thin films [10], and 2.89, 2.87, 2.86, 2.837 and 2.80 eV for casting Admantan Fulgide thin films [8], 2.87, 2.866, 2.837 eV for casting dicyclopropyle Fulgide (DCPF) thin films [11].

3.3. Electrical resistivity (\(\rho\)) of Aberchrome 670 nano thickness films.

The influence of the temperature on (\(\rho\)) of as-deposited Aberchrome 670 thin films with different nano thickness (423, 539, 614, 639, 717, 738, 794 and 850 nm) was measured from 298 to 373 K. Figure 4(a) exhibits the decreasing of \(\rho\) with both the temperature and the thickness in harmony with other semiconductor thin films [4, 19, 20]. The thin film growth mechanism can explain the exponential decreasing of it with the film thickness, and it can be introduced as following [14]:

\[
\rho = \rho_p + \rho_{im} + \rho_f + \rho_g + \rho_s
\]

(4)

Where \(\rho_p\), \(\rho_{im}\), \(\rho_f\), \(\rho_g\) and \(\rho_s\) represent the 'electrical resistivity' caused by phonon, impurity, defects, grain boundary and the surface scattering, respectively. For nearly large thickness, in comparison with the electron mean free path, the reduction in \(\rho\) is due to mainly, to the increase in the grain size [14]. The interpolated figure of figure 4(a) displays the logarithmic thermal dependence of \(\rho\). That figure exhibits the 'semiconductor behaviour' [4, 19, 20] with two activation energies \(\Delta E_1\) and \(\Delta E_2\), where \(\rho\) was introduced as follow [20]:

Figure 2. (a) 2D image, the inset: a zooming of a part of the surface, (b) 3D images and (c) the distribution of surface particles versus the mean radius (nm) of Aberchrome 670 thin film with 539 nm thickness, depending on AFM technique.
The analysis of the data depicted that there are two activation energies \( \Delta E_1 \) and \( \Delta E_2 \). The average value of \( \Delta E_i \) in the lower temperature range \((T < 360 \text{ K}) \approx 0.41 \pm 0.035 \text{ eV} \) and \( \approx 1.09 \pm 0.012 \text{ eV} \) in the higher temperature range \((T \geq 360 \text{ K}) \) compared with 0.32 eV and 0.95 eV, respectively for DCPF thin films [11].

### 3.4. The differential Seebeck coefficient(S) characterization

Because the designation of the thermoelectric power and Seebeck coefficient \((S)\) is a popular technique to portray organic semiconductors, [4, 20], they were measured for the prepared films and in the thermal range \((\sim 298–373 \text{ K})\). Figure 3(b) exhibits the thermal impact on \( S \), for films with different thicknesses \((423–850 \text{ nm})\).

The positivity of \( S \) over the entire temperature range, indicating the p-type conduction [4, 19, 20]. The bell shape of \( S \) referring to a negative shift of the Fermi-level [19–21]. It has explained in the lower temperature range \(<360 \text{ K}\) by the following equation;

\[
S = + \frac{K_B}{e} \left( \frac{\Delta E_1}{k_0 T} + A_1 \right).
\]

while in the higher temperature range \( \geq 360 \text{ K} \), according to the coming formula [19–21];

\[
S = - \frac{K_B}{e} \left( \frac{C - 1}{C + 1} \right) \left( \frac{\Delta E_2}{k_0 T} + A_2 \right)
\]

Where \( c \) is the mobility ratio, \( A_1 \) and \( A_2 \) are constants. The effective density of the states \( N_{\mu} \) is comparable to the density of molecules [20]. The using of both equations No.6 & 7 and the inset of figure 4(b) lead to investigate, \( N_{\mu} \), the concentration of charge carrier \( p \), and the value of \( c \) [20].

\[
p = N_{\mu} \exp \left( -\frac{eS}{K_0 T} \right)
\]

The mean values of \( p, c, \mu_0 \) and \( N_{\mu} \) are 3.58 \times 10^{20} \text{ m}^{-3}, 0.891 and 3.412 \times 10^{-12} \text{ m}^2 \text{ v}^{-1} \text{ s}^{-1} \) and 1.83 \pm 2.06 \times 10^{-4} \text{ m}^{-3}, respectively. The thermal depression in \( S \) may be assigned to the high mobility of the created electrons [20], correlated with the diversion of several molecules of unfolding E-isomer into bolted C-isomer, where the lone pair of electrons on the heteroatom in the furan ring has to share in the conjugated system. The Oxygen has two lone pair electron get in resonance with the ring to form six \( \Pi \)-electron parallel benzene. It is more reacting, extremely than benzene, due to the electron-donating leverage of the oxygen heteroatom. Inspection of the resonance contributors shows the augmented electron consistency of the ring, leading to increase in the rates of electrophilic substitution (see figure 5) [15]. The combination between the results of the thermal credence of \((p)\) and \((S)\) drive to infer that there are two impurity levels inside the gap of the films(423–850 nm) in the thermal zone (\(\sim 298–373 \text{ K}\)). The first one is shallow acceptor level with an activation.
Figure 4. (a): $\rho$ versus $d$ at fixed temperatures; the inset fig. $\ln(\rho)$ versus $\left(\frac{1000}{T}\right)$ for different thickness films. (b): $S$ versus $T$ for Aberchrome 670 nano thickness films with different thickness; the inset fig.: $S$ versus $\left(\frac{1000}{T}\right)$ at constants temperatures.
energy $\Delta E \sim 0.39 \pm 0.004 \text{eV}$. The second impurity level is deep donnered level with an activation energy $\Delta E \sim 0.98 \pm 0.012$ in thermal zone $\sim 360 \text{K}$. The determined electrical parameters of Aberchrome 670 thin films are listed in table 1.

3.5. Dielectric characterizations of Aberchrome 670 thin films.

The impact of both the temperature and frequency on the capacitance ($C$), the dielectric constant ($\varepsilon_1$), the temperature capacitance coefficient (T.C.C) and the dissipation factor tan(δ) was inspected in the frequency range (50–5MHz) and thermal zone ($\sim 298–373 \text{K}$) for films with 850 $\pm$ 25 nm thickness. Figure 6(a) displays the frequency dependence of $C$ at diverse steady temperatures. The figure manifests a decaying of $C$ with frequency to reach a minimum value $C_\infty$ at a higher frequency. The result can be interpreted of an equivalent circuit model of an original capacity element ($C$), a resistance ($R$) due to the film in parallel with $C$ and a series bulk resistance ($r$). The equivalent series capacitance ($C_S$) can be stated as follow $^{[22, 23]}$:

$$C_S = C_\infty + CD^2$$  \hspace{1cm} (8)

Where $D = \frac{1}{2\varepsilon_0}$

The leverage of charge redistribution can explain the behaviour of the capacitance, by carrier jumping on the defects $^{[22]}$. The increasing of $C$ with temperature may be attributed to the decrease of resistance element(R). The higher temperature is more active on the hopping of the carries due to swelling in the thermal resurrection $^{[22]}$.

Also, the temperature capacitance coefficient (T.C.C) was extracted according to the following simple formula $^{[24, 25]}$:

$$\text{T.C.C} = \frac{\Delta C}{\Delta T^*C_R}.$$  \hspace{1cm} (9)

where $\Delta C$ is the capacitance change in part per million(PPM), $\Delta T$ is the temperature change with concerning to the reference temperature, and $C_R$ is the capacitance at the reference temperature. Figure 6(b) accentuates the frequency dependence of T.C.C for Aberchrome 670 thin films at different fixed temperatures, where it was found to vary from 0.02 to 0.13 PPM K$^{-1}$ at 50 Hz and from 0.06 to 0.474 PPM K$^{-1}$ at 5MHz as the temperature increased from 298 to 373 K.

Also, the real dielectric permittivity ($\varepsilon_1$) could be obtained during the present work using the famous equation; $\varepsilon_r = \frac{CD}{\varepsilon_0A}$ $^{[22, 25]}$, $d$ is the thickness, and $A$ is the cross section area. Figure 7 shows the decreasing of $\varepsilon_1$ with increasing of the frequency and reduction of temperature. The decay of ($\varepsilon_1$) with frequency may be due to
Figure 6. (a) (C,F) versus Ln(ω, sec$^{-1}$) at different fixed temperatures for Aberchrome 670 nano films with 850 nm thickness. (b): (T.C.C, PPM/K) versus Ln(ω, s$^{-1}$) at different fixed temperatures for Aberchrome 670 nano films with 850 nm thickness.
the influence of Multi-component of polarization [25]. On the other side, increasing it with temperature perhaps related to the facility of the orientation of the dipoles [22, 25, 26].

Since the dielectric loss angle is an important parameter to describe the loss of power in a specimen [27], the effectiveness of the frequency on the dissipation factor (tan(δ)) of Aberchrome 670 film with 539 nm was investigated from 50 to 5 MHz and in the thermal zone from 298 to 373 K. Figure 8(a) exhibits the experimental results of it, where tan(δ) raised by the augmentation of the frequency to reach a maximum peak signalizing Debye-type relaxation procedure with the relaxation frequency (f_max) which is equal to (2πτ)^{-1} [22], where (τ) is the relaxation time. By the increment of the temperature from 298 to 373 K, the peak has shifted to a higher frequency (f_max) (see the inset of figure 8(a)). Tan(δ) can be dissected by the next relevance [23];

\[
\tan(\delta) = D\left(1 + \frac{L}{R}\right) + \omega_C
\]

This disposal alludes a temperature vassal relaxation time according to the following formula [22];

\[
\tau = \tau_\infty \exp\left(\frac{E_0}{k_B T}\right)
\]

Where τ_\infty is ‘the relaxation time at infinite temperature’, and E_0 is ‘the thermal activation energy’ for this process. The estimated values of them, depending on the intercept and the slope of the relation between ln(τ) and \(\frac{f_{\text{max}}}{T}\) (figure 8(b)), were 1.01 × 10^{-5} s and 0.049 eV, respectively, see table 2.

The obtained results deduced that the quality factor (Q = \frac{1}{\tan(\delta)} [27]) of an isolation portion of Aberchrome 670 thin films decreases as the frequency increases till f_max, where the losing in the power reach to maximum value. This reduction in the quality factor (increasing in Tan(δ)) may be due to the increasing in the reactive current and the conductivity. Behind f_max a short valley appeared and again the reduction in the power increases as the frequency increases. The reasonable region to use Aberchrome 670 thin films with nano thickness as a good insulator in the di-electrical applications is from f = 2.012 KHz to 12.7 KHz at room temperature and from f = 2.12 KHz to 25.9 KHz at 373 K.

4. Conclusions

The following results can draw from the current paper;
Aberchrome 670 (E-3(Admantan-2-ylidene)-4-[1-(2,5-dimethyl-3-furyl) Ethyldene] Dihydro -2,5-Furan-dione) thin films were prepared by thermal evaporation technique with different nano thicknesses, the mean radius of the surface particle $\approx 83$ nm, using AFM. The operating of cyclic-voltammetric technique deduced that the electrochemical gap $\approx 2.98$ eV.

The mean values of the density of states ($N_{\mu}$), the concentration of the holes ($p$), the ratio of the mobilities ($c$) and the mobility of the holes ($\mu_h$) are $1.83 \pm 2.06 \times 10^{24}$ m$^{-3}$, $3.58 \times 10^{20}$ m$^{-3}$, 0.891 and $3.41 \times 10^{-12}$ m$^2$ V$^{-1}$ s$^{-1}$, in the temperature zone (298-373 K), and for different thicknesses starting from 423 to 850 nm.

**Figure 8.** (a): Tan ($\delta$) versus ln($\omega$); the inset represents ($f_{\text{max}}$) versus ln($\omega$) at different steady temperatures for Aberchrome 670 nano films with 850 nm thickness. (b): ln (τ) versus ($\frac{1000}{T}$) for Aberchrome 670 nano films with 850 nm thickness.
Table 2. Dielectric parameters of (E-3(Admantan-2-ylidene)-4-[1-(2,5-dimethyl-3-furyl)Ethylidene] Dihydro -2,5-Furandione), nano thickness Films.

| T (K) | $f_{\text{max}}$ (KHz) | $C_{\infty}$ (pF) at ($f = 5$ MHz) | T.C.C (PPM/K) At $f = 50$ Hz | $f = 5$ MHz | $\varepsilon_1$ at $f = 50$ Hz | $f = 5$ MHz |
|-------|------------------|-------------------------------|-------------------------------|-------------|-----------------|-------------|
| 298   | 2.05             | 10.47                         | 0.059                         | 0.06        | 8.2             | 0.31        |
| 315   | 4.28             | 14.6                          | 0.064                         | 0.069       | 13.5            | 0.58        |
| 325   | 4.73             | 17.75                         | 0.077                         | 0.088       | 18.46           | 0.85        |
| 335   | 6.08             | 20.9                          | 0.081                         | 0.132       | 21.96           | 1.12        |
| 345   | 8.29             | 63.45                         | 0.088                         | 0.211       | 29.97           | 1.66        |
| 355   | 9.35             | 106                           | 0.108                         | 0.302       | 33.02           | 2.47        |
| 365   | 10.97            | 164                           | 0.112                         | 0.403       | 60.01           | 2.74        |
| 375   | 11.88            | 221                           | 0.130                         | 0.48        | 87.004          | 3.55        |

The frequency dependence of ($\tan(\delta)$) in the frequency band ($50 - 5$ MHz) and temperature range ($298 - 373$ K) revealed a Debye relaxation process with an infinite temperature relaxation time ($\tau_{\infty}$), and thermal activation energy ($E_0$) $\approx 1.01 \times 10^{-5}$ s and 0.049 eV, respectively.

Acknowledgments

The author wishes to express the sincere gratitude to Prof. Dr. M. M. El-Nahass, a professor of solid state and Prof. Dr. H. Hassanin, a professor of chemical physics, Ain Shams University for their excellent scientific guidance.

Compliance with ethical standards

The author announces that this work was not funded by any governmental or private bodies, as well as the author has no conflict of interest.

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