Properties of Iodine Doping In Amorphous Carbon Thin Films Grown by Chemical Vapor Deposition (CVD) Method

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***Abstract*—In this work, the influence of iodine doping on the electrical, optical, and structural properties of amorphous carbon (a-C) thin films that deposited on glass and quartz substrate at varied temperature ranging from 500 °C to 700 °C by thermal chemical vapor deposition (CVD) technique using argon (Ar) as carrier gas were reported. The films were characterized by Perkin Elmer (LAMBDA 750) UV/VIS/NIR spectrophotometer for optical properties, BUKOH KEIKI (CEP200) Solar Simulator for electrical properties, and Raman spectroscope for structural properties. The I-V measurement revealed that I-doping increased the conductivity as the temperature increased. The optical band gap decreased from 1.0 eV to 0.15 eV due to the condition of undoped and iodine doped of the thin films. The Raman spectral result which was measured using Raman Spectroscopy HORIBA Jobin Yvon (HR800) shows that graphitization of the films intensified after iodine doping is introduced. The surface morphology of the thin films was measured by Atomic force microscopy (AFM) which indicates the roughness of the films.**

***Keywords*—amorphous carbon (a-C) thin films; iodine doping; thermal chemical vapor deposition (CVD); electrical properties; optical properties; structural properties**

I. INTRODUCTION

The element carbon (C) is used to produce amorphous thin films for solar cells as it is an attractiveness element that can be distinguished in the forms ranging from insulator diamond to metallic graphite to semiconducting nanotubes [1,2]. Carbon is said has certain prominent properties such as high electrical resistivity, high thermal conductivity, high dielectric strength, and high hardness [3]. Camphor oil ($C_{10}H_{16}O$) was used as precursor as it act as the natural source of carbon that available in nature, which is a material of highly stable, cheap and non-toxic.

Carbon happens in the form like soot, carbon fiber, and evaporated carbon as it one of the attractive semiconductor element because of its semiconducting nature [4, 5]. The deposition by pyrolysis process of the evaporation of camphor oil as source of carbon has created the amorphous carbon (a-C) thin film on the substrates.

The conduction type of a-C is indispensable to be controlled when attempted to utilize a-C as an alternative material [7, 8]. The properties of semiconductor materials can be modified such that in optical band gap and photoconductivity by effective doping technique.

Doping technique is the method to enhance and establish the properties of undoped a-C and iodine (I) is p-type dopant that has good behavior in modifying optoelectronic properties such as conductivity and optical band gap values with the increment of electron or hole concentration [2, 4]. The characterization of I-doped a-C thin films was studied on optical and electric properties. The affects for the interband absorption of the films for the optical measurement at visible light caused decreasing optical band gap effects the density of states at the conducting band [5].

In this report, thermal CVD method was used to develop a-C thin films as CVD is the most economical in production. The deposition temperature was varying from 500 °C to 700 °C for a-C thin films deposition while deposition temperature of iodine doping was maintained at 300 °C for every sample.

II. EXPERIMENTAL PROCEDURE

A. Substrate Preparation

The quartz and glass substrate were immersed in acetone (C_3H_6O) and cleaned by using Ultrasonic Cleaner (Power Sonic 405) for 10 minutes. The same steps was repeated for methanol and deionized (DI) water. The glass and quartz substrate were blown with nitrogen gas (N_2).

B. Thermal CVD Preparation

The procedure to prepare the thin films is illustrated in the flow chart of Fig. 1. The Thermal CVD applied double furnace setup as shown in Fig. 2. The purpose of using double furnace CVD is to vaporize source material at first furnace and the pyrolysis process will take place at second furnace as the temperature is higher than the first furnace.

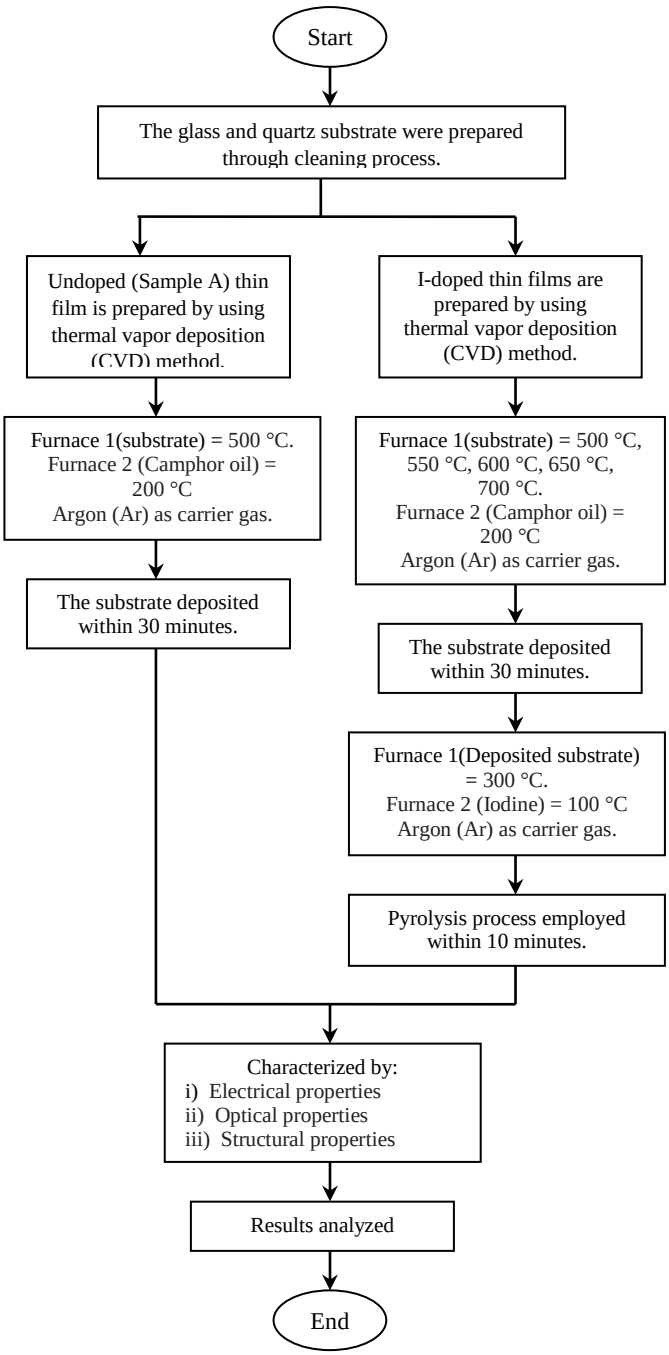

Fig. 1. Flow chart of experimental procedure

The undoped a-C thin film (Sample A) was prepared at 500 °C for first furnace (substrate) and 200 °C at second furnace (camphor oil). Amorphous carbon (a-C) thin-films were deposited on the substrate by the thermal CVD as a deposition method. For films deposition, argon (Ar) is used as carrier gas and the gas flow rate was maintained at 30 bubbles per minute and camphor oil ($C_{10}H_{16}O$) used as carbon precursor. The films deposition process was took place in duration of 30 minutes.

The same technique of deposition process as undoped thin film was committed for post-doping with the same deposition period at different temperature ranging from 500 °C to 700 °C for first furnace and 200 °C for second furnace in which located of substrate and camphor oil respectively. For iodine doping, the pyrolysis process was employed by the thermal CVD to deposit iodine which in solid form on the a-C thin films within 10 minutes with the evaporation of iodine. The first furnace was used to deposit the deposited substrate at temperature of 300 °C and the second furnace was used to place the materials which iodine (solid form) in the boat then put into the quartz tube and the temperature was maintained at 100 °C. Argon gas was supplied into both furnaces. Gold (Au) which is a good conductor material is used as metal contact and it been sputtered on the top surface of thin films by using sputter gold. The thin films were investigated by BUKOH KEIKI (CEP200) Solar Simulator for electrical properties while JASCO V-570 UV/VIS/NIR spectrophotometer (LAMBDA 750) was used to obtain optical properties. HORIBA Jobin Yvon (HR800) and Atomic force microscopy (AFM) were used to measured structural properties.

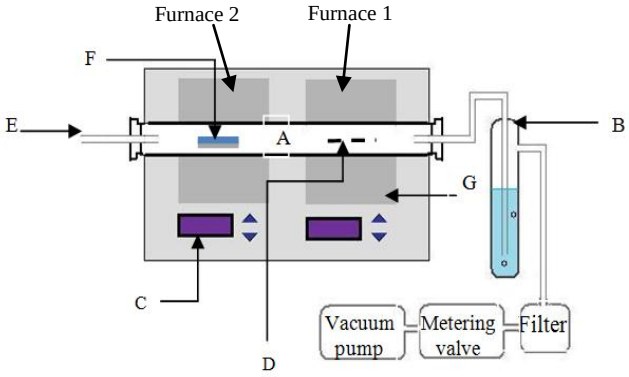

Fig. 2. Thermal CVD production apparatus (A) Quartz tube, (B) Water bubbling system, (C) Temperature controller, (D) Glass and guartz substrate, (E) Carrier gas (Ag), (F) Iodine (solid form), (G) Deposition furnace

III. RESULTS AND DISCUSSION

A. *Electrical Properties*

The electrical properties were investigated by using current-voltage (I-V) measurement that used gold (Au) as the metal contact with the thickness of 60nm. Fig. 3 shows the cross-sectional view of I-doped a-C thin film that deposited on substrate with the gold as the metal contact. Gold is said strongly acts as semiconductor material in which it consists of tiny atoms and to achieve ohmic contacts as well. According to Fig. 3 and 4, the current is increased as the deposition temperature increased which showing an ohmic behavior.

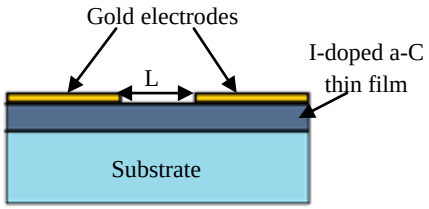

Fig. 3. Cross-sectional view of I-doped a-C thin film

Fig. 4 and 5 illustrate the characteristic of I-V under dark and illumination respectively. Both figures show that the current changes proportionally with the voltage and it increased under illumination that caused the by the generation of excess carriers. The I-V structure does not show the rectifying behavior or Schottky contact but ohmic contact is been formed between the gold (Au) electrodes and I-doped a-C layer. It is suggested happen due to the semiconductor characteristic of gold in which more metallic and might attributed to the existence of rich sp^2 bonding [9]. Microstructure of C consists of σ and π-sites and they lied closer to the Fermi level because π-sites are more weakly bonded. The characteristic of gap states is determined by the filled π-sites from the valence band and empty π-sites from conduction which influence the electrical conductivity when illuminated by the light [5].

The difference in I-V measurement and conductivity under dark and illumination show the characteristic of photoresponse which under illumination the properties are increased. When the light is strikes on the front surface of the I-doped a-C thin film, the high energy photons will absorbed by the layer and generate electron-hole pairs that contributed to quantum efficiency [6]. Thus, give results on increment of conductivity as the spin density is decreased that might related to the successful of doping process.

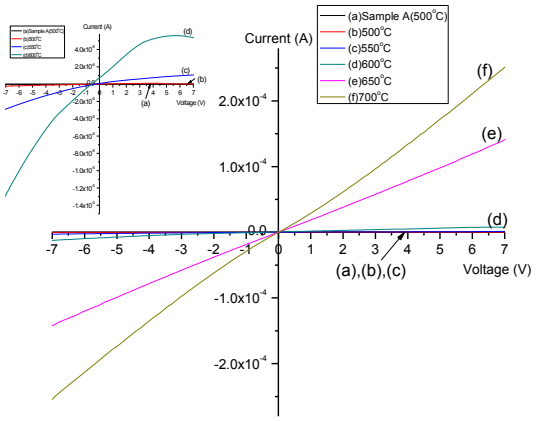

Fig. 4. I-V curves of undopd and I-doped a-C thin films deposited at different deposition temperature (in dark)

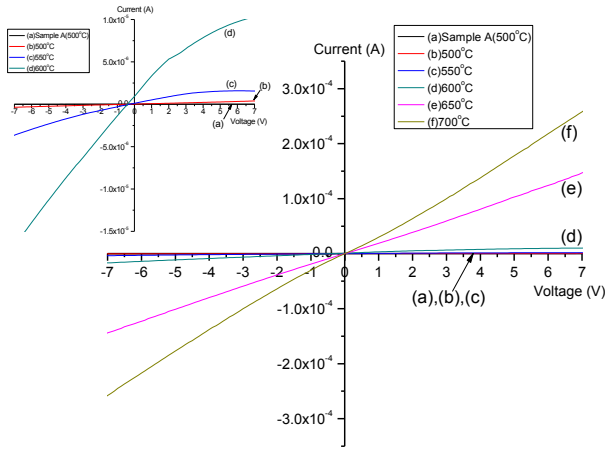

Fig. 5. I-V curves of undopd and I-doped a-C thin films deposited at different deposition temperature (under illumination)

The conductivity (σ) can be obtained from the calculation of resistivity (ρ). Resistivity was calculated by using equation (1), while conductivity was determined based on equation (2).

$$\rho = \frac{Rwt}{L} \qquad (1)$$

$$\sigma = \frac{1}{\rho} \qquad (2)$$

Where R is the resistance that obtained from relation of current and voltage, w is the width of the electrodes, t for the thin films thickness, and L is length between the electrodes.

The conductivity is increased as shown in Fig. 6 and it can be observed that the I-V curves and conductivity graph for doped thin films rapidly increased from 600 °C to 700 °C. It is suggested that the a-C thin films were transformed from insulator to semiconductor to semi-metallic materials due to the lower optical band gap such that inversely proportional to the temperature increment and might due to the increasing of sp^2 bonds and decrement on sp^3 bonds that related to graphitization [11,13].

The increment of conductivity as illustrated in Fig. 6 is tallying with the result of conductivity that reported by Latha et.al [14]. As the deposition temperature increase, the electrical conductivity also increased due to the electrical transport in the valence band which lead to the increase on formation of sp^2-bonded carbon and detraction of sp^3 bonding as well as resulted in increment of localized hopping state. When the deposition temperature increased, the localized hopping state also increased [15]. Details on the conductivity is shown in Table 1 and the conductivity of I-doped thin films at 500°C is higher than undoped sample A caused by content of iodine in the thin film in which decreasing of the optical gap that induced the graphitization and increase the conductivity [5].

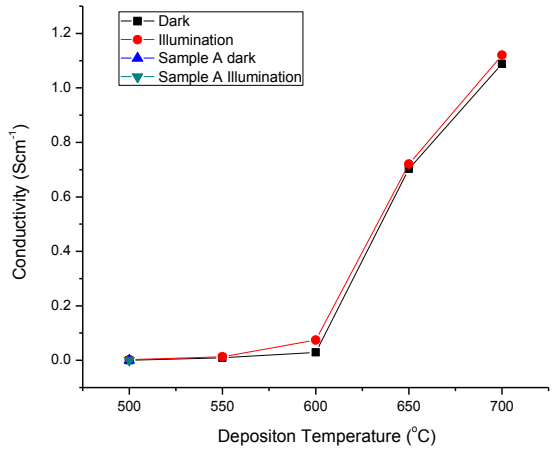

Fig. 6. Conductivity of undoped and I-doped a-C thin films deposited at different deposition temperature (under illumination)

TABLE 1.
CONDUCTIVITY OF UNDOPED AND DOPED a-C THIN FILMS DEPOSITED AT DIFFERENT TEMPERATURE

Temperature (°C)	Dark (Scm^{-1})	Illumination (Scm^{-1})
Sample A (Undoped)	8.85797E-5	1.02345E-4
500	3.41025E-4	0.00273
550	0.00963	0.01331
600	0.02909	0.07466
650	0.70258	0.72069
700	1.08799	1.12025

B. Optical Properties

The optical properties were carried out by using UV/VIS/NIR spectrometer with the wavelength in the range of 200 to 2000 nm. The transmittance (%T), absorption coefficient (α), and optical band gap (E_g) can be obtained by spectrometer measurement. Transmittance is defined as the ratio of light intensity that passed through a sample to the intensity of light that entered the sample. Transmittance result on Fig. 7 shown that the nearest to the 100% transmittance is undoped a-C thin film at 500 °C approximately 98%, followed by I-doped thin films at deposition temperature ranging from 500 °C to 700 °C that shown results on decrement in percentage of transmittance. For I-doped a-C thin films at 500 °C, 550 °C, and 600 °C, the transmittance were about 95, 90, and 80% respectively. Meanwhile, the transmittance decreased approximate to 40 and 30% for deposition temperature at 650 °C and 700 °C. The transmittance near to 100% indicates the imperfection of a-C thin films at the different deposition temperatures [10].

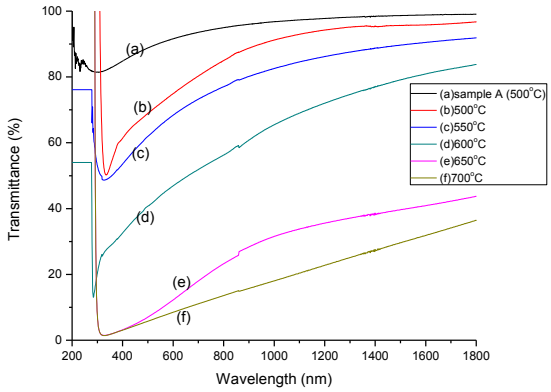

Fig. 7. Spectral of transmittance of undoped and I-doped a-C thin films deposited at different deposition temperature

The absorption coefficient (α) was obtained by the calculation of equation (3) with the transmittance (T) and thickness (t) data.

$$\alpha = \frac{1}{t}\ln\frac{1}{T} \qquad (3)$$

The result of absorption coefficient that been obtained is in the range of 10^5 cm^{-1} and corresponding with result that reported by Kalaga et.al [17]. The absorption coefficient of I-doped was higher than undoped thin films as shown in Fig. 8. The absorption coefficient of undoped thin film is lower compared to I-doped thin films that suggested the undoped sample possessing the amorphous nature of carbon. The absorption coefficient represents on how the penetration of the light can be onto the material before it is absorbed. The light is poorly absorbed for the material with low absorption coefficient and it will appear transparent if the material is thin enough. The highest absorption coefficient belonging to I-doped thin film at deposition temperature of 700 °C followed by 650 °C, 600 °C, 550 °C, 500 °C, and lastly undoped thin film at 500 °C. a-C material consist ratio of two bonding which sp^2 and sp^3. The increment of absorption coefficient is suggested due to the presence of more sp^2-bonds (graphite element) in the films [11].

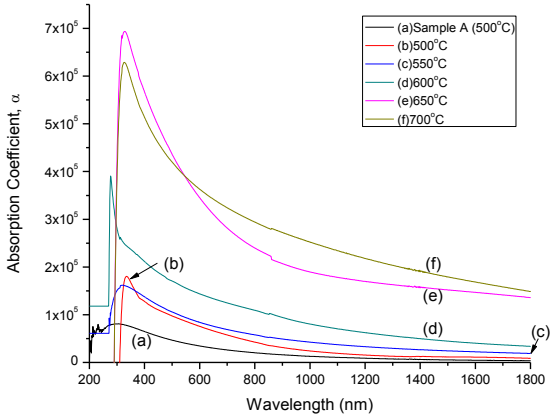

Fig. 8. Absorption coefficient of undoped and I-doped a-C thin films deposited at different deposition temperature

The optical band gap is the measurement of the gap between the extended state in the valence band and the conduction band. The graph of optical band gap energy ($(\alpha hv)^{1/2}$) versus photon energy (hv) shown in Fig. 9 by considering the pylorized of a-C thin films on undoped and I-doped. The optical band gap of each graph is acquired from the extrapolation of the linear section of the curves. The optical band gap energy can be obtained from Tauc equation (4).

Tauc equation,

$$(\alpha\, hv)^{1/2} = B(E_g - hv) \qquad (4)$$

The values for optical band gap for undoped and I-doped a-C thin films are clarified on table 1. The intercept of the curves have resulted on optical band gap of undoped a-C film with 1.0 eV and decrease to 0.6 eV when I-doping is introduced. The optical band gap of I-doped thin films decreased up to 0.15 eV as the deposition temperature increased from 500 °C to 700 °C. The results of detraction in optical band gap suggest that the iodine doping on a-C thin films given occasion to more conductivity because the composition of iodine on the films induced graphitization by decreasing the optical band gap compared to undoped thin films [9]. It is clear that the ratio of the sp^2 - sp^3 hybridized bonds change with difference of deposition temperature in which sp^2 or π-sites bonds increased and sp^3 bonds decreased as ascension of deposition temperature that strongly change the optical band gap because of decrement of Eg such that π-sites lies more closer to Fermi level then the σ-sites [11,12]. The films that deposited at higher temperature consist of large graphite cluster size since the large size of sp^2 bonded cluster contributes to decrement of Eg.

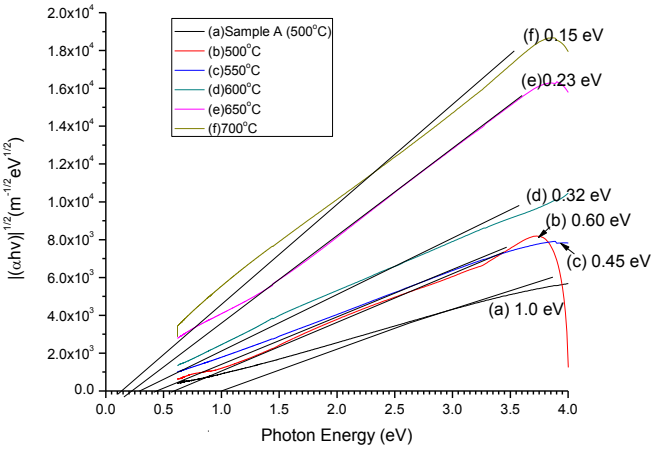

Fig. 9. Tauc plot $(\alpha hv)^{1/2}$ versus photon energy (hv) of undoped and I-doped a-C thin films at different deposition temperature

TABLE 2.
THE OPTICAL BAND GAP OF UNDOPED AND DOPED A-C THIN FILMS AT DIFFERENT DEPOSITION TEMPERATURE

Temperature (°C)	Optical Band Gap (eV)
Sample A (Undoped)	1.00
500	0.60
550	0.45
600	0.32
650	0.23
700	0.15

C. *Structural Properties*

i. Raman Characterization

Raman spectroscopy is a well known tool that frequently used to characterize and investigate the structural properties of a-C and even DCL thin films [8]. The illustration of Raman spectral in Fig. 10 was obtained for undoped thin film at 500 °C as well as 500 °C, 550 °C, 600 °C, 650°C, and 700 °C for I-doped thin films with the different deposition temperatures. The two peaks that been obtained indicate poly-crystalline graphite that known as disorder peak (D peak) and graphite peak (G peak) which are appeared at 1325 cm^{-1} and 1600 cm^{-1} respectively that commonly used to clarify disorder of carbon structure [10, 15]. The D peak indicates disorder sp^2-hybridized C with an amount of sp3- hybridized C, meanwhile the G peak is refer to graphite-like sp^2-hybridized C on the composition of thin films. The amorphous nature of undoped thin film was revealed according to its Raman spectroscopy that shown in Fig. 9 and its D and G peak is weaker compared to I-doped thin films [8]. The graphitization of the films was intensified after doped with iodine.

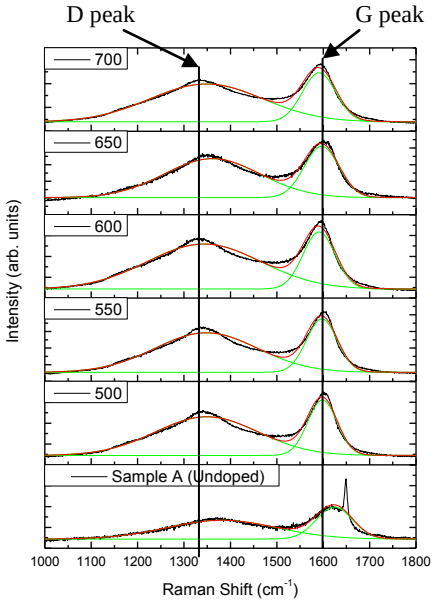

Fig. 10. Raman spectral of undoped and doped a-C thin films deposited at different deposition temperature

The data for peak position, peak full-width at half-maximum (FWHM), and the integrated intensity ratio of D and G peaks that been obtained from Gaussian fittings are tabulated in Table 3 and 4. It is can be observed that as the deposition temperature increased, the ratio of intensity (I_D/ I_G) also increased and G peak width decreased in which indicates that the size of graphite (sp^2) cluster becomes larger. Thus, increase the fraction of sp_2-bonded carbon atoms is resulted on the increment of conductivity [18]. The results of the Raman spectroscopy recommended that the films contain the mixture of sp^3-C bonds (tetrahedral bonded C) and sp^2-C bonds (trihedral bonded C) that act as localized conduction states. The iodine doping on the films has induced the graphitization thus it is proposed that the structural changed due to the relations between sp^2 and sp^3 bonds.

TABLE 3.
THE PEAK POSITION AND FWHM OF RAMAN SPECTRA OF UNDOPED AND I-DOPED a-C THIN FILMS AT DIFFERENT DEPOSITION TEMPERATURE

Deposition Temperature (°C)	D Peak		G Peak	
	Position (cm^{-1})	FWHM (cm^{-1})	Position (cm^{-1})	FWHM (cm^{-1})
Undoped (Sample A)	1416.74731	238.47536	1659.46607	77.88548
500	1349.04451	221.7451	1598.11636	63.15289
550	1347.95706	221.02248	1596.97581	66.18809
600	1355.94218	219.30478	1596.84842	69.25464
650	1360.78642	206.49056	1596.50711	74.91566
700	1343.03334	245.86684	1591.00549	71.93994

TABLE 4.
I_D/ I_G OF UNDOPED AND I-DOPED a-C THIN FILMS AT DIIFERENT DEPOSITION TEMPERATURE

Deposition Temperature (°C)	D Peak	G Peak	I_D/ I_G
	I_D	I_G	
Undoped (Sample A)	45.35133	76.75129	0.590886876
500	370.89543	531.32902	0.698052273
550	473.2462	639.2368	0.740329992
600	298.09725	391.48262	0.761457175
650	793.5472	1028.982	0.771196526
700	431.53377	547.19731	0.788625533

i. Atomic force microscopy (AFM)

The atomic force microscopy (AFM) was used to investigate the surface morphology of the thin film where it is a three-dimensional (3D) surface topography imaging technique. Fig. 11 shows the images of AFM for different deposition temperature and the data of AFM is tabulated in Table 5 in which the roughness of the thin films decreased as the deposition temperatures increased.

AFM investigates carbon films which it reveals the relation between roughness of surface and degree of diamond like sp^2 and sp^3 properties as it quite straightforward when concerning on amorphous carbon and nanocrystalline carbon [19]. The decrement of sp^3 bonding contributes to detraction of dangling bond which is the condition where the atoms not bonded each other or known as lone pair. Detraction of dangling bond due to the decompression of particles size proven by the decreasing of roughness value that contributes to increment of grain boundaries which increased the surface energy, thus make the electron easy to move and increase the electrical conductivity.

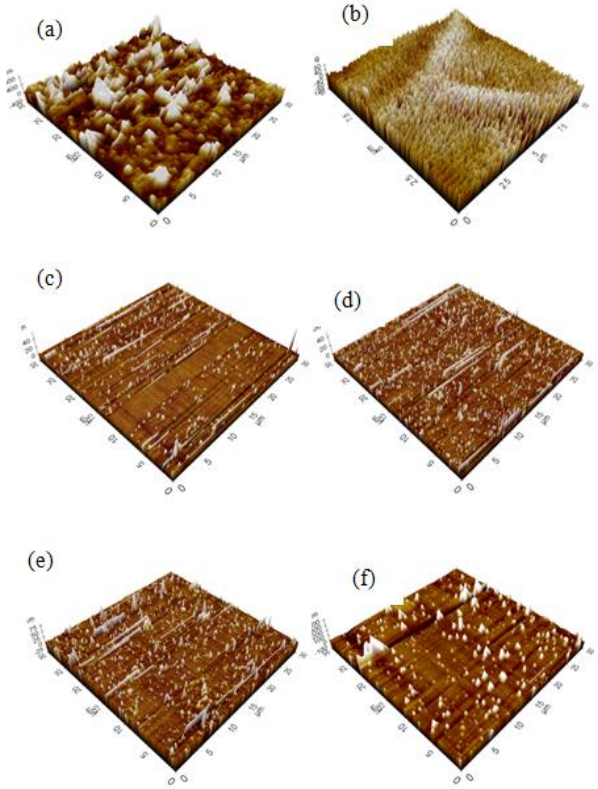

Fig. 11. Images of AFM for undoped and I-doped a-C thin film at different deposition temperature (a) Undoped (Sample A at 500°C), (b) 500°C, (c) 550°C, (d) 600°C, (e) 650°C, (f) 700°C

TABLE 5.
SURFACE ROUGHNESS OF UNDOPED AND I-DOPED a-C AT DIFFERENT DEPOSITION TEMPERATURE

Deposition Temperature (°C)	Roughness (nm)
Undoped (Sample A)	32.284
500	10.415
550	0.872
600	0.564
650	0.526
700	0.079

IV. CONCLUSION

The effect of iodine doped on amorphous carbon (a-C) thin films by different temperature ranging from 500 °C to 700 °C by using simple deposition technique, CVD have been investigated. The optical, electrical, and structural properties for undoped and I-doped a-C thin films were characterized and compared. The increment of conductivity was resulted from increment of deposition temperature which suggested that the sp^2 bonding on the sample was increased. The optical band gap of I-doped thin films was decreased from 0.60 eV to 0.15 eV while the optical band gap for undoped film was the higher which is 1.0 eV. The decrement of optical band happened due to large size of graphite cluster which make easy for electrons to move and increase conductivity. Raman spectra shows that the graphitization was intensified after I-doped on a-C thin films with ratio of I_D/I_G increased from 0.590886876 to 0.788625533 due to changes in sp^2 and sp^3 bonding in which increased the fraction of sp_2-bonded and contribute to ascension of electrical conductivity. The measurement of AFM gives results on surface morphology of thin films that clarified the decrement of surface roughness will decrease the sp^3 bonding and dangling bond. For the future research, it is recommended that the parameter can be varied according to rate of carrier gas flow and time deposition in order to analyze the effect of these parameters to the properties of the thin films. Besides that, thermal CVD technique can be exchanged to the Aerosol-Assisted CVD (AACVD) technique as it the tendency in producing the nanoparticles materials, as reported by A.Salaun et.al [20].

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