Angle dependent wavelength measurement using open spectrophotometer and optical characterization of thin films

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In this study, angle dependent optical transmittance measurement system supported by Arduino Uno has been developed to accurately determine the optical constants of thin films. Using this system, the angle of incidence light could be measured with an accuracy of ±0.01° and the relative optical transmittance with a precision of 0.01%. By taking into account, the accuracies in the designed system and the wavelength uncertainty of the spectrophotometer (0.15 nm), the factors that may adversely affect the optical transmittance measurements have been eliminated. With this designed system, optical transmittance spectra obtained for two different angles of incidence were obtained precisely for the a-SiN_x:H sample prepared using the glow-discharge system. These spectra and the genetic algorithm were used to determine the optical constants of the sample. With this new system, optical constants were determined faster and more accurately (± 0.05%) than the classical method. Measurement of the absolute optical transmission is no longer required.

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1. Introduction

To determine the optical constants of semiconductor thin films prepared with different techniques, the optical responses of the thin film at normal incidence light is usually taken into account. These responses are the optical transmittance (*T*) or optical reflectance (*R*), in which the intensity of light passing through the film or reflected from the surface of the thin film is measured without damaging the film. Taking accurate measurement of these responses at normal incidence light into account, optical constants can be calculated by evaluating optical transmission or optical reflection spectra or both for the visible wavelength (Vis) and near-infrared (NIR) wavelength regions [1-7]. In these calculations, usually empirical equations depending on wavelength or different model behavior (Forouhi-Bloomer, Cauchy, Sellmeier, classical oscillator, Drude) are used for the refractive index dispersion. The programs developed for these calculations are recommended to users as commercial products by various companies [8-10]. However, the presence of thin films that exhibit different behaviors depending on the materials and preparation techniques is not available in the database. This situation restricts the use of commercially available computer programs. Therefore, studies on the application of user-friendly software and algorithms developed for nonlinear equation systems to optical spectra leave room for further development. Genetic Algorithm (GA), which was first developed by Holland [11], was used by Jureca [12] and Güngör [4, 5] in determining the optical constants of thin films as well as in many engineering problems. The most important feature of GA is that it does not need gradient information like classical optimization methods and it explores the solution space globally.

The number of parameters is very important in calculations based on the solution of nonlinear equations. Decreasing the number of parameters decreases the uncertainty in the variables and increases the calculation speed. Also, having extra equations to be used in the solution of the problem will facilitate the solution of the problem. The evaluation of the optical transmission spectrum obtained at oblique incidence light can be given as an example. In such a case, the whole optical transmission spectrum shifts toward the shorter-wavelength region. Swanepoel determined the optical constants of thin films, taking into account the optical transmission spectra [13,14]. In addition, Fang used this method called as an improved Swanepoel method to determine the refractive index dispersion and thickness of hot-pressed chalcogenide thin films [15]. The biggest advantage of this method is that optical constants can be determined using nonlinear equations using a smaller number of parameters. The sensitivity of this method is based on obtaining accurate wavelength measurements only. Considering with the developments in semiconductor device technology, both detector sizes and microcontroller sizes have been reduced. In addition, the control electronics have become more compact. Hence, an open system spectrophotometer can be design using a microcontroller, a stepper motor driver with micro-step precision, a high sensitive light controller and charge-coupled detector (CCD) array. In this way, optical measurements can be performed more precisely and the angular and wavelength uncertainty required by the method is reduced. Although modern spectrophotometers have similar features, but their cost is considerably higher than such an open system spectrophotometer.

In this study, with the open spectrophotometric system designed, the wavelength shift between two spectra one under normal incidence and the other under oblique incidence angle of 54°, could be measured precisely. Using two spectra and the Genetic Algorithm, considering only the precisely determined wavelength values were evaluated, and the correct optical parameters such as film thickness, refractive index, and optical band gap values were calculated.

2. Materials and methods

2.1. Experimental details

In this study, the nitrogen-doped a-SiN_x:H thin film was prepared by adding a nitrogen gas line to the radio frequency plasma deposition system established [16]. Sample preparation parameters are given in Table 1. The optical measurements of the a-SiN_x:H thin film was carried out at room temperature using a spectrometer (S3000-UV-NIR spectrophotometer, Seemantech company.) with a CCD in the wavelength range 400 – 1000 nm. The wavelength uncertainty is 0.15 nm. For an open spectrophotometric system, a specially designed sample holder is placed on the step motor to obtain the optical transmittance spectrum for normal incidence and oblique incidence light (Fig. 1). With the micro-step motor driver (JK0230) used with Arduino UNO, the step motor completes one full turn in 25600 steps. Thus, the uncertainty in the angle is calculated as ±0.01°. In the experiment, the angle of incidence light was set as 54.00°. A 50 W halogen lamp is used as the light source. The luminous intensity is controlled by a voltage-controlled current source. A 12-bit resolution MCP4725 Digital-Analog Converter (DAC) with Arduino UNO was used to obtain the control voltage. A voltage value in the range of 0 - 5 V, corresponding to the appropriate light intensity that does not saturate the CCD detector, is sent to the lamp control unit via the DAC. In this way, the control voltage and thus the lamp current can be changed in 4096 different steps. The designed current source using with Op-Amp's keeps the lamp current constant (±0.001 A) for each set current. Hence, optical transmittance measurements can be measured with 0.01% sensitivity.

Table 1. Preparation parameters for a-SiN_x:H thin film. Atomic concentration of nitrogen (x), partial pressure of nitrogen ($r=PN_2/P(SiH_4)$), substrate temperature (T_s=320 °C), deposition time (t), film thickness (d) and optical band gap (E_g).

Sample	***x***	***r***	***t (hour)***	***d (nm)***	***E_g (eV)***
a-SiN_x:H	0.4	0.005	4.5	1227	1.77

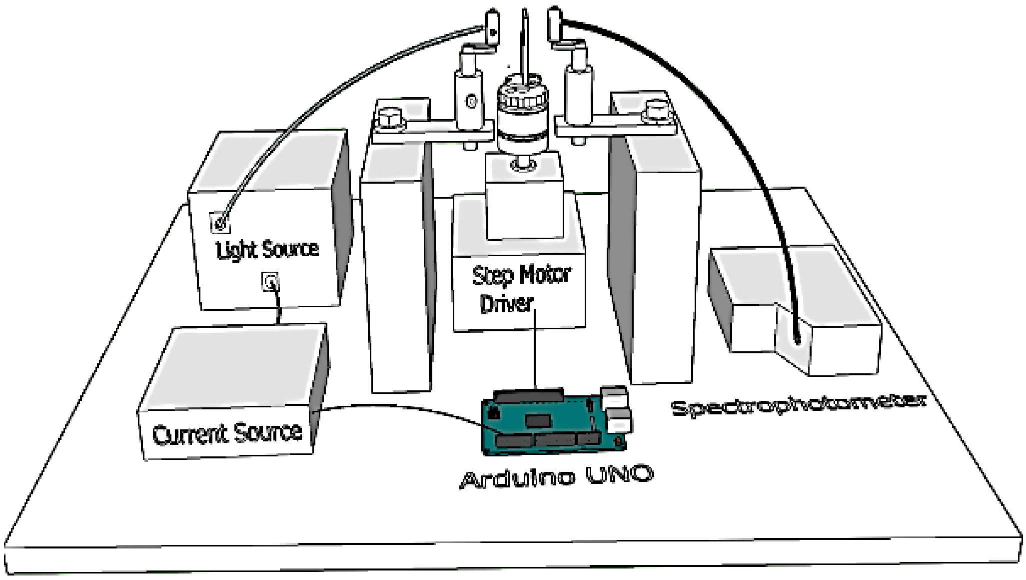

Fig. 1. An open spectrophotometric system and its components (color online)

2.2. The optical transmittance theory

When a monochromatic light beam comes perpendicular to the substrate surface where the thin film is homogeneously coated, multiple reflections occur at the system's interfaces. Three interfaces in the form of air/thin film/substrate/air are considered as the system (Fig. 2). Light passing through and reflecting through these interfaces creates interference fringes. These interference fringes were used to determine the optical constants and thickness of thin films. Optical transmittance (*T*) depends on the wavelength of the light (λ), the refractive index of the film (*n*), the extinction coefficient of the film (*k*), the substrate refractive index (s), and the film thickness (d) [1, 7].

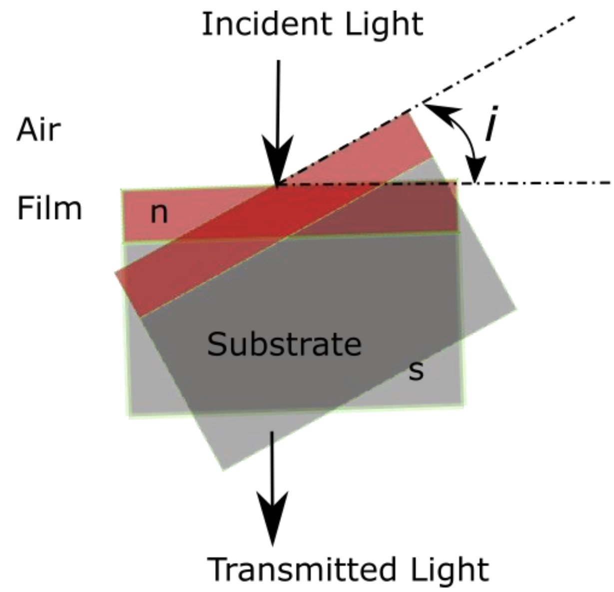

Fig. 2. The normal and oblique incidence of light on the thin film surface coated on a thick finite transparent substrate with thickness d (color online)

The refractive index, optical absorption coefficient, and film thickness of the film on a transparent substrate are mainly responsible for optical transmittance. Using the normal dispersion theory for an isotropic medium, the refractive index can be presented by three constants a, b, and x in the region of $k^2 << n^2$ by an equation of the form,

$$n^2 = a + \frac{b}{\lambda^x}. \quad (1)$$

Generally, the optical transmittance for normal incidence light is the most commonly used to determine optical constants. When the optical transmittance spectrum is an oscillating function of wavelength (λ) in the visible and near-infrared regions for the suitable product of refractive index n and thickness d, the maxima of the transmission spectrum T_M and the minima of the transmission spectrum T_m (influenced by interference effect) can be expressed with the following equations

$$2n_0\, d = m\lambda_0, \quad (2)$$

and

$$2n_0 d = (m + \tfrac{1}{2})\lambda_0, \quad (3)$$

where λ_0 and n_0 are the wavelength and refractive index of extrema transmittance for the normal incidence considering the order parameter m. In addition to these constants given Eq. (1), when the two more constants such as m and d come from Eq. (2) and Eq. (3) are taken into the account, optical transmittance can be expressed as $T(\lambda) = T(n, a, b, x, \mathrm{d}, \lambda)$. There are three methods for determining optical constants. The simple and direct method called the envelope method that uses transmittance extrema is the first method. If the substrate refractive index s is known, the optical transmittance spectrum is considered as a continuous function depending on the wavelength with two envelope curves $T_M(\lambda)$ and $T_m(\lambda)$ passing through the transmittance maxima and minima. For these envelope curves, the parabolic approach was used between the three consecutive extremes [17]. Thus, a pseudo-minimum (T_m') corresponding to the experimentally obtained transmission maximum (T_M) and a pseudo-maximum ($T_{M'}$) corresponding to an experimentally obtained transmission minimum (T_m) can be determined. With the measurement of accurate T_M and T_m values into the transparent region, the value of film refractive index n is given by

$$n = [N + (N^2 - s^2)^{\frac{1}{2}}]^{1/2}, \quad (4)$$

where

$$N = 2s\,\frac{(T_M - T_m)}{T_M T_m} + \frac{(s^2+1)}{2}. \quad (5)$$

Using Eq. (2), Eq. (3), Eq. (4) and Eq. (5), the film thickness (d) was calculated with the help of the Eq. (6)

$$d = \frac{\lambda_{01}\lambda_{02}}{2(\lambda_{01}n_{02} - \lambda_{02}n_{01})} \quad (6)$$

where, λ_{01}, n_{01} and λ_{02}, n_{02} are the wavelengths and refractive indices for two adjacent extrema transmittance for the normal incidence.

After that, using the refractive index, film thickness, and measured optical transmittance values, the optical extinction coefficient value calculated using the method proposed by Swanepoel [1].

The methods in the second group involve elaborate computer procedures to construct continuous $T(\lambda)$ complicated functions of many parameters [2, 5, 7, 12, 18]. In addition to these two methods, there is a third method in which the number of parameters used in calculations is reduced by increasing the number of experimentally measured quantities. This can be done by obtaining a second transmission spectrum for an incidence light of $i > 0°$. This method is based on the precise measurement of wavelength. In addition, the refractive index is only a function of wavelength, and the inhomogeneity of the film surface does not change the wavelength values of the optical interference extrema. This measurement provides auxiliary equations for determining refractive index and film thickness. In this case, considering the order parameter m is continuous, the equation for the interference extrema can be written as

$$2n_i d Cos(r) = m\lambda_i, \quad (7)$$

where r is the angle of refraction in the film. Hence, five constants can be determined from Eqs. (1) - (3), and Eq. (7) and used by the method given below. The shifting towards the shorter wavelength observed in the spectrum due to the oblique incidence of light related to the normal incidence of that as

$$2n_i d = (m + \Delta m)\lambda_i. \quad (8)$$

From Eqs. (7) and (8) and Snell's law it follows that

$$Cos(r) = \frac{m}{m+\Delta m}, \quad (9)$$

$$n_i = \frac{Sin(i)}{\mathrm{Sin(r)}}. \quad (10)$$

Considering two adjacent extrema of the normal incidence light of transmission spectrum at wavelength at λ_{01} and λ_{02} order parameter is given

$$m_1 = \frac{n_{01}\lambda_{02}}{2(n_{02}\lambda_{01} - n_{01}\lambda_{02})}. \quad (11)$$

Then, the quantity M is now defined as

$$M = \frac{\lambda_{02}}{2(\lambda_{01} - \lambda_{02})}. \quad (12)$$

Note that $M \geq m_1$. The equality $M = m_1$ is true only in the absence of valid dispersion. Such as the refractive index decreases with the increases in the wavelength of the incident light, and converges to a constant value at higher wavelengths. The optical band gap can be calculated according to the Wemple-DiDomenico single oscillator model [16] as follows

$$n^2 = 1 + \frac{E_d E_0}{E_0^2 - (hv)^2} \quad (13)$$

where E_o and E_d are the single-oscillator energy and dispersion energy parameters, respectively. E_0 is related to the optical gap E_g by the expression E_g=E_0/2.

M parameter can be expressed as a function of wavelength with the help of Eq. (2) and Eq. (3)

$$M = \frac{C}{\lambda_0}(1 + \frac{D}{\lambda_0^y}). \quad (14)$$

The first term in Eq. (14) reflects the wavelength dependence of m according to the Eq. (2). The second term in the parentheses indicates the dispersion given by Eq. (1). The value of m at each extremum is now approximately given by $m \approx C/\lambda_0$ are shown as m_y in Table 3. and Table 4. These values are, of course, only approximate but serve to assign the actual values of m to each extremum as shown in Table 3 and Table 4. Rewritten Eq. (14) is the first cost function for the GA:

$$M\lambda_0 = \frac{D}{\lambda_0^y} + C. \quad (15)$$

Substitution of Eq. (1) into Eq. (2) and Eq. (3) yields second cost function for GA as

$$m^2\lambda_0^2 = \frac{A}{\lambda_0^x} + B, \quad (16)$$

where A=4ad^2 and B=4bd^2. When the order parameters are known, the values of x, A, and B can be determined by GA. Substituting Eq. (1) into Eq. (8), using Eq. (16), and solving for Δm yield,

$$\Delta m = \left[m^2\frac{\lambda_0^2}{\lambda_i^2} + \frac{A}{\lambda_i^2}(\frac{1}{\lambda_i^x} - \frac{1}{\lambda_0^x})\right]^{1/2} - m. \quad (17)$$

The value of n_i at each extrema value of λ_i obtained from oblique incidence light can now be calculated using Eqs. (17), (9), and (10).

2.3. Genetic algorithm (GA)

GA use genes and array of genes called individuals or chromosomes. Genes, which are binary encoding of parameters, are the basic building blocks of GA. To apply the genetic algorithm to parametric equations, genes are first defined to correspond to unknown parameters. To generate the individuals randomly the genes are encoded by G bits. Depending on the desired resolution for the cost function, G can be taken as 10 bits, 12 bits, 14 bits or more can be used. For example, individual for the dummy parameter a can be expressed as 0010101010 for 10 bits representation. The real value of the uth individual of a is calculated by

$$a_u = a_{min} + \frac{a_{max} - a_{min}}{2^G - 1}\sum_{v=0}^{G-1} 2^v a'_{uv}. \quad (18)$$

Here a_{min} and a_{max}, are minimum and maximum limits of real valued parameter a, respectively. a'_{uv} is v^{th} binary number of G bits gene of u^{th} individual. Considering dummy function $W(a,b)$ with two parameters such as a and b, the cost function F can be defined as

$$F(a,b) = \left|W(a,b) - W(a,b)_{exp}\right| \quad (19)$$

where $W(a, b)_{exp}$ is an experimentally measured quantity. Then, GA proceeds by iteratively generating new individuals, which are derived from the previous individuals through the application of selection, crossover, and mutation operations. Considering the obtained cost function values, the individuals are ranked from smallest to largest. In our calculations, the number of individuals is chosen as 100. 6% of the individuals are mutated with randomly selected "1" bit in each gene of individuals per iteration to prevent the increase of the number of similar individuals in the population, and the crossover points are also randomly selected. The iterations are repeated until the predefined error for the cost function is obtained.

3. Results and discussions

In order to determine the optical constants of thin films, it is necessary to test the method and intermediate processes by repeating a reference optical transmittance spectrum and evaluation steps. Subsequently, the accuracy of the results obtained with the proposed new models or approaches can be compared. For this reason, firstly, optical transmittance spectra obtained for a-Si:H thin film under normal incidence and 30° oblique incidence were evaluated with the help of the GA which is the subject of this study. The proposed GA source code is given in Ref. 5 was used by adapting new objective functions such as $y(x) = \frac{b}{\lambda^t} + a$ ($y(x) = M\lambda_0$ and $y(x) = m^2\lambda_0^2$) and employed to find the optical parameters of reference a-Si:H thin film sample using transmission spectrum for normal incidence and oblique incidence with an angle of 30°. First, a, b and t correspond to the D, C, and y parameters of the first objective function given in Eq. (14) and then to the parameters A, B, and x of the second objective function given in Eq. (16) listed in Table 2. A good fit was obtained for the reference sample using GA results. The reference film thickness (1039 nm) was calculated as 1038 nm. To compare the value of n_i and Δm at each extrema value of λ_i obtained with GA and reference study are given in Table 3. In addition, it was observed that the variation of the refractive indices with the wavelength calculated by GA for oblique incidence light was in great agreement with the reference values (Fig. 3).

After that, the optical transmittance spectrum obtained from normal incidence for the a-SiN$_x$:H thin film was evaluated by the classical method. The parameters belonging to both objective functions were given in Table 4. Subsequently the film thickness was determined as 1212.28 nm for the a-SiN$_x$:H thin film. Considering the normal incidence and incidence angle of 54° wavelength shift was obtained precisely using the designed open spectrophotometric system (Fig. 4).

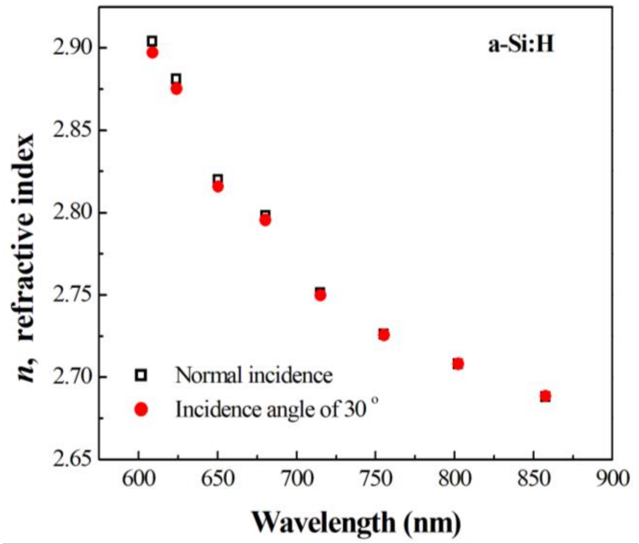

Fig. 3. Refractive index of reference a-Si:H sample [13] for normal incidence (black square, □) and incidence angle of 30 ° (filled red circle •) (color online)

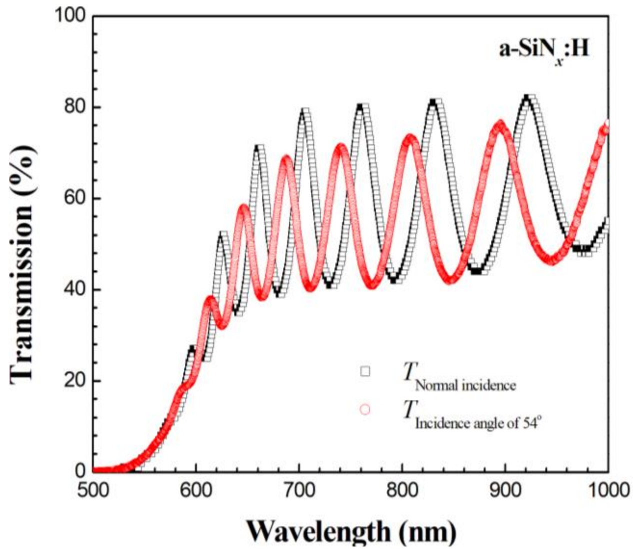

Fig. 4. Transmission spectra of a-SiN$_x$:H sample for normal incidence (□) and incidence angle of 54 ° (o) (color online)

These wavelength shifts and the parameters are evaluated by GA yielding the film thickness to be 1212.80 nm. A good fit was obtained for the first objective function ($R^2 > 0.965$) and the second objective function ($R^2 > 0.9962$). When the values of the refractive indices corresponding to the normal incidence and oblique incidence light on the sample surface were taken into account, the number of experimental data points used to determine the optical band gap with the help of the refractive index doubled (Fig. 5). After plotting $(n^2 - 1)^{-1}$ vs. $(h\nu)^2$, a linear fit was performed. A good straight line ($R^2 = 0.998$) is obtained as shown in Fig. 6. Using the single-oscillator model and Eq. (13), the optical band gap a-SiN_x:H film was calculated as 1.77 eV. A good agreement between this optical band gap and the reference value obtained from the optical transmittance spectrum for normal incidence light is supported by the Constant-Photocurrent Method [16].

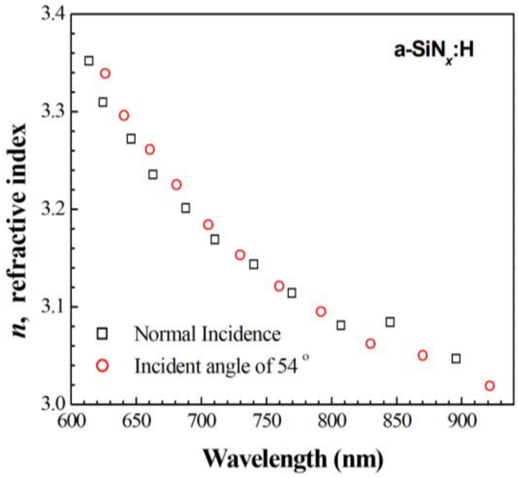

Fig. 5. Refractive index of a-SiN_x:H sample for normal incidence (□) and incidence angle of 54 °(o) (color online)

Table 2. Fitting parameters obtained from GA for the reference a-Si:H sample and a-SiN_x:H sample. Symbols defined in text

Sample	$D\times10^{16}$	C	y	d (nm)	$A\times10^{7}$	$B\times10^{18}$	x
*a-Si:H	0.9726	5709	4.5	1039	2.962	3.027	4.2
**a-Si:H	0.9585	5684	4.5	1038	2.959	4.890	4.28
**a-SiN_x:H	1.3116	7404	4.44	1212	5001	2.518	4.01

* Parameters taken from Ref [13]

** Parameters obtained from GA for reference sample

Table 3. The λ_0 and λ_i, are wavelength values in nm in case of normal and oblique incidence for the a-Si:H thin film. The parameter M, m_y and m obtained from reference [13]. Comparison of refractive indices (n) and Δm values obtained by reference and genetic algorithm for.a-Si:H thin film

λ_0	λ_i	M	m_y	m	Δm_{ref}	Δm_{GA}	$n_{i\text{-}ref}$	$n_{i\text{-}GA}$
857.6	844.0	7.25	6.627	6.5	0.1154	0.1153	2.688	2.685
802.3	790.1	8.05	7.083	7.0	0.1224	0.1223	2.708	2.705
755.4	744.4	8.90	7.523	7.5	0.1294	0.1294	2.726	2.729
715.2	705.3	9.78	7.946	8.0	0.1355	0.1355	2.751	2.756
680.4	671.6	10.80	8.352	8.5	0.1391	0.1392	2.798	2.785
650.3	642.3	11.82	8.739	9.0	0.1449	0.1452	2.820	2.819
623.9	616.8	13.00	9.109	9.5	0.1464	0.1469	2.881	2.855
600.8	594.3		9.459	10.0	0.1516	0.1522	2.904	2.989

Table 4. The λ_0 and λ_i, are wavelength values in nm in case of normal and oblique incidence. T_M, and T_m are the transmittance maxima and minima obtained from Fig. 4 for the a-SiN_x:H thin film. The parameter M, m_y and m defined in text. Calculated refractive index for oblique incidence is shown as n_i

λ_0	λ_i	M	m_y	m	Δm	n_i	T_M	T_m
1030.9	1003.3	8.480	7.182	7.0	0.209	2.982	-	-
973.5	946.3	8.861	7.606	7.5	0.238	3.019	81.9	44.6
921.5	895.5	8.429	8.035	8.0	0.262	3.050	81.9	44.6
869.9	845.1	10.374	8.511	8.5	0.290	3.062	81.0	44.6
829.9	807.1	10.420	8.922	9.0	0.303	3.095	80.5	43.0
791.9	769.5	11.797	9.350	9.5	0.339	3.121	80.4	42.1
759.7	740.1	12.162	9.746	10.0	0.334	3.153	80.4	41.6
729.7	710.3	14.453	10.147	10.5	0.374	3.184	79.8	41.0
705.3	687.9	13.953	10.498	11.0	0.372	3.225	78.6	40.3

λ_0	λ_i	M	m_y	m	Δm	n_i	T_M	T_m
680.9	662.9	16.027	10.874	11.5	0.431	3.261	75.8	39.4
660.3	645.9	16.174	11.213	12.0	0.379	3.296	71.0	37.8
640.5	624.3	21.740	11.560	12.5	0.475	3.339	62.3	35.3

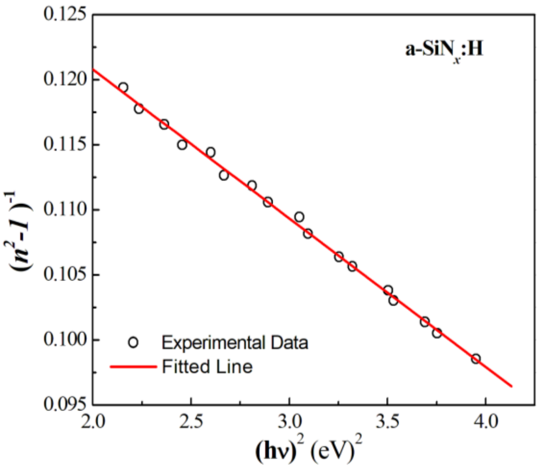

Fig. 6. Plot of $(n^2-1)^{-1}$ vs $(h\nu)^2$ of a-SiN$_x$:H thin film (color online)

4. Conclusions

In this study, an open spectrophotometric system was designed. The system consists of a mechanical system to minimize uncertainty in the angle of incidence, and a current source to provide constant illumination on the sample. Hence, the optical transmission spectrum was measured at normal and at oblique incidence light in a few seconds with an open system spectrophotometer. Then the correct film thickness and refractive index values were determined in a very short time with an input file containing these wavelength values and a template file using parameters calculated with the help of genetic algorithm. The optical transmittance spectrum obtained from normal incidence light was evaluated by the classical method and the film thickness was determined as 1212.28 nm for the a-SiN$_x$:H thin film. Considering the wavelength shift obtained in the case of oblique incidence light of the same sample was evaluated by a genetic algorithm and the film thickness was calculated as 1212.80 nm. The new method uses only the values of lambda to compute the accurate values for film thickness and refractive index. Also, obtaining a second transmittance measurement at oblique incidence light increased the number of refractive index values taken into account in the optical band gap calculations in the single-oscillator model. It was observed that there was an increase in the optical band gap of the nitrogen-doped sample compared to the a-Si:H sample. As a result, optical constants of thin films; with the designed open spectrophotometric transmittance measurement system based on the wavelength measurement at the oblique incidence light and the appropriate GA approach, were determined faster and more precisely. Measurement of the absolute optical transmission is no longer required.

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