

PRECISE MEASUREMENT OF THICKNESS DISTRIBUTION OF NON-UNIFORM THIN FILMS BY IMAGING SPECTROSCOPIC REFLECTOMETRY

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Abstract – A new method of imaging spectroscopic photometry enabling us to perform the complete optical characterization of thin films exhibiting area non-uniformity in optical parameters is presented. An original imaging spectroscopic photometer operating in the reflection mode at normal incidence is used to apply this method. A CCD camera serves as a detector in this photometer. Therefore the spectral dependences of the reflectance of the films characterized are simultaneously measured in small areas of the films surface corresponding to the individual pixels of the CCD camera. These areas form a matrix along a relatively large part of the films surface. The spectral reflectance measured by the individual pixels of the CCD camera is treated separately using the formulae for the reflectance corresponding to the uniform thin films. Using these formulae it is possible to determine the values of the local thickness and local optical constants for every small area of the matrix. In this way it is possible to determine distributions (maps) of the local thickness and local optical constants of the non-uniform films simultaneously in principle. The method described was used to characterize carbon-nitride thin films exhibiting only the thickness non-uniformity deposited by the method of dielectric barrier discharge onto the silicon single crystal substrates.

Keywords: non-uniform thin films, imaging spectroscopic reflectometry

1. INTRODUCTION

In practice you can often encounter thin films exhibiting non-uniformity along their area. For example, thin films prepared using various plasma-chemical methods often exhibit area non-uniformity in optical parameters, *i.e.* in optical constants or thickness. There are several reasons causing an origin of the area non-uniformity of the thin films created by these plasma-chemical methods. It is very difficult to perform a correct optical characterization of these films by means of the standard optical methods employed mostly in practice (e.g. photometric and ellipsometric methods). In many cases of the non-uniform thin films the standard photometric and ellipsometric method are not even usable. Therefore it is necessary to develop new optical methods enabling us to perform the

complete optical characterization of the films non-uniform in thickness and optical constants with a sufficient precision. Imaging spectroscopic reflectometry (ISR) represents such a new approach. Within this technique we developed a new method allowing us to perform the complete optical characterization of non-uniform absorbing thin films. Using this ISR method it is possible to determine the area thickness distribution along these non-uniform films together with the area distribution of the spectral dependences of their optical constants if a suitable dispersion model of these optical constants is selected. In principle, an important advantage of ISR consists in a possibility of using the formulae corresponding to uniform thin films in treatment of experimental data. This ISR method is applied using an imaging spectroscopic photometer containing a CCD camera as a detector. We developed an original imaging spectroscopic photometer (ISP) that can be employed in both reflection and transmission modes [1]. This ISP is a two channel instrument operating in *ex-situ* mode in contrast to a one channel ISP operating *in-situ* mode utilized for technological purposes [2]. In this paper ISR is applied to non-uniform carbon-nitride films.

2. EXPERIMENTAL

2.1. Sample preparation

The non-uniform carbon-nitride films were deposited by dielectric barrier discharge with CH₄/N₂ gas mixture onto silicon single crystal wafers at the pressure in the region from 300 mbar to 400 mbar. Details of preparing the carbon-nitride films are described in paper [3]. Note that the carbon-nitride films prepared in this way exhibited amorphous structure.

2.2. Experimental set up

A schematic diagram of the ISP employed is presented in Fig. 1.

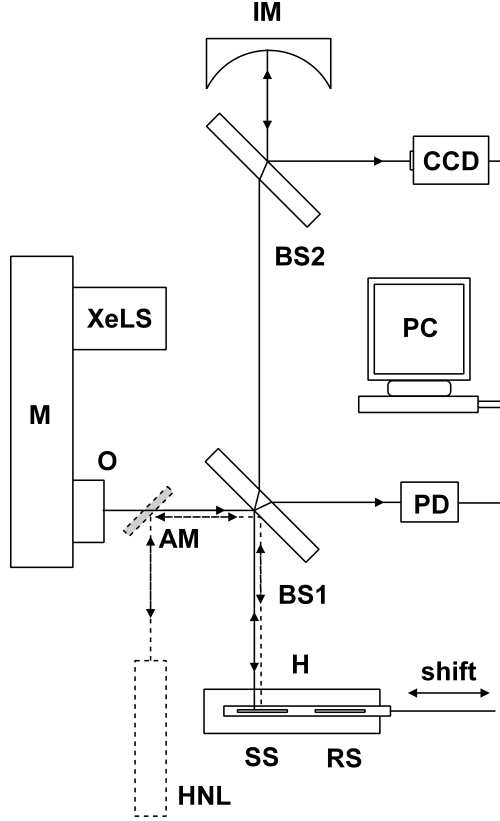


Fig. 1 Schematic diagram of the ISR experimental setup: M - monochromator, XeLS - Xenon light source, O - output of the monochromator, BS1, BS2 - beam-splitters, IM - imaging mirror, CCD - CCD camera, PC - computer, H - holder of samples, SS - studied sample, RS - reference sample, PD - photodetector, HNL - helium-neon laser, AM - auxiliary mirror.

The detector in the sample channel of the ISP is a CCD camera and so the spectral reflectance of small areas on the non-uniform thin film corresponding to all the individual pixels of this camera is measured. Thus, the spectral reflectance is measured in the small areas forming a matrix on this film. These areas are sufficiently small and therefore the reflectance measured for them is given by the known formulae for uniform thin films. A sample holder is constructed in such a way that the normal reflectance of the films is measured in the individual pixels of the CCD camera within the region of 320 – 810 nm. The concrete procedure for measuring the reflectance $R^{k,u}$ corresponding to the individual elements of the matrix, *i.e.* corresponding to the individual pixels, is as follows (for details see our earlier paper [1]):

1) The reference sample RS is situated into the position of the measuring channel. The signal $S_{MR}^{k,u}(t_1, \lambda)$ recorded by the $(k, u)^{\text{th}}$ pixel of the CCD camera at time t_1 and wavelength λ is given in the following way:

$$S_{MR}^{k,u}(t_1, \lambda) = I_0(t_1, \lambda) \eta_M^{k,u}(\lambda) R_0^{k,u}(\lambda), \quad (1)$$

where indices (k, u) belong to the $(k, u)^{\text{th}}$ pixel ($k = 1, \dots, A, u = 1, \dots, B$), where A, B are integers, $I_0(t_1, \lambda)$ is the intensity of the output beam of the monochromator at

time t_1 and wavelength λ , $\eta_M^{k,u}(\lambda)$ is so called apparatus function for the measuring channel of the experimental setup corresponding to the $(k, u)^{\text{th}}$ pixel (it includes the influence of the quartz wedge-shaped beam-splitters BS1, BS2, the spherical imaging mirror IM and parameters of the CCD camera), $R_0^{k,u}(\lambda)$ is the local absolute reflectance of the reference sample corresponding to $(k, u)^{\text{th}}$ pixel. In practice one can select the reference samples such as a silicon single-crystal wafer for which it holds that $R_0^{k,u}(\lambda) = R_0(\lambda)$. Signals $S_{MR}^{k,u}(t_1, \lambda)$ create the image of the reference sample for wavelength λ at time t_1 .

At the same time t_1 and the same wavelengths λ , the signal $S_{RR}(t_1, \lambda)$ is recorded by the integrating photodetector PD in the reference channel.

$$S_{RR}(t_1, \lambda) = I_0(t_1, \lambda) \eta_R(\lambda), \quad (2)$$

where $\eta_R(\lambda)$ is the apparatus function for the reference channel of the experimental setup. It includes the influence of the beam-splitter BS1 and parameters of the photodetector PD. The signals $S_{MR}^{k,u}(t_1, \lambda)$ and $S_{RR}(t_1, \lambda)$ occurring in (1) and (2) are measured at sufficient number of the wavelengths from the spectral region of interest.

2) The sample measured SS is situated into the position of the measuring channel. Then the steps of the measurements described above are repeated with this sample. Of course, these measuring steps must be performed in the same wavelengths as for the reference sample. In this way we obtain the following equations for the signals detected by the individual pixels of the CCD camera $S_{MS}^{k,u}(t_2, \lambda)$ and integrating photodetector $S_{RS}(t_2, \lambda)$ at time t_2 :

$$S_{MS}^{k,u}(t_2, \lambda) = I_0(t_2, \lambda) \eta_M^{k,u}(\lambda) R^{k,u}(\lambda) \quad (3)$$

$$S_{RS}(t_2, \lambda) = I_0(t_2, \lambda) \eta_R(\lambda) \quad (4)$$

where $R^{k,u}(\lambda)$ is the local absolute reflectance of the studied sample corresponding to the $(k, u)^{\text{th}}$ pixel. Signals $S_{MS}^{k,u}(t_2, \lambda)$ create the image of the studied sample for wavelength λ at time t_2 .

3) The values of ratio $P_R^{k,u}(t_1, \lambda)$ corresponding to situating the reference sample into measuring channel at time t_1 are determined in the following way:

$$P_R^{k,u}(t_1, \lambda) = \frac{S_{MR}^{k,u}(t_1, \lambda)}{S_{RR}(t_1, \lambda)} = \frac{\eta_M^{k,u}(\lambda)}{\eta_R(\lambda)} R_0^{k,u}(\lambda). \quad (5)$$

Similarly we obtain an analogous ratio $P_S^{k,u}(t_2, \lambda)$ of the signals for the situation when the studied sample is placed into the measuring channel at time t_2 :

$$P_S^{k,u}(t_2, \lambda) = \frac{S_{MS}^{k,u}(t_2, \lambda)}{S_{RS}(t_2, \lambda)} = P_R^{k,u}(t_1, \lambda) \frac{R^{k,u}(\lambda)}{R_0^{k,u}(\lambda)}. \quad (6)$$

In conclusion we find:

$$R^{k,u}(\lambda) = \frac{P_s^{k,u}(t_2, \lambda)}{P_R^{k,u}(t_1, \lambda)} R_0(\lambda). \quad (7)$$

Using the arrangement described, it is possible to determine the spectral dependence of the local relative reflectance $\frac{R^{k,u}(\lambda)}{R_0(\lambda)}$ of the studied sample corresponding to each pixel of the CCD camera owing to the reference sample since the ratios $P_s^{k,u}(t_2, \lambda)$ and $P_R^{k,u}(t_1, \lambda)$ are determined by means of this arrangement quantitatively. Because the absolute reflectance $R_0(\lambda)$ of the reference sample is known, it is possible to determine the local absolute reflectance of the sample measured corresponding to all the pixels too.

3. DATA PROCESSING

The reflectance $R^{k,u}$ is given as follows:

$$R^{k,u} = |\hat{r}^{k,u}|^2 \quad (8)$$

where

$$\hat{r}^{k,u} = \frac{\hat{r}_1^{k,u} + \hat{r}_2^{k,u} \exp(i\hat{x}^{k,u})}{1 + \hat{r}_1^{k,u} \hat{r}_2^{k,u} \exp(i\hat{x}^{k,u})}, \quad (9),$$

$$\hat{r}_1^{k,u} = \frac{n_0 - \hat{n}_1^{k,u}}{n_0 + \hat{n}_1^{k,u}} \quad (10)$$

$$\hat{r}_2^{k,u} = \frac{\hat{n}_1^{k,u} - \hat{n}}{\hat{n}_1^{k,u} + \hat{n}}, \quad (11)$$

$$\hat{x}^{k,u} = \frac{4\pi}{\lambda} \hat{n}_1^{k,u} d_1^{k,u}. \quad (12)$$

In the foregoing equations (8) – (12), symbols n_0 , \hat{n} , $\hat{n}_1^{k,u}$, $d_1^{k,u}$ and λ denote the refractive index of the ambient, complex refractive index of the substrate, local complex refractive index of the film, local thickness of the film and wavelength of incident light, respectively. Note that the ambient environment consists of air and therefore $n_0 = 1$. The complex refractive index of the substrate and/or film is expressed as $\hat{n} = n_s - ik_s$ and/or $\hat{n}_1 = n_1 - ik_1$. Symbols n_s and k_s are the real refractive index and extinction coefficient of the substrate, respectively, and symbols n_1 and k_1 denote the refractive index and extinction coefficient of the film, respectively. The foregoing mathematical expressions for the local reflectance $R^{k,u}$ belonging to the individual pixels of the CCD camera correspond to the reflectance of the absorbing thin film deposited onto an absorbing substrate. Strictly speaking, the optical model of the isotropic homogeneous absorbing substrate covered with isotropic homogeneous absorbing thin film was thus employed for expressing the local

reflectance corresponding to the individual pixels of the CCD chip.

The least-squares method (LSM) was used to treat the experimental data using (8). Of course, the LSM had to be applied for each local area on the film corresponding to the given pixels of the CDD camera. Within the LSM, the following merit function $S^{k,u}$ was employed:

$$S^{k,u} = \sum_{s=1}^K \left(\frac{R_s^{k,u} - R_s'^{k,u}}{\sigma_s^{k,u}} \right)^2, \quad (13)$$

where $R_s^{k,u}$ and/or $R_s'^{k,u}$ denotes the theoretical and/or experimental value of the local reflectance corresponding to the $(k, u)^{\text{th}}$ pixel and wavelength λ_s . Symbol $\sigma_s^{k,u}$ and/or K represents the standard deviation of $R_s^{k,u}$ and/or the number of the measurements of the reflectance, *i.e.* the number of wavelengths, for which the reflectance was measured in the individual pixels.

The dispersion model of the optical constants of the carbon-nitride thin films based on parameterization of the joint density of states (JDOS) was employed within the processing of the experimental data. The JDOS is defined, for example, in [4-6]. The detailed description of the parameterized joint density of states (PJJDOS) model is presented in paper [7]. Within the PJJDOS model the imaginary part of dielectric function $\varepsilon_i(E)$ is expressed using the following formula:

$$\varepsilon_i(E) = \frac{J(E)}{E^2} = \frac{1}{E^2} \sum_{j=1}^N J_{j \rightarrow j^*}(E), \quad (14)$$

where E , N and $J_{j \rightarrow j^*}(E)$ denote photon energy, number of transitions and non-normalized JDOS corresponding to $j \rightarrow j^*$ transition, respectively. Quantity $J_{j \rightarrow j^*}(E)$ is defined as follows [4-7]:

$$J_{j \rightarrow j^*}(E) = \left(\frac{eh}{m} \right)^2 \frac{|p_{j \rightarrow j^*}|^2}{4\pi\epsilon_0 B_0} \Im_{j \rightarrow j^*}(E), \quad (15)$$

where e , h , m , ϵ_0 and B_0 represent electron charge, Planck's constant, electron mass, dielectric function of vacuum and certain part of Brillouin zone of corresponding crystalline material, respectively. Symbol $|p_{j \rightarrow j^*}|^2$ denotes the squared momentum-matrix element. Symbol $\Im_{j \rightarrow j^*}(E)$ represents the JDOS function belonging to $j \rightarrow j^*$ transition. The explicit expression of $\Im_{j \rightarrow j^*}(E)$ is given in [7]. Symbol $J(E)$ in (14) expresses a sum of the above introduced $J_{j \rightarrow j^*}(E)$ corresponding to all the transitions in material forming the thin film.

The following parameterization of $J_{j \rightarrow j^*}(E)$ was employed:

$$J_{j \rightarrow j^*}(E) \propto (E - E_{gj})^2 (E - E_{hj})^2, \quad (16)$$

where E_{gj} and E_{hj} denote the minimum energy limit (band gap) and maximum energy limit of the transition $j \rightarrow j^*$, respectively.

Note that the following equation is fulfilled [7]:

$$\int_0^\infty J_{j \rightarrow j^*}(E) dE = Q_j^2 \quad (17)$$

where Q_j is the quantity proportional to the concentration of j electrons in material.

From the foregoing the following equation was derived:

$$\varepsilon_{i,j \rightarrow j^*}(E) = \frac{30Q_j^2 (E - E_{gj})^2 (E - E_{hj})^2}{(E_{hj} - E_{gj})^5 E^2} \text{ for } E_{gj} < E < E_{hj} \quad (18)$$

and

$$\varepsilon_{i,j \rightarrow j^*}(E) = 0 \text{ for } E \text{ out of } E_{gj} < E < E_{hj}. \quad (19)$$

Using the Kramers-Kronig relation [7], contribution of $j \rightarrow j^*$ transition to the real part of dielectric function $\varepsilon_{r,j \rightarrow j^*}(E)$ could be analytically expressed as

$$\varepsilon_{r,j \rightarrow j^*}(E) = \kappa \left[B \ln \left| \frac{E + E_{hj}}{E + E_{gj}} \right| + C \ln \left| \frac{E - E_{hj}}{E - E_{gj}} \right| - D \right], \quad (20)$$

where

$$\kappa = \frac{60Q_j^2}{\pi(E_{hj} - E_{gj})^5}, \quad B = \frac{Y(E) + X(E)}{2E^2}, \quad (21)$$

$$C = \frac{Y(E) - X(E)}{2E^2} \quad (22)$$

$$D = \frac{E_{gj}^2 E_{hj}^2}{E^2} \ln \left| \frac{E_{hj}}{E_{gj}} \right| + \frac{3(E_{hj}^2 - E_{gj}^2)}{2} \quad (23)$$

$$X(E) = 2E[E_{hj}(E_{gj}^2 + E^2) + E_{gj}(E_{hj}^2 + E^2)] \quad (24)$$

and

$$Y(E) = E^2(E_{hj}^2 + E_{gj}^2 + 4E_{gj}E_{hj} + E^2) + E_{hj}^2 E_{gj}^2. \quad (25)$$

Then the complete complex dielectric function of the material of the carbon-nitride thin films is given as follows [7]:

$$\hat{\varepsilon}(E) = 1 + \sum_{j=1}^N \hat{\varepsilon}_{j \rightarrow j^*}(E), \quad (26)$$

where

$$\hat{\varepsilon}(E) = \varepsilon_r(E) - i\varepsilon_i(E) \quad (27)$$

and

$$\hat{\varepsilon}_{j \rightarrow j^*}(E) = \varepsilon_{r,j \rightarrow j^*}(E) - i\varepsilon_{i,j \rightarrow j^*}(E). \quad (28)$$

Refractive index $n_1(E)$ and extinction coefficient $k_1(E)$ are connected with the real part $\varepsilon_r(E)$ and imaginary part $\varepsilon_i(E)$ of the dielectric function by means of the following equations:

$$\varepsilon_r(E) = n_1^2(E) - k_1^2(E) \quad (29)$$

and

$$\varepsilon_i(E) = 2n_1(E)k_1(E). \quad (30)$$

By using (29) and (30) it is possible to calculate the spectral dependences of refractive index $n_1(\lambda)$ and extinction coefficient $k_1(\lambda)$ of the thin films under study because $\lambda = \frac{hc}{E}$, where c is the light velocity in vacuum.

From the foregoing it is obvious that using the LSM the values of the local thickness $d^{k,u}$ and material parameters, *i.e.* parameters E_{gj} , E_{hj} and Q_j occurring in the dispersion model used, must be determined for each local area. Using these material parameters one can calculate the true spectral dependences of the refractive index and extinction coefficient of the carbon-nitride films within the local areas corresponding to the pixels of the CCD camera. This means that distributions (maps) of the thickness and optical constants are determined in this way. The spectral dependences of the optical constants of the silicon single crystal substrates were taken from the literature [8] and fixed within the LSM employed.

4. RESULTS AND DISCUSSION

It was found out that the best fit of the experimental data belonging to individual pixels was achieved for the dispersion model corresponding to two transitions of electrons, *i. e.* for $N=2$ (see (14) and (26)). The results presented in this paper represent the typical results of the optical characterization of the non-uniform carbon-nitride films studied using ISR. These results were achieved for the film prepared using ratio $\text{CH}_4 : \text{N}_2 = 1 : 10$. In Fig. 2 the image of this carbon-nitride film obtained by the CCD camera is presented for the wavelength of 440 nm.

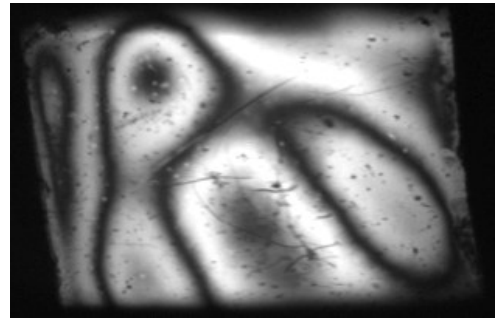


Fig.2: Image of the film investigated for the wavelength of 440 nm.

In Fig. 3 the local thickness distribution of the carbon-nitride film selected is presented.

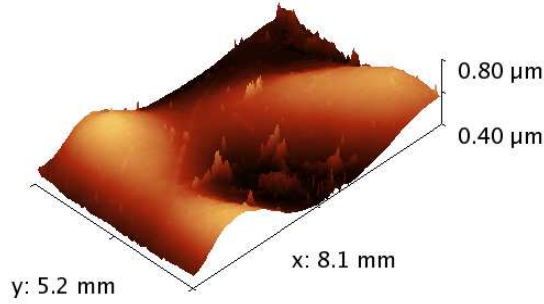


Fig.3: The 3D distribution of the local thickness of the carbon-nitride film selected

This distribution corresponds to the CCD camera image presented in Fig. 2. This figure shows that the film exhibited relatively strong thickness non-uniformity. For the film selected it was found out that the values of the material parameters of the dispersion model were practically identical for all the local areas belonging to individual pixels. Thus the non-uniform carbon-nitride film selected exhibited the area uniformity in the material parameters. The values of these material parameters were found as follows:

$$E_{g1} = (2.42 \pm 0.03) \text{ eV}, E_{g2} = (11.34 \pm 0.08) \text{ eV}, E_{h1} = (44.81 \pm 0.06) \text{ eV}, E_{h2} = (12.1 \pm 0.6) \text{ eV}, Q_1 = (32.9 \pm 0.7) \text{ eV}^{3/2}, Q_2 = (66.6 \pm 0.9) \text{ eV}^{3/2}.$$

This means that this film was also uniform in the optical constants along the area of the film. The spectral dependences of the refractive index $n_1(\lambda)$ and extinction coefficient $k_1(\lambda)$ of the film are plotted in Fig. 4. From this figure it is clear that the carbon-nitride film is absorbing film for shorter wavelength of 500 nm. For longer wavelengths this film is non-absorbing. Note that the same spectral dependences of the optical constants were found out for the other carbon-nitride films. These remaining films also exhibited the strong thickness non-uniformity.

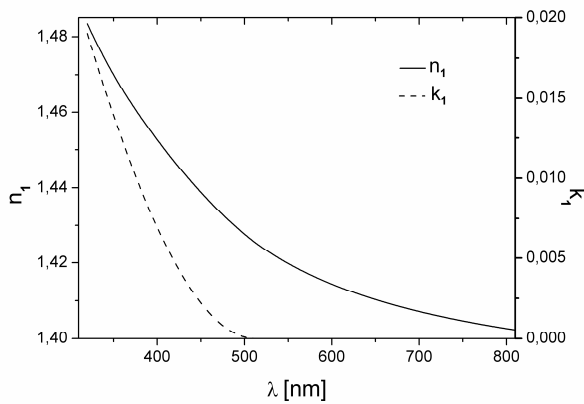


Fig.4: Spectral dependences of the refractive index n_1 and extinction coefficient k_1 of the non-uniform carbon-nitride film selected

Correctness of the results obtained within the optical characterization performed is supported by good agreement between the experimental and theoretical data. This fact is illustrated for the pixel selected in Fig. 5. Note that the theoretical data were calculated using (8) on the basis of the values of the parameters found using the LSM.

The reflectance values $R^{k,u}$ were measured with the relative accuracy about 1 %. Using a standard error analysis, we found out that the values of the local thicknesses in the area distributions were determined with a relative accuracy of 1-2 %. The same conclusion concerning the accuracy was found out for optical constants. Note that the precision presented above corresponds to the statistical precision.

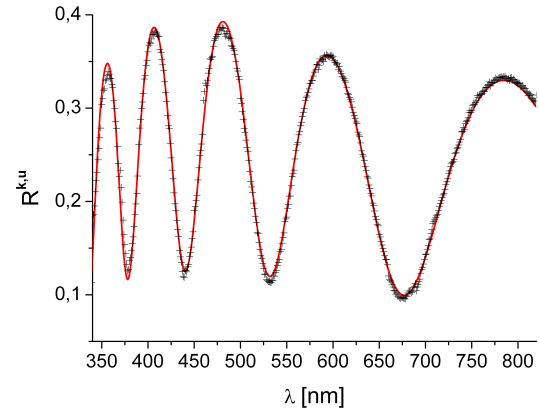


Fig. 5 Spectral reflectance of the carbon-nitride under study corresponding to a selected pixel: points denote the experimental values and the curve represents the theoretical data.

In conclusion of this section it should be pointed out that the transition characterized by material parameters $E_{g2} = 11.34 \text{ eV}$, $E_{h2} = 12.1 \text{ eV}$ and $Q_2 = 66.6 \text{ eV}^{3/2}$ obviously does not represent a real transition. This statement is mainly illustrated by the great value of E_{g2} . We therefore mean that the second transition ($j=2$) compensates some systematic error occurring in the experimental data. We mean that the influence of a certain non-linearity of the CCD camera or the influence of a light intensity background can cause this systematic error in the experimental data. An analysis of the systematic errors concerning the experimental data obtained for the individual pixels of the CCD camera will be presented elsewhere. However, the values of the material parameters of the second transition indicate the systematic error considered cannot be important, which is implied by the fact that the contribution of $\hat{\epsilon}_{2 \rightarrow 2^*}(E)$ to $\hat{\epsilon}(E)$ is relatively small.

Of course, the first transition ($j=1$) represents the real transition.

5 CONCLUSION

In this paper a new method of the optical characterization of the non-uniform absorbing thin films

based on imaging spectroscopic reflectometry (ISR) was presented. The original imaging spectroscopic photometer (ISP) operating in reflection mode was employed for obtaining the experimental data. The spectral dependences of the normal reflectance corresponding to the individual pixels of the CCD camera serving as the detector of the ISP were treated using the LSM. Within the LSM, a formula for the normal reflectance of the uniform absorbing thin film placed onto the absorbing substrate was used. When treating the experimental data, the dispersion model based on parameterization of the joint density of electronic states (JDOS) corresponding to amorphous materials was used. It was shown that in principle it is possible to determine both the thickness and optical constants distributions simultaneously in an independent way. This fact represents an important advantage of this method. The method was applied to the optical characterization of the non-uniform carbon-nitride films deposited by the dielectric barrier discharge method onto the silicon single crystal substrates. Using this method the local thickness distributions were determined for these films. Furthermore, it was shown that the carbon-nitride films were uniform in optical constants. The spectral dependences of the optical constants and area distributions of the local thickness for the carbon-nitride film selected were presented. It was also presented that the local thickness values and spectral dependences of the optical constants of the carbon-nitride films were determined with a relatively high accuracy. From this paper it is clear that the method of the ISR described here is also usable for the optical characterization of the other non-uniform absorbing thin films.

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