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# THE DETERMINATION OF THICKNESS AND OPTICAL CONSTANTS FOR PbSe FILM FROM IR REFLECTANCE SPECTRA

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ABSTRACT: The transmittance values measured in IR reflection-absorption (RA) spectra were used to determine the optical constants of dielectric films laid on solid substrates. When the recorded spectra show interference fringes, one can determine the film thickness. The PbSe film thickness was obtained by interpreting the interference fringes from the reflexion-absorption IR spectra recorded at two different incidence angles. In order to obtain the optical constants of PbSe films laid on steel we used dispersion analysis. The dispersion analysis offers the advantage of processing a large volume of data.
KEYWORDS: reflection-absorption, optical constants, IR spectra, dispersion analysis, interference fringes

## ❖ INTRODUCTION

Lead selenide (PbSe), is a semiconductor material used in manufacturing infrared detectors, in the solar cell fabrication, window screen and anti-reflection coatings. The study of optical properties in the IR is important for many of its applications.

If we assume the reflection of radiation on a flat surface coated with a thin film as shown in Figure 1, part of the radiation is reflected from the air-film interface (radius 1). The other part through the film, will be reflected at the film-substrate interface and then emerge from the film (radius 2). The recorded spectrum is a reflection-absorption (RA) spectrum and is very similar to the transmission spectrum of the film.

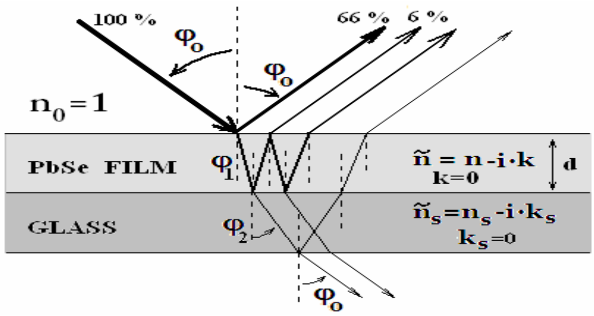

Figure 1. The specular reflection of radiation from a thin film deposited on glass surface

In order to determine the optical properties of the surface film we used dispersion analysis of reflection-absorption spectrum [1]. The spectra used should be free of interference fringes especially if absorption bands appear. The IR spectra showing interference fringes can be processed by dispersion analysis if they do not contain absorption bands.

Dispersion analysis is based on building an appropriate model for dielectric function and calculating the optical properties corresponding to this model. The best known is Drude-Lorentz model [2] which defines the electric permittivity:

$$\varepsilon(\nu) = \varepsilon_\infty + \sum_j \frac{\nu_{pj}^2}{\nu_{0j}^2 - \nu^2 - i\gamma_j \nu} \qquad (1)$$

It describes the optical response of a set of harmonic (damped) oscillators. In this relation, $\varepsilon_\infty$ is so-called "high-frequency dielectric constant", which represents the contribution of all oscillators at very high frequencies (compared to frequency range under consideration). The parameters $\nu_{pj}$, $\nu_{0j}$ and $\gamma_j$ are the "plasma" frequency, the transverse frequency (eigen-frequency), and the line-width (scattering rate), respectively of the j-th Lorentz oscillator. For the proposed model, from permittivity, we can calculate all optical quantities such as reflectance R and transmittance T. The spectrum of these theoretically calculated quantities is compared with those experimentally determined.

Suppose, we have a set of N experimental data points $\{x_i, y_i, \sigma_i\}$ (j = 1, .... N) that we want to fit. Here, $x_i$ is the light frequency, $y_i$ is the data value, and $\sigma_i$ is the data error bar. For a set of M internal parameters, the values y = f (x, $p_1$, .... $P_M$) are calculated based on the model.

The so-called Levenberg-Marquardt algorithm is used to minimize the value:

$$\chi^2 = \sum_j^N \left( \frac{y_j - f(x_j, p_j ....p_M)}{\sigma_j} \right)^2 = \chi^2 (p_1,....p_M) \tag{2}$$

Fitting process stops when the stopping criterion is met [3].

If the surface film is thin, non-absorbent (k = 0), with thickness between 1μm and 100 μm, the IR spectrum shows the interference fringes.

The radiation reflected at the air-film interface can interfere with the transflective radiation (which runs through the film twice and then is reflected at the film-substrate interface), and the interference fringes are obtained.

Certain conditions must be met to obtain interference fringes used in quantitative measurements:

- the film should have even thickness;
- the intensities of radiation that interfere should be comparable in value
- only spectral region should be used, in which the film is not absorbent;
- the film thickness must be comparable to the wavelength (between 2 μm and 20 μm for an IR spectrum) [4,5].

If the wavelength of incident radiation is changed continuously when the resulting spectrum contains maxima and minima for those wavelengths at which constructive or destructive interference occurs. When constructive interference occurs, we can write the condition for interference maxima:

$$2nd\cos\varphi = k\lambda \tag{3}$$

Because k can be any integer, there are only certain values of λ for which constructive interference occurs. The difference between two values of k and k′ corresponding to two wavelengths λ and λ′ is the number of cycles of maxima in the recorded spectrum. Thus we can write for the two wavelengths relations:

$$2nd\cos\varphi = k\lambda \text{ and } 2nd\cos\varphi = k'\lambda' \tag{4}$$

Since we can determine the difference between k and k′ by counting cycles of maxima, we can write:

$$k - k' = N = \frac{2nd\cos\varphi}{\lambda} - \frac{2nd\cos\varphi}{\lambda'} = 2nd\cos\varphi(\tilde{\nu} - \tilde{\nu}') \tag{5}$$

In this relation N is the number of inter-fringes in the spectral range $\Delta\tilde{\nu} = \tilde{\nu} - \tilde{\nu}'$ and φ is the angle of refraction of radiation at the air-film interface. If the surface film is optically non-absorbent (k = 0), the angle of refraction is a real parameter and is related to the angle of incidence by Snell's relationship:

$$\sin\varphi_o = n \cdot \sin\varphi \tag{6}$$

The two sizes n and d corresponding to surface film can be determined if we analyze the reflection-absorption spectra, showing interference fringes recorded at two different incidence angles $\varphi_{01}$ şi $\varphi_{02}$ [4,6].

In the spectrum recorded at $\varphi_{01}$ incidence angle, N1 fringes are observed for the spectral range $\Delta\tilde{\nu}_1 = \tilde{\nu}_1 - \tilde{\nu}_1'$ and in the spectrum recorded at $\varphi_{02}$ incidence angle, N2 fringes are observed for the spectral range $\Delta\tilde{\nu}_2 = \tilde{\nu}_2 - \tilde{\nu}_2'$. If we assume that the refractive index n of the surface film is constant for the two spectral ranges, we can write the system of equations:

$$N_1 = 2nd\cos\varphi_1(\tilde{\nu}_1 - \tilde{\nu}_1') \text{, respectively } N_2 = 2nd\cos\varphi_2(\tilde{\nu}_2 - \tilde{\nu}_2') \tag{7}$$

Film thickness d can be determined by solving the system of equations (7) using relationship:

$$d = \frac{1}{2 \cdot \Delta\tilde{\nu}_1 \cdot \Delta\tilde{\nu}_2} \sqrt{\frac{(N_1\Delta\tilde{\nu}_2)^2 - (N_2\Delta\tilde{\nu}_1)^2}{\sin^2\varphi_{02} - \sin^2\varphi_{01}}} \tag{8}$$

The refractive index of the surface film varies with the wavelength so that it can not be calculated from the system of equations (7). In order to determine the refractive index, the dispersion analysis is used.

## ❖ EXPERIMENTAL

The amorphous lead selenide film was obtained by chemical deposition on glass substrate from a solution of selenium sulfate and lead acetate by the method described in the literature [7].

The IR reflection-absorption spectra were recorded using the specular reflection device of the spectrograph UR-20 Carl Zeiss Jena. A non-polarized infrared radiation was used. Figure 2 shows the reflection-absorption spectra of PbSe recorded at 20 degrees and 55 degrees incidence angles. These spectra present interference fringes and were used to determine the thickness of the surface film.

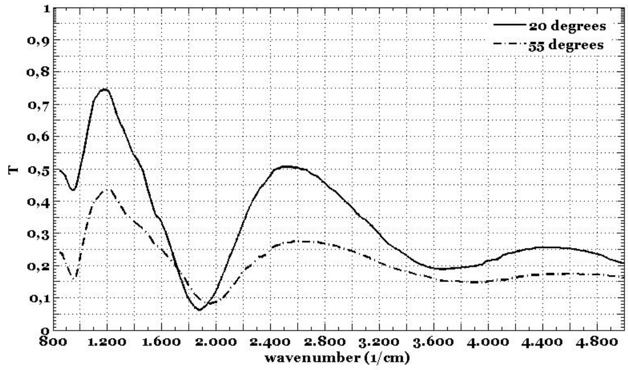


Figure 2. The reflection-absorption spectra of PbSe film deposited on glass, recorded at $20^0$ and $55^0$ incidence angles

The thickness of PbSe film was obtained using a MATLAB computer program that solves the system of equations (7).

The reflection-absorption spectrum recorded at $20^0$ incidence angle was processed using the RefFIT program to obtain the optical constants of PbSe film [2].

## ❖ RESULTS AND DISCUSSION

By processing the spectra shown in Figure 2, recorded at two different incidence angles, a value of 0.87μm was obtained for the PbSe film thickness.

In the spectral range $1000cm^{-1}\div5000cm^{-1}$ two complete cycles of change in amplitude can be observed due to interference. The refractive index of PbSe vary little with frequency so that it can be considered constant. It can thus use equation (8) to determine the thickness of the surface film. According to literature data, the refractive index changes with 0.136 in the spectral interval 1000 $cm^{-1}\div2000$ $cm^{-1}$ [8,9]. It is an error of 0.07 μm by assuming a constant refractive index in the spectral interval investigated.

The RefFIT program used for the dispersion analysis of the reflection-absorption spectrum of PbSe allows the spectra to be processed, which shows the interference fringes. In this case, the film thickness is used as an experimental variable parameter in fitting process. For the model of dielectric function we used 11 internal parameters. We have processed reflection-absorption spectrum containing a set of 2076 digitized experimental points. The model internal parameters and the experimental parameter are continuously adjusted to fit the theoretical values with experimentally measured data. In case of reflection-absorption spectra the theoretical transmittance and experimental transmittance spectra are compared. The parameter fitting process stops when the differences between theoretical and experimental spectra are minimal.

In order to improve the fitting process, we used alongside with the reflection-absorption spectra recorded at 20 degrees angle of incidence, the values of optical constants from literature [9]. I introduced a number of additional 1001 points. This spectrum shows weak interference fringes corresponding to a thickness of 0.86 μm for the surface film. The theoretical transmission spectrum corresponding to the physical model that best approximates the reflection-absorption spectrum is shown in Figure 3.

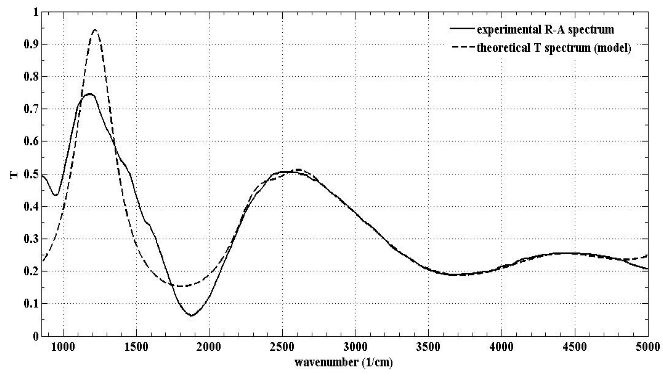


Figure 3 Experimental reflection-absorption spectrum at $20^0$ incidence angle and theoretical transmission spectrum corresponding to physical model

The two spectra are almost identical especially in the spectral interval $2200\div5000$ $cm^{-1}$ where the intensity of interference fringes is significantly smaller and does not disturb the appearance of the spectrum recorded.

Figure 4 shows the refractive index spectrum of PbSe film, obtained from the dispersion analysis by simultaneously fitting two data sets: reflection-absorption spectrum of PbSe, recorded at $20^0$ incidence angle and the values of optical constants of PbSe in the literature in the spectral range $1000cm^{-1}\div2000cm^{-1}$ [9].

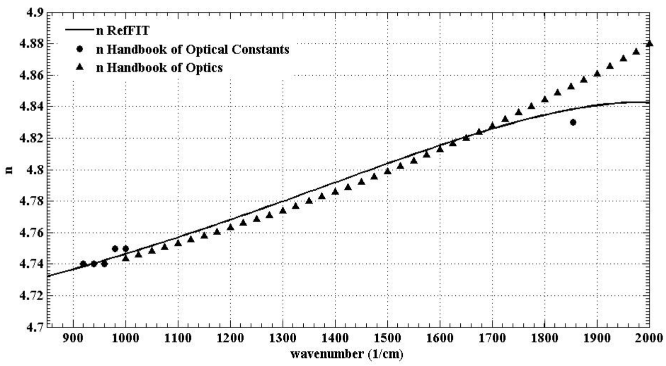


Figure 4. The refractive index spectrum of PbSe film obtained by dispersion analysis, compared with literature data [8.9]

For the PbSe film, the absorption index is approximately equal to zero so that the relations used to determine the film thickness from interference fringes can be considered correct. Figure 5 presents the corresponding absorbance spectra of PbSe film compared with literature values [8].

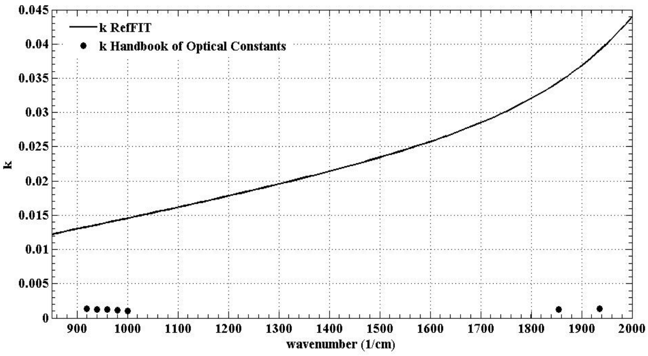


Figure 5. The absorption index spectrum of PbSe film obtained by dispersion analysis, compared with literature data [8]

## ❖ CONCLUSIONS

In the case of thin surface films, the specular reflectance spectra are reflection-absorption spectra and are similar to the transmission spectra.

The dispersion analysis of reflection-absorption spectra is more reliable and easier than the Kramers-Kronig analysis. RefFIT program determines both the refractive index and thickness of the surface film.

The thickness of the PbSe surface film can be determined by dispersion analysis and by the processing of the interference fringes present in the reflection-absorption spectra. The results from these two methods are comparably equal.

Knowing the spectrum n=f(v) is important for various areas of use of this semiconductor material.

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