

Electron beam induced changes in the reflectance of materials

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Abstract. In this work we examine the opportunity to combine the advantages of two well-known experimental techniques – ellipsometry and scanning electron microscopy (SEM). Ellipsometry has very high sensitivity in the direction normal to the sample’s surface while the lateral resolution is limited by the width of the probe beam, while the scanning electron microscopy has better lateral, but limited in-depth resolution. The electron beam can induce local changes in the electron density and the temperature of the sample, which alters the reflectance coefficient and can be potentially detected by optical methods and ellipsometry in particular.

1. Introduction

The ellipsometry is contactless and non-destructive method for researching of solid state or liquid surfaces and thin films. The information about the sample is achieved from the change of the polarization state of the probe light beam. Ellipsometry has high sensitivity for the optical properties and the thickness of layers – it can be used for detection of sub-monolayers. [1] However this resolution is in direction normal to the sample’s surface, while the resolution parallel to the sample’s surface is much lesser – it is limited by the width of the probe light beam.

The scanning electron microscopy has high resolution in the plane of the sample’s surface (10 – 30 nm). The detected signal can be secondary electrons, backscattered electrons, photons (cathodeluminescence), X-ray. [2] Every of these signals is consequence of interaction of the electrons with matter.

In recent years, the scanning electron microscopy is combined with other experimental techniques for more detailed research of the materials. This can be accomplished with using more channels of information from the interaction of the electrons with matter. One example is using acoustic signal generated by modulated electron beam and detected by piezoelectric sensor. This technique is called scanning electron acoustic microscopy (SEAM) and it is used for imaging of boundaries of different phases or ferroelectric domains.[3]

A part of the electron beam energy is converted to heat which increases the local temperature. The electron beam also injects free carriers in the sample. The carrier injection and local heating lead to local changes of the refractive index. [4-7] Here we examine the possibility to use this still unexploited channel of information. To achieve our aim we complete two tasks: to perform several simulations of the optical response due to the local heating and to conduct some preliminary experiments.



2. Simulations of optical response

To examine the possibility to combine the two techniques (ellipsometry and SEM) several simulations are made. The induced changes of the refractive index for many types of materials are very small. The thermo-optical coefficient is $dn/dT \approx 10^{-5}$ and $dk/dT \approx 10^{-3}$ for metals [8], and $dn/dT \approx 10^{-4}$ and $dk/dT \approx 10^{-5}$ for semiconductors [9, 10] and $dn/dT \approx 10^{-5}$ for dielectrics [11, 12]. The optical response depends heavily on the angle of incidence and the input polarization state and it is very important to find the optimal conditions for the largest optical response. For this purpose we simulate the changes of the ellipsometric angles ψ and Δ and the reflection coefficients R_p and R_s induced by a small change of the refractive index at different angles of incidence and polarization.

The derivatives $d\psi/dn$, $d\Delta/dn$, dR_p/dn and dR_s/dn are calculated for angles of incidence φ in the range $0.01^\circ < \varphi_i < 89.00^\circ$ with a step $\Delta\varphi_i = 0.1^\circ$. First, each of the parameters Ψ_0 , Δ_0 , R_{p0} , R_{s0} is calculated for varying angle of incidence (R_{p0} and R_{s0} are calculated using the scattering matrix approach [13]). Then a little value $-dn = 0.001$ is added to the refractive index n and the four parameters Ψ_1 , Δ_1 , R_{p1} , R_{s1} are calculated again. This addition dn simulates the change induced by the temperature alternation. In the end the numerical derivatives are obtained:

$$\frac{d\psi}{dn} = \frac{\psi_1 - \psi_0}{dn}, \quad \frac{d\Delta}{dn} = \frac{\Delta_1 - \Delta_0}{dn}, \quad \frac{dR_p}{dn} = \frac{R_{p1} - R_{p0}}{dn}, \quad \frac{dR_s}{dn} = \frac{R_{s1} - R_{s0}}{dn}.$$

For samples with complex refractive index $N = n - ik$ the derivatives are also calculated for the extinction coefficient k . The calculations are made for light source with wavelength $\lambda = 632.8$ nm. The derivatives of R_p and R_s are calculated to estimate the optical response in thermoreflectance and photoreflectance methods, where the relative change in the reflectance $\Delta R/R$ is measured. The thermoreflectance methods have very high sensitivity. The smallest detectable change of the reflectivity $\Delta R/R$ is in range less than 10^{-6} (10^{-7} range).[12, 14]

The simulations are made for three sample groups: with real refractive index, with complex refractive index and a layer with real refractive index on a substrate with complex refractive index. In figure 1 are shown the results for glass sample (real refractive index).

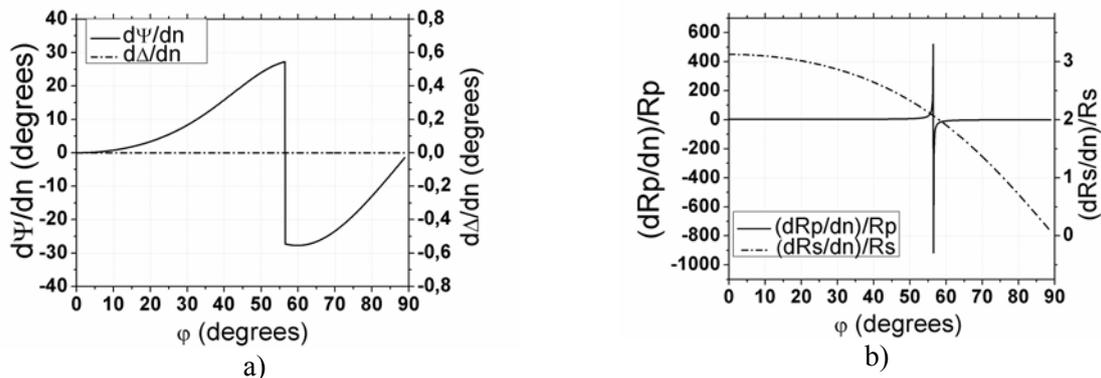


Figure 1. Derivatives of the ellipsometric parameters $d\psi/dn$, $d\Delta/dn$ a) and the reflection coefficients $(dR_p/dn)/R_p$, $(dR_s/dn)/R_s$ b) as function of the angle of incidence φ for glass sample $\lambda = 632.8$ nm; $N = 1.51$

It is obvious that the change of the reflection coefficient R_p is greatest close to the Brewster angle $\varphi = 57^\circ$. For s -polarization the sensitivity is higher close to the angle of incidence 0° (normal incidence).

The simulations for the second group of samples (complex refractive index) in this case gold are shown in figure 2.

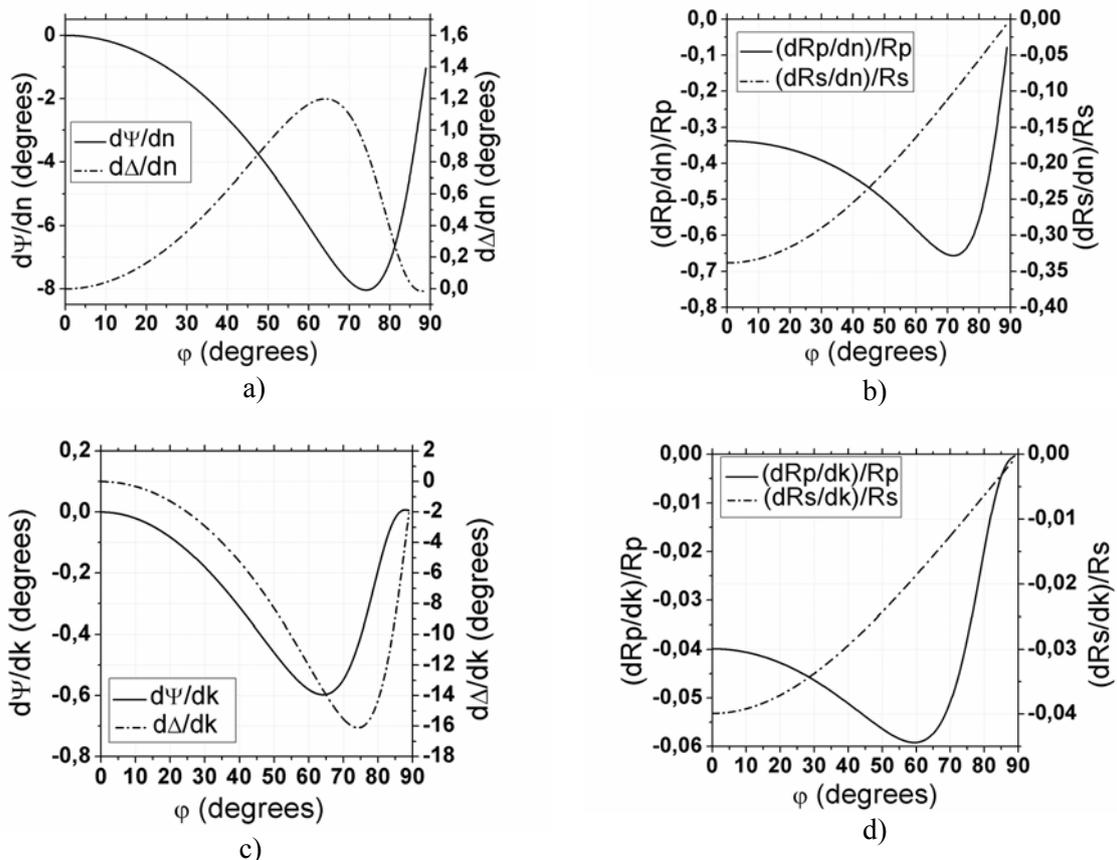


Figure 2. Derivatives of ellipsometric parameters $d\psi/dn$, $d\Delta/dn$ a), $d\psi/dk$, $d\Delta/dk$ c) and reflection coefficients $(dR_p/dn)/R_p$, $(dR_s/dn)/R_s$ b), $(dR_p/dk)/R_p$, $(dR_s/dk)/R_s$ d) as function of the angle of incidence for gold $\lambda = 632.8$ nm, $N = 0.21-3.272i$

The greatest changes in the ellipsometric parameters ψ and Δ are in the range $60^\circ < \varphi < 80^\circ$. The changes of the real part of the refractive index lead to greater changes in the reflection coefficients than the imaginary part. R_s changes mostly at angles close to 0° , while R_p changes mostly at angle $\varphi = 75^\circ$ for alternation of real part n and at angle $\varphi = 60^\circ$ for alternation of imaginary part k .

The third group of the model system is a layer with real refractive index on a substrate with complex refractive index. This simulation is more complex because there are six derivatives – four describing changes of the substrate and two describing changes of the layer. Representative results for a silicon dioxide layer (SiO_2 , with thickness 5nm and 180 nm) on silicon (Si) substrate are shown in figure 3.

The simulations show that for layer thickness 5nm, the optimal angle of incidence for the ellipsometric angles ψ and Δ is $\varphi \approx 75^\circ$. The useful range of incidence angles with acceptable optical response increases with increasing the thickness of the layer – see figure 6 c), d). The range where R_p and R_s have greater sensitivity also increases with thickness of the layer.

In order to estimate the detectable refractive index change induced by SEM irradiation, the power of the electron beam was calculated from the measured electron beam current at different accelerating voltages. The maximum power attainable in the SEM was 0.5 W. Using this value the effect of the electron beam heating onto different substrates was simulated. The results show that the temperature change is from several tens to several hundreds degrees.

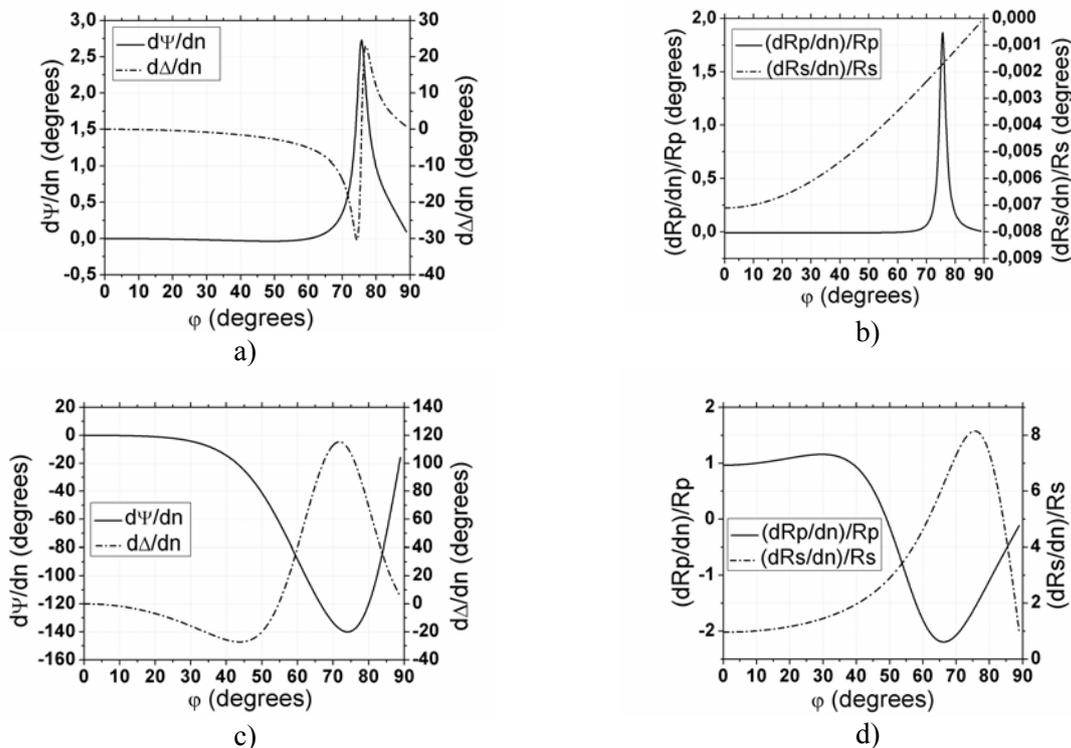


Figure 3. Derivatives of ellipsometric parameters $d\psi/dn_{SiO_2}$, $d\Delta/dn_{SiO_2}$ a) and reflection coefficients $(dR_p/dn)/R_p$, $(dR_s/dn)/R_s$ b) for layer with thickness 5nm as function of φ ; $d\psi/dn_{SiO_2}$, $d\Delta/dn_{SiO_2}$ c) and reflection coefficients $(dR_p/dn_{SiO_2})/R_p$, $(dR_s/dn_{SiO_2})/R_s$ d) for layer thickness 180 nm as function of φ

On the other hand the uncertainty of the ellipsometric parameters is $d\psi = 0.02^\circ$ and $d\Delta = 0.03^\circ$. This is used for estimation of the smallest detectable changes in the refractive index, which are in the order of $\Delta n = 0.002$ and $\Delta k = 0.002$. These changes in the (complex) refractive index could be achieved by rising the temperature with approximately 20 K for semiconductors and 200 K for metals and dielectrics, which is achievable by electron beam heating.

3. Experiments

Two proof-of-concept experiments were conducted. The heating by the electron beam is modelled with heating by electric current (the first experiment) and focused laser beam (the second experiment).

In the first, gold layer deposited on glass (thickness $d = 20$ nm) is heated by rectangle electric signal (figure 4 a)). Its resistance is $R = 76 \Omega$. The frequency and the duty cycle can be regulated. The maximum voltage amplitude is $V_{pp} = 10$ V and the maximum power is $P = 1.3$ W. The bifurcated optical fibre is used to illuminate the surface (semiconductor laser with $\lambda = 532$ nm) and to detect the reflected signal with a photo detector (Thorlabs PDA36A) – figure 4 a). The signal is fed to a lock-in amplifier with its reference channel connected to the output of the signal generator. The signal was detected with this set-up, but with very low signal to noise ratio. Strangely enough, a collateral result was far more stable detection of the acoustic signal generated by the heat modulation of the layer.

The second modelling experiment is similar to photoreflexive methods. A semiconductor laser ($\lambda = 532$ nm) was used for modelling the electron beam heating and another semiconductor laser ($\lambda = 659$ nm) - for probe beam. They are focused by microscope at one point with objective with magnification $\times 20$ (figure 4 b)). The heating beam is modulated with mechanical chopper with frequency $f = 723$ Hz and its power is $P = 130$ mW. Interference filters are used for blocking the heating beam. Measurements are made for two samples Si and InP. The results are in table 1.

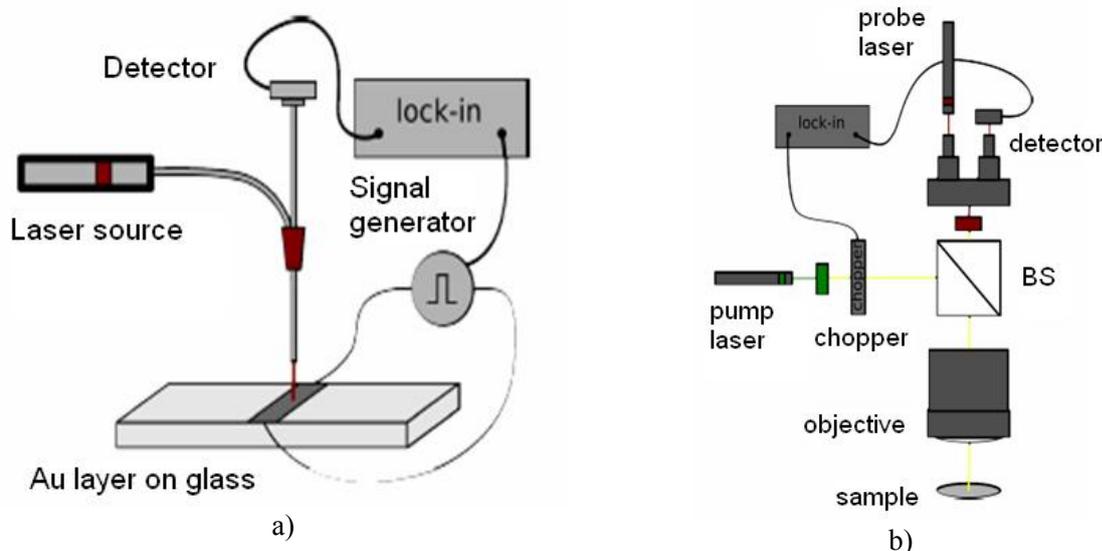


Figure 4. a) Modelling experiment – modulated heating of gold layer (thickness 20 nm) with rectangle electric signal and detection of the changes of the reflectance b) Modelling experiment – heating with laser. A microscope is used to focus the heating and probe beam. The pump laser is modulated by a mechanical chopper. The probe laser is placed at the one ocular and the detector at the other.

The temperature change is estimated for the two experiments. The temperature rise is of the order of 1 K in the first experiment and of the order of 10 K in the second experiment.

The power of the electron beam is greater than that of the laser beam and the electron beam can be focused on smaller area with diameter 1 μm or less, while the laser beam can be focused on area with diameter approximately 10 μm . Hence, the electron beam heating could be more effective than laser beam heating.

Table 1. Results from experiment - heating with laser beam. The modulation frequency is 723 Hz. $\Delta R/R$ is the relative change in the reflectance.

| Sample | $\Delta R/R$, $\mu\text{V}/\text{V}$ | Detector gain, dB |
|--------|---------------------------------------|-------------------|
| Si | 25 | 60 |
| InP | 10 | 50 |
| InP | 7 | 50 |
| InP | 20 | 60 |
| InP | 16 | 60 |

Good isolation from mechanical vibrations and better spectral isolation of the two laser beams are very important for this experiment to achieve stable results.

4. Conclusion

The possibility of combining two complementary experimental techniques – ellipsometry and scanning electron microscopy was demonstrated by theoretical calculations and simulations. The simulations show that the electron beam induces changes in the refractive index can be possibly detected experimentally by optical methods and in particular, by ellipsometry. Optimal conditions for this were estimated. Potential applications of the combined method are examining the thermal and

electronic properties of micro-objects. Another potential application is using the optical channel of information for imaging.

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