Ellipsometry in optical studies of thin films conducted at the Institute of Physics of Wrocław University of Technology

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Ellipsometry is a very powerful and totally nondestructive technique for determining optical constants, film thickness in multilayered systems, surface and interfacial roughness and material microstructure. Ellipsometric measurements can be made in vacuum, air and other environments.Ellipsometry has traditionally been used to determine film thickness and optical constants of dielectrics and optical coatings, semiconductors and heterostructures, magneto-optic, magnetic and opto-electronic materials, electrochemical, biological and medical systems and in surface modifications and surface roughness investigations. In situ measurements during crystal growth or material deposition are useful to study constituent fractions (including void fractions) in deposited or grown materials, surface oxide formation and film growth kinetics. Ellipsometric studies of metal, dielectric, semiconductor and organic layers carried out at the Institute of Physics of Wrocław University of Technology by members of Thin Films Group are presented.

1. Introduction

Ellipsometry is an optical technique which studies the polarization transformation that occurs as a beam of polarized light is reflected from or transmitted through the interface or film. The main adventages of this method are its nonperturbing character and that it may be used for in situ measurements to study kinetics of dynamic processes. The first reports of polarization and interference phenomena were made by Malus, who observed polarized light in 1810, and BREWSTER [1]. It should be noted that although it was not called by the name, ellipsometry was practiced in the late 1800's [2], and the term ellipsometry was introduced by ROTHEN in 1945 [3]. The main contribution to the scientific description of polarization and ellipsometry and to the theory of these phenomena was given by DRUDE [4], who used two mutually perpendicular polarized light beams with phase difference to measure the thickness of very thin films [5]. Later, ellipsometric method was tested in many specific problems of physics and surface chemistry. Ellipsometry as a method offering vast possibility of determining the optical parameters of thin and ultra-thin layers (also during their growing process) plays a predominant role in application to such new domains as optoelectronics, acoustooptics or hybrid optics.

In the early 1960's, Archer showed that for in situ measurements this technique can measure a fraction of a monolayer of adsorbed gas [6]. The first home-built ellipsometers appeared in scientific laboratories in the 1960's. Commercial instruments were designed in the late 1970's. In the first ellipsometers it was usually mercury or sodium arc lamps that were the sources of monochromatic light, at the present time the lasers are used. Since the early 1990's the spectroscopic ellipsometry began to be applied. Physical principles of theory and ellipsometry experiments are given in several textbooks [7] – [9]. The first monographic paper about ellipsometry in the Polish language was written by BRUDZEWSKI [10]. In 1993, Thompkins published a user's guide for single-wavelength ellipsometry [11], and in 1999, Thompkins and Mc Gahan wrote *Spectroscopic Ellipsometry and Reflectometry. A user's guide* [12]. Nowadays, ellipsometry develops extensively. The values and the changes of the optical constants caused by different external conditions could be controlled by ellipsometry in electronic industry. The propagation of the surface elastic waves in crystals and in multilayer systems or propagation of electromagnetic waves in optical fibers depend to a large extent on the solid state surface and interface conditions and can be studied by ellipsometry. It is very important that ellipsometry is a nondestructive and contactless method, ellipsometric measurements can be carried out in a wide temperature range, in vacuum or in different environments. The sensitivity and measuring accuracy are very high and since ellipsometers can be very precise, ellipsometry is competitive to other methods.

The aim of this paper is to present the results of investigations into the optical properties of metal, dielectric, semiconductor and organic layers with application of ellipsometry, carried out for over thirty years by the Thin Films Group at the Institute of Physics, Wrocław University of Technology.

2. Ellipsometric technique developed at the Institute of Physics of Wrocław University of Technology

The use of the ellipsometric method in the Thin Films Group of the Institute of Physics, Wrocław University of Technology, was initiated by Idczak. In 1971, Shklyarevskii, El-Shazly and Idczak proposed a new ellipsometric method for simultaneous determination of the thickness and dispersion of refractive index of thin transparent dielectric films on metallic substrate [13], [14]. The basic equation of ellipsometry is

$$\tan \Psi \exp i\Delta = f(n_1, k_1, n, d, \lambda, \varphi)$$

where: n_1 – refractive index of substrate, k_1 – absorption coefficient of substrate, n – refractive index of layer, d – thickness of layer, λ – wavelength of light, φ – angle of incidence, Ψ and Δ – ellipsometric angles, (Ψ is the angle whose tangent gives the ratio of the complex Fresnel reflection coefficients for the p and s polarizations, $\tan \Psi = |r_p|/|r_s|$, Δ gives the difference between the phase shifts

experienced upon reflection by the p and s polarizations, respectively, $\Delta = \delta_{rp} - \delta_{rs}$).

From the above equation, the relations $\Delta(d)$ and $\tan^2 \Psi(d)$ were calculated for given experimental values of λ, φ, n_1, k_1 and reasonably accepted values of refractive indices n of layer (Fig. 1 and Fig. 2). These calculations are performed on a computer. The intersection points of the curve sets $\Delta(d)$ and $\Psi(d)$ with the experimentally measured values Δ_{exp} and Ψ_{exp} give two new relations $n_\Delta(d)$ and $n_\Psi(d)$. The relations $\Delta(d)$ and $\Psi(d)$ are fulfilled simultaneously by one pair of values n and d.
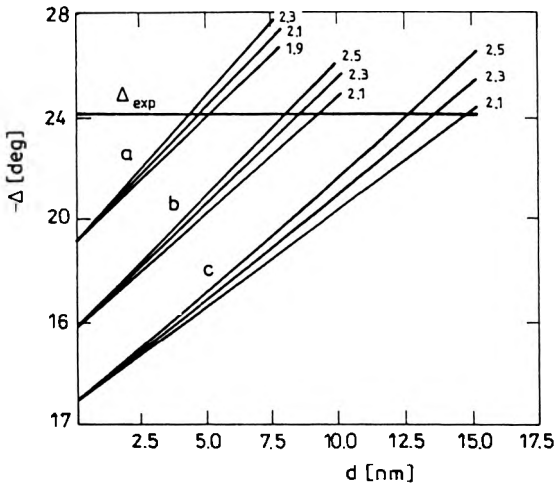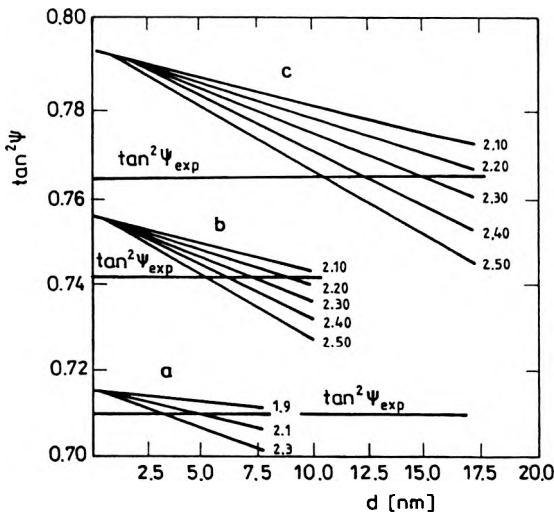

Fig. 1. Dependence $\Delta = f(d)$ for ZnS layers.
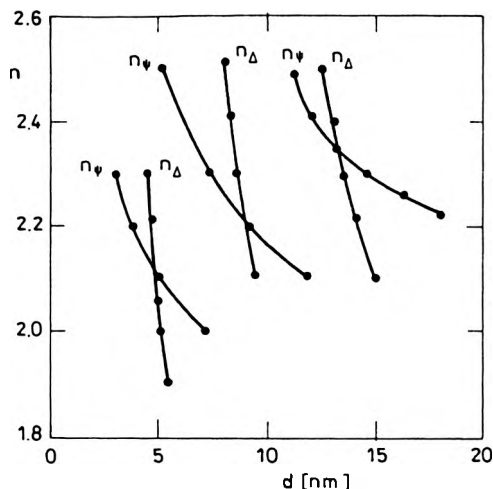

Fig. 2. Dependence $\tan^2 \Psi = f(d)$ for ZnS layers.

Fig. 3. Dependence of n_Δ and n_ψ on layer thickness for ZnS.

These values are found from the intersection of curves $n_\Delta(d)$ and $n_\psi(d)$, see Fig. 3.

Ellipsometric measurements were made for ZnS films on Sb substrates and for thin films of cryolite on chromium substrate. Thickness and refractive indices were determined in visible spectral range (0.45 – 0.65 µm) [14]. For very thin films of ZnS and cryolite the filling factor was also determined.
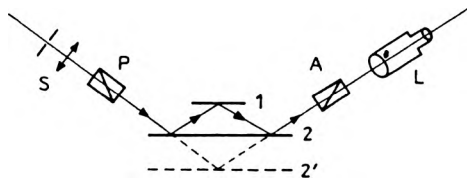

Fig. 4. Scheme of Shklyarevskii's method: 1 — sample under examination, 2 — reference sample, S — light source, P — polariser, A — analyser, L — detector.

In the ellipsometric method described earlier (1957) by SHKLYAREVSKII [15], the material under study was deposited onto two plates of sizes 70 × 20 mm (plate 2) and 20 × 20 mm (plate 1), which were placed in a special holder of goniometer (Fig. 4) Idczak modified the Shklyarevskii's method using only one plate of the material studied, the second plate was a reference sample (e.g., of Al). The sample under examination could then be of considerably smaller size (20 × 20 mm). The formulae for determining the optical constants of the material studied are described in paper [16], and the method proposed has been verified with Inconel layers, measured in visible spectral range, with aluminium layers as the reference sample. The results of these measurements have been compared with the ones obtained by classical Shklyarevskii's method, and these appear to be in a satisfactory agreement.
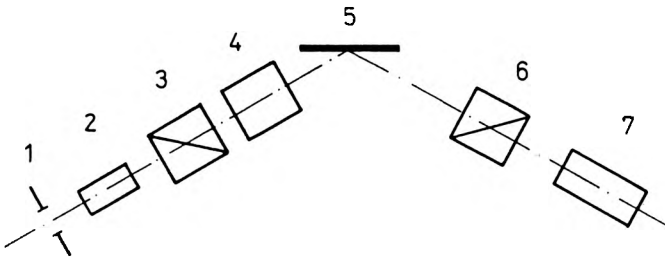
Fig. 5. General view of the ellipsometer: 1 — light source, 2 — collimator, 3 — polariser, 4 — compensator, 5 — sample, 6 — analyser, 7 — detector.

In 1972 — 74, an ellipsometric spectroscopy stand was set working at the Institute of Physics, for which the Archer method was applied. A general view of the ellipsometer is presented in Figure 5.

The ellipsometer and the calculation methods were tested by IDCZAK and OLESZKIEWICZ [17] by means of measuring the ellipsometric angles Δ and Ψ of anodic oxidised aluminium layers. The ellipsometric angles Δ and Ψ have been measured both for the aluminium layers and the Al_2O_3 on the Al substrate. Based on the ellipsometric measurements the optical constants for aluminium layers have been calculated by a numerical method, taking into account the existence of the natural oxide layers as well as the refractive index and different thicknesses of the aluminium oxide layers deposited on aluminium. It has been found that the refractive index of the aluminium oxide on aluminium layers does not practically
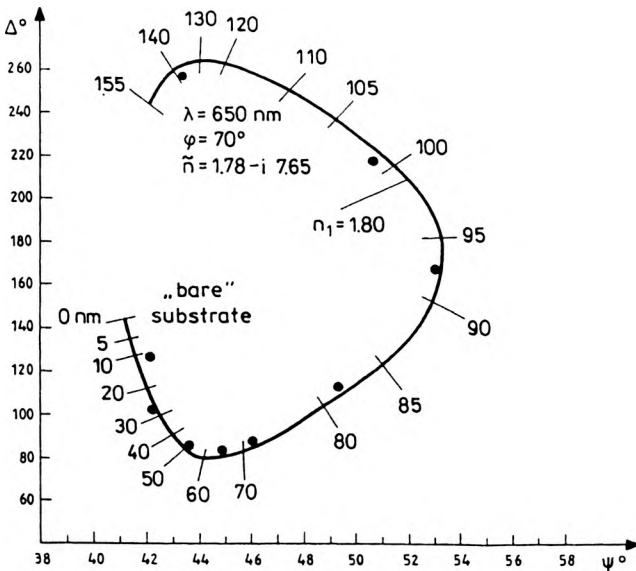

Fig. 6. Experimental ellipsometric measurements $\Delta = f(\Psi)$ for the anodized aluminium layers ($\varphi = 70°$, $\lambda = 650$ nm, $n = 1.78$, $k = 7.65$).

depend upon the oxide thickness and that the aluminium layers exhibit normal dispersion within the visible spectral range. The results of ellipsometric measurements of the aluminium oxide layer thickness for a chosen sample are shown in Figure 6.

The measurements start at the point which represents the non-anodized aluminium layers and as the oxide layer thickness grows, the measurement points are shifted along the ellipsometric curve in the counterclockwise direction. The computer programs which were worked out at that time enabled calculation of the refractive index n and the dielectric layer thickness d on the absorbing substrate characterised by definite optical constants [18], [19]. It was possible to calculate the optical constants n, k and the thickness d of the absorbing substrate with specific optical constants. The RIAD computer was applied for the calculation [20].

3. Optical studies of ytterbium oxide and terbium oxide layers

Thin films of ytterbium oxide on chromium substrate have been investigated in the spectral range $0.45-0.65$ μm by IDCZAK and ŻUKOWSKA [21]. The ellipsometric measurements were made for Yb_2O_3 layers of various thicknesses, with the refractive index and thickness of layers being calculated by using the Shklyarevskii's method [14]. It has been shown that while for the Yb_2O_3 layers of thickness greater than 20 nm the refractive index n is almost the same as for thick layers ($n = 1.85$ [22]), for very thin Yb_2O_3 layers n is much greater. Such a non-typical dependence could be explained by the presence of a thin chromium oxide layer situated between Cr and Yb_2O_3 layers [23]. Given the optical constants n_3 and k_3 of the chromium substrate, the refractive index n_2 and thickness d_2 of the chromium oxide layer as well as n_1 and thickness of the ytterbium oxide layer, we could calculate the ellipsomertic angles Δ and Ψ for such a system of layers. By comparing the calculated ellipsometric angles of the assumed model with those measured experimentally, the best agreement was obtained for a system with a chromium oxide layer of refractive index $n = 2.55$ and thickness 5 nm. On the basis of this comparison we can conclude that the assumed model for the layer under examination is satisfactory [24].

Optical properties of terbium oxide films were investigated by MARCINÓW et al. [25]. Terbium oxide films were deposited by an electron gun evaporation onto heated substrates (up to 200 °C) and TbO_x ($1.5 < x < 1.75$) layers were obtained. Then terbium oxide layers were subjected to the oxidation process in air at temperatures ranging from 400 °C to 500 °C and next to the reduction process in hydrogen at 700 °C; in consequence Tb_4O_7 and Tb_2O_3 films were formed. The optical constants n and k for Tb_4O_7 and Tb_2O_3 layers have been determined from $R = f(\lambda)$ and $T = f(\lambda)$ (Fig. 7) characteristics measured by SPECORD UV VIS, SPECORD 61 NIR and UR 10 spectrophotometers in the wavelength range from 0.2 to 2.5 μm using methods described by VRIENS and RIPPENS [26] and SCHULTZ and TANGHERLINI [26]. The Tb_2O_3 layers can be used for optical purposes in the range from 0.4 to 2.5 μm because of the normal dispersion of the refrac-
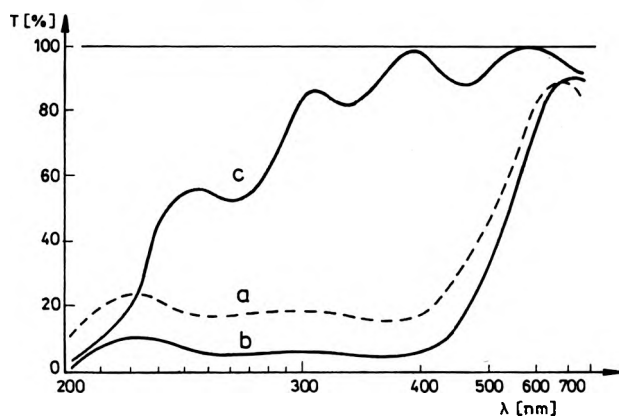
Fig. 7. Transmission coefficient T of terbium oxide layers: a — after evaporation, b — after annealing in air, c — after reduction in hydrogen.

tive index and a good transparency in this spectral region. The Tb_4O_7 layers can be used as filters cutting down the visible and ultraviolet ranges of spectrum or in a multilayer system in near-infrared region for $\lambda > 0.64$ μm.

4. Ellipsometric investigations of semiconductor layers

The ellipsometric method was also used for studies of the optical properties of implanted semiconductor layers by Żukowska and Kulik (Kulik from the Institute of Physics, Maria Curie-Skłodowska University, Lublin). Optical constants, the thickness of disordered layer and the degree of amorphousness of GaP layers implanted with Ar^+ ions were determined [28]. A four phase model consisting of the ambience, a native oxide, an implanted layer and a crystalline substrate was assumed; optical constants and the thickness of implanted layer were calculated using Multiple Angles of Incidence Ellipsometry (MAIE) method [8]. It has been shown that optical constants n and k rapidly increase for the dose of implanted ions equal 10^{14} ions/cm^2 (Fig. 8). This can be explained by the fact that the point defects tend to form into clusters and then the layer under study becomes totally amorphous. Figure 9 shows the degree of amorphousness as a function of dose. Similar results were obtained by GÖTZ [29] from the RBS measurement for GaAs. The thickness of disordered layer was compared with the projected range of ions R_p in the LSS profile. The results obtained have proved that ellipsometry can be a good experimental technique for non-destructive determination of the thickness and the degree of amorphousness of disordered layer in samples of semiconductors after implantation.

The multilayer model was also applied in the ellipsometric investigations of implanted GaAs layers. Ellipsometric measurements of layers unimplanted and implanted with Ar^+ ions were carried out at five angles of incidence for three wavelengths. Using the four-phase model (ambience, native oxide, transition layer,
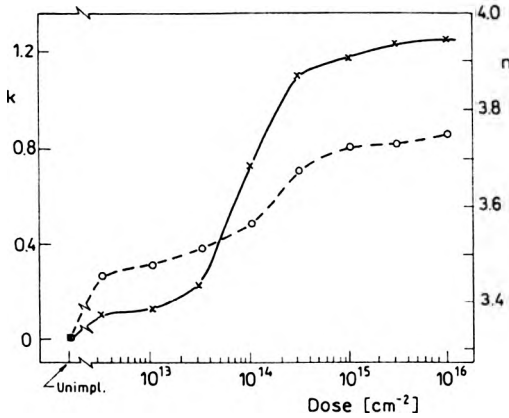
Fig. 8. Optical constants n (solid line), and k (dashed line) of GaP samples implanted with 100 keV Ar^+ ions as a function of a dose ($\lambda = 632.8$ nm)
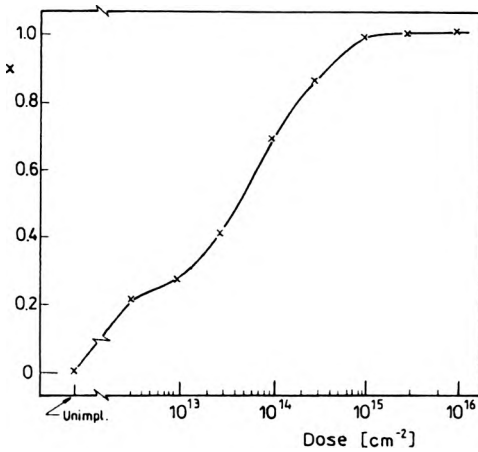

Fig. 9. Degree of amorphousness versus a dose of 100 keV Ar^+ ions implanted to GaP.

amorphous phase) and the MAIE method, the refractive index, extinction coefficient and thickness of the amorphous phase and the thickness of transition layer were determined (Tab.) [30]. The depth profiles of the optical constants in the transition layer (Fig. 10) were calculated by the effective medium approximation (EMA) [31], [32].

5. Ellipsometric studies of chromium layers

The ellipsometric measurements of chromium have been performed since 1975. The chromium layers were prepared in different technological conditions. The metal was evaporated at 6.6×10^{-6} Pa and 1.2×10^{-3} Pa [33], deposited with an electron gun and ion plated [34]. It has been found that the evaporated or electron gun deposited
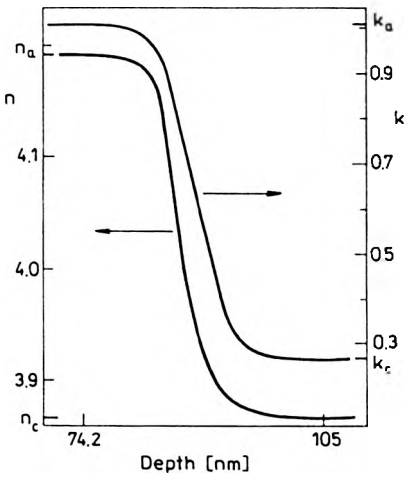
Fig. 10. Distribution of optical constants in the transition layer of GaAs implanted with 100 keV Ar^+ with the dose 3×10^{14} ions/cm^2, $\lambda = 632.8$ nm.

T a b l e. The multilayer model of implanted GaAs.

Dose [cm^{-2}]	λ [nm]	n	k	d'_{ox} [nm]	n'_{ox}	d_a [nm]	d_t [nm]
	625	4.25	1.01				
3×10^{14}				4.15	2.180	70.20	30.80
	500	4.73	1.50				
	625	4.31	1.06				
1×10^{15}				4.18	2.182	71.30	35.00
	500	4.84	1.59				
	625	4.39	1.07				
3×10^{15}				4.35	2.184	74.80	38.50
	500	4.84	1.60				
	625	4.40	1.08				
1×10^{16}				4.40	2.184	76.10	39.20
	500	4.91	1.69				

λ — wavelength, n — refractive index of the transition layer, k — extinction coefficient of the transition layer, d'_{ox} — thickness of the native oxide on the amorphous phase, n'_{ox} — refractive index of the native oxide layer, d_a — effective thickness of the amorphous phase, d_t — thickness of the transition layer.

chromium layers have a regular, body centred cubic structure (b.c.c.). The ion plated chromium layers are a regular form of chromium with a primitive unit cell. The chromium layers evaporated in high or low vacuum and with an electron gun have nearly the same refractive index n of values within the range $2.66 - 2.95$ for $\lambda = 450$ nm and $3.58 - 3.60$ for $\lambda = 650$ nm. The absorption coefficient k appeared more sensitive to the deposition method. The values k ranged from 1.91 (electron gun) to 2.81 (high vacuum). The ion plated chromium layers strongly differed from the afore-mentioned ones as regards n and k: $n = 4.44 - i\,5.67$ for $\lambda = 450$ nm and $n = 6.21 - i\,4.72$ for $\lambda = 650$ nm.

All the values mentioned above were calculated on the assumption that the chromium layer was covered with a natural oxide film ($d = 5.0$ nm thick, $n = 2.23$). The experiments proved that the refractive index and the absorption coefficient of pure chromium are higher than the values obtained from ellipsometric measurements of layers for which a natural oxide film was not taken into account. Optical constants of chromium layers do not depend on the kind of substrate (glass BK7, quartz). The chromium oxide was obtained by heating thin chromium films at a temperature of 673 K in the atmosphere of oxygen or air [33] or deposited with an electron gun [33], [35], [36].
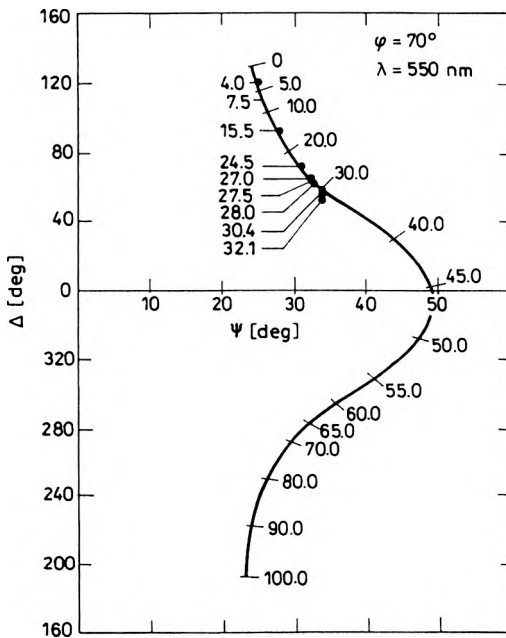

Fig. 11. Dependence of experimentally measured values Δ on Ψ (points) for chromium oxide layers of different thicknesses growing by oxidation in O_2 atmosphere at $T = 673$ K on chromium layers deposited by thermal evaporation at pressure 6.6×10^{-6} Pa. Solid curve is calculated theoretically ($\varphi = 70°$, $\lambda = 550$ nm).

Figure 11 presents an experimentally obtained characteristics of ellipsometric angles Δ and Ψ vs. chromium oxide film thickness. These curves are also compared with the theoretical curve. The results show that the oxidation rate and the oxidation process features depend on the kind of substrate. The oxide film growth on the ion plated chromium layers on the BK7 glass substrates is a parabolic time function, while the same process performed on the quartz glass substrate is inversely logarithmic.

Independent of the oxidising atmosphere composition (oxygen, air), the Cr_2O_3 film grows on the chromium layer at a temperature of 673 K. However, other oxygen Cr compounds cannot be ruled out. The Cr oxide film grew thicker in air than in oxygen, which corresponds to results obtained by the other authors. Nitrogen dissolved in the

layer is supposed to cause the oxidation rate increase. The refractive index n of the oxide films on the chromium layers, which were evaporated at 6.6×10^{-6} Pa or deposited with an electron gun, increases with the oxide film growth. It reaches a constant value when the oxide is thicker than 30 nm.

The refractive index of chromium oxide films on ion plated layers is, in a wide range, practically independent of their thickness. Regardless of the chromium layers technology and the heating conditions the refractive index of the growing oxide shows a normal dispersion. The refractive index of thin deposited Cr oxide layers is low (2.00). A value characteristic of the bulk material is reached when the film is thicker than 70 nm.

The optical properties of chromium layers were also studied by Idczak and Rachwalska. The influence of the substrate temperature on optical constants for chromium layers deposited by thermal evaporation was investigated by ellipsometry in visible spectral range. The examined chromium layers of about 200 nm in thickness were deposited with a constant rate (1 nm/s) at a pressure of about 1.33×10^{-5} Pa onto quartz substrates at temperatures ranging from 308 K to 873 K.
The optical constants of chromium layers, both the refractive index and the absorption coefficient, increase with the substrate temperatures and are higher than for layers deposited onto unheated substrate (Fig. 12). This indicates a more perfect crystal
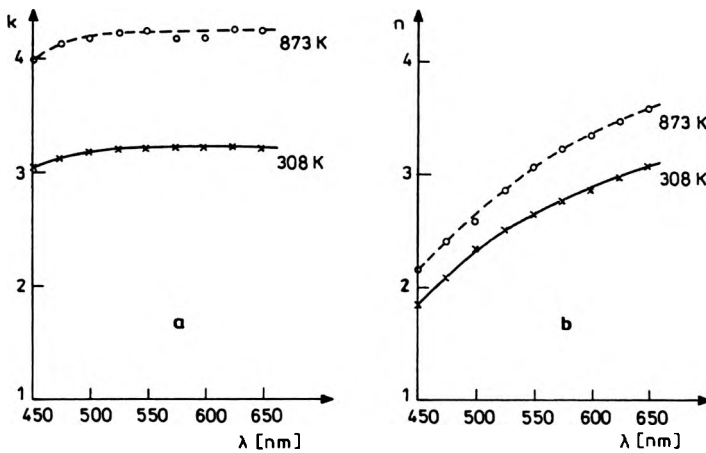
Fig. 12. Spectral dependence of the absorption coefficient k for chromium layers (deposition rate $v = 1$ nm/s) (a), spectral dependence of the refractive index n of chromium layer (b).

structure of layers deposited on the heated substrates [37]. The influence of evaporation conditions, *i.e.*, deposition rate $(0.1 - 5$ nm/s) and substrate temperature $(308 - 873$ K), on both the structure and the optical constants of the chromium layers in a wide spectral range of $0.2 - 25$ μm has been examined [38]. The chromium layers are of polycrystalline structure ranging from fine-grained (2 nm) for layers evaporated onto unheated substrates to the coarse-grained (100 nm) one for the layers deposited onto substrates of 873 K temperatures. No influence of the evaporation rate within

the applied range on the structure of the layers obtained has been found. The chromium layers deposited onto substrates of higher temperatures exhibit both higher coefficient of reflection and higher coefficient of absorption for the whole spectral range (0.2 – 25 μm). The refractive index of these layers remains practically constant within the visible and ultraviolet spectral range while it increases with the substrate temperature within the infrared region. When the optical constants of chromium layers in the wide spectral range and the electric conductivity at both room and helium temperature are known, the electron microcharacteristics of the examined layers can be determined. The electron characteristics of chromium layers produced under various technological conditions were determined [39].

6. Ellipsometric studies of organic layers

The co-operation with the Institute of Electron Technology of the Wrocław University of Technology in 1984 – 89 concerned research on organic layers applied as masks, especially poly(methyl metacrylate) (PMMA). The PMMA, like other organic materials, dielectrics (SiO_2, Si_3N_4) and metals (Al), are used in the form of thin layers as masking materials in the process of controlled and selective implantation of impurities into substrates. The required mask thickness depends on the effective ion implantation range. The range is established with electric and non-electric methods. The CV is a commonly applied electric measuring technique. Other non-electric methods include: the Auger Electron Spectroscopy (AES), the Rutherford Backscattering Ion Spectroscopy (RBS) and the Secondary Ion Mass Spectroscopy (SIMS). However, applicability of these methods is restricted in the case of polymer films, because of their low sensitivity and undesirable influence of the radiation applied on the film features.

Therefore new methods are required to enable measurements of particle range in polymers. It was found that the ion lithographic technology can be applied to the range measurements. The method takes advantage of the dependence of depolymerisation level on the irradiation level. The cut polymer chains are more easily dissolved in developers. The polymer structure change indicator is the polymer layer thickness change after being irradiated and developed. The layer thickness was measured with an ellipsometer. The advantage of this method is that there is no need of the sample preparation. It also allows us to find changes in refractive index values n of a layer as well as the refractive index n and absorption coefficient k of the silicon substrate as a function of the implanted ion dose.

The minimum useful dose and contrast can be calculated when the polymer layer thickness is known. The research was done for pure PMMA and modified with dibutyl maleate in a wide range from 5% to 20%. The layers were irradiated with light ions (B^+, Li^+, H^+) with energies of 30 and 50 keV. The doses were $10^{12} – 10^{14}$/cm^2. The results have been published in [40] – [42].

7. Application of ellipsometric measurements to extrapolation procedures in the analysis of dispersion relations

When optical constants are determined from the reflectivity measurements in a wide spectral range by using Kramers-Kronig dispersion relations it is necessary to know the reflected wave phase over the entire frequency range. Part of the reflected wave phase in the measurable frequency range could be calculated from experimentally measured reflectivity values $R(\omega)$. The contribution of the unmeasurable spectral range to the phase of reflected wave could be estimated using appropriate extrapolation. If the optical constants of the material under study are known in some narrow spectral range, the reflected wave phase can be calculated by addition of appropriate amendment. IDCZAK [43] studied optical properties of thin chromium layers in the spectral range $0.2 - 25$ µm. The spectral dependence of optical constants n and k of chromium was determined by Kramers–Kronig relations. The optical constants in the spectral range $0.4 - 2.1$ µm were determined from ellipsometric measurements based on the Beattie method [44].

The Kramers-Kronig dispersion relations were also used by Żukowska and Idczak for determining optical constants of thermally evaporated ytterbium layers. The reflectivity measurements were carried out in the spectral range $0.2 - 25$ µm, and ellipsometric measurements in the range of $0.45 - 0.65$ µm. The extrapolation procedure applied was based on the knowledge of the reflected wave phase shift in the visible spectral range (measured by ellipsometry). A detailed description of this procedure can be found in [43]. From K–K relations the optical constants of Yb were calculated and important microcharacteristics such as conduction electrons concentration and effective frequency of electron collisions were determined [45]. The same extrapolation procedure was applied in investigations of the optical properties in the spectral range $0.2 - 50$ µm of vanadium films studied by ŻUKOWSKA *et al.* [46].

8. Automatic ellipsometer

Studies of dynamic processes, *e.g.*, the growth of layers or the oxidation kinetics, require in situ measurements by an automatic ellipsometer (Fig. 13) [47]. Ellipsometric measurements were carried out during the vacuum deposition of thin ytterbium layers and during the exposure of ytterbium layers to the atmosphere. Rare earth metal films (such as ytterbium) are highly reactive to atmospheric gases. Ellipsometric measurements in situ allow us to determine the optical constants of the "clean" metal layers and to follow the earliest stages of metal oxidation after exposure of the sample to air. From the ellipsometric in situ measurements the optical constants of ytterbium metal film as well as the thickness of the native oxide layer were estimated (Fig. 14) [48].
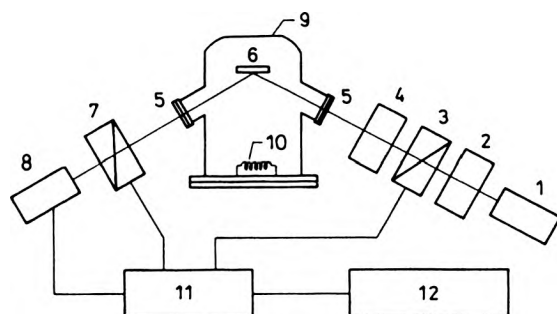
Fig. 13. Schematic representation of the automatic ellipsometer with ultrahigh vacuum system. 1 — He-Ne laser, 2 — quarter-wave plate, 3 — polariser, 4 — compensator (achromatic quarter-wave plate, 5 — optical windows, 6 — sample, 7 — analyser, 8 — photodetector, 9 — vacuum chamber, 10 — evaporating boat, 11 — control system, 12 — microcomputer.
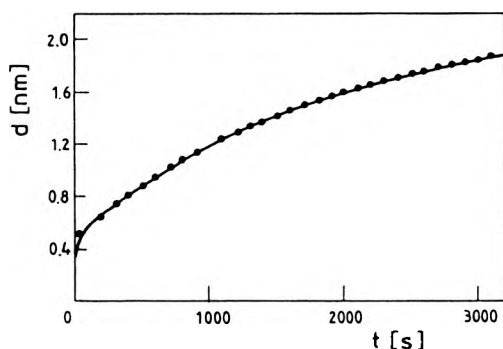

Fig. 14. Thickness of the ytterbium oxide layer in the earliest stage of ytterbium metal oxidation.

9. CrN layers

The ellipsometric method was used in the optical investigations of CrN layers obtained by Ion Beam Assisted Deposition (IBAD) process. Thermally evaporated chromium reacted with nitrogen ions generated in a Kaufman ion source was deposited as a CrN layer onto negatively biased plane parallel BK7 glass substrates. Ellipsometric measurements were made in the 0.45−0.65 μm spectral range, and measurements of reflectivity were carried out in the spectral range 0.2−0.8 μm. Optical investigations of CrN films obtained by IBAD allow us to estimate the influence of ion bombardment on physical properties of layers under study. The values of reflectivity and extinction coefficients of CrN films .increase with the ion energy (Fig. 15 a, b). The value of reflectivity increases with the bias voltage, which also indicates the improvement of the film surface quality [49].

Chromium nitride layers were also obtained by magnetron sputtering in IBAD process [50]. The source of material was Cr target subjected to magnetron sputtering. The Cr and CrN layer deposition processes were carried out in the presence of Ar and Ar+N_2 ions atmosphere. The quality of deposited layers (including their optical
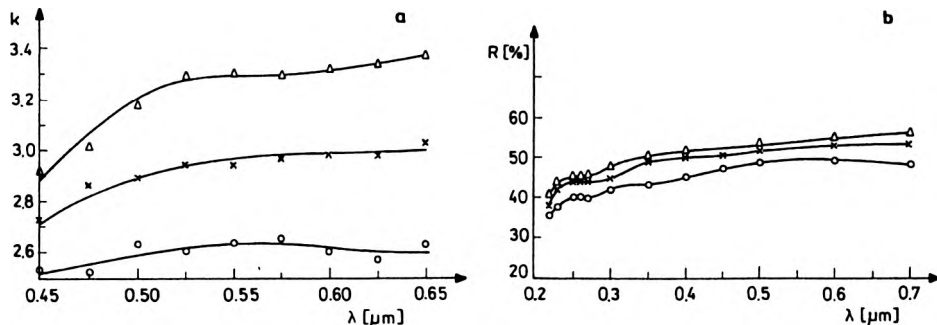
Fig. 15. Spectral dependence of extinction coefficient of CrN layers — a (o — U_p = −100 V, × — U_p = −300 V, Δ — U_p = −500 V), b — reflectivity dependence on wavelength for CrN layers deposited at different substrate bias voltage values (o — U_p = −100 V, × — U_p = −300 V, Δ — U_p = −500 V)

properties) depends on ion beam parameters such as density, energy and homogenity as well as mutual spatial situation of ion gun, source of deposited material and substrate. The CrN layers under examinations were deposited under such conditions in which the anode current reached maximum value at magnetron power of 30 W/cm², causing the most effective N_2 ionization. Optical constants of Cr and CrN layers were determined by ellipsometry. The extinction coefficient k of CrN layers deposited by IBAD with magnetron sputtering does not reveal any significant dependence on the deposition process parameters.
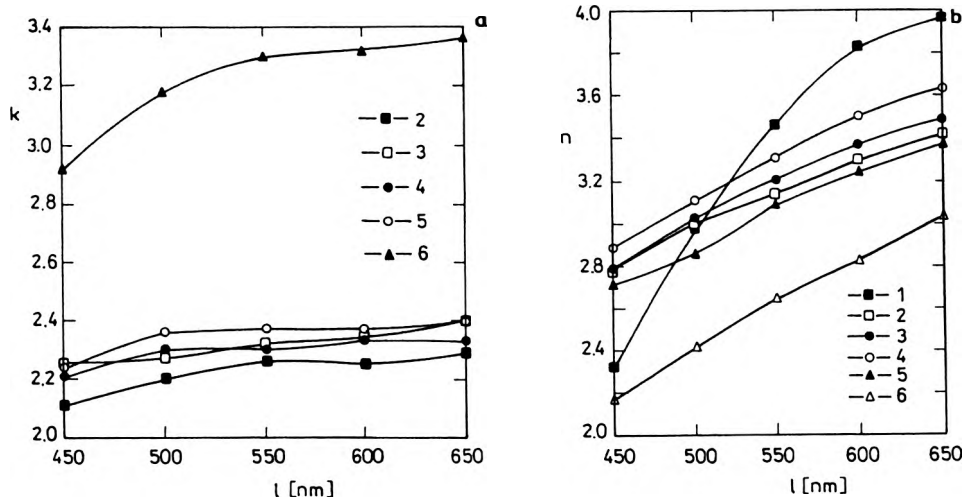

Fig. 16. Extinction coefficient k (a) and refractive index n (b) of Cr layer and CrN layers obtained in different deposition conditions on the wavelength of incident light (1 — Cr layer, 2 — CrN layer deposited onto grounded substrate, p_{N_2} = 0.2 Pa, 3 — CrN on substrate at floating potential, p_{N_2} = 0. 2 Pa, 4 — CrN on grounded substrate, p_{N_2} = 0.4 Pa, 5 — CrN on substrate biased of U_s = −500 V, p_{N_2} = 0.2 Pa (samples 1−5 obtained by IBAD with magnetron sputtering of Cr), 6 — CrN layer on substrate biased of U_s = −500 V obtained by IBAD with thermal evaporation of Cr.

Deposition rate in magnetron sputtering is lower than in thermal evaporation which explains the smaller values of k of CrN layers deposited by magnetron sputtering (Fig. 16 a). The refractive index n of magnetron sputtered CrN layers changes inconsiderably upon bias voltage of substrates, while the pressure of working gas influences significantly the n value of CrN layers deposited onto grounded substrates (Fig.16 b). For the layers formed on negatively biased substrates the refractive index of thermally evaporated layers is lower than for magnetron sputtered layers when the magnetron effect is evident.

10. Conclusions

The ellipsometric method was applied at the Thin Films Group of the Institute of Physics, Wrocław University of Technology, in optical studies of different materials such as metal, dielectric, semiconductor and organic layers. Ellipsometry was used for characterization of optical properties as well as the control method in thin films technology. Ellipsometric measurements are helpful in the choice of such deposition conditions of thin films which allow us to obtain layers of required physical properties. In the more detailed analysis of the physical properties and structure of materials under study additional techniques for ancillary measurements are used. Specifically, Auger Electron Spectroscopy (AES), X-ray Photoelectron Spectroscopy (XPS), Rutherford Backscattering Spectroscopy (RBS), Infrared Spectroscopy (IR) and UV interferometry, SIMS (Second Ions Mass Spectroscopy), LEED (Low Energy Electron Diffraction) could be used as a supporting experiments. The development of traditional ellipsometry consists in application of Fourier Transform Infrared spectroscopy (FTIR). A slightly modified ellipsometer setup with IR optical components is coupled to an FTIR spectrometer. This system allows IR spectroscopy of thin films with all the advantages of ellipsometry: it can be used on any substrate (unlike any other IR spectroscopy) and offers high sensitivity down to surface passivation layers. Information on the refractive index of materials and thin films in the IR, IR absorption and extinction coefficient, chemical structure (identification of chemical bonds), chemical content analysis, control of chemical interactions during processing, adhesion of thin films, film thickness can be derived from the measurements. It seems to be a very promissing and perspective technique for characterization of the properties of thin films.

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