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Ellipsometric examinations of thin ytterbium** oxide layers on chromium

Thin films of ytterbium oxide on chromium substrate have been examined within the spectral range 0.45 μ m-0.65 μ m. The ellipsometric angles Δ and ψ have been measured for layers of various thicknesses and both the refractive index and the thickness of ytterbium oxide layers have been calculated from the experimental data by using the ellipsometric method of Shklyarevskii.

1. Introduction

The examinations of thin dielectric layers on an absorbing substrate are interesting and important for investigation of metal oxidation, in tunnel spectroscopy, and for examination of surface states of materials in the ultrahigh vacuum. These layers can be either naturally developed or artificially deposited. The basic parameters determining the optical properties of such dielectric layers are: refractive index and thickness. The ellipsometric methods allow to determine both the magnitudes simultaneously.

The same two quantities may be also determined with the help of the approximate DRUDE-TRONSTAD formulae [1-3] frequently used in practice but the latters are valid only for very thin dielectric layers (5-7 μ m) [4,5].

Recently Shklyarevskii [6–8] has suggested a new ellipsometric method for simultaneous determination of the refractive index and the thickness of dielectric (both absorbing and non absorbing) layers on the absorbing substrate. In the Shklyarevskii's method the refractive index and the thickness of the layers is determined from the general interference formulas for the substrate-dielectric layer system. In the present paper the Shklyarevskii's ellipsometric method has been applied to determine the refractive index and the thickness of the thin layers of ytterbium oxide.

The determination of the refractive index (n) and the thickness (d) of a thin dielectric layer on the absorbing substrate (n_1, k_1) with the help of an ellipsometric method consists in measuring the phase difference Δ between the *p*- and *s*-components of the reflected light and the azimuth ψ (tan ψ determines the ratio of *p*- to *s*-components of the reflected light) for the absorbing substrate examined layer system for the given incidence angle φ and a given wavelength λ . The optical constants (n_1 , k_1) of the layer substrate must be known.

From the basic equation of ellipsometry [6-8]:

$$\tan \psi \exp i\Delta = f(n_1, k_1, n, d, \lambda, \varphi)$$
(1)

where: n_1 – refractive index of substrate,

	k_2	-	absorption index of substrate,
•	n	—	refractive index of layer,
	d	—	thickness of layer,
	λ		wavelength,
	φ	—	incidence angle
the set	s of	' cu	rves illustrating the relations $\Delta(d)$ and
$\psi(d)$ as	re c	alcı	lated for given experimental values of

 $\psi(d)$ are calculated for given experimental values of λ , φ , n_1 , k_1 and reasonably accepted values of refractive indices (n) of layers. These calculations are performed on a computer. The intersection points of the curve sets $\Delta(d)$ and $\varphi(d)$ obtained in this way with the experimentally measured values of 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. These values are found from the intersection of curves $n_{\Delta}(d)$ and $n_{\psi}(d)$.

2. The determination of thickness and refractive index for the ytterbium oxide layers

A better accuracy in determining *n* and *d* for dielectric layer may be obtained for a steeper dependence of Δ and ψ upon the layer thickness. This dependence is influenced (for given refractive index (*n*) of the layer) mainly by optical constants (n_1 , k_1) of the substrate

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used. Chromium proved to be the best substrate for the vtterbium oxide layers. The chromium layers were obtained by thermal evaporation of spectrally pure chromium (produced by JMC 703) from a tungsten basket in a Edwards Vacuum Unit. The layers were condensed on a substrate from BK-7 glass at a room temperature. The substrates were cleaned chemically according to a routine method applied in the laboratory and next subjected to an ion bombardment (physical cleaning) in vacuum at a 10^{-2} Tr pressure. During the evaporation of chromium layers the vacuum was 10^{-6} Tr and the evaporation rate 20 Å/s. From the measurements of ellipsometric angles \varDelta and ψ made immediately after evaporation of layers the optical constants (n_1, k_1) of the chromium layer were calculated [9]. The opaque layers obtained in this way were employed as the substrates for the examined ytterbium oxide layers. The ytterbium oxide layers of different thickness were evaporated in the same vacuum unit by an electron gun onto unheated chromium substrates. The starting material was the spectrally pure ytterbium oxide of Ferak firm (Berlin), the evaporation rate being ~ 1 Å/s and the pressure in the vacuum unit $8-9 \cdot 10^{-5}$ Tr.

The measurements of ellipsometric angles (Δ, ψ) have been carried out with the help of an ellipsometer of EL-6 type [9] employing the ARCHER's method [10-12]. In this method the azimuth of components is fixed and amounts to $-\pi/4$. The polarizer and analyzer rotate to obtain the extinction of light reflected from the sample under test. To increase the accuracy of the azimuth determination by light extinction it is measured twice at two polarizer azimuths P_1 and P_2 differing from each other by $\pi/2$. From the angles measured at extinguishing state of analyzer (A_1, A_2) and polarizer (P_1, P_2) the ellipsometric angles (Δ and ψ) are determined in the following way

$$\psi = \frac{A_1 + (\pi - A_2)}{2},$$
 (1)

 $\Delta = P_1 + P_2 \text{ for } 0 \leqslant P_1 \leqslant \pi/2, \text{ and } \pi/2 \leqslant P_2 \leqslant \pi;$ $\Delta = P_1 + P_2 - 180 \text{ for } 3\pi/4 \leqslant P_1 \pi, \text{ and } \pi/4 \leqslant P_2 \leqslant \pi/2;$

$$\Delta = P_1 + P_2 + 180 \text{ for } \pi/2 \leqslant P_1 \leqslant 3\pi/4, \text{ and } 0 \leqslant \\ \leqslant P_2 \leqslant \pi/4.$$
 (2)

The measurements of ellipsometric angles were made for different thickness of ytterbium oxide layers deposited on chromium within the visible spectral range (0.45–0.65 μ m), and at the 70° incidence angle. The latter was measured with the accuracy of $\pm 1'$ while the accuracies of measuring the ellipsometric angles Δ and ψ achieved $\pm 3'$. The results of ellipsometric measurements for one wavelength have been collected in fig. 1. Similar curves have been obtained for the whole examined spectral range. After having performed the experimental measurements for each



Fig. 1. The results of ellipsometric measurements of Δ and ψ for thin ytterbium oxide layers on chromium ($n_1 = 2.32$, $k_1 = 2.10$) for $\psi = 70^\circ$ and $\lambda = 550$ nm

of the wavelength within spectral range 0.45–0.65 μ m, ellipsometric angles Δ and ψ have calculated theoretically from the basic equation of ellipsometry for all the assumed values of *n* and *d* of ytterbium oxide layers.

The sets of calculated relations $\Delta(d)$ and $\psi(d)$ have been shown in figs 2 and 3. The horizontal lines intersecting these sets of curves correspond to the values Δ_{exp} and ψ_{exp} measured experimentally for an ytterbium oxide-chromium set; analogical relations were drawn for other wavelengths. The relations $n_{A}(d)$ and $n_{w}(d)$ found from those graphs are shown in fig. 4. The intersection points give the sought values of the refractive index (n) and thickness (d) of the examined ytterbium oxide layers. In table the calculated values of the refractive index (n) and thickness (d) for Yb_2O_3 layers on Cr have been collected for several wavelengths. In fig. 5 an examplified dispersion $n(\lambda)$ has been shown for Yb₂O₃ layers of various thicknesses, while in fig. 6 a dependence of the refractive index upon the layer thickness is given for one wavelength of light.

As it follows from table the values of thickness calculated for different wavelengths are well consistent.



Fig. 2. Calculated sets of curves $\Delta(d)$ for three Yb₂O₃ layers. The incidence angle 70°, $\lambda = 500$ nm. The optical constants of chromium for $\lambda = 500$ nm, $n_1 = 2.11$, $k_1 = 2.10$. The horizontal layers – experimental values of Δ for the examined values of Yb₂O₃ on Cr, the numbers in the vicinity curves – the values of n



Fig. 3. The calculated sets of curves $\psi(d)$ for three Yb₂O₃ layers. The notation identical with that in fig. 2



Fig. 4. The determination of the refractive index (n) and thickness (d) of the ytterbium oxide layers

 $1 - n_{\Delta}(d)$ found from fig. 2; $2 - n_{\varphi}(d)$ found from fig. 3



Fig. 5. Dispersion $n(\lambda)$ of thin ytterbium oxide layers. 1-3 corresponds to d = 8.5, 47.3 and 80 nm $\Delta - 1$, O - 2, X - 3

The refractive index of Yb_2O_3 layer of thickness greater than 20 nm coincides with that for thick layers to 450 nm thickness [13]. However, with decreasing thickness of very thin layers the refractive index increases instead of decreasing (fig. 6). For very thin layers of a series dielectrics, such as ZnS, CdS, cryolite, the refractive index of these layers diminishes with the decreasing thickness, which indicates an increase in the porosity of the layers [6, 7, 14]. The nontypical

Table	1
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λ [nm]	n	d [nm]	n	d [nm]	n	<i>d</i> [nm]	n	d [nm]	n	d [nm]	n	d [nm]	n	<i>d</i> [nm]	n	d [nm]
450	-	- 14	120	_	2.20	8.70	-		1.89	22.70	1.84	44.60	2.12	64.00	1.86	47,20
475	2.56	3.39	2.10	7.69	2.15	7.65	1.98	12.10	1.89	23.10	1.82	44.80	1.95	76.00	1.84	48.00
500	—	_	2.04	7.81	2.08	6.62	2.03	11.00	1.83	23.90	1.81	44.70	1.85	81.80	1.82	47.90
525		-	1.93	8.12	2.02	8.05	1.87	11.90	1.84	22.40	1.83	44.10	1.86	80.40	1.83	47.50
550	2.46	2.80	1.92	7.18	2.08	8.15	1.76	14.10	1.84	23.30	1.80	45.30	1.86	80.00	1.83	47.90
575	2.45	3.21	1.68	8.50	1.90	9.25	1.62	15.50	1.79	25.30	1.81	44.70	1.82	84.00	1.82	47.30
60 0	2.23	3.62	1.81	8.94	2.02	8.80	1.62	15.65	1.84	24.20	1.85	43.40	1.85	78.20	1.85	43.40
625	2.09	3.83	1.72	11.63	1.88	10.20		-	1.81	26.70	1.81	44.50	1.80	84.6	1.82	47.20
650	1.97	4.71	1.70	9.05	2.06	8.70	1.60	17.00	1.81	26.60	1.83	44.20	1.80	85.0	1.76	49.20

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character of the dependence of n on d for Yb₂O₃ layer found in this paper may indicate the existence of an intermediate layer between Cr and Yb₂O₃



Fig. 6. Dependence of the refractive index (n) upon the thickness (d) for thin ytterbium oxide layers ($\lambda = 525$ nm)

caused by the diffusion of oxide and by formation of thin chromium oxide layer; its refractive index is being greater than that of the Yb_2O_3 layer. A similar of refractive index increase with the decrease of the layer thickness has been observed for selenium layer deposited on gold [15].

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Ellipsometric examinations ...

Эллипсометрическое исследование тонких пленок окиси иттербия на хроме

В видимой области спектра 0,45-0,65 мкм исследованы тонкие пленки окиси иттербия на хроме. Для пленок разной толщины измерены эллипсометрические углы Δ и ψ , а на их основе вычислены, эллипсометрическим методом Шеляревского, коэффицент преломления и толщина пленок окиси иттербия.

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