

Optical Properties of Aluminium

Intensity and polarization measurements of the light reflected from the surface of evaporated opaque aluminium films have been carried out. The optical constants (refractive index n and absorption coefficient k) have been determined in atmospheric air at room temperature in the wavelength region 0.4–2.5 μm .

A correction of the optical constants has been made for an assumed 20 Å thick oxide layer overcoating the aluminium film. Such oxide layer causes an increase of the value of n by 12% and the value of k by 6% in the whole spectral range.

The analysis of spectral distribution of the real and imaginary components of the complex dielectric constant enables to identify the regions of absorption of light either by free carriers or by bound electrons.

The microcharacteristics of conduction electrons give: concentration of free electrons = $8.8 \times 10^{22} \text{ cm}^{-3}$, plasma frequency = $16.8 \times 10^{15} \text{ s}^{-1}$, relaxation time = $4.2 \times 10^{-15} \text{ s}$, and the optical d.c. conductivity = $0.95 \times 10^{17} \text{ s}^{-1}$.

1. Introduction

The optical properties of aluminium have been thoroughly studied for the last several years under different experimental conditions [1–15]. From the electron theory point of view aluminium is one of the most interesting metals. This is due to the successful application of the nearly free electron model in interpreting the Fermi surface experiments [16, 17]. For practical reasons evaporated aluminium films are most frequently used as coating for front surface mirrors and for interferometry in the ultraviolet.

As aluminium is a highly oxidizable metal, it is accordingly a great task to obtain a oxide-free film, even in high vacuum [15].

The problem of preventing the oxidation of the aluminium layer may be solved by:

a) attempting to prevent the formation of that layer by evaporating and measuring in ultra high vacuum [15].

b) measuring the optical constants of the composite surface and numerical evaluating of the influence affected by the oxide layer [9, 11, 18, 19].

The aim of this study is to measure, in atmospheric air, the optical constants n , k of evaporated opaque aluminium films in order to determine:

a) the effect of the surface oxide films on the ellipsometric measurement of n and k when correcting for the oxide overcoating layer,

b) the microcharacteristics of conduction electrons and interband transitions.

2. Experimental Procedure

Sample preparation

Opaque aluminium films have been prepared by vacuum deposition [20]. In the present study evaporated samples of aluminium of initial purity 99.99% have been used (**). During evaporation the vacuum was maintained at the level of 10^{-4} – 10^{-5} mm Hg. The evaporated sample remained in the vacuum chamber as long as it was necessary to its cooling down to room temperature.

Applied methods

In the present study the optical constants have been measured with the aid of the ellipsometric Beattie method [21].

This method consists in an analysis of an elliptically polarized radiation reflected from the metallic mirror film if the linearly polarized incident beam falls under an angle Φ (in our case $\Phi = 80^\circ$). The quantities characterizing the elliptically polarized reflected beam are:

Δ — the phase difference between the two components of the electric vector measured parallelly and normally to the plane of incidence,

Ψ — azimuth of the reduced polarization.

Basing on the measured parameters the optical constants: n , k could be calculated by using PRICE'S [22] equations:

(**) The material was provided by Degussa Company (Frankfurt am Main, West Germany) in the form of wires of diameter 1.2 mm.

*) Solid-State Physics Laboratory, National Research Centre, Cairo.

$$-\operatorname{Re}(\epsilon) = \frac{2\sin^2\Phi \tan^2\Phi [\cos\Delta + \sin 2\Psi]}{\sin 2\Psi [\cos\Delta + \operatorname{cosec} 2\Psi]^2} - \tan^2\Phi, \quad (1)$$

$$\operatorname{Im}(\epsilon) = \frac{2\sin^2\Phi \tan^2\Phi \sin\Delta}{\tan 2\Psi [\cos\Delta + \operatorname{cosec} 2\Psi]^2}, \quad (2)$$

where $\operatorname{Re}(\epsilon)$, $\operatorname{Im}(\epsilon)$ are the real and imaginary components of the complex dielectric constant $\epsilon^* = (n - ik)^2$.

This method has been applied by many authors, especially for examination of alkali metals [23]. In our case the accuracy is about 5% in n and 3% in k .

3. The Effect of Surface Oxide Film on the Ellipsometric Determination of the Optical Constants of Aluminium

Oxide coatings on aluminium films have been discussed by many authors [3, 8, 9, 15, 24]. A freshly evaporated aluminium film, if exposed to atmospheric air, is immediately coated with an oxide layer. Aluminium films are covered by these oxide layers even if kept in typical vacuum. As it has been shown by HASS [7] and BENNETT [9], the oxide layer increases even above 30 Å when aluminium films are exposed to normal atmosphere. Field emission experiments [24] showed that there is an appreciable oxide coverage on a clean surface after one minute exposure to oxygen under the pressure of 3×10^{-7} mm Hg. BERNING [7] found that the oxide layer formed on aluminium exposed to atmospheric air at room temperature for one hour was 10–12 Å thick. One day later the oxide layer thickness increased to 15 Å to reach about 20 Å after ten days. Berning has also shown that the subsequent growth was very slow and ceased almost completely after one month when the oxide layer was about 22 Å thick.

The optical constants of pure aluminium films are affected by the presence of the oxide coating [15]. For example, BENNETT [9] found that the reflectance in the visible and ultraviolet regions was affected by the aluminium oxide and gas molecules trapped on the films during deposition, but neither he nor PADALKA [25] noticed any effect on the infra-red reflectance. FANE and NEAL [15] pointed out, that the phase shift Δ and the azimuth Ψ was considerably affected by the oxide layer. They have shown that the changes in n, k due small amounts of oxide are greater than those caused by variations of the incidence angle [26].

In the present study there have been applied DRUDE'S [18] and ARCHER'S [27] formulae in order to calculate the correction in Δ and Ψ , by using the

method of successive approximation. Similar methods have been used by SHKLAREVSKY [11] applying Drude's formulae. The Drude's and Archer's equations are a first order approximation. It has been supposed that they are valid for oxide layers not thicker than 50–100 Å.

In our calculations we assume that aluminium films are covered with 20 Å thick oxide layers (corresponding to 100 hours exposure to atmospheric air) with refractive index equal to 1.6 [11]. As reported by MALITSON [28] the refractive index of aluminium oxide lies between 1.834 and 1.596 in the wavelength range 0.265–5.577 μm. The results of our calculations*) restricted to the third order approximation are presented in Fig. 1. As we can see, the corrected values of the optical constants are approximately the same independently of whether DRUDE'S [18] or

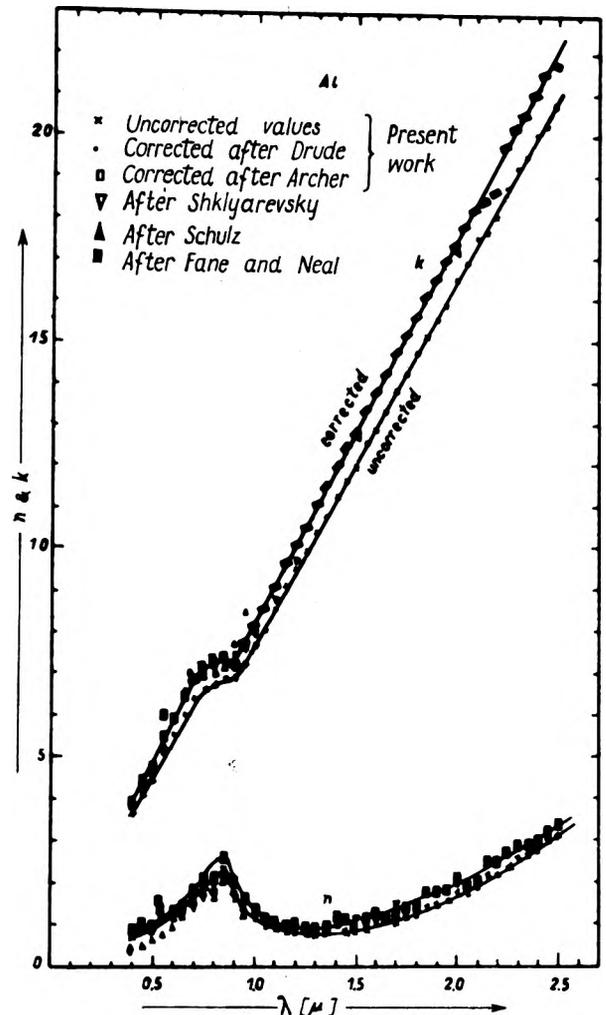


Fig. 1. Relation between the optical constants n, k and the wavelength λ for Al films without and with correction for the oxide layer

*) The calculations have been carried out in Scientific Computation Centre of the Cairo University using ICL 1905E computer.

ARCHER'S [27] formulae are employed. We have found that the correction in Δ lies between 4 and 2 degrees and the correction in Ψ between 0.5 and 0.02 degrees in the wavelength range 0.4–0.5 μm . This shows that the presence of the oxide layer leads to an increase by 12% in n and by 6% in k over most of the wavelength range. For comparative reasons there have been also presented in Fig. 1 the results obtained by SCHULZ [3], SHKLAREVSKY [12], FANE and NEAL [15] as well as MOTULEVICH [29]. We see that, our results for the corrected values of the optical constants do not differ very much from those obtained by other authors. It is worth mentioning that Fane and Neal have performed their measurements in ultrahigh vacuum at $\lambda = 0.549 \mu\text{m}$. They came to the conclusion that in aluminium, values of $n < 1.5$ and $k < 6$ are regarded as optical constants for a surface consisting of aluminium contaminated by oxide. As it can be seen from the reference to Fig. 1, the same wavelength our corrected optical constants are $n = 1.4$ and $k = 5.5$ i. e. close to the limit set by Fane and Neal as a criterion for purity. A small difference are presumably caused by preoxidation during the formation of the films in vacuum. Therefore, the correction for n, k measured in air, enables to determine values not much different from those obtained at a pressure 10^{-9} mm Hg for samples rapidly evaporated at 10^{-8} mm Hg. It appears clearly from Fig. 1 as well that the general behaviour of the curves representing both corrected and uncorrected values of n, k are the same. This means that the presence of oxide layer does not lead to any change in the qualitative analysis of the curves. In chapter IV presenting our experimental data as well as their discussion we shall refer only to the corrected values of n and k .

4. The Microcharacteristics and Quantum Absorption of Al

In order to determine the microcharacteristics and quantum absorption of aluminium the real ($-\text{Re}(\epsilon)$) and the imaginary ($\text{Im}(\epsilon)$) components of the complex dielectric constant $\epsilon = (n - ik)^2$ have to be plotted as a function of the wavelength [10, 12, 34]. DRUDE [30] deduced expressions for the refractive index and absorption coefficient as a function of frequency based on the classical free electron model of metals. In this theory the values of n, k are given in terms of the density of free electrons N_f and their relaxation time τ . As the absorption of light in metals is caused also by bound electrons [31,32], the $\text{Re}(\epsilon)$, $\text{Im}(\epsilon)$ are given as the sum of two parts related to bound and free electrons and expressed as:

$$-\text{Re}\epsilon(\omega) = k^2 - n^2 = -1 + \text{Re}^b\epsilon(\omega) + \text{Re}^f\epsilon(\omega), \quad (3)$$

$$\text{Im}\epsilon(\omega) = 2nk = \text{Im}^b\epsilon(\omega) + \text{Im}^f\epsilon(\omega), \quad (4)$$

where

$$\text{Re}^b\epsilon(\omega) = \frac{[4\pi N_b e^2/m^*](\omega_r^2 - \omega^2)}{(\omega_r^2 - \omega^2)^2 + \omega_b^2 H_b^2},$$

$$\text{Re}^f\epsilon(\omega) = \frac{4\pi N_f e^2/m^*}{\omega^2 + H_f^2},$$

$$\text{Im}^b\epsilon(\omega) = \frac{[4\pi N_b e^2/m^*] H_b \omega}{(\omega_r^2 - \omega^2)^2 + \omega_b^2 H_b^2},$$

$$\text{Im}^f\epsilon(\omega) = \frac{4\pi N_f e^2/m^*}{\omega^2 + H_f^2} \cdot \frac{H_f}{\omega},$$

where b and f are symbols for bound and free electrons respectively, H_f denotes collision frequency of electrons ($H_f = 1/\tau$), m^* is effective mass, ω_r the resonance frequency and ω the angular frequency of light used.

In the infra-red frequencies, where the free electron absorption predominates, the optical properties may be adequately described by Drude's equation. However, in the ultraviolet and visible regions the pronounced deviations from Drude's theory are likely to occur due to interband transitions.

Figure 2 presents the dependence of $\text{Re}(\epsilon)$, $\text{Im}(\epsilon)$ versus λ^2 . The plot of $(k^2 - n^2)$ against λ^2 may be

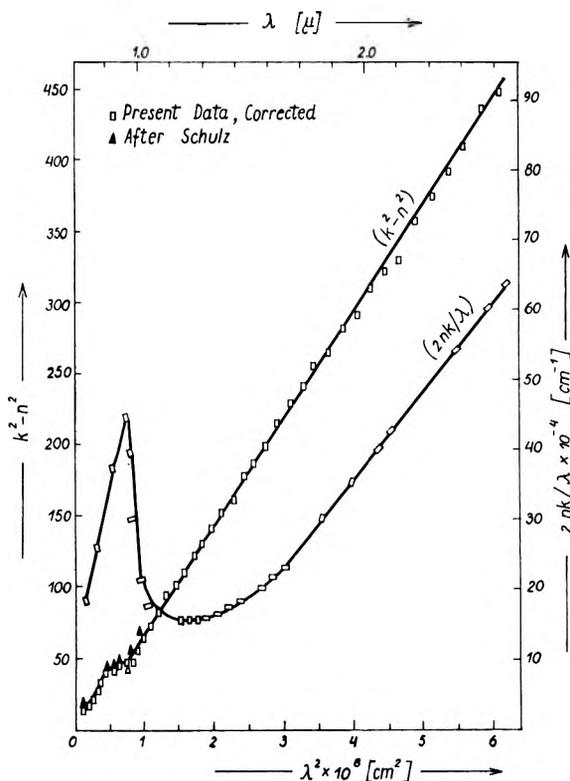


Fig. 2. Relation between $k^2 - n^2$, $2nk/\lambda$ versus λ^2 for Al with correction for the oxide layer

regarded as two parallel straight lines separated by an interband transition region from 0.7 to 1.0 μm . The existence of the nearly flat parts of the curves indicates the interband transition as it was expected by MENDLOWITZ [6]. The straight line in the long wavelength region (curve 1) satisfies the free electrons theory. So that, the observed linearity of $\text{Re}\epsilon(\omega)$ versus λ^2 may be explained satisfactorily by equation (3) (for $\omega^2\tau^2 \gg 1$). In the limiting case of extremely large $\omega\tau$ (but ω is still small enough so that we do not exhibit interband transition) condition of a free electron gas without any interaction is approached. In this case, the plasma frequency ω_p ($\omega_p = \sqrt{4\pi N_f e^2/m^*}$ -- the frequency at which free electrons in the metal oscillate collectively due to coulomb interaction) is found to be equal to 16.8×10^{15} radians s^{-1} . This value agrees with the values 16.1, 15.7 and 16×10^{15} radians per s, which are calculated from the results obtained independently by HODGSON [1], BEATTIE and CONN [4] as well as GOLOVASHKIN [5]. For the spectral range the number of free electrons/ cm^3 N_f is found to be $8.8 \times 10^{22} \text{ cm}^{-3}$ corresponding to 1.46 free electrons/atom. This value of N_f , obtained from the optical measurements, is considerably smaller than that of $18.1 \times 10^{22} \text{ cm}^{-3}$ calculated basing on free electrons atom. The density of free electron determined by MENDLOWITZ [6] was found to be equal to $14.4 \times 10^{22} \text{ cm}^{-3}$ corresponding to 2.4 free electrons/atom. This density is considerably higher than that obtained by Hodgson (1.35), Beattie and Conn (1.28) as well as Golovashkin (1.33) for evaporated and annealed films as well as MOTULEVICH [14] (1.12) for sputtered and annealed ones. Such comparison leads to the conclusion that there exists a large disagreement between the concentration of conduction electrons determined by help of optical methods and the concentration of valence electrons. Many attempts have been made in order to explain this discrepancy. It has been shown by MOTULEVICH [14] that the periodic lattice potential strongly affects the values of the optical constants of metals in the visible as well as in the infra-red regions of the spectrum. There exist three physical magnitudes which are affected by this periodical potential in the infra-red region: concentration of conduction electrons, area of Fermi surface and velocity of electrons on Fermi surface [33]. Considering the effect of lattice potential upon the microcharacteristics of the conduction electrons we cannot overlook the concept of pseudopotential [34, 35]. According to this concept, the electron does not "feel" the whole potential of the ion, but is sensitive only to a weak pseudopotential, which means that the electrons are approximately free. GURZHI [33] calculated the effect of the periodic lattice potential on

the concentration of conduction electrons determined from the optical measurements. He indicates that the difference between theoretical and experimental values of electrons concentration is determined to great extent by the periodic lattice potential.

The discrepancy between the values of N_f results from the assumption that $m = m^*$. Such explanation is due to EHRENREICH [36] who reported a value of $m^*/m = 1.5$. Theoretical calculation carried out by BURST [37], basing on Ashcroft's two parameter potential model, gives the ratio $m^*/m = 1.45$. However, the effect of interelectron interaction upon the optical constants in the infra-red region is negligible as it is restricted by Pauli principle [29]. It was reported by GINZBURG [40] that the smaller value of the free electron density may be due to electron-electron interaction and the assumed value of $m^*(m^* = m)$.

Study of the wavelength dependence of $\text{Im}(\epsilon)$ represents a great interest, as the quantity $2nk$ is proportional to the conductivity σ , and therefore, also proportional to the energy absorbed by the metal. This imaginary component of the dielectric constant is more sensitive to interband transitions. Fig. 2 (curve 2) shows the plot of $\text{Im}\epsilon(\lambda^2)$. The curve exhibits a peak at $\lambda = 0.85 \mu\text{m}$ corresponding to 1.48 eV. Beyond $1.5 \mu\text{m}$, the curve shows a continuous increase in the remaining part of the spectral range under test. This behaviour of $\text{Im}\epsilon(\lambda^2)$ in the long-wavelength region agrees with the free electron theory for $\omega^2\tau^2 \gg 1$. The peak observed at $\lambda = 0.85 \mu\text{m}$ suggests the presence of interband transition. Similar peaks have been observed by BURST [37], HUGHES [38], EHRENREICH [36] and LENHAM [39]. Burst found two peaks in the interband part of $\text{Im}\epsilon(\omega)$ at 0.5 and 1.6 eV for aluminium. The origin of these peaks has been discussed by Burst on the basis of band structure and are related to zone-boundary effects in an extended zone picture.

As mentioned by HARISON [41] and MOTULEVICH [29] there should exist interband peaks associated with Fourier coefficients of the pseudo-potential (V_g). The presence of Bragg reflections leads to the appearance of maxima in the interband conductivity as well as to the imaginary component of the complex interband dielectric constant. Aluminium is a face-centered cubic lattice, where the planes $\{111\}$ and $\{200\}$ cross the sphere of free electrons [31]. This leads to the appearance of two bands of interband conductivity. During our experiments we obtained only one band for $\text{Im}\epsilon(\omega)$ at $\lambda = 0.85 \mu\text{m}$. The other band had been predicted by BURST [37] at $\lambda = 2.45 \mu\text{m}$. In our measurements we have examined the case when $|V_{200}| > |V_{111}|$ ($|V_{200}|, |V_{111}|$ -- the Fourier components of the pseudopotential). The fact that $|V_{200}| > |V_{111}|$

results also from the Haas van Alphen effect on aluminium [31].

SHKLYAREVSKY and YAROVAYA [12] showed graphically the quantum absorption on the diagram of Brillouin zone using the representation of energy bands in aluminium given by HARISON [41]. Similar graphical representation of quantum observation has been introduced by EHRENREICH [36].

The analysis of $\text{Re}, \text{Im}\varepsilon(\omega)$ performed according to the model of free electrons and interband transitions applies for the spectral dependence of n, k (see Fig. 1). The curves have a similar behaviour as those represented by SCHLUZ [3], SHKLAREVSKY [12] and MOTULEVICH [29], where the theory of free electrons predicts a linear increase of k and a quadratic increase in n with λ (Fig. 1) does not support this prediction at short wavelengths due to the existence of interband transitions of electrons with quantum absorption of light [36]. Calculations carried out by EHRENREICH [36] show that k is slightly dependent on interband transitions. FANE and NEAL [15] have confirmed that k is in fact slightly dependent on interband transitions but n is dependent more strongly on quantum absorption.

The relaxation time τ and the d. c. conductivity σ_0

$$\sigma_0 = \lim_{\omega \rightarrow 0} \left(-\frac{2k\omega}{2\pi} \right) = N_f e^2 \tau / m^*$$

are calculated from the data presented in Fig. 2. The value of τ is found to be 4.2×10^{-15} s and the optical d. c. conductivity = 0.95×10^{17} esu. The values of τ , as determined by BEATTIE [4], MENDLOWITZ [6], HASS [8] and MADDEN [10], are 6.1, 1.2, 1.2 and 0.7×10^{-15} s⁻¹, respectively.

The value of σ_0 does not differ very much from that obtained by HODGSON [1] (0.73×10^{-17} s⁻¹). Therefore, the fitting of parameters must be such that the quantity $N_f e^2 \tau / m^*$ is approximately of the same order of magnitude for all optical measurements i.e. when N_f increases τ decreases (for the same value of m^*). The values of σ_0 are about 1/4 to 1/3 the accepted value of the electrical conductivity for the bulk material. This fact is not a surprise as we deal with surface layers whose characteristics are different from those for bulk bodies. The discrepancy may be related to an assumption that there are more than one kind of free electrons having different ratios N_f / m^* and different collision frequencies [31]. DINGLE [42] and ROBERTS [32] showed that the d. c. conductivity at the surface is less than σ_0 for the bulk material due to the surface collisions (the mean free path of electrons near the surface is shorter than that at the deeper layers).

On the basis of the Drude's theory the relaxation time is a constant value independent of λ (for $\omega^2 \tau^2 \gg 1$). Fig. 3 presents the plot of τ as a function of ω , where

$$\tau = \frac{1 - \text{Re}\varepsilon(\omega)}{\omega \text{Im}\varepsilon(\omega)}$$

It is clear that for the long wavelengths (the lowest frequencies) τ is constant (for wavelengths $\geq 2 \mu\text{m}$).

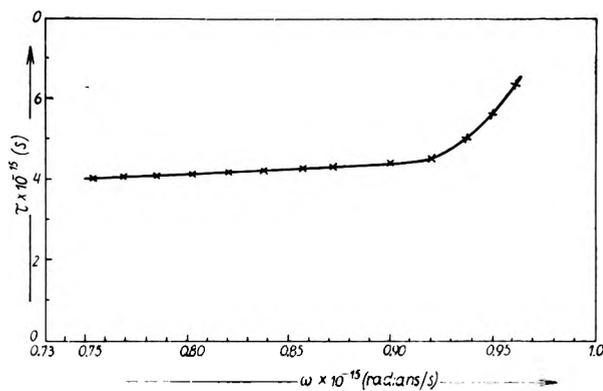


Fig. 3. Frequency dependence of relaxation time

A non-linear course of the dependence $\tau(\omega)$ starts for $\lambda < 2 \mu\text{m}$. Therefore in our particular case we are allowed to calculate τ according to Drude's theory for wavelengths $> 2 \mu\text{m}$.

5. Conclusions

1. A correction of the optical constants, measured ellipsometrically, has been made for an assumed 20 Å thick oxide layer covering the surface of opaque aluminium films. Such correction showed an increase of the value of the refractive index n by 12% and the value of the absorption coefficient k by 6% in the wavelength range 0.4–2.5 μm .

2. The qualitative analysis of wavelength dependence of the optical constants as well as of the real and imaginary components of the complex dielectric constant ε^* showed, that absorption due to free electrons is predominant in the long wavelength region. In the visible and near infra-red indications of optical transition have been noticed.

3. The microcharacteristics of conduction electrons have been determined.

4. Interband transitions have been detected by a marked peak at $\lambda = 0.85 \mu\text{m}$ (1.48 eV) in the spectral of $\text{Im}(\varepsilon)$. This agrees with the results obtained from calculations of the pseudo-optical potential in aluminium.

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Sur les propriétés optique de l'aluminium

A l'aide de la méthode de Beatti on a déterminé, dans le domaine spectral de 0,4 μm à 2,5 μm , les constantes optiques n , k des couches non-transparentes de l'aluminium évaporées thermiquement dans le vide. On a donné les microcaractéristiques des électrons de conduction, on a calculé aussi les parties réelle et imaginaire des perméabilités électriques. On a discuté l'influence de la couche superficielle sur les résultats de mesure.

Оптические свойства алюминия

Определены, по приспособленному методу Бити, оптические постоянные n , k непрозрачных алюминиевых слоев, испаряемых термически в вакууме, в области спектра 0,4–2,5 μm . Приведены микрохарактеристики электронов проводимости; рассчитаны действительная и мнимая части диэлектрической проницаемости. Обсуждено влияние поверхностного оксидного слоя на результаты измерений

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