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# DETERMINATION OF REFRACTIVE INDEX AND THICKNESS OF ANODIC OXIDE FILMS ON ALUMINIUM BY A GALVANOLUMINESCENT METHOD

LJUBIŠA D. ZEKOVIĆ[1,2], VLADETA V. UROŠEVIĆ[1], BRANISLAV R. JOVANIĆ[1], BRATIMIR PANIĆ[1] and ALEKSANDAR ŽIKIĆ[2]

*1. Institute of Physics, P. O. Box 57, 11001 Beograd, Yugoslavia*

*2. Department of Physics, Faculty of Sciences, P. O. Box 550, 11001 Beograd, Yugoslavia*

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In this work a galvanoluminescent method is described which makes possible determination of both thickness (or growth rate) and refractive index of thin oxide films formed by anodic oxidation of valve metals. The method is based on the analysis of positions of interference maxima which appear during the anodization on $I_\lambda(t)$ curves when the emitted radiation is detected both normal and parallel to the oxide film surface within a small ($\alpha \leqq 5°$) acceptance angle. For anodic alumina formed in 0.1 M oxalic acid at constant current density ($j = 12.5$ mA/cm$^2$) and temperature ($T = 300.6$ K), the refractive index $n_2 = 1.63 \pm 0.02$ (mean value for wavelength interval 400—580 nm) has been obtained in a rather good agreement with Huber and Gaugler ($1.65 \pm 0.02$). The value of anodizing rate $a = (0.468 \pm 0.005)$ nm/s/mA/cm$^2$) is about 10% smaller than the results obtained by Golubiev and Diggle et al. under rather different anodizing conditions.

## *1. Introduction*

Galvanoluminescence (GL), i. e. the emission of optical radiation during anodic oxidation of valve metals (Al, Ta, Ti, Si, etc.), is a well known phenomenon (see,

This work is devoted to the memory of our friend and teacher, late Professor Aleksandar B. Milojević whose permanent interest in the development of original experimental and measurement methods was a great challenge to us all.

for instance, review articles by Tajima[1] and Ikonopisov[2]). In our previous papers we demonstrated that spectroscopic and time resolved investigation of GL shows a well pronounced interference effect which can be used for determination of oxide film thickness $d$ if the refractive index of the oxide $n_2$ is known, or *vice versa*. The precise knowledge of refractive index is also needed for many other purposes, but the corresponding data are not always available. Moreover, the values of $n_2$ can be influenced by preparation conditions (purity and previous treatment of valve metal, electrolyte composition and temperature, forming voltage, duration of anodizing process, etc.). So, it is desirable to measure $d$ and $n_2$ in the same experiment.

In this work we used a combination of two interference methods described in Refs. 3 and 4 for the investigation of barrier oxide films in order to perform independent measurements of both thickness and refractive index of porous anodic alumina under the same experimental conditions.

## 2. *Experimental*

Pure aluminium foils (99.99%) have been annealed at 350°C for 5 hours, slowly cooled and then electropolished in a perchilorc acid-ethanol mixture[5], DC anodization at constant current density ($j = 12.5$ mA/cm$^2$) has been performed in 0.1 M oxalic acid solution at $T = (300.6 \pm 0.5)$ K, using a platinum wire as a cathode. Under the above conditions, a porous oxide film is formed, its thickness $d$ being linear function of time. This film consists of a thin barrier layer near the Al surface, with thickness $x_0$ which depends upon the chosen $j = 12.5$ mA/cm$^2$ (corresponding to the forming voltage $U_f = 51.7$ V) and a much thicker porous layer ($d - x_0$), with thickness depending upon anodizing time. Detailed investigation of the film structure, which is schematically presented in Fig. 1, is given in Refs. 6 and 7.

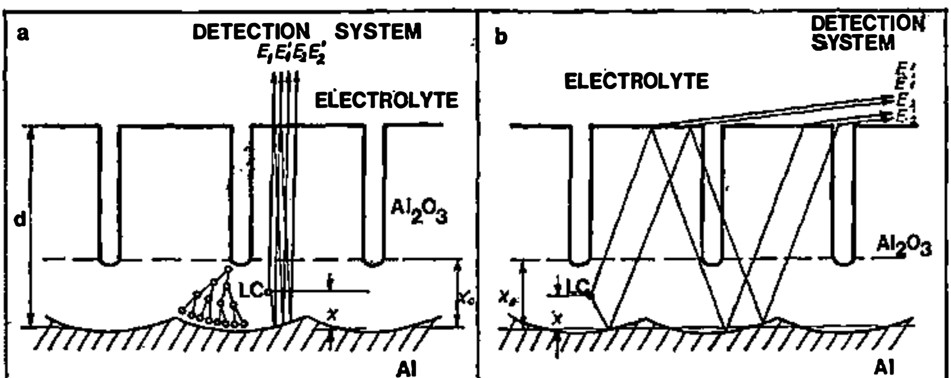


Fig. 1. Multiple reflections of galvanoluminescent radiation emitted from porous oxide film
a. The axis of detection system is *normal* to the oxide film surface (case $A$).
b. The axis of detection system is nearly parallel to the oxide film surface (case $B$).

During the anodization, the cell voltage $U$ whose stationary value $U_f$ depends upon $j$ and the spectral intensity of emitted light (at a chosen wavelength near the emission maximum*) $I_\lambda$ have been plotted vs. time. For $I_\lambda(t)$ measurements we used a sensitive detection system consisting of a quartz achromate, Zeiss SPM-2 optical monochromator and a photon counter (cooled RCA 31034 A photomultiplier, ORTEC counting electronics and a multiscaler).

The anodized Al surface has been positioned either normal (case A), or parallel (case B) to the axis of the optical system. The acceptance angle of the condensor was about 5° and spectral resolution $\Delta\lambda \approx 2$ nm.

## 3. *The principle of the method*

It is generally assumed[9] that GL is excited by collision of electrons, injected into the oxide film at the electrolyte-oxide interface and accelerated by intense electric field ($10^6$—$10^7$ V/cm), with luminescent centra inside the film. In porous films, the light is emitted from the barrier layer (as shown in Fig. 1), i. e. at the bottom of the pores, in the high field region. Regardless of the nature of luminescent centra and their spatial distribution in the film, there is an interference between monochromatic rays reflected at the metal-oxide and oxide-electrolyte interfaces[3,4]. Due to this effect, some interference maxima and minima at $I_\lambda(t)$ curves appear, the positions of which depend upon $d$ and $n$ (at given $\lambda$). The shape of these maxima depends upon the geometry of measurement.

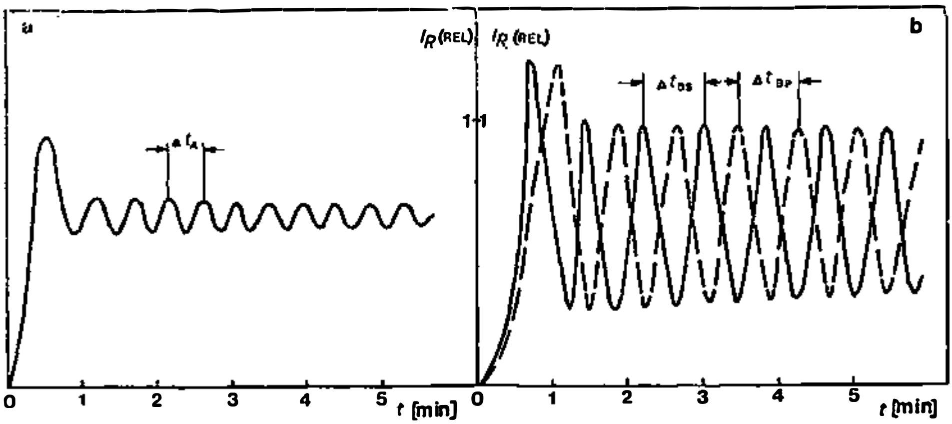


Fig. 2. The dependence of spectral intensity $I_R$ (relative units) on anodization time $t$ ($c = 0.1$ mol/dm$^3$ oxalic acid; $j = 12.5$ mA/cm$^2$; $\lambda = 500$ nm).
a) Case *A*.
b) Case *B*; full line — *s* component; broken line — *p* component.

### 3.1. *Case A*

In case A (detected radiation is nearly normal to the oxide film surface) the ray paths are presented in Fig. 1A and $I_\lambda(t)$ curves in Fig. 2A. The obtained

* In this case, neglecting the interference effect, the emitted spectrum consists of a broad maximum centered at about 440—480 nm[8].

interference patterns can be explained as follows: An excited luminescent centrum at distance $x$ from the metal-oxide interface emits the ray $E_1$ (in direction of the detector) and the ray $E_1'$ (in opposite direction) within the acceptance angle of detecting system ($\alpha \approx 5°$). Owing to the reflection at the oxide-electrolyte interface and further reflection at the metal-oxide interface, instead of the primary $E_1$ ray we obtained a set of rays:

$$E_1 = (1 - r) E_0 \exp(\mathrm{i}\,\omega\, t)$$

$$E_2 = (1 - r)(r R) E_0 \exp\left[\mathrm{i}\left(\omega t + \pi - \varphi - \frac{4\pi n_2 d}{\lambda}\right)\right] \tag{1}$$

$$E_3 = (1 - r)(r R)^2 E_0 \exp\left\{\mathrm{i}\left(\omega t + 2(\pi - \varphi) - \frac{8\pi n_2 d}{\lambda}\right)\right\}$$

$$E_{p+1} = (1 - r)(r R)^p E_0 \exp\left\{\mathrm{i}\left[\omega t + p(\pi - \varphi) - \frac{4\pi n_2 d}{\lambda} p\right]\right\}$$

where:

$E_0$ — the amplitude of the emitted radiation
$r$ — reflection coefficient at the oxide-electrolyte interface, given by

$$r = \frac{n_2 - n_1}{n_2 + n_1} \approx 0.112 \tag{2}$$

$n_2$ being the refractive index of the oxide film ($n_2 \approx 1.65$)[10,11], and $n_1$ — the refractive index of the electrolyte ($n_1 = 1.3336$, as measured with Abbé refractometer at $\lambda = 590$ nm) $R$ — reflection coefficient at the metal-oxide interface, given by Fresnel formula:

$$R = \sqrt{\frac{(n - n_2)^2 - k^2}{(n - n_2)^2 + k^2}} \approx 0.92 - 0.95, \tag{3}$$

$n$ and $k$ being the real and imaginary parts of the complex refractive index of aluminium ($n^* = n - \mathrm{i}k$), respectively. In this work, for $n$ and $k$ the values obtained in Hass[12] experiment have been used.

The phase shift at the metal-oxide interface is given by $(\pi - \varphi)$, where

$$\varphi = \mathrm{arctg}\left(\frac{2 n_2 k}{n^2 + k^2 - n_2^2}\right). \tag{4}$$

By summation one obtains:

$$E = \lim_{p \to \infty} \sum_{i=1}^{p+1} E_i = \frac{(1 - r) E_0 \exp(\mathrm{i}\omega t)}{1 - r R \exp\left(\pi - \varphi - \frac{4\pi n_2 d}{\lambda}\right)}. \tag{5}$$

Owing to the multiple reflection at the metal-oxide and oxide-electrolyte interface from the ray $E_1'$ we have:

$$E_1' = E_1 R \exp\left[\mathrm{i}\left(\pi - \varphi - \frac{2\pi n_2}{\lambda} \cdot 2x\right)\right]$$

$$E_2' = E_1' (r R) \exp\left[\mathrm{i}\left(\pi - \varphi - \frac{4\pi n_2 d}{\lambda}\right)\right] \tag{6}$$

$$E_3' = E_1' (r R)^2 \exp\left[2\mathrm{i}\left(\pi - \varphi - \frac{4\pi n_2 d}{\lambda}\right)\right]$$

$$E_{p+1}' = E_1' (r R)^p \exp\left[p\mathrm{i}\left(\pi - \varphi - \frac{4\pi n_2 d}{\lambda}\right)\right]$$

and after summation:

$$E' = \lim_{p\to\infty} \sum_{i=1}^{p+1} E_i' = \frac{(1-r) E_0 R \exp\left[\mathrm{i}\left(\omega t + \pi - \varphi - \frac{4\pi n_2 x}{\lambda}\right)\right]}{1 - (r R) \exp\left[\mathrm{i}\left(\pi - \varphi - \frac{4\pi n_2 d}{\lambda}\right)\right]}. \tag{7}$$

The total amplitude of radiation coming from a luminescent centrum at the distance $x$ from the *metal-oxide* interface is:

$$E_t = E + E' = \frac{(1-r) E_0 \exp(\mathrm{i}\omega t)\left\{1 + R\exp\left[\mathrm{i}\left(\pi - \varphi - \frac{4\pi n_2 x}{\lambda}\right)\right]\right\}}{1 - r R \exp\left[\mathrm{i}\left(\pi - \varphi - \frac{4\pi n_2 d}{\lambda}\right)\right]}, \tag{8}$$

and the corresponding measured spectral intensity is proportional to the square of the amplitude:

$$I_\lambda(x) \approx E_t \cdot E_t^* \tag{9}$$

i. e.

$$I_\lambda(x) \approx \frac{1 + R^2 - 2R\cos\left(\varphi + \frac{4\pi n_2 x}{\lambda}\right)}{1 + (r \cdot R)^2 + 2r R\cos\left(\varphi + \frac{4\pi n_2 d}{\lambda}\right)}. \tag{10}$$

The total measured spectral intensity from the oxide film as a whole can be obtained by integration of relation (10):

$$I_\lambda \approx \frac{\int_0^{x_a} I_{0,\lambda} \cdot n_c(x)\left[1 + R^2 - 2R\cos\left(\varphi + \frac{4\pi n_2 x}{\lambda}\right)\right] \mathrm{d}x}{1 + (r \cdot R)^2 + 2r R\cos\left(\varphi + \frac{4\pi n_2 d}{\lambda}\right)} \tag{11}$$

where:

$x_a$ — the mean range of electron avalanches by which the luminescent centra are excited ($x_a \leq d$),

$n_c(x)$ — the spatial distribution of luminescence, which includes spatial distribution of luminescent centra, spatial and energy distribution of avalanche electrons, etc.,

$I_{0,\lambda}$ — the initial spectral intensity of luminescent radiation (without interference effect).

In this experiment we are not interested in the determination of absolute values of spectral intensities emitted by the oxide film, but only in their relative values as a function of anodizing time $t$ or oxide film thickness $d$ ($d \sim t$). As shown in Fig. 2A, the curves $I_\lambda(t)$ exhibit some interference maxima with positions (according to Eq. (11)) corresponding to the film thicknesses $d_m$:

$$\varphi + \frac{4\pi n_2 d_m}{\lambda} = (2m + 1)\pi; \quad m = 0, 1, 2, .... \tag{12}$$

For $m$-th and $(m + f)$-th maxima we have:

$$d_{(m+f)} - d_m = \frac{f \cdot \lambda}{2n_2}. \tag{13}$$

Taking into account the proportionality between the film thickness $d$ and anodizing time $t$ in oxalic acid[13]:

$$d = a j t \tag{14}$$

where $a$ is the oxide film growth rate and $j$ is the current density (which is constant in our experiment), we can write:

$$a = \frac{\lambda}{2n_2 j} \cdot \frac{f}{(t_{(m+f)} - t_m)_A} = \frac{\lambda}{2n_2 j \Delta t_A} \tag{15}$$

where index $A$ corresponds to case $A$.

From the curves $I_\lambda(t)$ (as presented in Fig. 2A) and from Eq. (15) we can determine either the film growth rate $a$, or its refractive index $n_2$ (for different wavelengths $\lambda$) .

### 3.2. *Case B*

In case $B$ (the detected radiation is nearly parallel to the oxide film surface, i. e. the angle of incidence at the oxide-electrolyte interface is nearly equal to the total reflection angle) the ray parths are presented in Fig. 1B, and the shape of $I_\lambda(t)$ curves in Fig. 2B. In this case a Lummer-Gehrke type of interference takes place leading to the separation of normal ($s$) and parallel ($p$) components of the electric field vector, as shown in our previous works[14]. Using a similar analytic procedure as in case $A$, we obtain:

$$I_{\lambda,s} \approx \frac{\int_0^{x_a} I_{0,\lambda}\, n_c(x)\, [1 + R_s^2 - 2R_s \cos(\varphi_s + K_s x)]\, \mathrm{d}x}{1 + (r_s R_s)^2 + 2r_s R_s \cos(\varphi_s + K_s \cdot d)} \tag{16}$$

for normal ($s$) component, and

$$I_{\lambda,p} \approx \frac{\int_0^{x_a} I_{0,\lambda}\, n_c(x)\, [1 + R_p^2 - 2R_p \cos(\varphi_p + K_p \cdot x)]\, \mathrm{d}x}{1 + (r_p R_p)^2 + 2r_p R_p \cos(\pi + \varphi_p + K_p d)} \tag{17}$$

for parallel ($p$) component, where:

$$K_s = \frac{4\pi}{\lambda}\sqrt{n_{2s}^2 - n_1^2}; \qquad K_p = \frac{4\pi}{\lambda}\sqrt{n_{2p}^2 - n_1^2}. \tag{18}$$

Contrary to case $A$, the reflection coefficients at the oxide-electrolyte interface are close to unity ($r_s \approx r_p \approx 1$), and the interference maxima are very sharp for both components. There is also a difference in phase shifts between the two components: the reflection at the oxide-electrolyte interface does not change the phase of $s$ component, while for $p$ component the phase shift is equal to $\pi$.

Regardless of the shape of function $n_c(x)$, the maxima of $I_\lambda$ curves appear at:

$$K_s d_m + \varphi_s = (2m + 1)\pi; \qquad m = 0, 1, 2 \ldots \tag{19}$$

for $s$ component, and

$$K_p \cdot d_m + \varphi_p + \pi = (2m + 1)\pi; \qquad m = 0, 1, 2 \ldots \tag{20}$$

for $p$ component.

Using a similar procedure as in case $B$, we can obtain the oxide film growth rate $a$:

$$a = \frac{\lambda}{2j\sqrt{n_{2s}^2 - n_1^2} \cdot \Delta t_{sB}} = \frac{\lambda}{2j\sqrt{n_{2p}^2 - n_1^2} \cdot \Delta t_{pB}} \tag{21}$$

where $\Delta t_s$ and $\Delta t_p$ are time intervals between two successive maxima in $I_{\lambda,s}(t)$ and $I_{\lambda,p}(t)$ curves, respectively.

In our experiment we obtained $\Delta t_{sB} \approx \Delta t_{pB} \approx \Delta t_B$, which means that the investigated oxide films are nearly isotropic in the investigated spectral range 360—580 nm.

Hence,

$$K_s = K_p$$

$$n_{2s} = n_{2p} = n_2$$

$$a = \frac{\lambda}{2j\,\Delta t_B \sqrt{n_2^2 - n_1^2}}. \tag{22}$$

3.3. *Final formulae*

By measuring $I_\lambda(t)$ curves for both geometries ($A$ and $B$), at some $\lambda$ and $j$, and using relations (14), (15) and (22), it is possible to obtain the following formulae for $n_{2,\lambda}$, $a$ and $d$:

$$n_{2,\lambda} = \frac{\Delta t_B}{\sqrt{\Delta t_B^2 - \Delta t_A^2}} \cdot n_1 \tag{23}$$

$$a = \frac{\lambda \sqrt{\Delta t_B^2 - \Delta t_A^2}}{2 n_1 j\,(\Delta t_A \cdot \Delta t_B)} \;[\mathrm{nm/s(mA/cm^2)}] \tag{24}$$

$$d = \frac{\lambda \sqrt{\Delta t_B^2 - \Delta t_A^2}}{2 n_1 \,(\Delta t_A \cdot \Delta t_B)} \cdot t \;[\mathrm{nm}] \tag{25}$$

where $\lambda$ is given in nm and $j$ in $\mathrm{mA/cm^2}$.

## 4. *Experimental results and discussion*

A set of curves $I_{\lambda,A}(t)$ and $I_{\lambda,B}(t)$ has been measured at $\lambda = 360$; 400; 420; 440; 460; 480; 512.5; 550 and 580 nm, $j = 12.5\ \mathrm{mA/cm^2}$, $T = 27.5 \pm 0.5$°C, using for each measurement a new Al foil. Two of these curves are presented in Fig. 2, as examples.

The mean $\Delta t_A$ and $\Delta t_B$ values, determined from the distance between several (7—10) successive maxima at $I_\lambda(t)$ curves have been presented in Fig. 3 as a function of the emission wavelength $\lambda$. These results have been used for the calculation of refractive index $n_2(\lambda)$, relation (23), and anodizing rate $a$, relation (24), which are presented in Table 1. As refractive index of the electrolyte $n_1$ we used the value $n_1 = 1.336$ determined by Abeé refractometer at 590 nm.

As expected, the anodizing rate $a$ does not vary with measuring wavelength $\lambda$, and the experimental error (~2%) is mainly due to the variation of the samples area, i. e. current density $j$ (at constant current $i$). The mean value $\bar{a} = 0.486 \pm$ $\pm\, 0.006\ \mathrm{nm/(mA/cm^2)} \cdot \mathrm{s}$ is about 10% smaller than the results given by Golubiev[15)] and Diggle et al.[13)] which are obtained by a different measuring method and under rather different anodizing conditions. Our mean value of refractive

TABLE 1.

| $\lambda$/nm | $\Delta t_A$/s | $\Delta t_B$/s | $n_2^*$ | $a$/nm (smA/cm$^2$)$^{-1}$ |
|---|---|---|---|---|
| 360 | 18.75 | 32.5 | 1.636 | 0.470 |
| 400 | 20.55 | 36.1 | 1.622 | 0.480 |
| 420 | 21.95 | 38.0 | 1.637 | 0.468 |
| 440 | 23.4 | 40.1 | 1.645 | 0.458 |
| 460 | 24.4 | 42.1 | 1.639 | 0.461 |
| 480 | 25.6 | 44.3 | 1.637 | 0.460 |
| 512.5 | 27.2 | 48.0 | 1.621 | 0.466 |
| 550 | 28.8 | 51.3 | 1.614 | 0.474 |
| 580 | 30.5 | 54.5 | 1.612 | 0.473 |
| | | | $\bar{n}_2 = 1.63 \pm 0.01$ | $\bar{a} = 0.468 \pm 0.006$ |

Experimentally measured mean distances between two successive maxima ($\Delta t_A$, $\Delta t_B$), refractive indices ($n_2$) and anodizing rates ($a$) at different wavelengths ($\lambda$).

* This are »effective« $n_2$ values for a film formed at $j = 12.5$ mA/cm$^2$, corresponding to a fixed porosity. The dependence of $n_2$ on film porosity is discussed in Ref. 14.

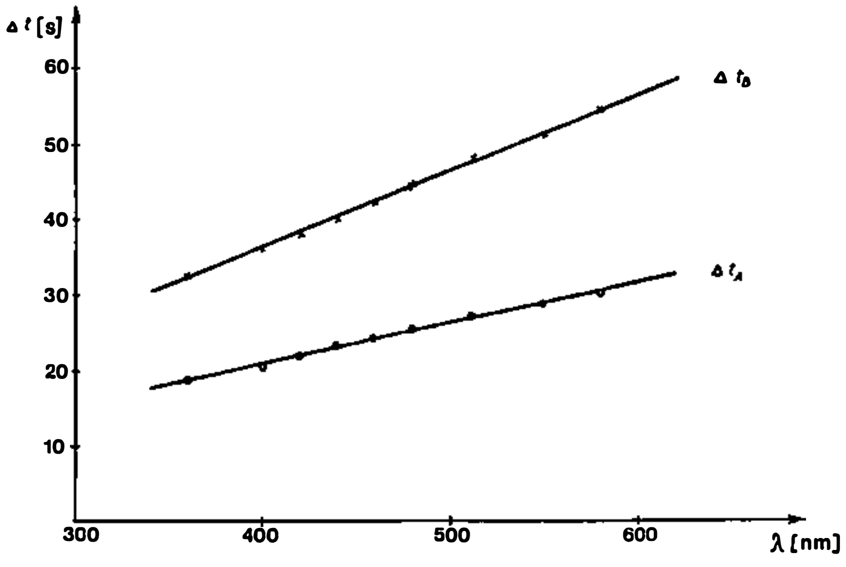


Fig. 3. The dependence of mean time interval between two successive interference maxima; $\Delta t_A$ — case $A$; $\Delta t_B$ — case $B$.

index is $\bar{n} = 1.63$. It seems that there is a decrease of $n_2$ with the increase of $\lambda$ for $\lambda > 500$ nm. For the barrier oxide films formed on aluminium, the published data[16–19] are scattered between $n = 1.53$ and $n = 1.68$. For porous oxide films on aluminium, which were investigated in this work, Huber and Gaugler[10,11] gave $n = 1.65$ for a film formed in 2% oxalic acid solution at $U = 48$ V. We estimate that the error of our $n_2$ measurement is 1.0—1.5%, mainly do to the fact, that at a given wavelength we measured $\Delta t_A$ and $\Delta t_B$ with two different samples with possible different effective areas $A_1$ and $A_2$, i. e. different current densities $j_1$ and $j_2$ at fixed current $i$.

It can be shown that in this case, instead of relation (23), one has:

$$n_{2,\lambda} = \frac{\Delta t_B}{\sqrt{\Delta t_B^2 - (j_1/j_2)^2 \cdot \Delta t_A^2}} \cdot n_1 \tag{26}$$

and, by comparing (26) and (23)

$$\frac{n_2\,(j_1 \neq j_2)}{n_2\,(j_1 = j_2)} = \sqrt{\frac{\Delta t_B^2 - \Delta t_A^2}{\Delta t_B^2 - \left(\frac{j_1}{j_2} \cdot \Delta t_A\right)^2}}. \tag{27}$$

A realistic estimation of $j_1/j_2$ ratio in our case is about 1.02 which gives:

$$\frac{n_2\,(j_1 \neq j_2)}{n_2\,(j_1 = j_2)} \approx 1.01.$$

This additional error can be avoided by a more carreful determination of the sample area, or (better) by using the same sample of simultaneous $\Delta t_A$ and $\Delta t_B$ measurement. An experimental apparatus which makes possible such measurement is under construction.

## 5. *Conclusion*

It is shown that the light emitted during the anodization of valve metals can be used for precise determination of both thickness (or growth rate) and refractive index of anodic oxide films. Both quantities can be measured within $\pm 1\%$ (or better, with some modification of the described method). For routine measurements much simpler apparatus, consisting of a narrow band interference filter, photomultiplier and an $X - t$ recorder, can be used.

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# ODREĐIVANJE INDEKSA PRELAMANJA I DEBLJINE OKSIDNOG SLOJA NA ALUMINIJUMU GALVANOLUMINISCENTNOM METODOM

LJUBIŠA ZEKOVIĆ[1,2], VLADETA UROŠEVIĆ[1], BRANISLAV JOVANIĆ[1], BRATIMIR PANIĆ[1] i ALEKSANDAR ŽIKIĆ[2]

*1. Institut za fiziku, P.O.B. Box 57, 11001 Beograd*

*2. Prirodno-matematički fakultet, Fizički fakultet, P. O. Box 550, 11001 Beograd*

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U radu je opisana galvanoluminiscentna metoda koja omogućava istovremeno (nezavisno) određivanje debljine (brzine rasta) i indeksa prelamanja tankih oksidnih slojeva dobijenih anodnom oksidacijom ventilnih metala. Metoda se zasniva na analizi položaja interferentnih maksimuma radijacije $I(\lambda)$ emitovane tokom anodizacije u pravcu normalnom i paralelnom površini oksidnog sloja. Za anodnu aluminu formiranu u 0,1 M oksalnoj kiselini ($T = 300{,}6$ K, $j = 12{,}5$ mA/cm$^2$) dobijena vrednost za indeks prelamanja sloja $n_2 = 1{,}63 \pm 0{,}02$ (srednja vrednost u intervalu talasnih dužina od 400 nm do 580 nm) se dobro slaže sa Huber-ovim i Gaugler-ovim merenjima ($n_2 = 1{,}65 \pm 0{,}02$). Dobijena vrednost za brzinu anodizacije $a = (0{,}486 \pm 0{,}005)$ nm/s/mA/cm$^2$ je oko 10% manja od merenja Golubijeva i Digglea pod nešto različitim uslovima anodizacije.