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Thickness-dependent iridescence of one-dimensional photonic crystals based on anodic alumina



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HIGHLIGHTS

GRAPHICAL ABSTRACT

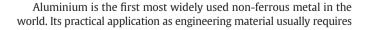
- Iridescent anodic alumina coatings with sharp photonic bandgaps were designed by cyclic anodizing of aluminium.
- Enhancement of the number of anodization cycles leads to both a blue shift and the increase in colour depth.
- The effective refractive index of anodic alumina lies in the range from 1.48 to 1.59.
- At the stop band of anodic alumina films, transmittance below 1% and reflectance above 80% were achieved.
- Alumina coatings with >150 structure periods show deep colours suitable for aluminium finishing.

ARTICLE INFO

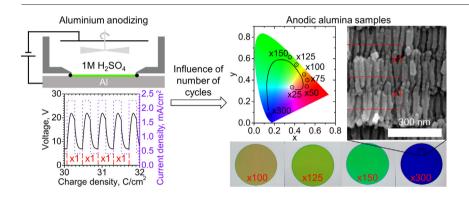
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1. Introduction



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ABSTRACT

One-dimensional photonic crystals based on anodic alumina are of high practical importance in modern materials science and photonics owing to wide variety of their applications: from optoelectronics to decorative coatings. Although the variety of anodizing regimes allows one to design the periodic modulation of the porosity of oxide film, the information about the optimal number of layers needed for the appearance of intense photonic band gaps is absent. Here the variation in the spectral properties of one-dimensional photonic crystals on the thickness of porous anodic alumina is examined. The samples consisted of up to 300 periods are obtained by anodizing of aluminium under square-wave current profile in sulfuric acid electrolyte. Fabry–Pérot optical interference of the porous film at different incident angles is used for the calculation of the wavelength dispersion of the effective refractive index of anodic alumina. The influence of the number of layers in anodic alumina photonic crystals on the intensity of photonic band gap and colour of oxide coating is discussed.

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anodizing of the metal surface to increase corrosion durability and hardness, and to offer a decorative finish with desired colour. Four most important methods of aluminium surface colouration are: (i) dyeing of porous anodic alumina, (ii) appearance of the colour during anodizing due to the constituents of the alloy (self colouring) or due to the inclusions of the electrolyte species into oxide (integral colouring), (iii) electrolytic colouring based on electrodeposition of metals at the pore bases, and (iv) interference colouring.

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In 2007 the possibility of the creation of layered porous anodic alumina structures possessing photonic band gaps (1D photonic crystals) was demonstrated [1]. If a photonic band gap lies in optical wavelength region, the 1D photonic crystal becomes coloured. It is worth noting, that in this case the colour of the material surface depends on the observation angle. Hence, 1D photonic crystals based on anodic aluminium oxide (AAO) can be considered as novel and advanced coating for the decoration of aluminium alloys. In comparison with alternative methods of preparation of photonic crystal coatings [2–6], anodizing is a simple, highly reproducible, and easily scalable approach, which allows one to design the infinite variety of photonic crystal structures just by variation of current and/or voltage profiles.

In order to obtain 1D photonic crystals based on anodic alumina, periodic modulation of anodizing conditions should be applied. The broader the range of anodizing conditions changing is, the greater is the modulation of porosity and the effective refractive index within one period of the AAO structure. On the other hand, rapid increase in voltage/current can result in electrical breakdown of a barrier oxide layer and non-uniform formation of AAO film, whereas rapid decrease in voltage can lead to the termination of pore growth. A great number of voltage-time or current-time transients have been offered to maintain continuous growth of oxide layer with modulated porosity along the surface normal: sinusoidal increase and linear decrease in voltage [1,7–9], square-wave current profile [10], step-like increase and linear decrease in current [11], periodic current signal with a sine wave [12,13], sinusoidal pulse mode with variable amplitude [14], superposition of sine waves of current [15], linear rising and falling of voltage [16,17].

Spectral position and width of a photonic band gap are determined by period and refractive index contrast, respectively. These parameters of porous AAO can be easily tuned by anodizing conditions (e.g. the electric charge spent for the formation of one period of AAO structure, electrolyte composition, temperature, and anodizing voltage) and by pore widening using post-anodizing chemical treatment [1,7,13,16,17]. In order to achieve high reflectance in a photonic band gap, a large number of anodizing cycles are usually applied for the formation of 1D photonic crystals. To the best of our knowledge, the quantitative data on the influence of the number of AAO layers on the parameters of photonic band gaps are still absent. The increase in the intensity of the photonic band gap for as-prepared AAO structures with the number of periods has been demonstrated in Ref. [17], but only two photonic crystals consisted of 50 and 150 periods have been studied. On the other hand, the thicker sample exhibited lower transmittance due to light scattering on the irregular interfaces inside the layered AAO structure.

Anodic alumina films with modulated porosity along the surface normal have been used for the development of various optical elements, such as bandpass filters [11,13,14,18], sensors [12,19], and encoders [15].

Here the spectral properties of one-dimensional photonic crystals based on porous anodic alumina films with different thicknesses have been studied. Reflectance and transmittance spectra for the samples with up to 300 periods have been collected and analysed for various incident angles. Fabry–Pérot optical interference has been used for the calculation of wavelength dispersion of effective refractive index of anodic alumina. It has been demonstrated, that the number of periods in AAO photonic crystals has strong influence on intensity, width and position of the photonic band gaps, as well as on a background intensity.

2. Experimental

2.1. Samples preparation

High-purity aluminium foil (99.99%, 0.1 mm thick) was used as the starting material. Before anodizing Al foils were electrochemically polished in solution containing 880 ml/l H_3PO_4 (IREA 2000, 85%) and 185 g/l CrO₃ (Vecton, 99.7%) at 80 °C to a mirror finish. Electropolishing

was carried out in an impulse mode. The aluminium electrodes were polarized 40 times for 3 s at anodic current density of 0.4 A/cm². Delay time between pulses was 40 s.

1D photonic crystals were obtained by anodizing of Al foils in 1.0 M H_2SO_4 under square-wave current oscillation (see Table 1). Electrolyte was continuously stirred and the environment temperature was kept constant at 2 \pm 1 °C during anodizing. Anodizing area (5.45 \pm 0.05 cm²) was restricted by o-ring with the internal diameter of 27 mm. The initial charge of low current stage (2.25 \pm 0.05 mA) and of high current stage (12.35 \pm 0.05 mA) was 1.2 C for all samples. The charge of each subsequent anodizing cycle was reduced by 0.1% to compensate the effect of chemical etching of upper porous oxide layers in acidic electrolyte [10].

After anodizing, porous oxide films were washed repeatedly in deionized water and then dried in air. The residual Al substrate was dissolved in the centre of the samples using a mixture of bromine with methanol (1:10 by volume).

2.2. Samples characterization

Morphology characterization of the porous oxide films was carried out using the field-emission scanning electron microscopes Leo Supra 50VP and Carl Zeiss NVision 40. Samples before SEM investigations were covered with a 5 nm thick conductive layer of chromium using a Quorum Technologies Q150T ES sputter coater.

Optical properties of the samples were analysed using Perkin Elmer Lambda 950 spectrometer in the range from 200 to 890 nm with the slit width of 4 nm. Reflectance spectra were collected at incident angles from 8 to 68°. The spot size at the sample position of $4 \times 2.5 \text{ mm}^2$ was used. The transmittance spectra were recorded at normal incidence (0°) of light on AAO film surface using integrating sphere. The sample was placed in the focal plane.

3. Results and discussions

3.1. Morphology

It is well known that anodizing voltage, at least under steady-state conditions, determines many of geometrical parameters of anodic oxides of valve metals, such as the thickness of the barrier layer, the interpore distance and the pore diameter at the pore basis [20,21]. Thus, information about the voltage on the cell during anodizing process is of high importance. Fig. 1a shows a part of square-wave current oscillation profile applied during aluminium anodizing and corresponding variation of the measured voltage. Anodizing voltage changes periodically due to the periodic alternation of current density between low (0.41 mA/cm²) and high (2.27 mA/cm²) values.

It is worth noting that measured voltage-charge profile is more complex than the applied square-wave current-charge profile. After steplike increase in current density, the anodizing voltage starts to increase linearly, similar to the behaviour observed usually at the initial stage of galvanostatic anodizing [22]. Then, at the stage of constant high current

Table I	
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Processing parameters.							
Sample	<i>I</i> ₁ , mA	Q ₁ , C	<i>I</i> ₂ , mA	Q ₂ , C	Number of cycles	Duration of anodizing, min	
$\times 1$	2.25 ± 0.05	1.2	12.35 ± 0.05	1.2	1	11	
$\times 25$					25	264	
$\times 50$					50	518	
$\times 75$					75	764	
×100					100	1007	
×125					125	1255	
×150					150	1530	
×300					300	2736	

 I_1 – current on the 1st stage, I_2 – current on the 2nd stage, Q_1 – initial charge of the 1st stage, Q_2 – initial charge of the 2nd stage.

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