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Optical transmission spectra of ordered nanoporous alumina films fabricated by Hard Anodization with different thicknesses

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Abstract: We report on the structural and optical properties of self-ordered nanoporous anodic alumina (NAA) films fabricated by hard anodization in different time. The morphology of NAA films is examined by scanning electron microscopy (SEM). The results show that the porous structure have hexagonally ordered arrays of nanopores with interpore distance about 260 nm and pore size 60 nm. The optical transmission spectra of the NAA films with different thickness are measured in the wavelength range of 800–900 nm. Base on SEM and optical transmission spectra measurements, effective optical thickness was evaluated and compared.

Keywords (11 Bold): Hard anodization, nanoporous alumina, transmission spectra, Fabry-Pe´rot fringes, effective optical thickness

1 Introduction

In recent years there has been a tremendous growth in the field of nanotechnology. Nanoporous anodic alumina (NAA) films have attracted a great scientific and technological interest as a material for nanotechnology applications. NAA is fabricated by electrochemical anodization of aluminium in acidic electrolyte. Recently, a new method based on the hard anodization (HA) process was reported [1] for fast fabrication of highly self-ordered NAA with a wide range of pore sizes and interpore distances for practical application. In this new method, in a controllable procedure, an excellent array of nanopores was obtained applying a high current density (400/800 mA cm^{-2}) for a relatively short time.

The unique geometrical properties of these NAA films provide a wide range of potential applications such as magnetic storage [2], sensors [3], light emitting diodes [4], solar cells [5] or photonic crystals [6,7]. Recently, NAA films have been used as template or host material for preparation of nanowires [8], nanotubes [9] or metal nanohole arrays [10].

In order to extend the applicable fields of the NAA films a deep knowledge of the physical properties is required to understand the obtained results and to improve the derived devices. Specifically, the optical properties of NAA films are essential from the point of view of optoelectronic devices [11] .

Recent studies on the optical properties of NAA films have been focused on photoluminescence (PL) and Fourier transform infrared spectroscopy (FTIR) properties [12,13]. However, there are few detailed studies of wavelength dependence on refractive index and extinction coefficient and their dependence on structural properties [14,15].

The principles of optical and structural properties, that are both dependent on each other, still need to be clarified .Therefore in this work; we focus on optical features of nanoporous alumina related to increase the hard anodization time. The optical transmission spectra of NAA film with different thickness were measured by using ultraviolet- visible transmission spectra.

2 Materials and method

The samples were prepared from high purity (99.999%) aluminium (Al) foils with 0.25 mm thickness. First, the samples were cleaned in an ultrasonic bath of acetone, washed in deionized water and then the samples were electropolished in ethanol and perchloric acid solution (4:1) to obtain smooth-surfaced samples for anodizing. The anodization was performed in 0.3M oxalic acid solution cooled to 1℃. The system was connected to a potenciostatic control that has the following profile: first, the mild anodization (MA) is set to 40V for 5 min, then the voltage increases quickly from 40V to 130V at a rate of 0.4 V/s, and finally the high voltage is maintained constant at 130V for different time to perform the so-called hard anodization. In order to control hard anodization the applied voltage must be set below the upper limit of 150 V, otherwise the current density rises above a critical value and the sample is burnt. The anodized sample was then washed in deionized water. For the optical characterization of the porous alumina, the remaining aluminium substrate was immersed in mixture of 0.1 M copper dichloride and 20 wt% hydrochloric acid ($CuCl_2$+HCl) until the freestanding NAA membrane was achieved and then transfers the NAAs onto the transparent glass substrate. The optical transmission measurements were performed on a cintra spectrophotometer for the NAA with different thickness (by different hard anodization time) in the wavelength range of 800-1100 nm.

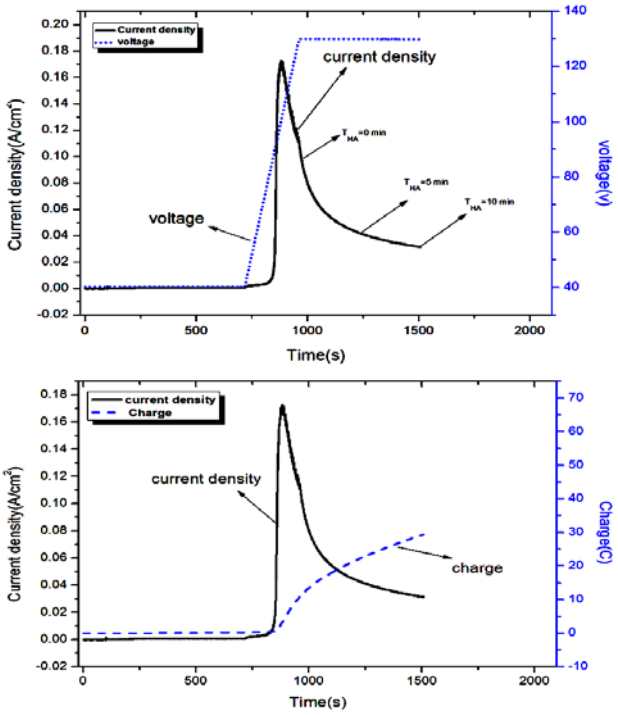

Figure 1.(a) Typical current density and voltage-time curves and (b) current density and charge-time curves of samples made in 0.3M oxalic acid at the hard anodization.

3 Results and Discussion

The evolution of the current density and voltage of the NAA film was investigated for all the sampes. Typical cuvers of current density and its corresponding voltage as well as anodization charge against time are shown in figure 1. After MA at 40V, the anodization voltage was increased at a rate of 0.4 vs^{-1} until reaching a final anodization voltage of 130V, which then continued for different time. As seen in figure 1, the current density increased rapidly at the threshod voltage and reached a maximum, where in decreased slightly, subsequently showed an approximately exponential decay as a function of time. Typical plane view cross sectional scanning electron microscopy (SEM) micrographs of the sample after 1h hard anodization are shown in figures 1(a) and (b). The templates exhibit hexagonally arranged pores. The pore spacing and pore size of the NAA film are about 260 nm and 60 nm respectively. The cross sectional SEM image presents high aspect ratio of parallel cylindrical nanopores. The spectra, obtained using a spectrophotometer in a 90° backscatter configuration, display a series of interference fringes. These fringe patterns result from Fabry-Pe´rot interference of light reflected from the various interfaces present in the structures [16]. The fringe maxima are described by the Fabry-Pe´rot relationship given in eq 1

$$m\lambda=2nL \qquad (1)$$

where m is an integer, L is the thickness of the porous NAA layer, n is the average refractive index, and λ is the wavelength of incident light. The term 2*nL* is thus the optical path, referred to as the effective optical thickness (EOT) here. According to SEM micrograph of pores in figure 2b, the pore dimensions in these structures are too small to effectively scatter light, and approximately each porous layer is behaved as a single medium with a single refractive index value.

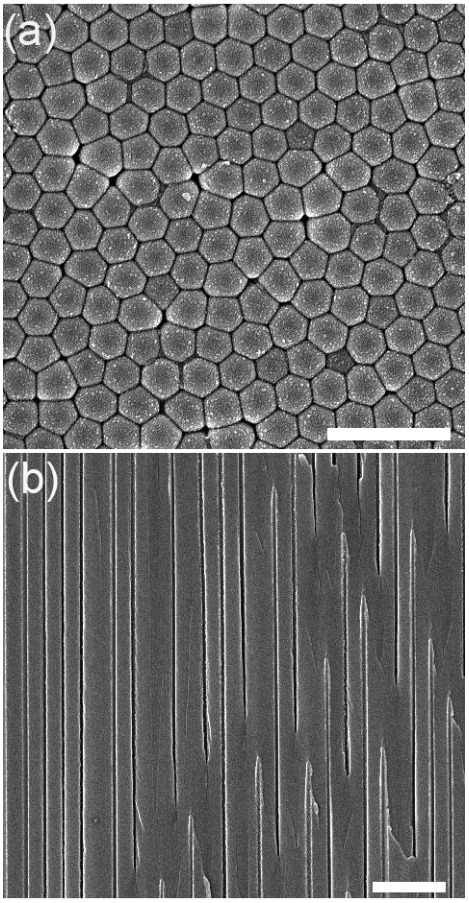

Figure 2. Plane view of NAA bottom surface (a) and (b) Cross-sectional SEM images of template that fabricated by hard anodizing methods (scales bar is 1μm).

As estimated from the relationship of eq 1, the series of Fabry-Pe´rot fringes observed in the all samples, are spaced evenly in frequency (Figure 3). As it is seen in this figure by increasing the anodization time, the oscillation frequency of transmitted spectra increases. As we khown [16], increasing the anodization time lead to increases of film thickness, subsequently the effective optical thickness (EOT) increases. Furthermore this increament, increases the amplitud of oscillation spectra. A plot of m vs $1/\lambda_{max}$, where λ_{max} is the wavelength of each peak maximum and m is an integer, numbering each successive peak (increasing from long wavelength to short), gives a straight line, whose slope is equal to the quantity **2nL [16].**

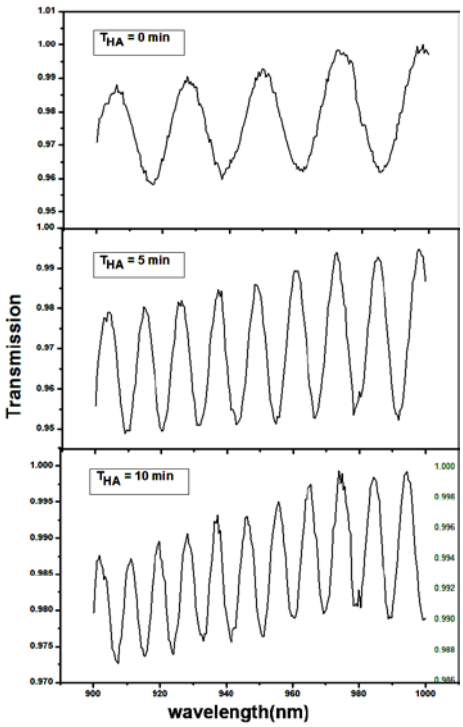

Figure 3. Transmition spectra of nanoporous anodic alumina sample with different hard anodization time.

effective optical thickness was evaluated directly through the 2nL and in indirect way by the slope of $m\text{-}1/\lambda_{max}$ curves. In direct manner, n=1.6 was considered [17] and L was evaluated through the cross sectional SEM of nanoporous anodic alumina layer. Figure 4 displays the effective optical thickness vs anodizaion time that evaluated in two manner. As it is shown in this figure by increasing the anodization time evalution of these two manner is different. This difference may be is due to the alteration of refractive index during the anodization. As it is clear from figure 1, by increasing the anodization time, the anodization current reduced. This reduction, able to change the chemical and structural properties of nanoporous alumina films and consequently their refractive index.

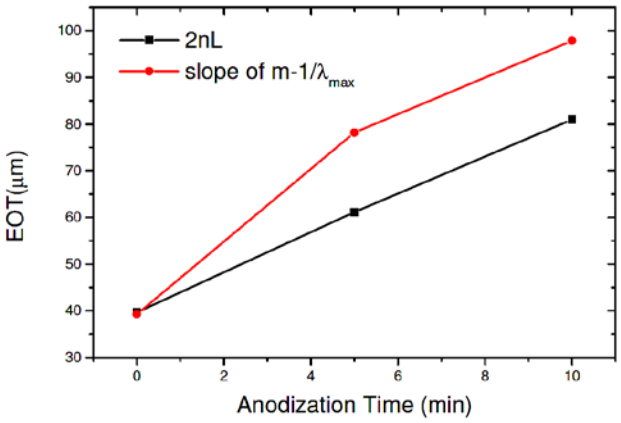

Figure 4. Comparison of methods used to evaluate effective optical thickness (EOT): 2nL base on SEM measurment vs slope of m-1/λmax.

4 Conclusions

We have employed the optical transmission measurements for the study of optical properties in highly ordered NAA films with different thicknesses and the following conclusions were obtained.

1. According to SEM micrograph of film pores, the pores dimension are too small to effectively scattered the light.

2. increasing the anodization time lead to increases of film thickness and effective optical thickness (EOT), subsequently oscillation frequency of transmitted spectra increases.

3. The reduction of anodization current during the anodization process able to change the chemical and structural properties of nanoporous alumina films and consequently their refractive index.

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