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THE VISIBLE WAVELENGTH DIFFRACTION FROM TWO-DIMENSIONAL GRATING FABRICATED BY MILD ANODIZATION TECHNIQUE

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Abstract. Two-dimensional diffraction grating was fabricated on aluminum plate on the basis of anodization technique. The reflective grating with interpore ranging 500 nm was prepared using mild anodization. In order to make the porous alumina arrays a single step mild anodization in 0.4 M phosphoric acid with 195 anodization voltage was employed. Interpore distances were seen 500 nm when the mild anodization technique was used. The domain size of hexagonal ordered arrays was observed with over 25 μ^2 area. Optical properties of gratings were investigated using visible light sources thereby the structural details of gratings were determined.

1. Intorduction

Self organized porous alumina nanostructures fabricated by the anodization of aluminum have attracted considerable attention in electronic, magnetic, photonic, and biosensors devices [1-10]. To facilitate different applications, fabricating of highly ordered porous anodic alumina films with simple process and low cost in a large scale is an essential and urgent. Self ordered porous aluminum oxide (PAO) are obtained by mild anodization (MA) in three regimes with H_2SO_4, $H_2C_2O_4$, and H_3PO_4 acids at 40 V, 25 V, and 195 V respectively [11-13]. It is found that the formation of a periodic pore structure is sensitive to anodizing conditions. The type of electrolyte (sulfuric acid, phosphoric acid or oxalic acid), applied voltage and anodizing temperature can affect the pore distribution. Though various electrolytes and applied voltages can produce different pore sizes and interpore distances, it has been found that the pore distribution with a high symmetry can be achieved at lower anodizing temperatures [13]. Explanations of the pore formation have been proposed according to the experimental observations of porous structures under various conditions [12, 14].

Diffraction gratings have played a vital role in many fields of science and technology, including spectroscopy, astronomy, beam-sampling for high power lasers, and filtering [15-19]. The most commonly used grating is the metallic reflection grating. One of the main requirements for this kind of grating is the so-called blazing effect, e.g., the concentration of the reflected light in one scattered beam only, at a given wavelength. High-efficiency gratings are desirable for several reasons. A grating with high efficiency is more useful than one with lower efficiency in measuring weak transition lines in optical spectra. A grating with high efficiency may allow the reflectivity and transmissivity specifications for the other components in the spectrometer to be relaxed. Moreover, higher diffracted energy may imply lower instrumental stray light due to other diffracted orders, as the total energy flow for a given wavelength leaving the grating is conserved (being equal to the energy flow incident on it minus any scattering and absorption). Control over the magnitude and variation of diffracted energy with wavelength is called blazing, and it involves the manipulation of the microgeometry of the grating grooves [20]. As early as 1874, Lord Rayleigh recognized that the energy flow distribution (by wavelength) of a diffraction grating could be altered by modifying the shape of the grating grooves. It was not until four decades later that R.W. Wood showed this to be true when he ruled a grating on which he had controlled the groove shape, thereby producing the first deliberately blazed diffraction grating [21].

In this paper, the mild anodization techniques were employed to fabricate the two-dimensional gratings in porous alumina films. In the type of reflective, the self-ordering and ordered domain size of polydomain grating were evaluated using full width at half maximum (FWHM) of the curves in which the intensity of reflected beam was plotted as a function of the relative angle.

2. Experimental

In our procedure, high purity aluminum foil with 99.999 % purity, 1 mm thickness, bought from Aldrich was used as starting material. Prior to anodizing the aluminum foil was annealed at 400 c in the air for 2 h and degreased in acetone. Then the samples were electro polished at room temperature in 25 : 100 volume mixture of $HCLO_4$ and C_2H_2OH at constant current density at 25 mA/cm^2 for 3 min. The aluminum foils were mounted on a copper plate serving as the anode and exposed to the acid in a thermally isolated electrochemical cell. The diameter of the exposed area was 8 mm. While the anode temperature was kept constant at 0 °C, the temperature of the electrolyte deviated from that value by less than 0.1 °C. The electrolyte was rigorously stirred during anodization. In the experiments, 0.1 and 0.4 M phosphoric acid electrolytes were used [11, 13]. The porous oxide layers were obtained by anodizing of aluminum at the constant cell potential 195 V. The time of the anodization was 18 h for each cell potential. The aluminum oxide formed in the anodization procedure was then removed by chemical etching in a mixture of 6 wt.% H_3PO4_4 and 1.8 wt.% H_2CrO_4 at 60 ºC. The resultant Al surface had the periodic roughness corresponding to the morphology of the underside of a nanohole array. We examined the surface morphology of these samples by scanning electron microscopy. The fast Fourier transformation (FFT) of the SEM images for the qualitative and quantitative analysis of pore arrangement was performed. The observed patterns were corresponded to the underside of the nanopore array which was a direct consequence of the periodic curvature of the bottom barrier layer of individual nanopores.

3. Results and discussion

A schematic diagram of experimental setup is shown in Fig. 1. The optical measurements were conducted using He-Ne laser as optical source.

To study the optical behavior of the gratings the following well known equation were used:

$$m\lambda=d(\sin\theta_i - \sin\theta_m),$$

in which m is a diffraction index, λ is a wavelength of laser beam, d is a lattice constant, θ_i is an angle of incident beam, θ_m is an angle of diffracted beam, and n is a refractive index. The interpore distance of the fabricated grating (d) is 500 nm. For such gratings only the first order diffraction could be measured. In a comparison between the optical measurements and the obtained results from FFT images the grating quality (the ordering and domain size) were studied. The self-ordering is correlated to the FWHM of the intensity curve against diffracted angle and the smaller FWHM is an indication of the better self-ordering. The degree of self-ordering is directly proportional to intensity of the spots and inversely proportional to the spot size of FFT patterns. Current curve versus time for mild anodization in 0.4 M phosphoric acid at 195 V and 0 ºC for 20 hours is shown in Fig. 2.

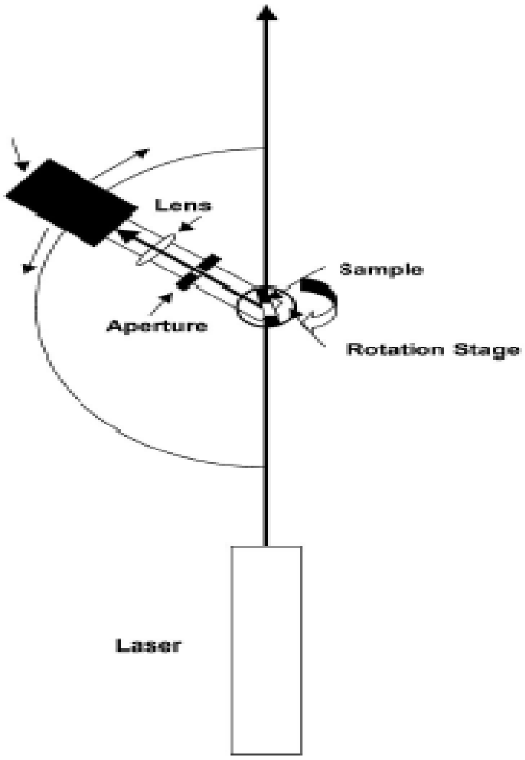

Fig. 1. The visible wavelength diffraction measurements utilized a sample mounted on a rotation stage with an adjustable aperture detector mounted on a rotating arm.

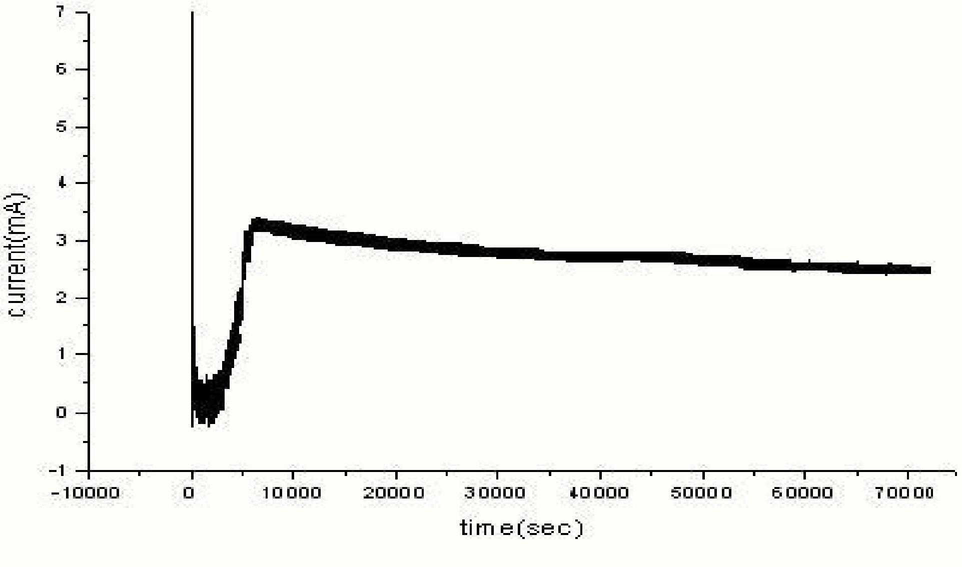

Fig. 2. Current curve versus time for mild anodization in 0.1 M phosphoric acid at 195 V and 0 °C for 20 hours.

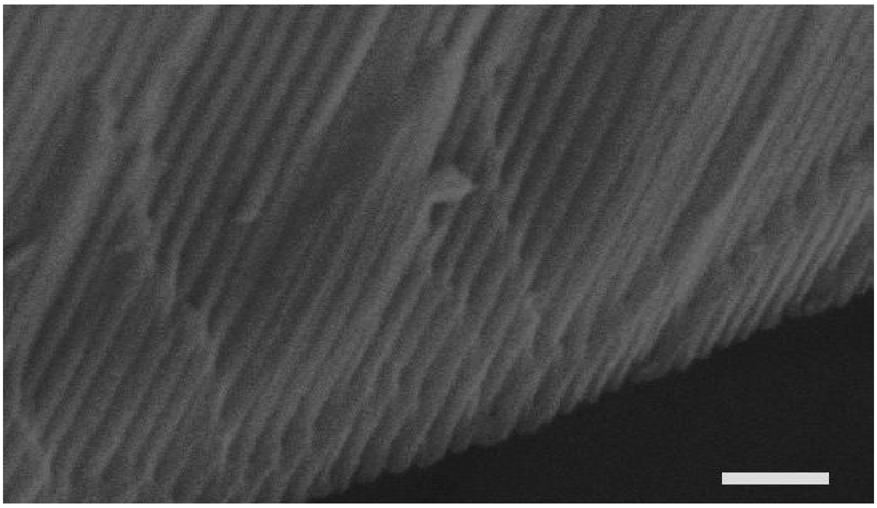

Fig. 3. The cross section SEM micrographs of nanopore configurations of sample made in 0.4 M phosphoric acid at the anodization voltage of 195 V. The scale size is 2 μ.

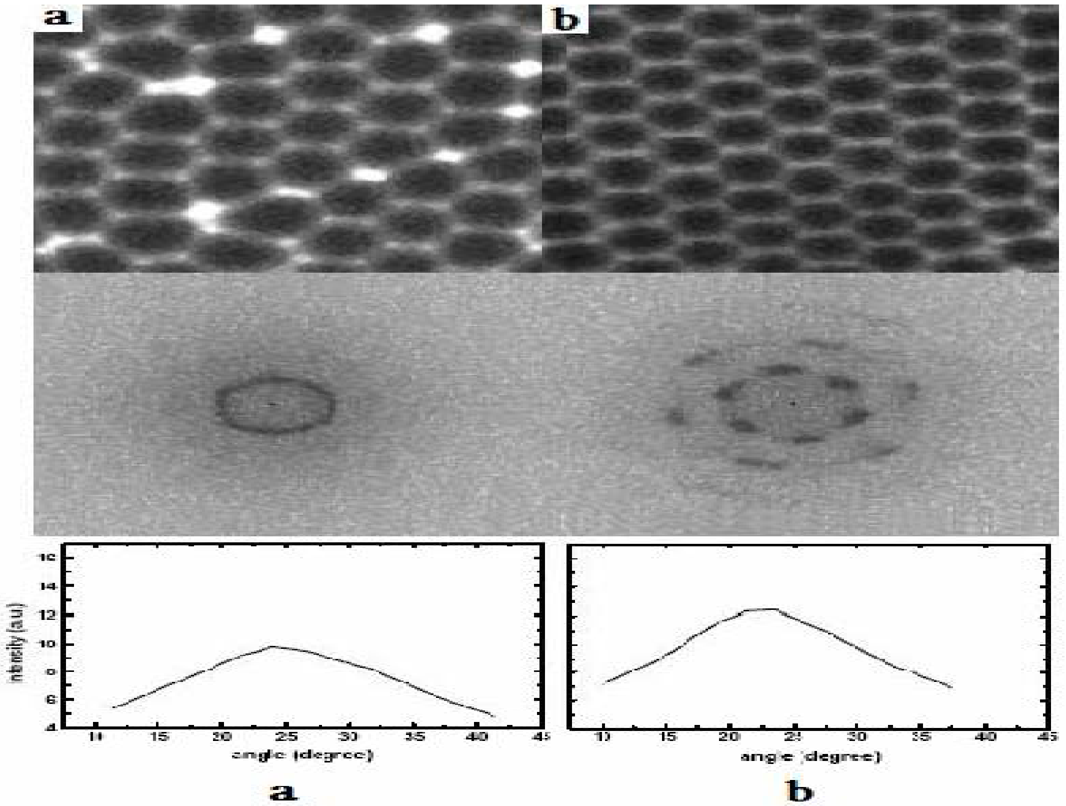

Fig. 4. 5 μm×5 μm SEM images, FFT and diffraction curve of anodized samples surface in (a) 0.1 M phosphoric acid at 195 V,
(b) 0.4 M phosphoric acid electrolyte at the anodization voltage of 195 V.

In Figure 3 the cross section SEM micrographs of nanopore configurations of sample made in 0.4M phosphoric acid at the anodization voltage of 195 V. The scale size is 2 μ. SEM micrographs, diffraction curves and FFT images of two samples fabricated in 0.1 M and 0.4 M phosphoric acid at anodization voltage of 195 V are shown in Fig. 3. The FFT pattern is a spotty ring for the sample made in 0.1 M phosphoric acid as seen in Fig. 4 which interprets that the domain size is relatively small and the arrayness of the sample is low. As seen with an increase in phosphoric acid concentration the FFT image changes to a six-fold symmetry pattern with relatively low spot size which implies an increase in self-ordering. We see a good agreement between optical evaluation and FFT pattern. As shown the diffracted intensity peak reduces to 10 from 12.5 and the FWHM increase to 16 from 14 degree for the samples made with 0.4 M and 0.1 M phosphoric acid electrolyte respectively.

4. Conclusion

We have observed self-organization of two-dimensional pore arrays with about 500 nm interpore distances in porous anodic alumina. The experimental results have shown that the electrolyte concentration have a significant effect on the quality of the nanoporous arrays. We see a good agreement between optical evaluation and FFT pattern.

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