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MORPHOLOGY INVESTIGATION OF NANOPOROUS ANODIC ALUMINA FILMS WITH IMAGE ANALYSIS

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Abstract. Aluminum films approximately 100 nm thick were deposited on silicon substrates (SiO_2 / Si) by thermal evaporation in a vacuum. Porous anodic alumina films were obtained in a potentiostatic mode at 20 V in 0.3 M aqueous solution of oxalic acid and 1.8 M aqueous solution of sulfuric acid. The main pore diameter was determined using the ImageJ software from SEM images. An algorithm determining the pore diameter in porous anodic alumina films was developed. In the nanoporous alumina films formed in sulfuric acid at 20 V the average pore diameter was 12.3 ± 0.1 nm. In the case of the oxalic acid electrolyte, the nanoporous alumina films formed at 20 V had an average pore diameter of 14.8 ± 0.1 nm. The obtained results are in a good agreement with the literature.

Keywords: anodic alumina, pore diameter, SEM image, surface morphology

1. Introduction

Porous anodic aluminum oxide (AOA) is of great interest because of its unique properties, primarily, such as a highly ordered porous structure, nanosized pores, and the ability to control structural parameters at the stage of their formation using aluminum anodizing [1,2]. High mechanical hardness, thermal and chemical resistance allows using anodic alumina for chemical and biochemical separation (filtration), as well as for the synthesis of various nanomaterials, such as nanowires and nanotubes. For expanding the application areas of anodic alumina films and increasing the reproducibility of their properties, it is necessary to know the nanostructure obtained in various modes of aluminum anodizing. It is well known that the basic properties of anodic aluminum oxide depend on the pore diameter, so there is a need for precise regulation of their geometric dimensions during its formation. In this regard, it is important to apply statistical analysis methods to process large arrays of nanosized pores and to develop a methodology and algorithm for studying the surface morphology and nanoporous structure of anodic films [3,4].

2. Experimental

The aluminum films approximately 100 nm thick were deposited on silicon substrates (SiO_2 / Si) by thermal evaporation in a vacuum. Then the square samples of 4 cm^2 were cut out and anodized in a potentiostatic mode at 20 V in an aqueous solution of 0.3 M oxalic acid and 1.8 M aqueous solution of sulfuric acid. The anodizing area of aluminum (about 0.22 cm^2) was set with Viton-o-ring. The aluminum anodizing process was carried out in a two-electrode fluoroplastic cell at a constant temperature of 18ºC using the thermostat F 12 (Julabo). As DC power source, PS-2403D (Voltcraft) was used. The cathode was a platinum

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grid. The electrolyte during anodizing was vigorously stirred using a mechanical stirrer. The main pore diameter was calculated from the SEM images using the ImageJ software.

3. Results and discussion

For processing SEM images of the nanoporous anodic alumina surfaces, the ImageJ software was used. The program includes all the necessary functions for digital image processing: correction of brightness and contrast, selection of image limits, high-frequency and low-frequency filtering, etc. The following algorithm in the ImageJ program (Fig. 1) consists of:

1) Transforming the image into 8 bits to improve the contrast and simplify the subsequent analysis.

2) Filtering the image to eliminate random noise (which forms small porous structures and unnecessary pore bonds).

3) Preliminary segmentation to isolate homogeneous regions.

4) Finding the threshold value for image segmentation (separation into foreground and background). Segmenting the image based on this threshold value that allows fully define the object.

5) Analysis of the selected objects.

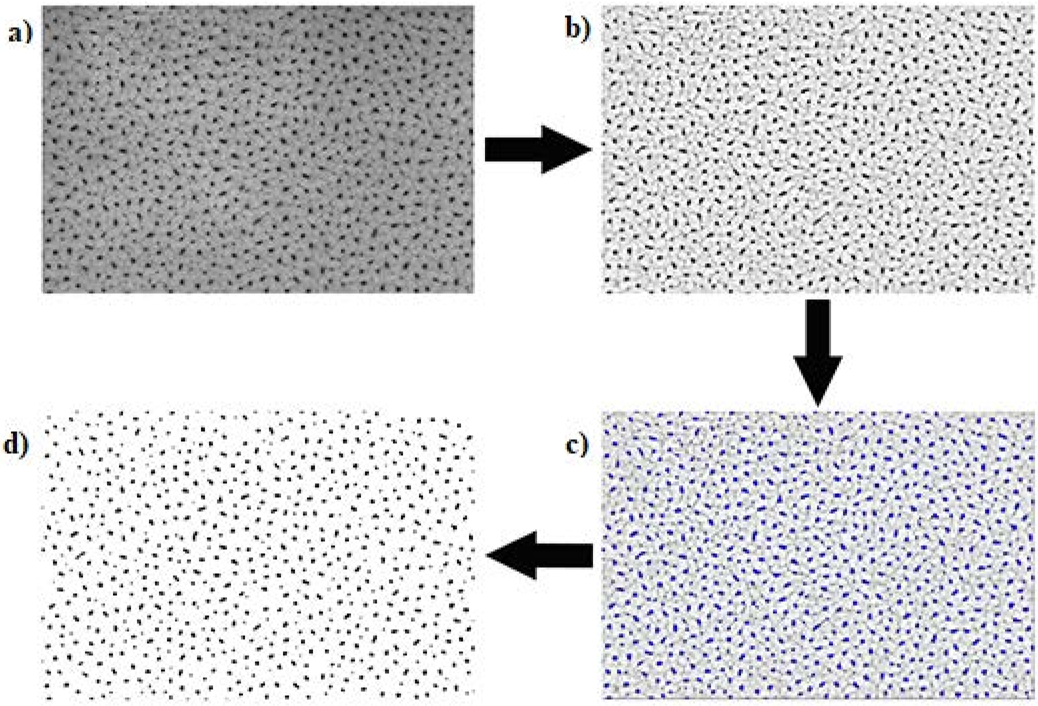
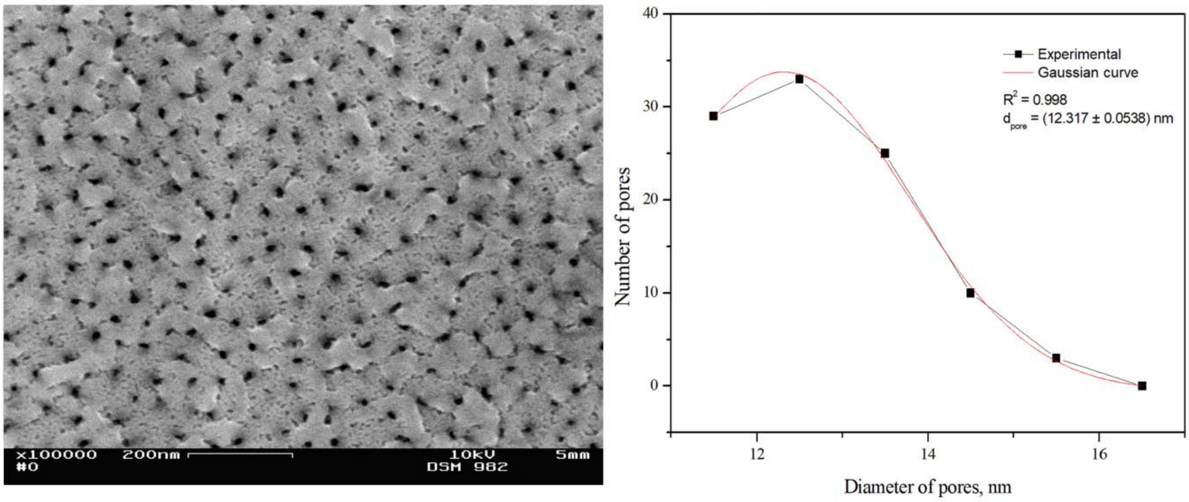


Fig.1. Processing SEM images using ImageJ software: a) original SEM image, b) contrast enhanced image, c) threshold image, d) final image

The ImageJ software allows calculate the areas and statistical values of the pixel values of various areas selected manually or by means of threshold functions on the images. It supports standard image processing functions, such as logical and arithmetic operations between images, contrast manipulation, convolution, Fourier analysis, sharpening, smoothing, border detection. The program also allows perform various geometric transformations, scaling, rotation and reflection. The ultimate task of image analysis is statistical processing the data obtained at measuring characteristics of an object having a porous structure, i.e. determining the average value of pore diameters and plotting graphs to visualize the analysis process.

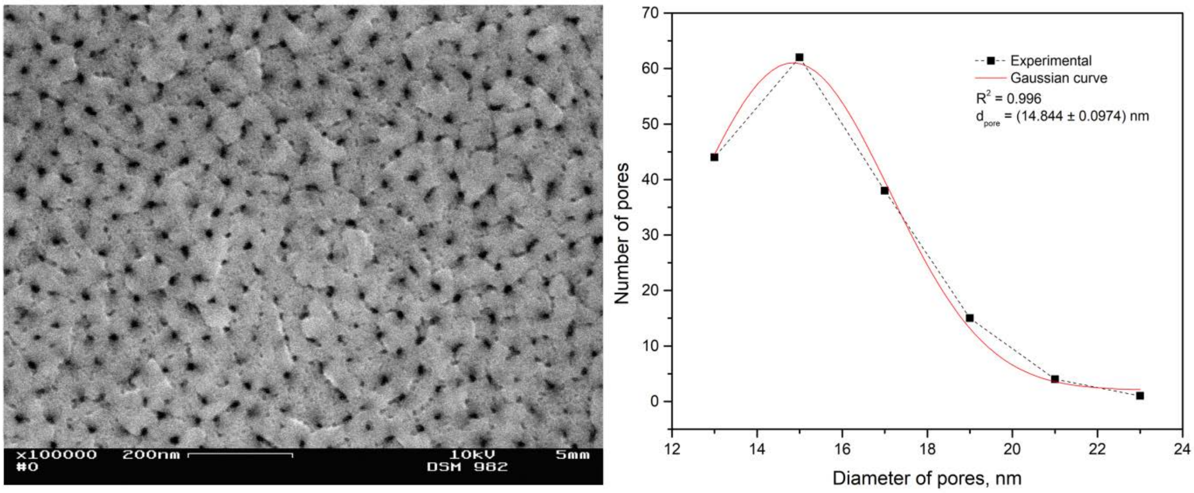
The main pore diameter (d_{pore}) was determined from the surface images of porous anodic aluminum oxide obtained by SEM, the pore size distribution curves being approximated with the Gaussian functions. It was assumed, as in Ref. [5], that the initial pore size distribution contained both initial small-diameter pores and major pores of a larger diameter. To obtain correct results, the SEM images were processed at least ten times and a new time was selected each time for comparison. Since only the data on the main pores are of practical importance, and the presence of the initial pores interferes with the analysis, some of the pores with a small diameter were not taken into account at activities of approximating. At this, only one smaller value was left to the maximum on the pore size distribution curve. The maximum on the Gaussian curve corresponds to d_{pore}.

We have found that d_{pore} = 12.3 ± 0.1 nm for porous alumina films obtained in sulfuric acid (Fig. 2b) and d_{pore} = 14.8 ± 0.1 nm for the anodic alumina films obtained in oxalic acid (Fig. 3b). The structure parameters of the porous alumina films obtained coincide with the data reported in Ref. [6].



a) b)

Fig. 2. Porous alumina films obtained in 1.8 M aqueous solution of sulfuric acid: (a) SEM image of surface; (b) results of processing image with ImageJ software



a) b)

Fig. 3. Porous alumina films obtained in 0.3 M aqueous solution of oxalic acid: (a) SEM image of surface; (b) results of processing image with the ImageJ software

4. Conclusions

Methods of modern software image processing have a full set of functions necessary to obtain information about the surface morphology of nanoporous films. However, the general picture after processing images contains also side shapes that arise from surface defects and scratches that make analysis difficult. Therefore, for processing images of nanoporous structures, algorithms that take into account the patterns of growth of the nanoporous films are necessary. Using the regularities of anodic oxide film growth at the analysis of histograms showing the distribution of pores over the diameter, it is possible to identify the region that relates to the oxide growth and to determine the pore diameter of nanoporous structures.

Porous alumina films obtained in sulfuric and oxalic acids at 20 V have d_{pore} = 12.3 ± 0.1 nm and d_{pore} = 14.8 ± 0.1 nm respectively.

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