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Fast Fourier transform based arrangement analysis of poorly organized alumina nanopores formed via self-organized anodization in chromic acid

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ABSTRACT

Poorly arranged nanopores focus attention of the researchers due to the many fundamental aspects. Thus, quantitative arrangement analysis of non-ideally arranged pores is demanded. In the paper, radial average fast Fourier transform based arrangement analysis of the nanopores has been reported. This arrangement analysis tool has been applied in the quantification of arrangement of anodic alumina formed in 0.3 M chromic acid at voltage ranging from 20 to 50 V and temperature ranging from 20 to 50 °C. Presented approach, in contrary to typically applied methods, is based on the radial average of the fast Fourier transform (FFT). Averaged regularity ratio, estimated with the use of the reported tool, gives more repeatable results with smaller spread. Thus, reliable dependencies between operating conditions and the averaged regularity ratio can be also obtained for poorly arranged pores. It was found that the greater the voltage and temperature, the better nanopores arrangement.

1. Introduction

Highly-ordered alumina is being applied for template nanofabrication and is obtained in sulfuric [1], oxalic [1,2] and phosphoric acid [1,3,4]. However, alumina with less ordered pores also attracts researchers, due to many other benefits. Fabrication of anodic alumina with ultra-small pores with diameter below 10 nm provides worse nanopores arrangement [5] than oxide growth conducted at highly-ordering regimes [6]. The arrangement is also spoiled after the addition of ionic liquid into the electrolyte, which enhances significantly the anodic oxide growth rate [7]. Introduction of chelate ions into the electrolyte allows to obtain anodic aluminum oxide (AAO) with luminescent properties, however an incorporation of these ions into the AAO worsens the pores arrangement [8]. Thus, there is a need of the nanopores arrangement quantification. Typically, highly-ordered alumina is being characterized with three intensity profiles conducted through fast Fourier transform (FFT) of the FE-SEM image along the three major, hexagonal directions [9,10]. This method is useful, when FFT is a hexagon with six distinct dots at its corners [9,10]. Only in this situation intensity profile based approach brings reliable information. In the case of poorly ordered AAO [7,8], FFT is a blurred ring and selection of three intensity profiles bringing information about arrangement is rather hard to perform and may bring unreliable or strongly dispersed results. Therefore, researching new FFT based arrangement analysis approaches is demanded.

The main objective of the paper is application of modified quantitative arrangement analysis method to assess the ordering of the nanoporous AAO formed in chromic acid. Reported analysis method is dedicated to the periodic structures with poorly arranged nanopores.

2. Materials and methods

Fabrication of nanoporous alumina was done in accordance to the methodology described previously [11]. Briefly, high-purity aluminum foil (99.9995% Al, Puratronic, Alfa-Aesar) was cut into samples (0.5 cm × 2.5 cm), degreased in acetone and ethanol and electropolished (ethanol: $HClO_4$ 4: 1 vol. mixture, 10 °C, 0.5 A/cm^2, 1 min). By using acid-resistant dye, exposure area of electropolished aluminum sample was limited to 0.5 cm^2. A two-step self-organized anodization was performed in 0.3 M chromic acid at various voltages (20, 30, 40, and 50 V) and temperatures (20, 30, 40, and 50 °C). After 60-min long first step of anodization, obtained alumina was chemically removed in a mixture of 6 wt% phosphoric acid and 1.8 wt% chromic acid at 60 °C for 90 min. Next re-anodization was performed at the same set of operating conditions as in the first step.

Images of nanoporous alumina were taken with field-emission scanning electron microscope (FEI, Quanta D FEG). Fast Fourier transforms (FFT) were generated and taken into further calculations

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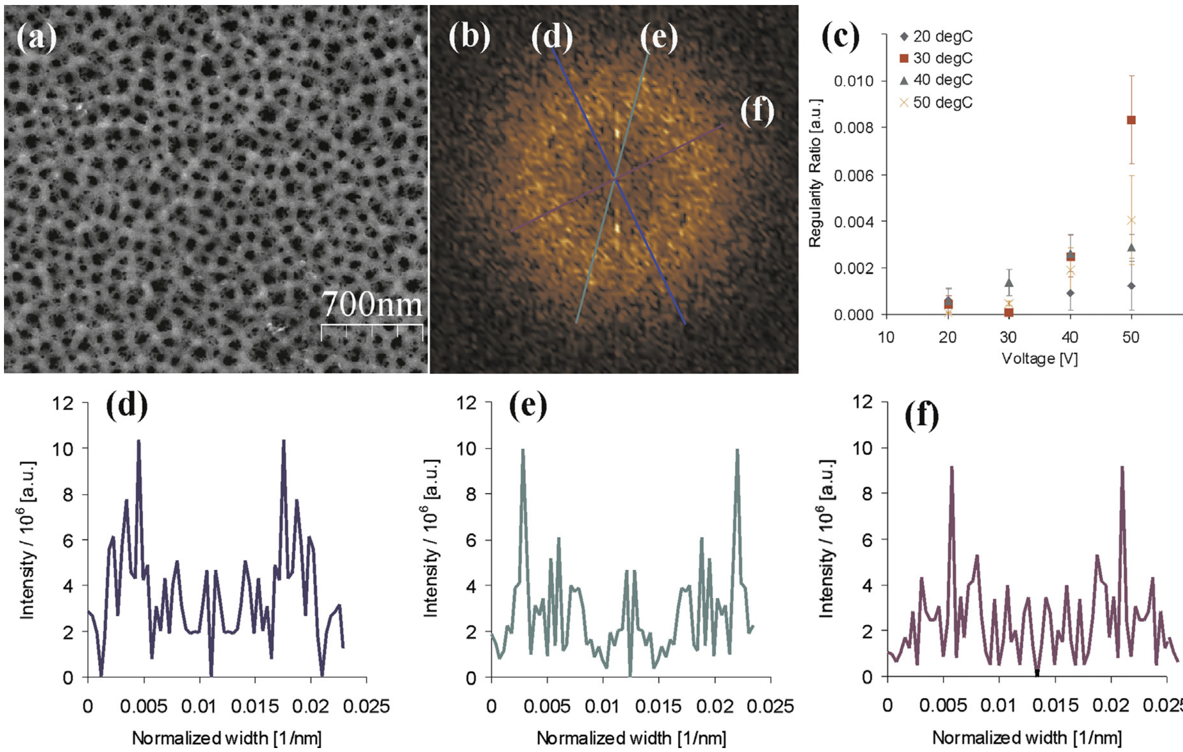

Fig. 1. An exemplary top-view FE-SEM image (a) and its fast Fourier transform (b) with three intensity profiles (d–f) as well as the influence of operating conditions on the regularity ratio (c).

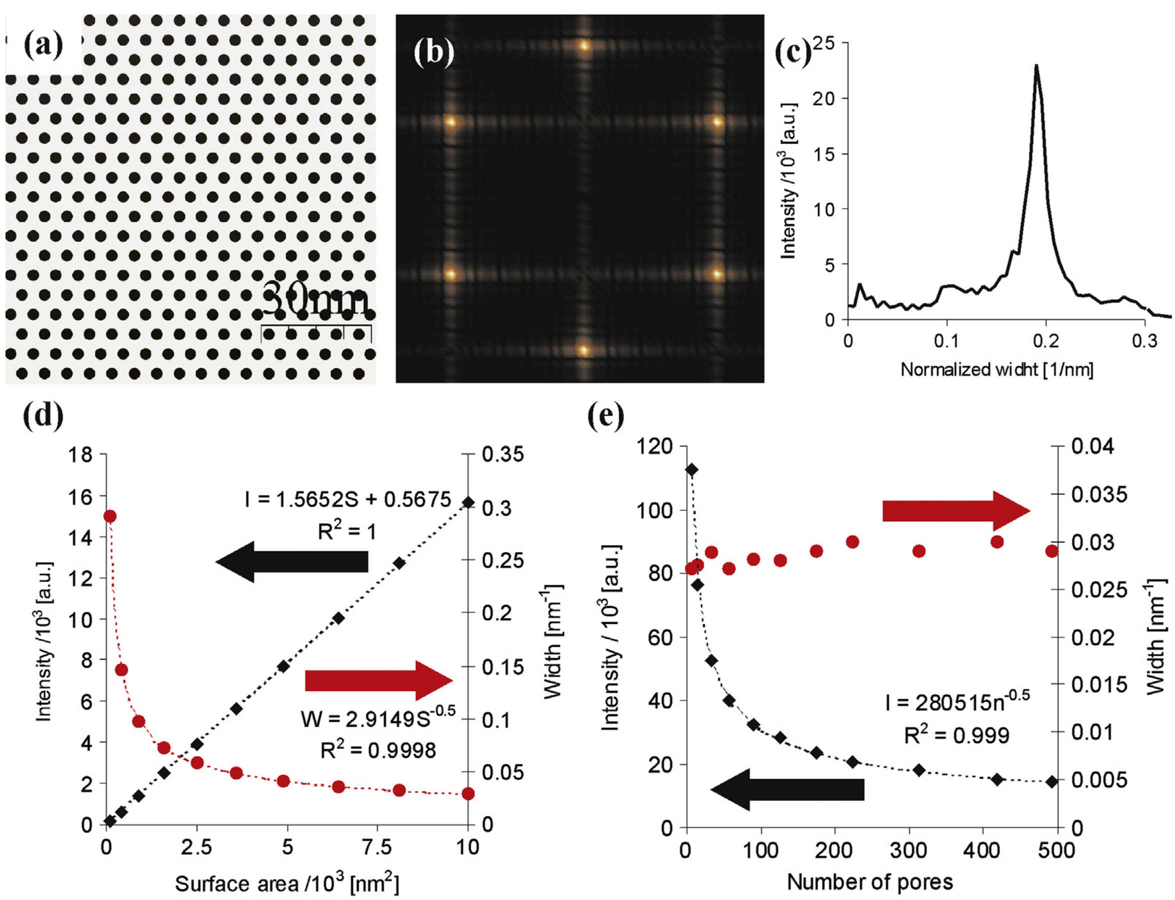

Fig. 2. An exemplary computer model of ideally arranged pores (a) and its fast Fourier transform (b) with radial average (c) as well as the influence of surface area (d) and number of pores (e) on intensity and width of the radial average peak evaluated for ideally arranged computer models.