

Post-processing of SiO₂/Si ion-track template images for pores parameters analysis

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Abstract

The use of porous templates for controlled formation of nanostructures and nanosystems requires reliable method of pore size, shape, quantity per unit area (porosity), distance to nearest neighbors and porous matrix thickness monitoring. The scanning electron microscopy method allows to estimate these parameters, however, manual processing of obtained images is a very time-consuming task. The use of ImageJ software makes it possible to significantly simplify the process of obtaining detailed statistical information about templates surface. Taking that into account, the work describes in detail the methodology for analyzing of scanning electron microscopy images of SiO₂/Si ion-track templates surface to control pore size, shape, unit area and distance to the nearest neighbors. The method for estimating thickness of SiO₂/Si ion-track template based on processing of data about surface is also proposed. It is important to note, that the techniques described in this paper not only allow to effectively control SiO₂/Si ion-track template pore parameters, but also suitable for other types of porous templates.

Keywords: Ion-track technology; SiO₂/Si template; ImageJ; surface analysis.

1. Introduction

Nowadays nanoindustry is involved in many fields: environmental protection, biotechnology, medicine, electronic devices and etc. [1–6]. Depending on potential applications, materials with unique physical properties are required. This determines relevance of searching of methods for controlled synthesis of nanoobjects with predetermined characteristics. Among the wide variety of techniques template synthesis can be distinguished due to its simplicity, low price and high quality

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[7–14]. The method is based on using of porous matrices (which are templates based on polymer, silicon or oxide materials) as a form-building basis for realization of nanostructures [15–22]. By using such approach structures are formed by controlled self-organization of matter in pores. It makes possible to obtain arrays of nanostructures with different morphologies: particles, rods, tubes, dendritic structures [23–26], which can be useful for creation of new types of electronic devices, sensors, systems of biological control and etc. [19,27]. Due to simple adaptation to standard of silicon technology silicon-based templates are of great interest. The ion-track technology is a simple method of templates based on silicon and its oxide layers forming [7]. With such approach, it is important to control such templates parameters as height, external and internal pore diameters and their density. For this purpose various software solutions can be used. In particular, one of the most effective methods is using ImageJ software in analysis of scanning electron microscopy images. This method allows to determine a wide range of pore parameters for obtaining a large amount of statistical information. Therefore, in this paper we demonstrate the possibility of surface analysis by SEM with ImageJ post-processing for SiO₂/Si ion-track template parameters control.

2. Methods

Amorphous SiO₂ films were thermally grown on (100) *n*-type (P-doped) silicon wafers of 524 nm thickness in a pyrogenic environment at 1050 °C. Irradiation of the SiO₂/Si substrate was carried out at the DC-60 accelerator (Astana, Kazakhstan). Samples were bombarded at normal incidence with ²⁰⁸Bi ion with an energy of 1.75 MeV/nucleon, at fluence 2×10^7 cm². Low fluences were chosen in order to minimize the spatial overlap of the individual conical pores appearing after etching of latent tracks in SiO₂. Conversion of ion tracks into pores was carried out by etching in hydrofluoric acid at a concentration of 1% for 58 minutes at room temperature.

Morphology of the porous samples was characterized by field emission scanning electron microscopy (SEM), using a JEOL JSM-7000F microscope at 5 kV accelerating voltage. At higher accelerating voltage the sample surface will charged which greatly complicates the processing of information. At a lower accelerating voltage, it is difficult to obtain a high-quality image. This approach allows defining all the characteristic dimensions of the pores. However, only few pores can be analyzed in this way, leading to poorer statistical representation. Image analysis was performed with ImageJ software with ImageJ2 version [30]. The ImageJ package is a free cross-platform software with an open architecture that allows calculating areas, statistical indicators of pixel values of different areas on images, extracted manually or using threshold functions [31].

3. Algorithm of porous templates SEM images post-processing by ImageJ software

Surface analysis by ImageJ post-processing of SEM images was carried out on images of the SiO₂/Si ion-track template surface, typical for the selected irradiation and etching modes (Fig. 1a). From Fig. 1a it is seen that in connection with the random nature of the ion distribution in the irradiating beam, the pores in the oxidized silicon layer are stochastically distributed. Determination of pores characteristic parameters was carried out by using of *Analyze Particles* (*Analyze* → *Analyze Particles*) command of ImageJ software. Running the *Set Measurements* command (*Analyze* → *Set Measurements*) allows to set analyzed parameters. The selection of parameters in menu (*Measure* → *Set Measurements*), *Analyze Particles* determines which statistical data about image (area and perimeter of selection, tilt angles, pore coordinates and etc.) will be displayed in a special window *Results*.

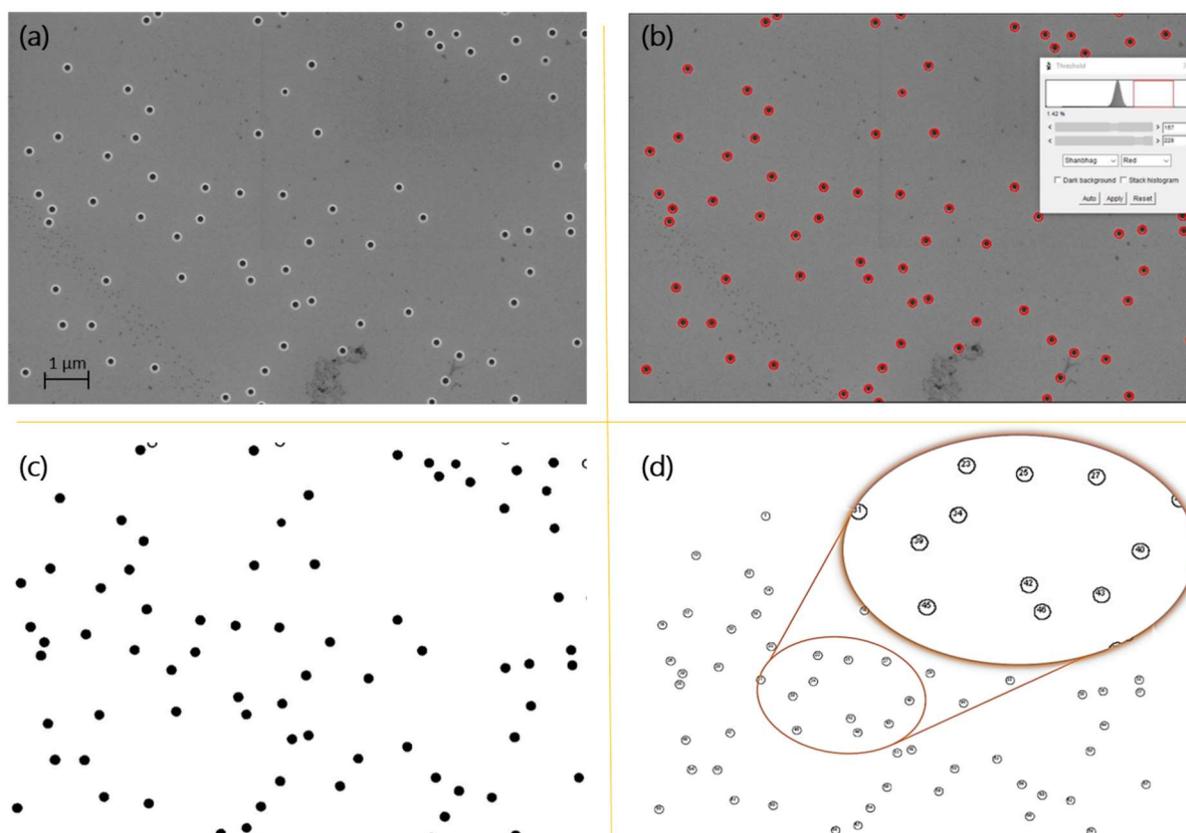


Fig. 1. (a) SEM image of porous SiO₂/Si template surface, (b) setting the threshold by using of ImageJ command *Threshold*, (c) image after binarization and (d) result of command *Analyze Particles* execution.

For efficient data processing, preliminary preparation of images by standard command of ImageJ package *Threshold* (*Image* → *Adjust* → *Threshold*) and image binarization (*Process* → *Binary* → *Make Binary*) is necessary. Using of *Threshold* command allows you to interactively adjust upper and lower threshold values for segment of area of interest and image background (Fig. 1b). The

threshold level is determined by analysis of histogram of current selection or full image if there is no selection. Setting of minimum and maximum exceptions ranges from different parameters after binarization (Fig. 1c) allows to extract information from binary image.

A more detailed image description can be obtained by using of BioVoxel Toolbox [29]. The *Extended Particle Analyzer* is based on ImageJ command *Analyze Particles* and allows to restrict particle analysis in accordance with many parametric descriptions of shape and angular orientations. The results output types are the same as for *Analyze* → *Analyze Particles*, namely: converted image and two-dimensional array of described above statistical data. It is possible to obtain information about the pores like area, extent perimeter, circularity, roundness, solidity, compactness, aspect ratio, Feret aspect ratio, ellipsoid angle (degree), max and min Feret, Feret angle and coefficient of variation by using of *Extended Particle Analyzer*. Information about function, which is realized by separate line, is shown in Fig. 2.

The output form of the results can be also set for working with them (display results, summarize, reset after analysis, clear results, add to manager) and for include/exclude of objects located on border of image and on area inside segmented regions (include holes, exclude edges).

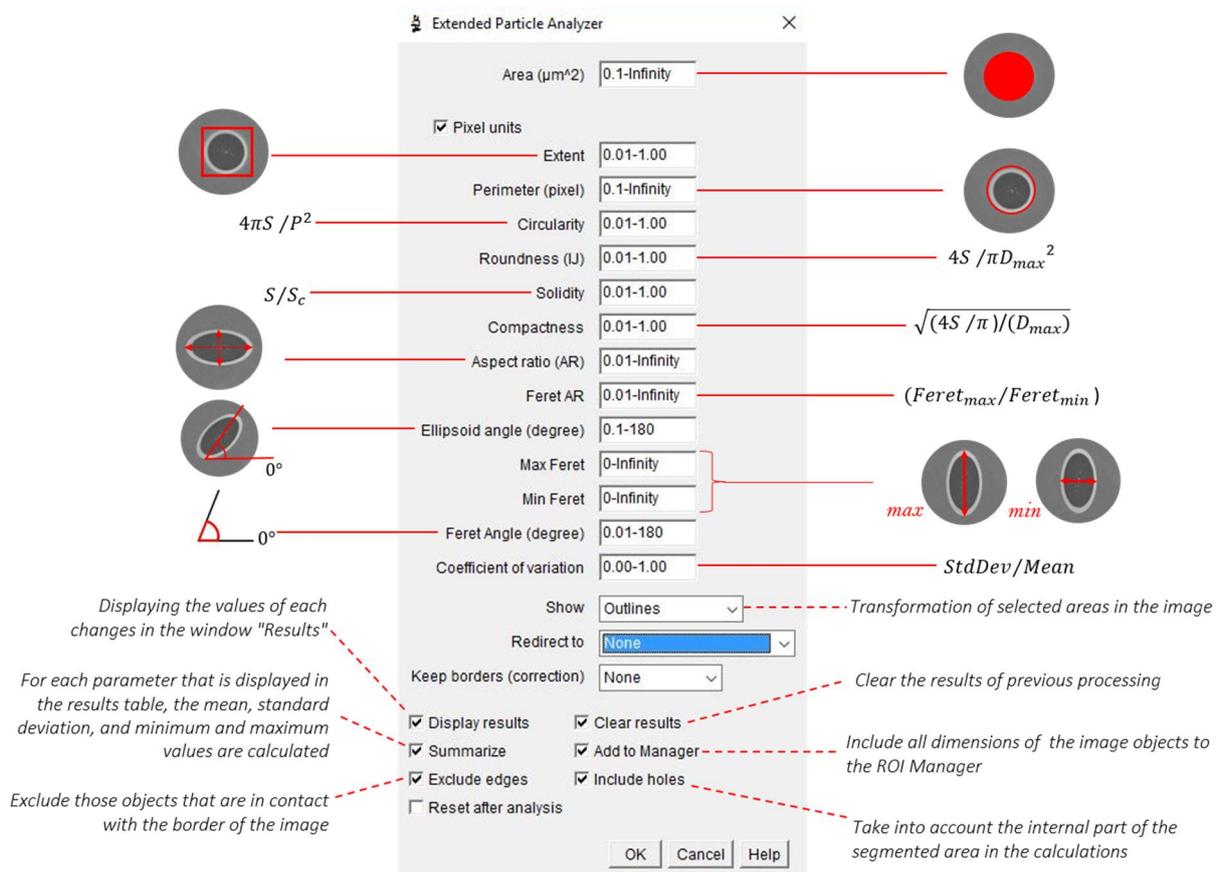


Fig. 2. Setting parameters in *Extended Particle Analyzer*, where S – area, S_c – area of a convex domain, P – perimeter, D_{max} – maximum diameter, $StdDev$ – standard deviation of the area, $Mean$ – mean value of the area.

It is possible to obtain information about distance between the nearest pores with the plugin *Nnd* (*Plugins* → *Nnd*), which is added to built-in functions of ImageJ. As a result, the screen displays an image with pore contours and their numbering (Fig. 1d), as well as a window with a table, which contains information about distance to the nearest neighboring pore. The distance between the nearest neighbors is determined on the basis of following approximations. In tightly-packed configuration of pores having a circular cross-section in 2D space, there are 6 neighbors surrounding each particle. In random system order the coordination number depends on visual perception and can be lower or higher. Estimation of particle's distance to its neighbors is performed by following algorithm: determination of coordinate of each particle gravity center (X, Y) based on the results table of built-in plug-in *Analyze Particles* constructing circle on each particle with center (X, Y) and radius r (Fig. 3) → determination of interval (wall thickness) between a pair of particles (d_p) according to the correlation:

$$d_p = \sqrt{(Y_2 - Y_1)^2 + (X_2 - X_1)^2} - (r_1 + r_2) \quad (1)$$

Further, the distance from each pore relative to all other pores is stored in array and sorted, and finally, average distance is calculated depending on coordination number [19].

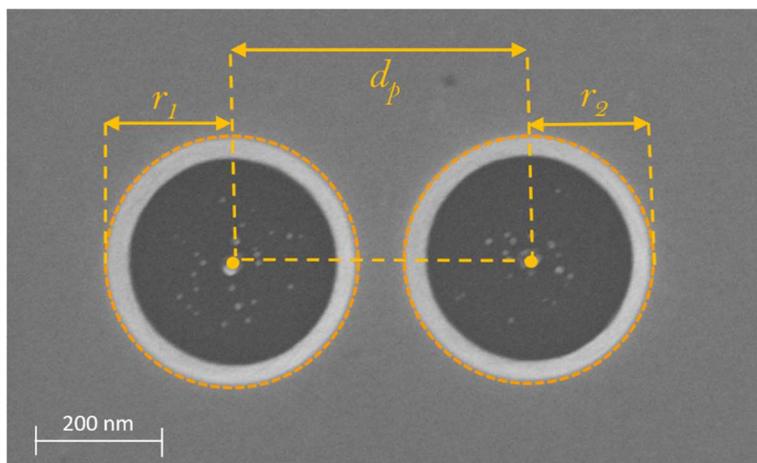


Fig.3. Determination of pores radius and distance between pores by using of plugin *Nnd*.

4. Analysis of SiO₂/Si ion-track template parameters

The results of determination of pores number, their total area, average size and standard deviation from this value, substrate porosity, as well as pore deviations from the round shape and average pore distance by using of *Analyze Particles* command and *Nnd* plugin are given in Table 1.

Table 1. Basic pore parameters determined by using of ImageJ.

Number of pores	Total pores area, μm^2	The average pore size, μm^2	Average standard deviation, %	Porosity, %	Pore deviation from round shape, %, %	Average distance between pores, μm
69	8,2	0,12	4	2,2	5	1,45

On the basis of obtained results, the average value of upper pore diameter (D) – $0.39 \mu\text{m}$ is easily determined. The total area of image was $371.84 \mu\text{m}^2$, it allows to calculate number of pores per unit surface – $1.85 \times 10^7 \text{ cm}^2$. The obtained value is lower than irradiation fluence ($2 \times 10^7 \text{ cm}^2$) because selected parameters of command *Analyze Particles* were set so as to automatically exclude pores, which are partially outside the image. The presence of such pores is easy to see during comparing of SEM image and its processed version (Fig. 1a and 1d). The average distance to the nearest neighbor of each pore was $1.45 \mu\text{m}$, it agrees well with theoretical calculations for the chosen fluence, given in [7]. It is important to note that standard deviation from average area value for each pore does not exceed 4%, and shape deviation from an ideally round is less than 5%. The small values of these parameters indicate the high quality of the SiO_2/Si templates obtained by ion tracks chemical etching.

Typically, the determination of pore parameters such as silicon oxide thickness after etching (l) and internal pore diameter (d) requires study of SiO_2/Si ion-track template cross section, which is time-consuming task. In addition, information on thickness of template obtained by using of SEM depends strongly on quality of prepared SiO_2/Si ion-track template cross section and orthogonally of sample cleavage plane electron beam. An example of SiO_2/Si ion-track template cross section with schematic representation of main pore parameters is presented in Fig.4.

It should be noted that for l and d determination, the method for estimating of thickness of SiO_2/Si ion-track template based on information on upper pore diameters obtained during post-processing of SiO_2/Si ion-track template surface images can be used. It is known that etching parameters of irradiated SiO_2/Si substrates and characteristics of resulting pores are interrelated through a half-pore angle θ , which is determined by irradiation parameters. Earlier, in work [7] it was shown that θ at high energies of irradiating ions (electronic energy value $(dE/dx)_e > 16 \text{ keV} \times \text{nm}^{-1}$), pore characteristics do not depend on irradiating ion type, and the half-angles of etching cone are in the range of $17^\circ \pm 2^\circ$. Calculation of silicon oxide thickness after etching and internal pore diameter of SiO_2 can be performed by correlations:

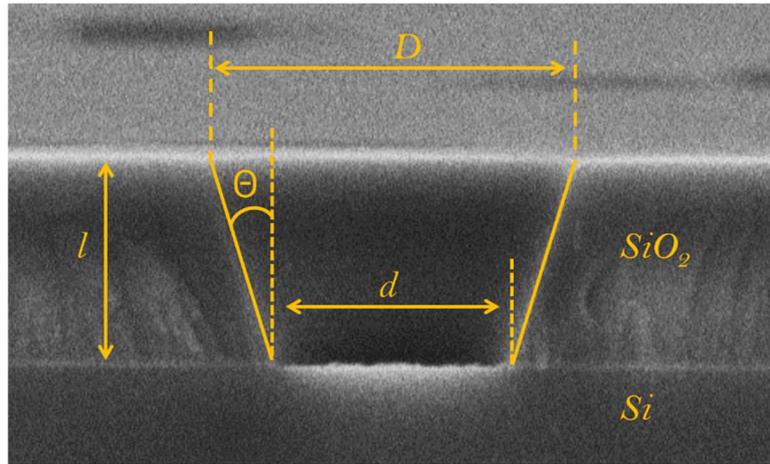


Fig. 4. SEM image of the SiO₂/Si ion-track template cross section with schematic representation of main pore parameters: D – upper pore diameter; l – silicon oxide thickness after etching; d – internal pore diameter; θ – half-pore angle.

$$l = L_{SiO_2} - \frac{D}{2} \sqrt{\frac{1+\sin\theta}{1-\sin\theta}} \quad (2)$$

$$d = D - 2l \cdot \tan\theta. \quad (3)$$

During estimating of l and d , half angle was assumed equal to 17° and pore characteristic values determined as a result of calculations were $l = 0.257$ and $d = 0.231 \mu\text{m}$. It should be noted that information on θ is not known in advance, when irradiation energy of corresponding electronic loss energy value $16 \geq (dE/dx)_e \geq 4 \text{ keV} \times \text{nm}^{-1}$, but once obtained information on this parameter it can be extrapolated to all experiments of pore SiO₂/Si ion-track template etching with the selected energy and ion type by using of equations (2) and (3). Thus, the conducted studies visually demonstrate that using of ImageJ for processing of SEM images allows to obtain a large amount of statistical data on template parameters and significantly saves time during research.

Conclusion

Ion-track SiO₂/Si templates were formed by irradiation with swift heavy ions and subsequent treatment in hydrofluoric acid. The study of oxide pores containing surface of silicon was made by using of scanning electron microscopy. The technique of post-processing of obtained SEM images by using of ImageJ software package for obtaining and analyzing statistical data is described. The possibility of determining such template parameters as number of pores, their total area, average size and standard deviation, porosity, as well as deviations of pore shape from the circular and average distance between pores are demonstrated. It is shown that standard deviation from the mean value of average area for each pore does not exceed 4%, and the deviation of shape from an ideally round less

than 5%, it indicates high quality of SiO₂/Si templates obtained by ion tracks chemical etching. Taking into account univocal mathematical relationship of upper pore diameter determined by SEM-study with thickness of silicon oxide after etching and internal pore diameter of SiO₂ pores through a half-pore angle, method for their evaluation is proposed. Since length and diameter can't be directly determined in the usual analysis of template surface, the described estimation method greatly simplifies the process of SiO₂/Si templates parameters analyzing and significantly saves time for obtaining the data by eliminating the stage of complex sample preparation. Thus, due to the high precision and low cost of research, the SEM method for studying of SiO₂/Si templates surface is an effective tool for characterization of pore parameters and, importantly, can be used for other types of porous matrices.

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