

# Determination of the size of nanoparticles in photonic nanostructures from AFM images

**A A Akhmadeev and M Kh Salakhov**

Kazan Federal University, 420008, Kremlevskaja str., 18, Kazan, Russian Federation

E-mail: Albert.Akhmadeev@kpfu.ru

**Abstract.** In this paper we propose a relatively simple algorithm for determining the size of ellipsoidal particles on atomic force microscopy (AFM) image. The proposed algorithm frees researchers from the “manual” measuring of the size of a large number of particles. This algorithm is based on a three-dimensional approximation of the segmented images by ellipsoids and it has several advantages over similar methods. One of important advantages is the ability to determine the parameters of particles the surface of which is not completely visible on the image. Proposed method has been tested on simulated images as well as on the experimentally obtained AFM images of silica particles. Especially the method is very useful for the study of opal-like photonic crystals, in which the particles are packed tightly.

## 1. Introduction

Recently, advanced materials consisting of micro- and nanoparticles have received considerable attention of researchers. These materials are widely used in various areas including applied physics, chemistry and biology. Such materials include photonic nanostructures and photonic crystals [1]. The structure of photonic crystal is characterized by periodic distribution of refraction index in the spatial directions so far it has its own scheme of electromagnetic states and the photonic band gaps in the spectrum. With the advent of devices such as a scanning electron microscope (SEM) and atomic force microscope (AFM), it was possible to visualize the surface of nanostructured materials and investigate their properties. The particle size is one of basic characteristics of these materials because it influences a large variety of physical, electronic, and optical properties. Also, the size of the particles of photonic crystal could be crucial for the manifestation of a new quantumelectrodynamical effect predicted in earlier works [2–4]. Thus, accurate determination of the size is an important task [5–8]. In this paper we developed a relatively simple algorithm that allows determining the size of a large number of ellipsoidal particles on AFM images.

## 2. Algorithm for determining the particle size

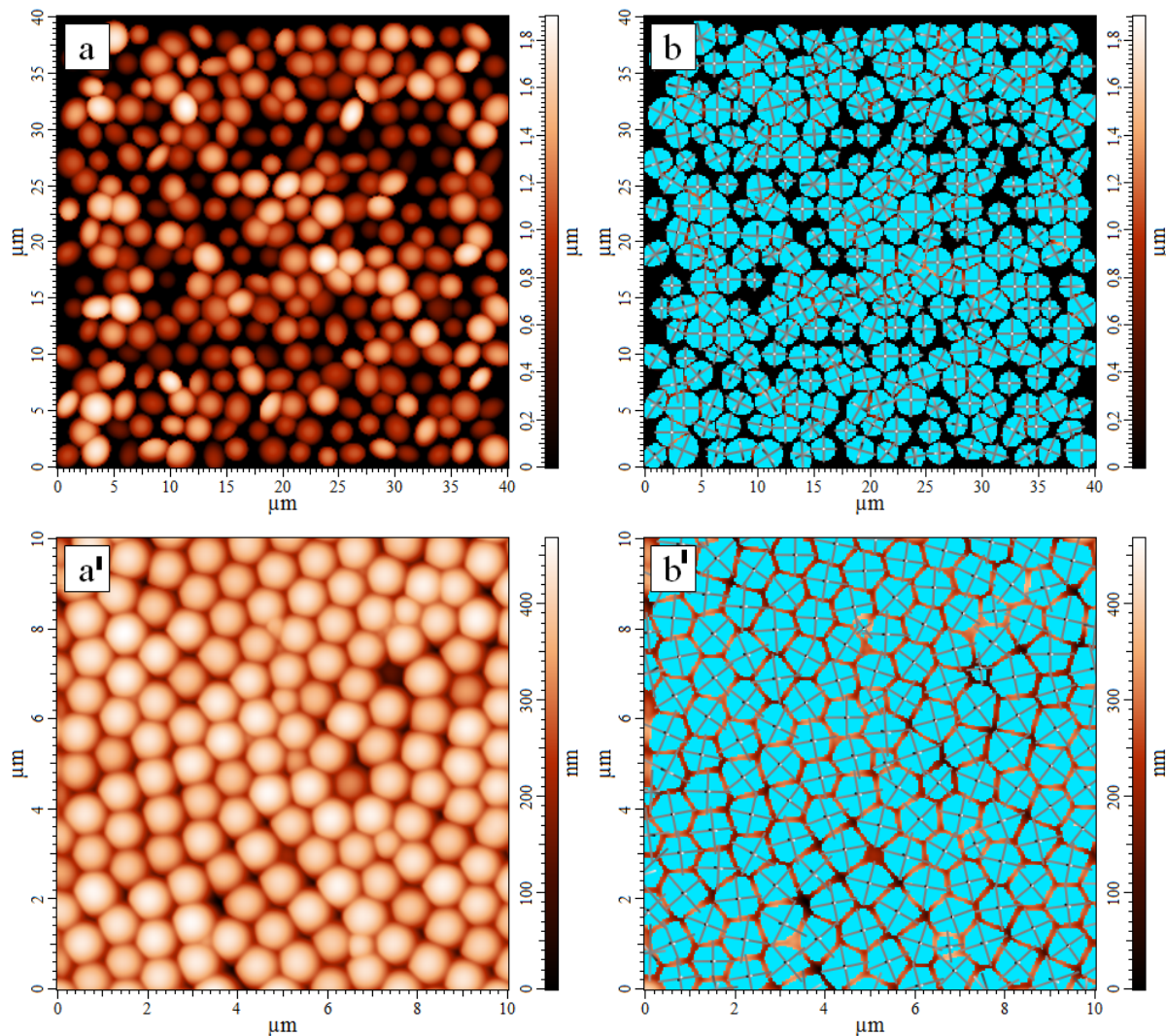
Many existing methods for determining the size of spherical particles on microscopy images include two basic steps:

- 1) Transformation of the original image (usually obtained by SEM) to monochrome using special filters (e.g., Canny [9], Sobel [10] filters). Result of this transformation is an image containing only the outlines of particles.



- 2) Recognition of the outlines by Hough transform [11], or analogous methods. Thus, the outline of each particle is mapped to a circle whose diameter is approximately equal to the particle size.

The considered approach is not free from drawbacks. In the case of some of the particles on the image are not completely visible (e.g., the particles are covered by other particles) their observable outline will not match the actual boundaries of the particle thus leading to incorrect results.



**Figure 1.** Processing of AFM image on the basis of three-dimensional approximation by quadric surfaces. The simulated and real AFM image of particles (a) and (a') correspondingly and its segmentation into approximated ellipsoids (b) and (b').

The technique proposed in this paper does not have such a drawback, since it utilizes an important advantage of AFM – 3D-visualization of sample surface. Thus, scanning the sample surface by atomic force microscopy provides an image that represents two-dimensional array of heights  $Z_{i,j}$ , corresponding to the sample topography. We propose determining the particle sizes on these images via the geometric curvature, instead of particle boundaries. To this end the set of points of  $Z_{i,j}$  are approximated by quadric surfaces (sphere and ellipsoid are special cases), described by the equation:

$$a_{11}x^2 + a_{22}y^2 + a_{33}z^2 + 2a_{12}xy + 2a_{13}xz + 2a_{23}yz + b_1x + b_2y + b_3z + c = 0 \quad (1)$$

The origin of coordinate system is chosen so that  $c \neq 0$ . Thus, the approximation is reduced to finding the nine independent parameters ( $a_{ij}$ ,  $b_j$ ) in the equation (1). Using the method of least squares (OLS) these parameters can be found by solving a system of linear equations (case of linear regression).

But primarily, the input image should be segmented, where each segment represents a set of points in the array  $Z_{ij}$ . At the same time, these segments must conform to the individual particles. Since the particles have a convex surface, then the following conditions are satisfied:

$$\frac{\partial^2 Z}{\partial x^2} < 0, \quad \frac{\partial^2 Z}{\partial y^2} < 0 \quad (2)$$

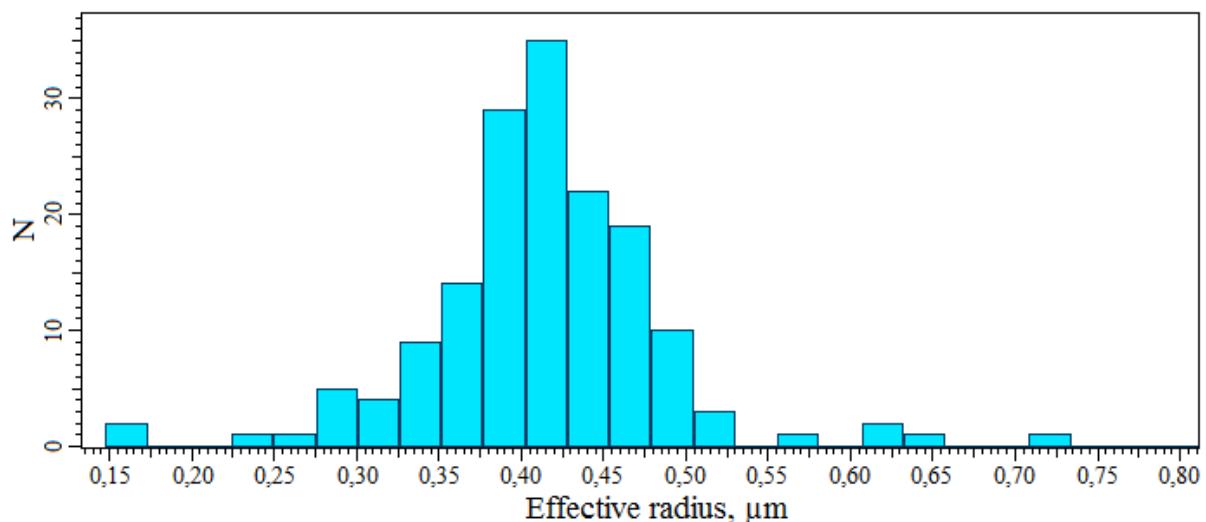
Because the particle surface on AFM image is represented as an array  $Z_{ij}$  instead of a continuous function, derivatives in (2) are replaced by the differences:

$$Z_{i-1,j} - 2Z_{i,j} + Z_{i+1,j} < 0, \quad Z_{i,j-1} - 2Z_{i,j} + Z_{i,j+1} < 0 \quad (3)$$

All points ( $i,j$ ) satisfying the condition (3) are distributed on separate groups, each group corresponds to a single particle. So the proposed method consists of the following steps:

- 1) Obtaining AFM images of micro- or nanoparticles (as an array).
- 2) Segmentation of the array into groups of points satisfying the condition (3).
- 3) Approximation of each group of points by quadric surface using OLS.
- 4) Finding the transformation to a canonical form for a quadric surface.

As a result of this procedure the following parameters are determined for each particle on the AFM image with the shape close to an ellipsoid: coordinates of the center, length of the semiaxes and their orientation in space.



**Figure 2.** Distribution of effective radius of silica particles.

### 3. Verification of the algorithm on simulated and experimental images

To verify the described method, images containing spheres and ellipsoids have been simulated (figure 1 a), each particle has a predetermined parameters. Filled areas in figure 1 (b) correspond to groups of points in the array  $Z_{ij}$ , which satisfy the condition (3), the approximation of which yielded the output

parameters: the length of the semi-axes, coordinates of the centers and three orthonormal vectors showing the orientation of the axes of the ellipsoid. Output parameters with high precision coincide with the input parameters of the ellipsoids; the difference in particle size does not exceed  $10^{-8}$  nm. Figure 1 (b) also shows the orientation of each ellipsoid, obtained from the image processing by our method.

As can be seen from figure 1, this technique is able to handle with particles which are covered by other particles, and particles that cling together. By applying this algorithm to real AFM image of silica particles ( $\text{SiO}_2$ ) the sizes and orientations of the particles were determined (figure 1 a' and b'). The particles of  $\text{SiO}_2$  were obtained by hydrolysis of orthosilicic acid (tetraethoxysilane, TEOS), under the action of a catalyst (Stober method [12]). Such particles work as a building material for the preparation of colloidal photonic crystals [13, 14]. Distribution of effective radius of these particles is shown in figure 2, where the effective radius is the geometric mean of semiaxes of the ellipsoid ( $R = \sqrt[3]{abc}$ ).

#### 4. Conclusions

Thus, we have proposed a relatively simple algorithm that allows rapid determining of the size and spatial orientation of the particles whose shape is close to an ellipsoid. The results show that the algorithm allows determining the parameters of particles that are not completely visible on the image.

#### Acknowledgements

This work was funded by the subsidy allocated to Kazan Federal University for the state assignment in the sphere of scientific activities.

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