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Determination of the fractal size of titanium films at different scales

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Abstract. The morphology of the relief of nanosized titanium films on the mica surface was studied using a scanning probe microscope at various scales. The characteristic features of the nanorelief of the surface of the investigated films, including fractal properties, are described. The obtained data on the fractal dimension are compared with the available experimental data, as well as the data obtained using scanning tunneling microscopy. Recommendations for the development of technology for «growing» structures with a given surface morphology are proposed.

1. Introduction
The application of scanning probe microscopy methods to the study of nanosized films' morphological characteristics on dielectric surfaces is an urgent problem for the intensive development of the physics of surface phenomena at the nanoscale [1]. For probe microscopy, the problems of analyzing and identifying the boundaries of objects and the problems of developing and testing methods for calculating the structural characteristics of nanocoatings – nanosized (in thickness) films – are currently relevant. In particular, titanium films with a thickness from several to tens of nanometers have potential applications in intelligent materials, micro-electro-mechanical systems, designing spintronic devices, and optical coatings that can significantly depend on fractal properties.

Our previous works were experimentally established [2-4] that films of gold, silver, nickel and copper on dielectric substrates can form fractal structures. In order to comprehensively study the morphological characteristics of the resulting coatings and individual agglomerates on the surface, including the fractal dimension, we consistently apply various methods of surface investigation to obtain and accumulate statistical data. There is an opinion [2] that the fractal dimension parameter can and should be used as an effective characteristic of the development of the micro- and nanostructure of a rough surface, and the fractal dimension equivalently replaces a whole complex of amplitude and step characteristics of the surface roughness.

However, as a rule, in technological processes it is required not only to use nanoscale films with a given value of the fractal dimension but also to control specific altitude parameters. Sometimes a comprehensive account of all the above parameters is required. Modern technologies for the artificial creation of relief details [5, 6] already make it possible to provide reproducibility of conditions for forming of nanosized metal films with a fractal structure on solid surfaces.

2. Experimental

Investigations of titanium films' surface topography were carried out at room temperature on a SolverNext scanning probe microscope (SPM) (NT-MDT SI LLC). A cantilever of the MFM10 series, designed for measurements with a high spatial resolution, was used as a probe. A special protective coating helps to avoid oxidation and significantly increases the service life. Simultaneously, the cantilever's radius of curvature remains relatively small for obtaining images with a high spatial resolution - about 20-30 nm. The typical value of cantilever resonant frequency is 150 kHz, typical force constant is 5.1 N/m. The cantilever is coated with aluminum on the reflective side to increase the laser signal. When scanning in the semi-contact mode (atomic force microscopy), we recorded the surface relief. Titanium films were formed on a mica substrate by electron beam sputtering on an A700QE/DI12000 setup. The value of the vacuum during the deposition was 10^{-4} Pa. Spraying speed 2.0±0.2 Å/s. Substrate size 50×40 mm^2. The thickness of the films obtained 90 nm with an error of ±2 nm. The substrate temperature during the deposition was 70°C. Six IR heaters are installed in the chamber, which heat the substrates before spraying to improve the adhesion properties of the surface. The chamber also has a quartz oscillator that controls the deposition rate in real-time with angstrom precision. Note that earlier in our works [2-4], films were produced only by thermal vacuum deposition.

3. Experimental results

The Figure 1 shows the surface profiles at different scales and 3D-images of titanium nanocoatings obtained using an atomic force microscope. As a rule to characterize the basic properties of fractal cluster aggregates – the self-similarity of their internal structures – the fractal dimension D_c is determined using the equation [7]:

$$N = \left(d / a\right)^{D_c}, \tag{1}$$

where N is the number of particles in the cluster (the number of monomers), d is the linear size, i.e. cluster (aggregate) diameter, a is the size of the particles that the cluster consist of (average monomer size). The fractal dimension of the profile D_L can be determined as follows using the fractal dimension D_f of the surface:

$$D_L = D_f - 1. \tag{2}$$

In this paper, the estimation of the fractal dimension and processing of AFM images of the surface was performed in the Image Analysis software package (version 3.5.30.19856). The proposed method for determining the fractal dimension is described in [8]. Below a brief overview of its provisions is presented. The following relation is used to determine the fractal dimension

$$D_c = 3 - \alpha, \tag{3}$$

where α is the scaling coefficient called the roughness index (Hurst index H). The Hurst index is determined through the slope of the initial section $tg\beta$ of the height-height correlation function for the selected direction, constructed in logarithmic coordinates according to the equation (4):

$$H = \tan\beta / 2\ . \tag{4}$$

Moreover, as was shown in [9], the fractal dimension of the surface D_f can be identified with the corresponding cluster dimension of the three-dimensional aggregates.

In our opinion, the technique [8] has several advantages, such as simplicity of calculation for the user and standardized error. However, it is known [2] that when determining the fractal dimension using mathematical image processing, it is necessary to take into account the «instrumental» error of two types: 1) image distortion; 2) own error of the method for determining the fractal dimension. The first error is associated with distortions introduced by the device itself and is removable, as well as with the image digitization procedure – «pixel effect». The previously used method for determining the fractal dimension [2-4] is primarily associated with determining a cluster's belonging to a selected section of the «grid» of measurements. In our opinion, this method provides a decrease in the measurement error.

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In this case, the researcher once takes into account the belonging of the image object to the «grid» section. It is absolutely not crucial that any section (even smooth) of the object boundary (that does not lie at an angle 0°, 90°, 180°) will be worse represented after digitization. Note that the use of software [8] does not remove the questions of the adequacy of the method for determining the boundaries of the object, the use of appropriate boundary conditions in the process of image processing, as well as the method for calculating the height-height correlation function for the selected direction. Using SPIP [10], a diagram of the angular distribution of the fractal dimension was constructed based on the analysis of the amplitude Fourier spectrum (see Figure 2). The results obtained and, in general, a fairly uniform angular distribution of fractal dimension confirm the applicability and adequacy of the methodology [8].

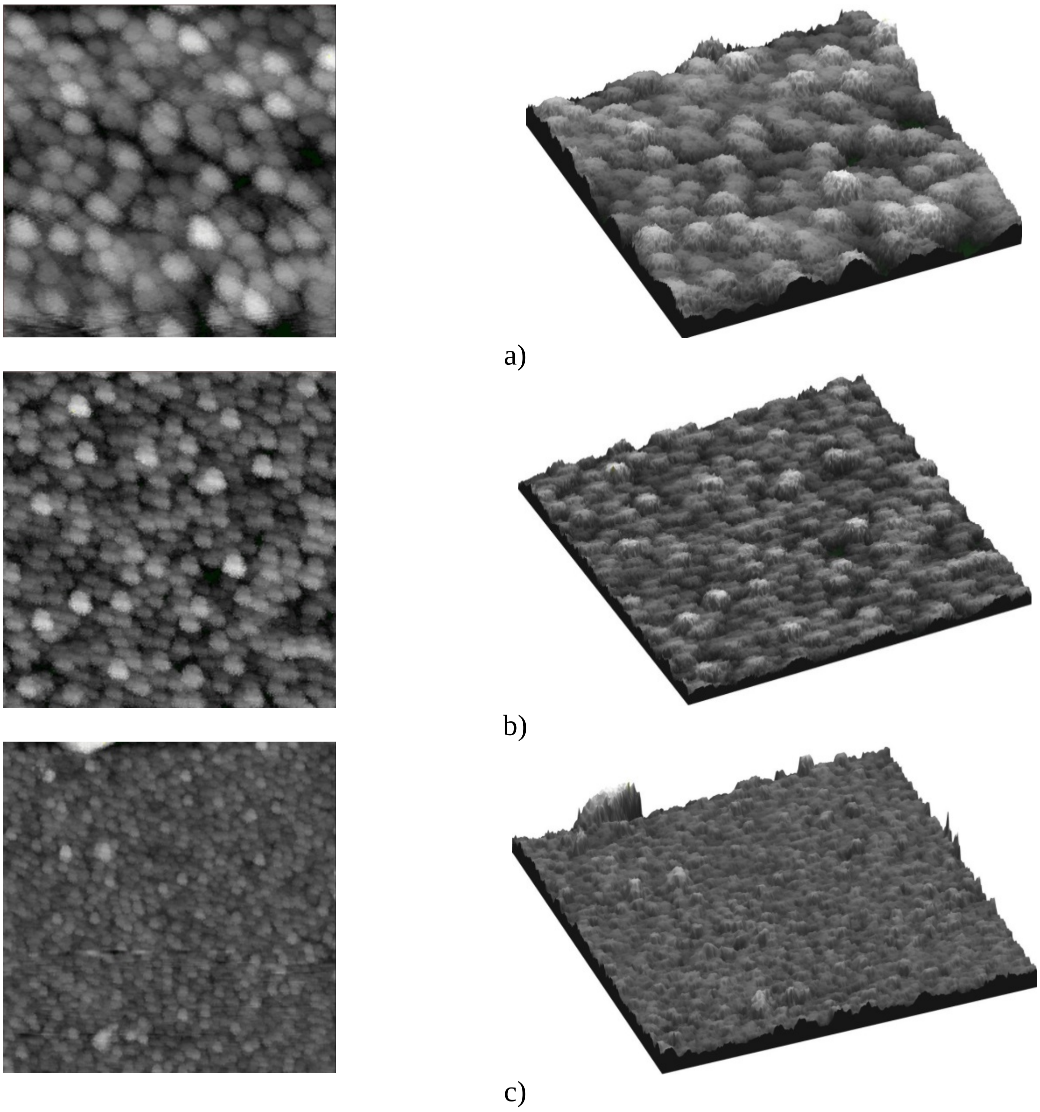
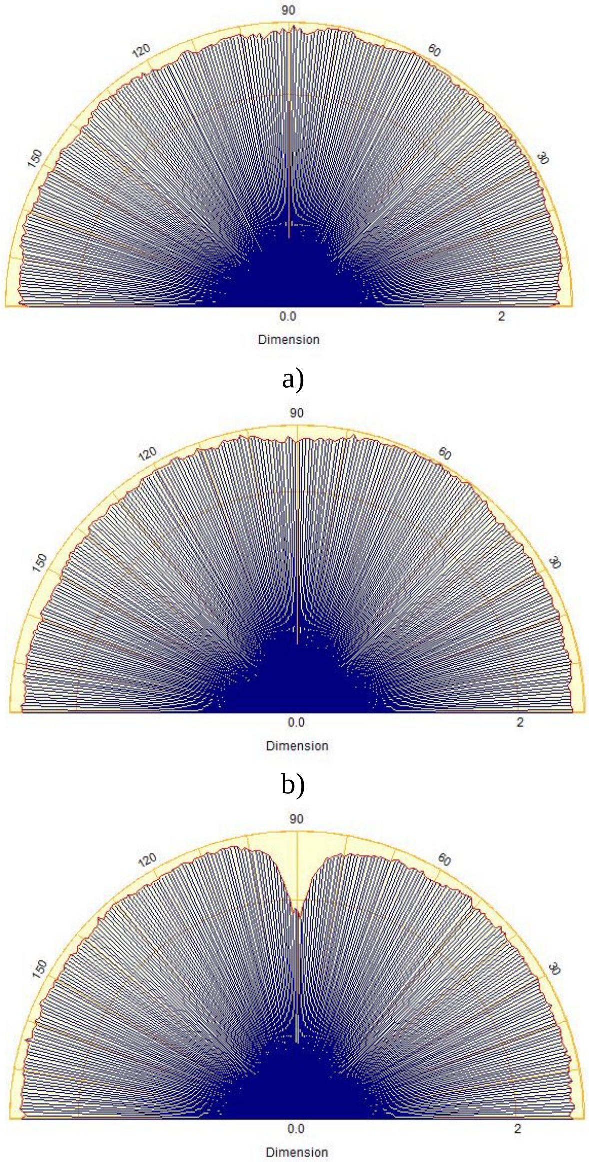


Figure 1. Surface images at different scales (left column) and 3D-images (right column) of titanium nanocoatings obtained using an atomic force microscope: a – scan area size 500×500 nm^2, b – 1000×1000 nm^2, c – 2000×2000 – nm^2.



c)

Figure 2. Angular distribution of fractal dimension of titanium nanocoating at different scales: a – scan area size 500×500 nm^2, b – 1000×1000 nm^2, c – 2000×2000 – nm^2.

The Table shows the results of calculations of altitude parameters: the arithmetic mean of the absolute values of the profile deviations within the base length S_a, the standard deviation S_q, the sum of the average absolute values of the heights of the five largest profile protrusions and the depths of the five largest profile valleys within the base length S_{10z}, the average fractal dimension D_c.

Table 1. Average values of the morphological characteristics of the titanium film on the studied scales.

Scale, nm	S_a, nm	S_q, nm	S_{10z}, nm	D_c
500	1.428	1.801	13.012	2.44
1000	1.219	1.535	11.136	2.52
2000	1.391	1.913	24.144	2.55

An analysis of the data in the diagram in Figure 3 shows that the chosen technique for obtaining titanium films of the nanometer thickness makes it possible to obtain thin films with a sufficiently developed fractal relief, while only moderate degradation of the relief is observed when passing from the largest of the considered scales to the smallest (see Table). Interestingly, for titanium dioxide the fractal analysis revealed that the value of the fractal dimension of the samples decreases slowly from 2.23 to 2.15 following the annealing process [11]. In [12] the fractal characteristics of titanium dioxide nanocrystalline films were studied and related to the efficiency of light absorption. The fractal dimension of the film surface was computed using atomic force microscopy and found the mean value $\boldsymbol{D_f}$ =2.33±0.02. Thus, for pure metals, characteristic values of the fractal dimension correspond to a sufficiently developed fractal relief. The values of the fractal dimension obtained by us are in good agreement with the value of 2.5 obtained for films with a thickness of 400-450 nm [13].

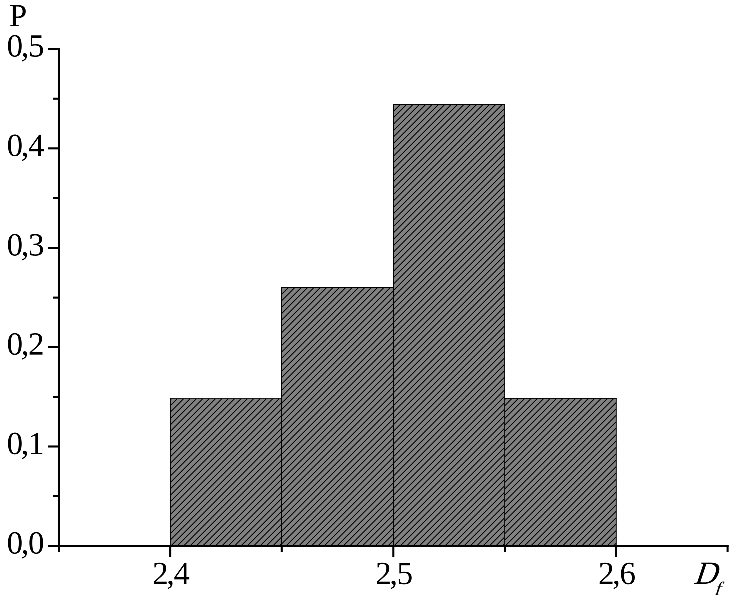


Figure 3. Generalized distribution of the probability $\boldsymbol{P}$ of detecting clusters with a certain fractal surface dimension on the titanium film surface.

4. Conclusion

The main technological solution for the creation and improvement of methods for «growing» structures with a given surface morphology, as well as structures with certain physical properties (conductivity, optical characteristics and etc.) is the selection of external factors (temperature substrates, external pressure, the presence of subsequent chemical treatment, etc.), and the use of certain techniques for preparing nanosized films [5, 6].

In this case, a computer experiment [14] can help to ensure reproducibility of results under fixed conditions (film preparation method, external factors, selected metal), description of specific features of nanoscale films (relief type (for example, «plateau» type), area of local zones in which the «fractal relief» has not formed, the limits of values for the fractal dimension of the profile and surface). Based on a comprehensive analysis of micro- and nanostructure together with magnetic properties, it was determined that the pinning mechanism governs the magnetic behavior of the explored alloys. It can be argued that a combination of the technique for obtaining nanoscale coatings and external factors, as well as taking into account the physicochemical characteristics, in particular, surface and interfacial tensions [15, 16], should ensure the formation of a «fractal relief» for metal films on dielectric surfaces, even if not over the entire sample area, but although would be locally on nanoscale. Figure 2 data predict a fairly isotropic profile at scales less than 1000 nm, it means that the surface has the same properties without considering of the direction. In this case, the surface texture can be investigated using 2D profile only.

Our preliminary studies show that the fractal dimension can slightly increase with increasing film thickness. Apparently, an increase in the deposition time makes it possible to activate to a greater extent the processes that determine the formation of the relief: spreading, spontaneous coalescence. This conclusion agrees with the data [13]. In contrast to [14], in our work the study of titanium films was carried out not at the microscale, but already at the nanoscale. We managed to achieve good resolution at scales 4 times smaller than investigated in [14]. In addition, the data of [14] do not allow one to analyze the angular distribution of the fractal dimension for different values of the deposition time. However, it should be understood that the growth of the fractal dimension will occur in a relatively narrow range of values if the control parameter is only the deposition time (or as a result the film thickness). For the formation of a high developed fractal relief, this parameter alone is clearly not enough, since, along with the fractal agglomerates growth, the process of degradation will also occur, which can only be slowed down by other external factors (temperature, beam density, the presence of stabilizing additives, etc.). In [14] degradation of high-rise surface irregularities – motifs, according to the authors, should suggest the formation of a more developed fractal relief. (The «motif» is used instead of «dale» and «hill» to determine a texture on a surface [17]).

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