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Small Angle X-ray Scattering in Thin Iron Films

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By small angle X-ray scattering (SAXS) and atomic force microscopy characteristic sizes are determined, structural features of thin iron films deposited by magnetron evaporation onto substrates from pyroceramics are established. It is shown that morphologically the film is characterized by disorder. It is formed from columnar nano crystallites that are oriented either perpendicular to the substrate or situated in its plane, which dictates polydispersity of those coatings. It is shown that SAXS may be thought of as nondestructive technique for analyzing structure and composition and conducting quality control of magnetron films.

Keywords: Small angle X-ray scattering, Atomic force, Scanning electron, Digital holographic microscopy, Thin magnetron films.

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1. INTRODUCTION

Thin-film materials that feature unique physicochemical properties, which are due in large part to dimensional effects and phenomena, find very broad application in both nanomaterial science and micro- and nanoelectronics that are being developed most intensively [1], including development of sensor devices, magnetic data carriers, protecting coatings, etc. Study of structural features of such entities presents a profound scientific problem. Transmission electron microscopy (TEM), as a rule, does not provide validity of obtained data any more. In this connection, small-angle X-ray scattering (SAXS) technique, which does not cause secondary effects due to X rays, can be successfully applied for studying the regularities of growth of thin-film materials, and the influence of their synthesis parameters on the structure and properties. The chief merit of this technique is that only this method, in fact, makes it possible to obtain statistical values of size, volume, surface area and forms of nanoparticles of substance. Unlike granulometric analysis, SAXS excludes the influence of human factors [2].

2. EXPERIMENTAL SECTION

Magnetron iron films were evaporated with facility GENCO onto substrates from pyroceramics the size of 40 × 60 mm, which provided high surface uniformity of films. Working pressure of argon in a chamber was 1 Pa. Power of plasma discharge was not greater than 400 W. These parameters of the facility are significantly distinct from those given in [3]. Topology of film surface and determination of an average size of the granular structure was investigated with scanning probe microscope (SPM) SmartSPM (AIST NT). Morphological features and changes in surface structure of materials studied were investigated with scanning electron microscope (SEM) (JEOL JSM-6610LV) with a magnification of up to 100000x at an accelerating voltage of 20 kV. Morphology of film surface was also studied with digital holographic microscope (Lyncee tec) with a height measuring accuracy of 0.1 nm.

Studies with SAXS were conducted with the help of facility SAXSessmc[2] (Anton Paar) in the linear collimation mode, which makes it possible to characterize structural features of objects with the size of 0.1 to 100 nm. To this end a typical X-ray tube was used with copper anticathode and a monochromator with X-ray radiation of 0.154 nm K_α line.

3. RESULTS AND DISCUSSION

Typical scattering curve according to SAXS is given in a logarithmic scale in Fig. 1.

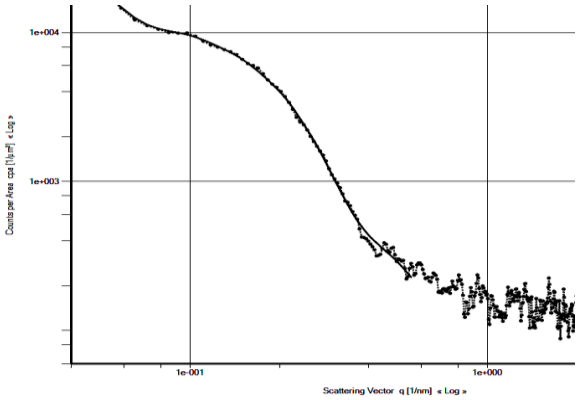

Fig. 1 – SAXS curve of a magnetron iron film at the substrate from pyroceramics (logarithmic scale)

Figure 2 shows SEM-image of an iron film with characteristic sizes of nanodimensional objects. It is seen some surface disorder when structures in both evaporation plane and as usual perpendicular to it are observed [3-5] with the formation of columnar nano crystallites. Disorder of thin-film structure is attested by its profile along the line of 350 μm length obtained with holographic microscope according to which the maximum height difference was 26.24 nm with an average roughness of not greater than 3.5 nm.

The shape observed was of two types: elongated in the substrate plane and regular of a circle form, which was probably the tip of a columnar nanocrystal structure

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perpendicular to the substrate surface. Granulometric analysis of AFM-images revealed that coatings studied had clearly polydisperse structures (Fig. 2b). The given particles had sizes varied within broad ranges from several tens to several hundredths of nm, among which prominent are formations of a cylindrical shape (up to 360 nm) composed from individual likely disk-like blocks of order 20 nm. It should be noted that sizes obtained with AFM-images are, as a rule, laterally overestimated due to spherical form of a cantilever probe (R = 15 nm). Real-world size form (Fig. 2) can be reduced by 30 %.

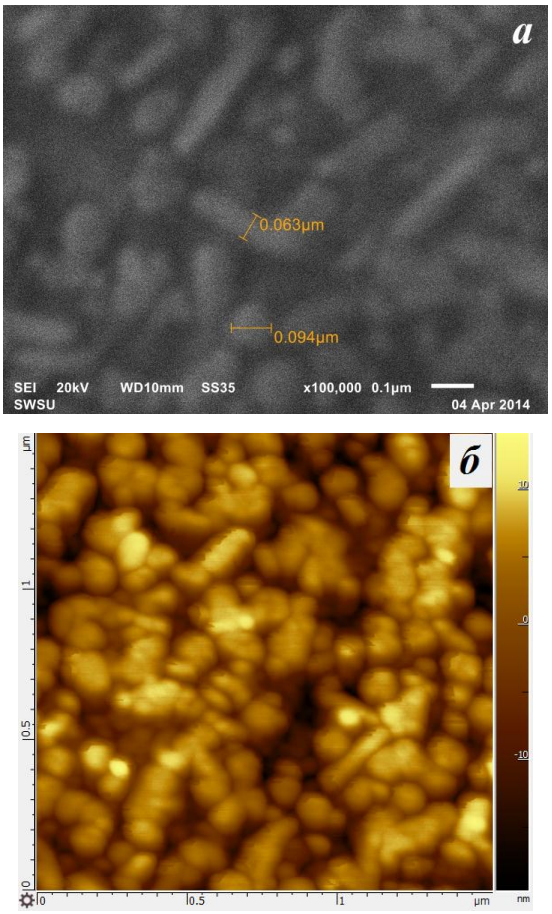

Fig. 2 – Electron-microscopic and atomic-force image of the iron film

As was noted, for studying disperse systems in such thin magnetron layers SAXS can be utilized. However, unlike the situation shown in [6] it is excluded in our case. Moreover, as AFM- and SEM-data reveal film magnetron layers on substrates from pyroceramics were formed from polydisperse nanoparticles and their agglomerates as columnar nano crystallites.

Processing of SAXS data (Fig. 1) was made with the program GIFT (PCG Software Package), which made it possible to compute the volume size distribution of particles $D_V(r)$ (Fig. 3) in analogy with [7]. The average sizes of particles forming the magnetron films were found to be 6, 22, and 114 nm. Evaluation of normalized number of various-size nanoparticles determined from the ration of their areas under their distribution reveals that particles with a radius of 11 nm take the large part of the volume in the film studied. Comparison of these sizes with data found with microscopic techniques suggests correspondence.

The validity of this approach stems from the following considerations. It is obviously that for monodisperse film there is a maximum size of nanoparticles that is determined from the distance distribution curve $p(r)$. The curve is computed from intensity of SAXS with the program of indirect Fourier transformation

$$p(r) = \frac{1}{2\pi^2} \int_{s=s_{\min}}^{s_{\max}} I_{\exp}(s) \frac{\sin(sr)}{sr} ds .$$

The length of the scattering vector – **s** (the vector equal to the difference between the vectors of scattered and incident waves) is determined from:

$$|\mathbf{s}| = 2|\mathbf{k}_0| \sin\theta = \frac{4\pi \sin\theta}{\lambda} ,$$

where 2θ is the, I_{exp} is experimentally observed intensity, and r is the distance between two scattering electrons. Whereas for polydisperse system one can determine the function of volume size nanoparticle distribution from the integral equation:

$$I(s) = \int_{R_{\min}}^{R_{\max}} D_V(R) m^2(R) i_0(sR) dR$$

Here $R_{\min}$, $R_{\max}$ and R are maximum and minimum sizes and the particle size, $i_0(x)$ and $m(R)$ are form-factor of particle (the particle shape is set a priory) and its volume. In computing $_V(R)$ the value $R_{\min}$ is taken equal to zero and the quantity $R_{\max}$ is chosen individually for each specific case by the most appropriate fitting. The function $D_V(R)$ may be thought of as appropriate if it does not provide negative spikes and approaches zero at a maximum value of R without a sharp fall-off, i.e. the computed scattering intensity curve coincides with the curve from experiment.

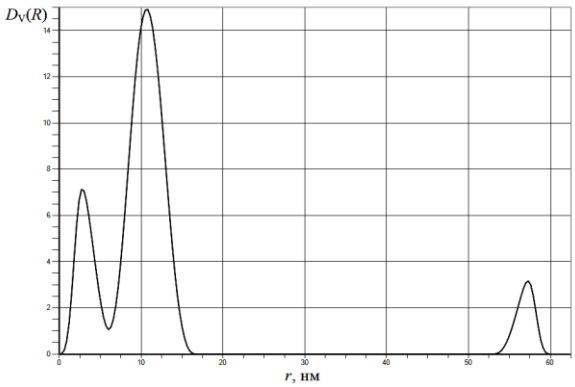

Fig. 3 – Volume size distribution of particles in iron magnetron film at the substrate from pyroceramics

4. CONCLUSIONS

The given inference is also corroborated by similar studies with copper magnetron films when the formation of columnar nano crystallites is observed with aspect

ratio $L = l / d$, whose value with characteristic magnitudes of diameter (d) and length (l) is 15-20. Such magnetron films also feature developed morphology; include columnar nanoparticles, which are oriented both perpendicular to the substrate and situated in its plane. This dictates polydispersity of those coatings and makes its analysis and examination difficult. In this connection, SAXS technique may be thought of as nondestructive technique for analyzing structure and composition and conducting quality control of magnetron films.

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