

Short Communication

Features of Structure of Magnetron Films  $\text{Si}_3\text{N}_4$  and  $\text{SiC}$

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By small-angle X-ray scattering and atomic force microscopy shows the features of the structure of thin films of  $\text{Si}_3\text{N}_4$  and  $\text{SiC}$ , deposited by magnetron sputtering on glass substrates.

**Keywords:** Small-angle X-ray scattering, Atomic force microscopy, Silicon nitride, Silicon carbide, Thin films.

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1. INTRODUCTION

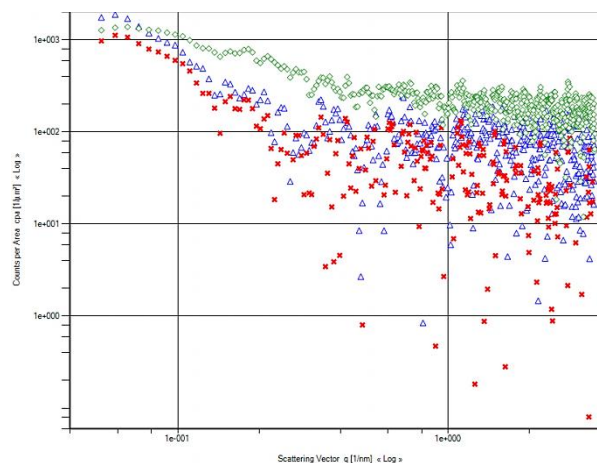
Nitride and silicon carbide have become the center of attention for designers of new generation of diodes and transistors, nonvolatile storage devices, which is due to unique combination of their characteristics (the energy gap width in  $\text{Si}_3\text{N}_4$  is 9 eV and  $\text{SiC}$  – 3.02 eV, concentration of electrons and holes is  $10^{19} \text{ cm}^{-3}$ , critical field intensity is 4 MV/cm, Debye temperature up to 1430 K and thermal conductivity up to  $4.9 \text{ W/cm} \times \text{K}$ , velocity of volume electron charge is  $2 \times 10^7 \text{ cm/s}$ ) [1, 2]. In addition to this, these compounds are ceramic, feature a number of strength properties in terms of mechanics, and are noted for a variety of synthesis techniques [3]. However, this circle of technologies becomes considerably narrower applied to materials of microelectronics. For these goals techniques that provide materials with high stoichiometry values become practically important, namely, CVD, ALD, and others to which there belongs the magnetron deposition method [3, 4, 5].

2. EXPERIMENTAL SECTION

The structure of thin films  $\text{Si}_3\text{N}_4$  and  $\text{SiC}$  deposited with this method onto glass substrates was studied with small-angle X-ray scattering (SAXS) with the apparatus SAXSess mc<sup>2</sup> (Cu K $\alpha$   $\lambda = 0.154 \text{ nm}$ ) [6], atomic force microscopy (AFM) SmartSPM, and changes in chemical structure were detected with Raman microspectrometer (OmegaScope,  $0.8 \text{ cm}^{-1}$ , 50 nm). The thickness of deposited films determined with interferometric technique was several nanometers, which limited the potential to study them with electron scanning microscopy. Raman spectrum of  $\text{SiC}$  corresponded to a cubic structure, did not have amorphous halo, and slight widening of characteristic peaks 984, 970, and  $953 \text{ cm}^{-1}$  may be due to small thickness of the films studied. Films  $\text{Si}_3\text{N}_4$  were deposited on the substrate both at room temperature and heated to 300° C.

3. RESULTS AND DISCUSSION

Grain-size analysis of AFM-images for  $\text{Si}_3\text{N}_4$  has revealed that coatings under study are of polydisperse



**Fig. 1** – Curve of SAXS for the magnetron film  $\text{Si}_3\text{N}_4$  on a glass substrate:  $\Delta\Delta\Delta$  – at room temperature;  $\blacksquare\blacksquare\blacksquare$  – heated to 300° C;  $\circ\circ\circ$  –  $\text{SiC}$  (logarithmic scale)

structure (Fig. 2a, b). The particles have the size of several to hundreds of nanometers. Large formations of nanoparticles present nanocrystallites. The structure of film  $\text{SiC}$  (Fig. 2) is monodisperse and formed by nanoparticles the size of  $r_c = 15 \text{ nm}$ . It should be noted that  $r_c$  obtained by the AFM-image presents overestimated value since was found with a probe with a curvature radius  $R = 7 \text{ nm}$ . So in determining real-world sizes one needs to take into consideration the relation:  $r = (2R)^{1/2}$ , according to which the particle size equals around 4 nm.

Solving the inverse scattering problem in analyzing mono- and polydisperse films  $\text{SiC}$  and  $\text{Si}_3\text{N}_4$  with SAXS curves must be made with consideration for their structural distinction. By analyzing curves of distance distribution at SAXS in monodisperse film the maximum nanoparticle size can be found according to expression.

$$p(r) = \frac{1}{2\pi^2} \int_{S=S_{\min}}^{S_{\max}} I_{\text{exp}}(s) \cdot \frac{\sin(sr)}{sr} ds$$

The length of scattering vector  $s$  (the difference

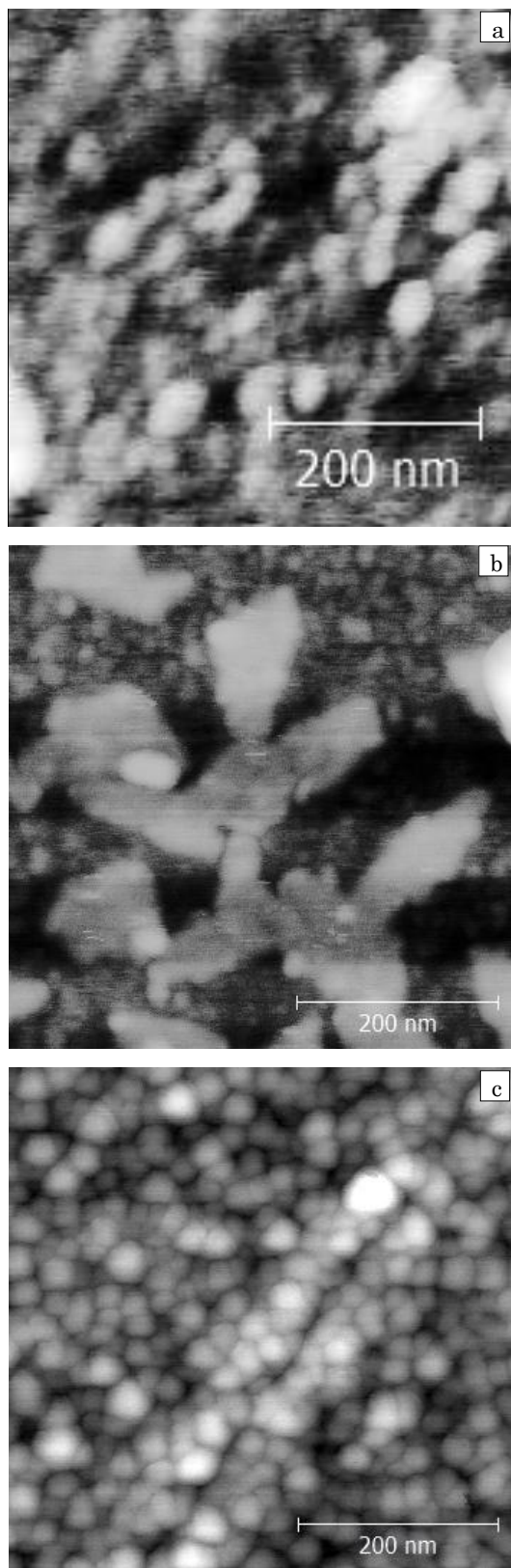


Fig. 2 – AFM-image of Si<sub>3</sub>N<sub>4</sub> films: a – at room temperature; b – at 300 °C; c – SiC

between vectors of scattered and incident waves) can be determined from equation

$$|s| = 2|k_0| \sin \theta = \frac{4\pi \sin \theta}{\lambda},$$

where  $2\theta$  is Bragg scattering angle,  $I_{\text{exp}}$  is experimentally observed intensity, and  $r$  is the distance between two scattering electrons. For polydisperse system one can determine the function of size distribution of nanoparticles

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

Here  $R_{\min}$ ,  $R_{\max}$  and  $R$  are minimum and maximum size and particle size, respectively,  $i_0(x)$  and  $m(R)$  are form-factor and volume of the particle (the particle shape is given a priory). In calculating  $D_V(R)$   $R_{\min}$  is taken equal to zero and  $R_{\max}$  is chosen for each individual case by adjustment. The function  $D_V(R)$  is thought to be appropriate if it does not have negative spikes and approaches zero at a maximum size of  $R$  without an abrupt break, and when the calculated curve of intensity scattering coincides with experimentally observed one.

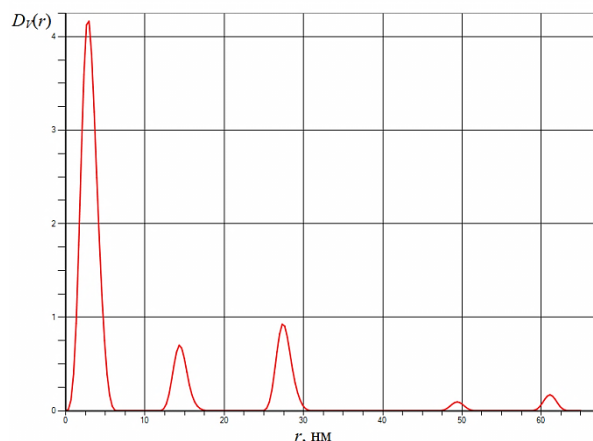


Fig. 3 – Volume size distribution of particles in magnetron film Si<sub>3</sub>N<sub>4</sub> at a substrate at room temperature

Treatment of obtained curves of SAXS was carried out with the program GIFT (PCG Software Package), which made it possible to compute the volume size distribution functions of nanoparticles  $D_V(r)$ , of which characteristic form is given in Fig. 3. Obtained particle sizes according to SAXS are provided in Table 1. The estimation of normalized number of particles with various sizes found from the ratio of areas under their distributions indicates that the great part of volume in

Table 1 – Particle radius in magnetron films Si<sub>3</sub>N<sub>4</sub> and SiC

Film and substrate temperature	Particle radius, nm
Si <sub>3</sub> N <sub>4</sub> at $T_r$	2.8, 14.5, 27.5, 49.4, 61.2
Si <sub>3</sub> N <sub>4</sub> at $T = 300^\circ\text{C}$	4.8, 15.2, 26.5, 68.4
SiC at $T_r$	1.3, 10, 25.8

$\text{Si}_3\text{N}_4$  at a substrate at room temperature is taken by particles with a radius about 2.8 nm. Whereas in this film at a hot substrate the minimum size increased to 4.8 nm, and in SiC the minimum size equals 1.3 nm and is really dominant.

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#### 4. CONCLUSIONS

Thus, a statistical distribution size of particles in magnetron films SiC and  $\text{Si}_3\text{N}_4$  obtained by small angle X-ray scattering data agrees with results of atomic force microscopy.