Investigation of Structure Formation Modes for Metasurfaces and Confocal Optical Systems

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Polycrystalline silicon/silicon oxide on silicon substrate structures are obtained by plasma chemical vapor deposition. The effect of rf power, gas mixture pressure, and vacuum annealing temperature on the roughness, mechanical stresses, and refractive index of layers has been studied. It is shown that vacuum annealing makes it possible to substantially correct the values of material parameters. The conditions for formation of structures suitable for further fabrication of metasurfaces and optical elements and systems based on them are established.

Keywords: metasurface, silicon on insulator, silicon oxide, plasma-enhanced chemical vapor deposition.

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1. The use of metamaterials is one of the promising ways to solve the problems of light control inherent in the refractive and diffractive elements of optical devices [1–3]. Metasurfaces, which are two-dimensional metamaterials, do not have the disadvantages of bulk materials. By rationally designing metasurface elements (metaatoms) and cells with them, it is possible to control the amplitude, phase and polarization of reflected, transmitted or diffracted waves. This makes it possible to effectively control the radiation, for example, by collecting plane wave into a point at the focal length, which allows to produce metalenses with a thickness less than the radiation wavelength [1,4]. Various methods are used to form metasurfaces, including deposition, lithography, and etching [5–7]. These techniques make it possible to create metasurfaces and confocal optical systems with various characteristics, as well as to optimize their production [5–10]. However, the main problem of their formation remains the lack of serial technologies that allow reproducing the metasurfaces of the required functionality, parameters and size, and such situation limits the possibilities of their use for solving practical problems. The most suitable material for the formation of such metasurfaces are „silicon on an insulator" (SOI substrates) structures [6–8], as well as similar structures, in which the device layer of silicon is replaced by a layer of polycrystalline silicon [9,10].

The purpose of this paper is to study the modes of plasma-chemical deposition and subsequent vacuum heat treatment of polycrystalline silicon structures on insulator, and to study their physical, mechanical and optical properties for the development of fabrication technology of metasurfaces and confocal systems.

2. The paper studied the formation of structures for the production of metasurfaces consisting of layers of silicon dioxide (up to $2\,\mu$m thick) and polycrystalline silicon ($1.2\,\mu$m thick). Silicon wafers KEF (100) with diameter 100 mm, $460\,\mu$m thick were used as substrates. The substrates were subjected to standard RCA cleaning. A layer of silicon oxide $2\,\mu$m thick was formed using plasma-enhance chemical vapor deposition (Oxford Instruments PlasmaLab System 100 PECVD) [11] with the following parameters: temperature 350°C, ratio of flows of monosilane, argon and nitrous oxide 1 : 28 : 84, pressure 1 mmHg., plasma power 20 (sample # 1) and 50 W (sample # 2), pressure 2 mmHg., plasma power 50 W (sample # 3). In this case, the deposition rate was $66.7-128.1$ nm/min. The resulting structures were annealed using equipment for rapid heat treatment (ZAO „NTO" STE RTA79) in vacuum at a temperature of 450 600°C.

The morphology was studied using atomic force microscopy (SPM NTegra Vita) and stylus profilometry (KLA Tencor AlphaStep D-100). Based on morphology data, we calculated the roughness parameters in the field $5 \times 5\,\mu$m (AFM) and scanning length $983\,\mu$m (profilometry) and mechanical stresses in films using the technique [12] at scanning length 10 mm. The refraction index values were obtained from ellipsometric measurements (LEF-3M, wavelength 632.8 nm).

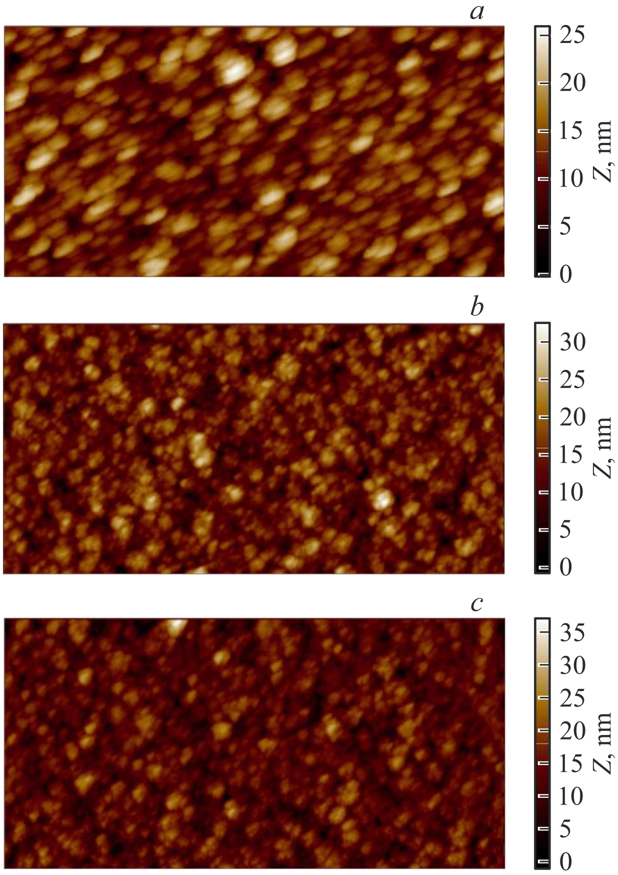

Figure 1. AFM-images (field $2.5 \times 5\,\mu$m) of silicon oxide films after deposition (*a*), annealing at 450°C (*b*) and 600°C (*c*) of sample # 1.

Table 1. Average (Ra) and root mean square (Rq) values of the surface roughness of silicon oxide films (sample type # 1) in the field $5 \times 5\,\mu$m

Parameter	Temperature, °C		
	Deposition	Annealing	
	350	450	600
Ra, nm	3.01	3.34	3.43
Rq, nm	3.80	4.20	4.31
Average grain size, nm	187	113	104

Table 2. Average (Ra) and root mean square (Rq) values of surface roughness of silicon oxide films along scanning length $983\,\mu$m

Sample type	Parameter	Temperature, °C		
		Deposition	Annealing	
		350	450	600
# 1	Ra, nm	7.59	5.71	5.11
	Rq, nm	9.79	8.55	7.91
# 2	Ra, nm	7.48	6.87	6.60
	Rq, nm	13.22	12.21	10.61
# 3	Ra, nm	7.94	7.41	6.80
	Rq, nm	14.10	13.20	13.07

3. A series of samples of three types with the structure of a layer of silicon oxide on silicon wafer (8 pieces in total) were manufactured. Studies of the surface morphology of formed films after plasma-chemical deposition and heat treatment (annealing) showed that with temperature increasing, the average grain size and surface roughness (along the scanning length) decrease (Figure 1, Table 1, 2). However, according to AFM data (see Table 1), with annealing temperature increasing, the roughness undergoes a slight (up to 3%) increase. This may be due to the difference in the study techniques used, namely different scanning areas (a line in profilometry and a square area in AFM) and the tip radius of the probes used. At that, the values of roughness parameters calculated from the profiles are approximately by two times higher in comparison with AFM data. They are also characterized by a greater deviation from the average value. This is partly due to the more likely inclusion of artifacts in the analyzed area. In previous studies, the deposition temperature had a similar effect on the surface structure of the film less than 100 nm thick [11].

Mechanical stresses in silicon oxide films $2\,\mu$m thick, calculated from profilometry data, were compressive and averaged from -165 to -240, from -50 to -115 and -5 to -140 MPa after deposition and annealing at 450 and 600°C, respectively (Figure 2). The obtained values are consistent with the data [12,13]. For all types of samples, stress relaxation was observed during annealing. The least stressed films were obtained at the annealing temperature of 450°C. However, with a further increase in temperature in samples formed at higher plasma power, the stress relaxation curve experiences saturation, and in the case of pressure of 1 mmHg. There is a tendency towards their accumulation.

The effect of plasma deposition conditions and heat treatment on the refraction index is also shown in Figure 2. With increase in temperature, not only the grain size in the silicon oxide film decreases, but also the stoichiometry and associated optical properties change. An assessment of the stoichiometric index of experimental films based on the dependence of the refraction index obtained in [14] supposes that the formation of films can occur under conditions of some excess of oxygen (lack of silicon), especially at low plasma power, which affects to some extent on the structure density reduction. This can be confirmed by the higher etching rate of such a film in liquid solutions in comparison with a film subjected to thermal annealing. Upon annealing (with temperature increasing), the composition of the oxide films was restored

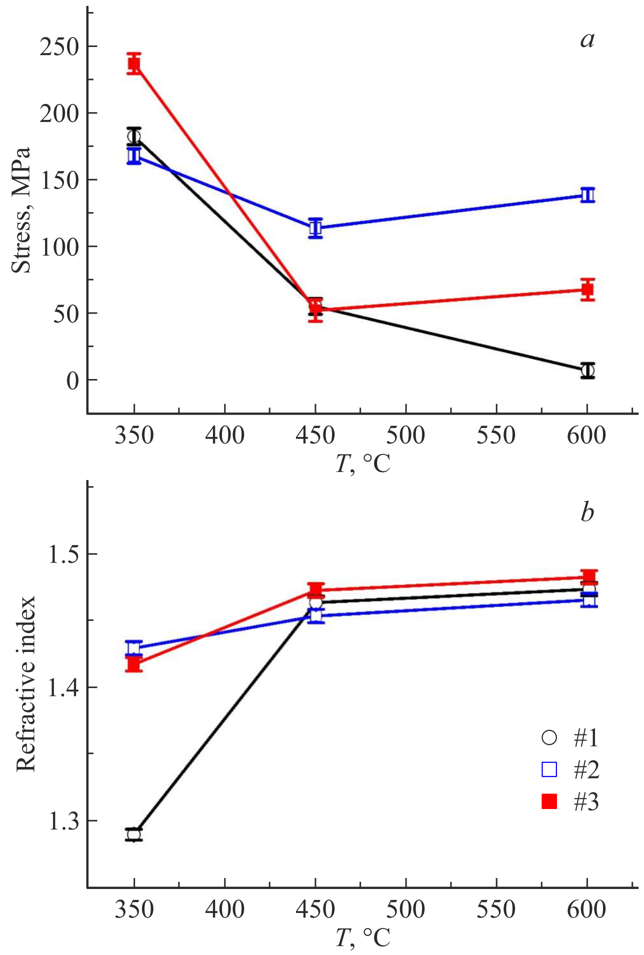

Figure 2. Change in the modulus of compressive stress (*a*) and refraction index (*b*) in silicon oxide films during annealing of samples ○ — # 1, □ — # 2 and ■ — # 3.

towards stoichiometric silicon dioxide, which corresponds to the refraction index values 1.46–1.48 (SiO_2 1.45–1.48) in Figure 2.

The noted nature of the influence of the conditions for the silicon oxide layer formation with subsequent heat treatment in vacuum, as well as the numerical values of the studied parameters, should be taken into account when developing a technology for (amorphous, polycrystalline) silicon on insulator structure fabrication by plasma-chemical deposition. Thus, before direct plasma-chemical deposition of silicon, the wafers with the oxide are kept in vacuum for several minutes at temperatures 450–700°C [12,14], at that the composition, structure and properties of the films change.

Previously obtained study results on the influence of pressure and temperature of plasma-chemical deposition of silicon on mechanical stress [12,14] allowed to complete the study with the formation of silicon-on-insulator structures with stresses less than ±100 MPa. The refraction index of polycrystalline silicon, depending on the deposition conditions, was in the range of 3.71 to 4.86.

4. Using plasma-chemical deposition, polycrystalline silicon structures on insulator with controlled roughness and internal stresses were fabricated. By changing the deposition conditions and subsequent heat treatment in vacuum, the physical, mechanical and optical parameters of the materials were modified in layers, which determine the possibility of adjusting the parameters of the metasurface elements. Roughness and mechanical stress in films (2 μm thick) can be reduced to ones of nm and from −50 to −10 MPa, respectively. The refraction index of silicon oxide and silicon was 1.29–1.48 and 3.71–4.86, respectively. It was established that high-temperature treatment makes it possible to correct the parameter values of deposited silicon oxide films on average by 12–25%.

The obtained results can be taken into account when modeling metasurfaces and developing promising optical elements — metalenses and confocal optical systems based on silicon-on-insulator structures.

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Conflict of interest

The authors declare that they have no conflict of interest.

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