Surface Morphology and Topography of Silicon Dioxide Nanostructures Prepared by DC Reactive Sputtering

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Abstract
In this work, highly-pure silicon oxide nanostructures were prepared by a closed-field unbalanced magnetron plasma sputtering technique. These nanostructures were characterized by field-emission scanning electron microscopy (FE-SEM) and atomic force microscopy (AFM) in order to determine the optimum preparation conditions. These conditions are optimized to control the structural characteristics, mainly surface morphology and topography, of such nanostructures and hence to satisfy certain requirements and purposes in spectroscopic and photonic applications of SiO_2 nanostructures.

Keywords: Silicon dioxide; Nanostructures; Magnetron sputtering; Reactive sputtering
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1. Introduction

It was experimentally revealed that amorphous silicon dioxide (SiO_2) preserves much of the ordering present in the crystalline forms on a short or intermediate length scale [1,2]. For example, the coordination of atoms and the first and second nearest neighbor distances are very similar in the amorphous and crystalline forms, suggesting that both materials have similar building blocks. It is therefore instructive to start our review with the various crystalline allotropes [3-5].

All forms of silica are constructed from the corner-sharing tetrahedra, such as the SiO_4 building block [6]. High-temperature cristobalite and tridymite possess the largest bond angles, and have the most open structures of the crystalline forms of SiO_2 [7]. The smaller the bond angle, the denser is the possible packing, reflected in measured density variations [8].

The temperature- and pressure-stability of the various forms of SiO_2 determine which might be present - as microcrystallites - in thermally grown oxide films or silica glass. The most likely form is tridymite, which is stable at low pressure up to 1470°C [15,16]. High-temperature quartz might not be expected in a typical thermal oxide, as it transforms to tridymite at 870°C [17]. However, due to the large stresses possible during oxidation, the high-temperature quartz form and its associated bonding configuration should not be ruled out entirely [18].

Silicon dioxide films are extensively used as low-index films in multilayer optical devices [19], passivated and/or protective layers of silicon devices [20], scratch resistant coatings for plastic ophthalmic lenses [21] and so on. Stoichiometry and stability of the films are important during applications [22]. The usual methods employed for forming SiO_2 films involve oxidation of silicon at elevated temperatures (T>900°C), however, the high-temperature processing results in junction degradation [23].

In this work, silicon oxide nanostructures are prepared by dc reactive magnetron sputtering. The surface morphology and topography of the prepared nanostructures were determined.

2. Experimental Part

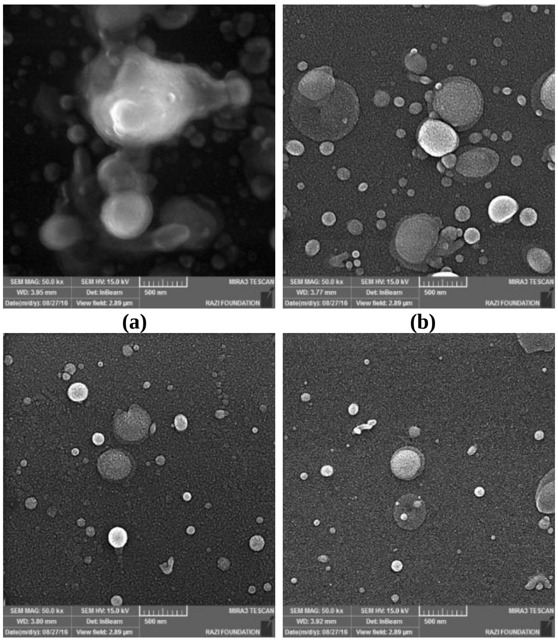
A p-type silicon wafer of 10cm in diameter and 300μm in thickness was used as the sputtering target. It was cleaned by HF acid, ethanol and distilled water, dried and then used for deposition process. This target was maintained carefully on the cathode.

Highly-pure argon and oxygen gases were used as discharge and reactive gases, respectively. The silicon dioxide films were deposited on glass substrates. Before using them in sputtering experiments, these substrates were first cleaned with ethanol to remove any oil layers or residuals may exist on their surfaces, rinsed and washed with distilled water to remove ethanol, and then dried completely before being kept in clean case or placed inside vacuum chamber. More details on magnetron sputtering system can be introduced in references [24-28].

The samples prepared in this work were characterized in order to determine their structural characteristics. The measurements and characterization tests include atomic force microscopy (AFM) and field-emission scanning electron microscopy (FE-SEM).

3. Results and Discussion

Figure (1) shows the SEM images for the SiO_2 film sample prepared using different mixing ratios of Ar:O_2 gases after deposition time of 3 hours. The minimum particle size for each sample was determined as shown in table (1) and found to be 20nm for the sample prepared using mixing ratio of 70:30. The samples show agglomeration as large grains are formed.


(a) (b)

(c) (d)

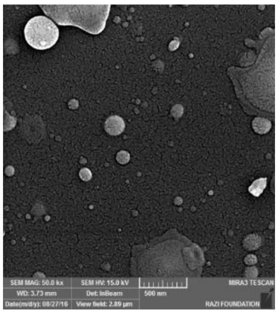
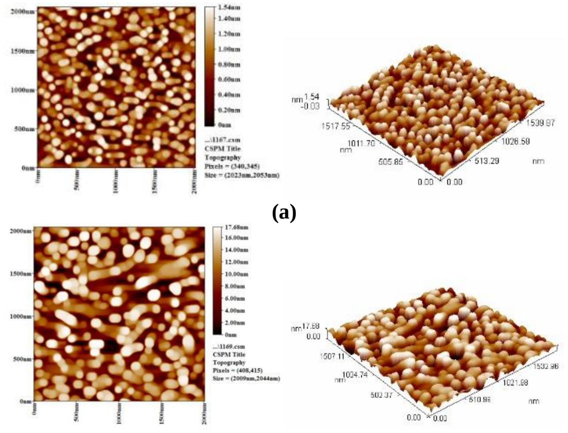

(e)

Fig. (1) SEM images of the nanostructured SiO_2 films prepared in this work using different Ar:O_2 gas mixing ratios (a) 50:50, (b) 60:40, (c) 70:30, (d) 80:20 and (e) 90:10

Table (1) Minimum particle size obtained from SEM results for the samples prepared using different mixing ratios of Ar:O_2 gases

Ar:O_2 ratio	Minimum Particle Size (nm)
50:50	90
60:40	40
70:30	50
80:20	20
90:10	35

Figure (2) shows the 2D and 3D AFM images for the nanostructured SiO_2 thin films prepared using different mixing ratios of Ar:O_2 gases after deposition time of three hours, while table (2) shows the average diameter of nanoparticles prepared at different deposition times using Ar:O_2 mixing ratio of 70:30. As can be seen, the agglomeration on the surface of prepared sample is sufficiently low. The average diameter of particles is 91.83nm. Certain applications of nanostructures, such as photodetectors, energy conversion devices, gas sensing and ultra-hard coatings, require as high as possible surface area. Therefore, the prepared samples can be efficiently used for such applications.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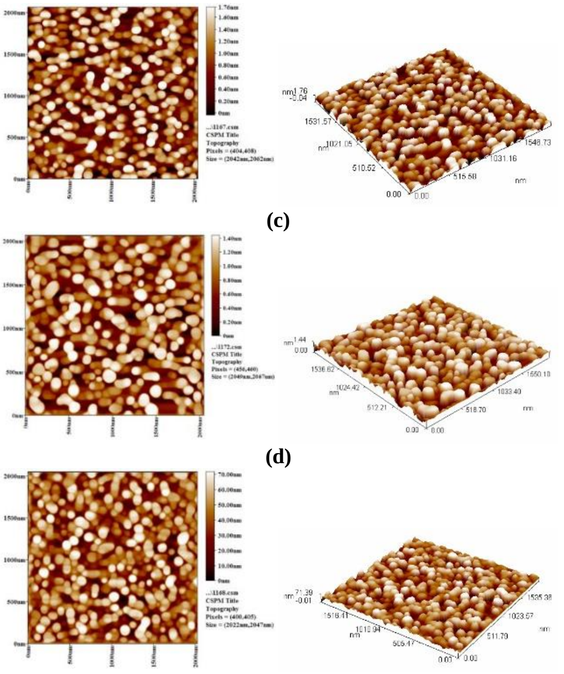

(c)

(d)

(e)

Fig. (2) The 2D and 3D AFM images for the nanostructured SiO_2 films prepared in this work using different $Ar:O_2$ gas mixing ratios (a) 50:50, (b) 60:40, (c) 70:30, (d) 80:20 and (e) 90:10

Table (2) The average diameter of nanostructured SiO_2 thin films prepared at different deposition times

Deposition time (hours)	Average diameter (nm)
2	97.35
2.5	108.79
3	91.83
3.5	91.30
4	78.10

4. Conclusions

As conclusions obtained from these results, the optimum conditions to prepare 20nm nanostructured silicon dioxide films are inter-electrode distance of 4cm, $Ar:O_2$ gas mixing ratio of 70:30, total gas pressure of 0.08torr, discharge voltage of 2.5kV, discharge current of 35mA, anode temperature of 27°C (room temperature) and cathode temperature of about 40°C. The excellent control of these parameters enabled very good control of the structural characteristics of the prepared SiO_2 nanostructures.

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