Surface Topography of Silicon Nitride Nanostructured Thin Films Prepared by Reactive Sputtering

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Abstract

In this work, silicon nitride nanostructures were prepared by a reactive sputtering technique. The optical properties of the prepared nanostructures were studied by their absorption and transmission spectra in the range of 200-800nm. As well, the structural properties of these structures were studied to determine the optimum preparation conditions. The produced nanostructured thin films showed average particle size of 20-30nm, average particle diameter of 99.22 nm, and average roughness of 0.777nm.

Keywords: Silicon nitride; Thin films; Reactive sputtering; Nanostructures

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1. Introduction

Amongst all deposition methods, magnetron sputtering and CVD techniques (CVD, PECVD, LPCVD) can be considered as the most appropriate to deposit silicon nitride. Due to their columnar microstructure, silicon nitride (Si_3N_4) films are effectively used in piezoelectric applications in spite of their amorphous structures [1]. In magnetron sputtering, a silicon target of high purity is sputtered in argon-nitrogen gas mixture environment. Since silicon nitride (Si_3N_4) is a ceramic material, it can be used as a target to prepare ceramic thin films by reactive sputtering in the same gas mixture environment [1]. Substrates, on which the silicon nitride films are deposited, can be biased and heated to 1300°C in order to increase the density of the deposited films. However, films of good density can be deposited on unheated substrates.

Reasonably high number of interconnect levels are resulted by increasing the integration of semiconductor devices due to the extreme reduction in particle sizes. As well, such integration is consequently obtained by improving the dependencies on the properties of the dielectric materials and their performance [2]. Due to vast use of silicon nitride as passivation layer, inter-level isolation layer, oxidation or diffusion barrier, in manufacturing of microelectronic devices, it continuously attracts research and technical interests [3-4].

This work aims to form silicon nitride nanostructures by a reactive sputtering using a dual closed-field unbalanced magnetron technique. In order to produce Si_3N_4 nanostructures with low cost, high purity and homogeneous surfaces, the operation parameters included in such technique are required to be effectively controlled and optimized.

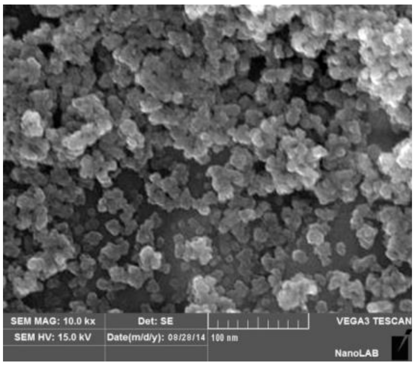
2. Experimental Part

Silicon target was sputtered in presence of nitrogen gas by plasma generated between two electrodes employing the closed-field unbalanced dual magnetron (CFUBDM) system. The design of the CFUBDM system is novel due to the shape of the magnets used for this purpose as well as the arrangements of these magnets at both discharge electrodes. The optimum pressure of nitrogen was $2x10^{-2}$ mbar and the electrodes were separated by 4 cm. The cathode electrode, on which the silicon target was maintained, was cooled down to 4°C to prevent the emission of secondary electrons while the anode's temperature was kept at 20-23°C. More experimental details on the magnetron sputtering system used in this work can be found in references [16-18].

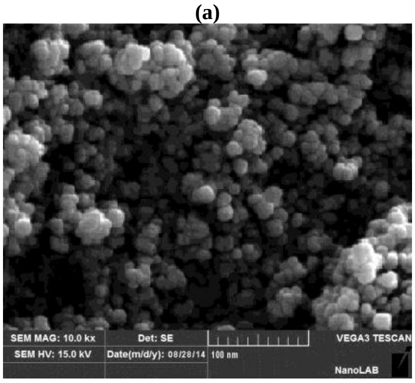
Structural tests such as XRD (Bruker, 1.54.5Å CuKα radiation), SEM (TESCAN Vega EasyProbe), AFM and SPM (Angstrom AA3000 SPM), and FTIR (Shimadzu FTIR-8400S), were performed on the prepared samples to introduce the structural properties of the silicon nitride nanostructures prepared in this work.

3. Results and Discussion

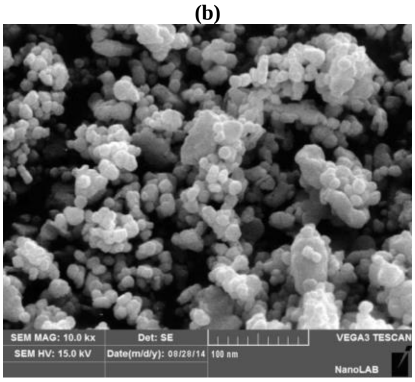
Figure (1) shows the SEM micrographs of the Si_3N_4 nanostructures prepared at different inter-electrode distances (2, 4, and 6cm).



(a)



(b)



(c)

Fig. (1) SEM micrographs of the Si_3N_4 nanostructures prepared at inter-electrode distance of (a) 2cm, (b) 4cm and (c) 6cm

The agglomerations were formed, as shown in Fig. (1a), due to the higher deposition rate and the average particle size was critically determined to be 30nm. These agglomerations were reasonably decreased and the Si_3N_4 nanoparticles prepared at inter-electrode distance of 4cm (Fig. 1b) could be relatively recognized as individuals with average particle size of 27nm. The distribution of particle sizes was inhomogeneous in the Si_3N_4 sample prepared at inter-electrode distance of 6cm, as shown in Fig. (1c), however, particles of 20nm in size could be determined over reasonably larger grains. This inhomogeneity may be attributed to the disturbed deposition at this distance (6cm) as the sputtered Si atom had to traverse longer distance to react with nitrogen atom and then deposit on the substrate after subjecting the effects of gas flow and high density of particle near both electrodes.

As shown in Fig. (2), the AFM images shows that the surface roughness of the prepared Si_3N_4 nanostructure was higher for the samples prepared at inter-electrode distance of 4cm as the average roughness was 0.777nm and the R.M.S roughness was 1.03nm. These values were lower for the samples prepared at smaller and larger distances. However, the distribution of grains over the surface was noticeably much uniform for the sample prepared at distance of 2cm when compared to those prepared at larger distances. This may be attributed to the spatial disturbance effects at larger distances. When working to produce such nanostructures, we look for higher ratio of surface area to volume in order to benefit from this parameter in photonic and tribology applications. Figure (3) shows the granularity cumulation distribution chart of the silicon nitride (Si_3N_4) nanostructures prepared at inter-electrode distance of 4cm. The average diameter is 99.22nm.

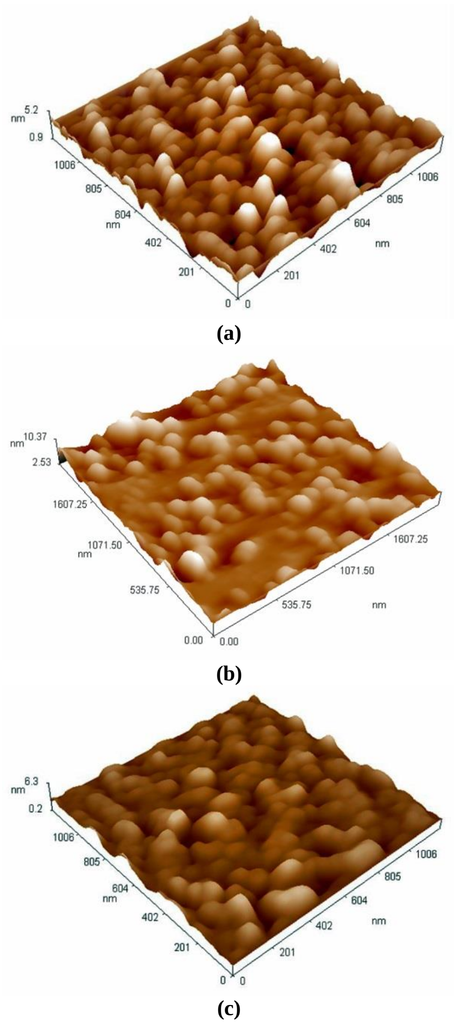


Fig. (8) AFM results of the Si_3N_4 nanostructures prepared at inter-electrode distance of (a) 2cm, (b) 4cm and (c) 6cm

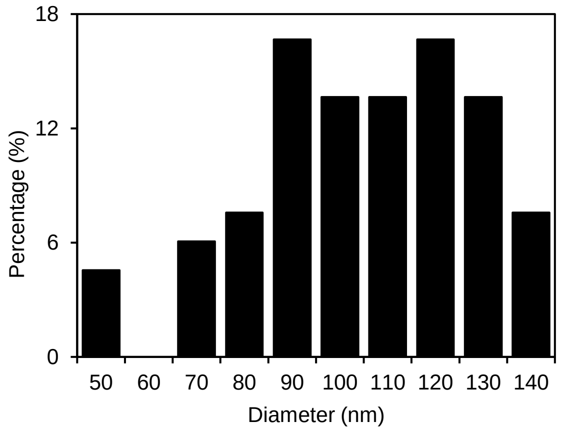


Fig. (9) Granularity accumulation distribution chart of the Si_3N_4 nanostructure prepared at inter-electrode distance of 4cm. The average diameter is 99.22nm

4. Conclusions

Results showed that the nanoparticle size was decreased from 30nm to 20nm by increasing the inter-electrode distance from 2cm to 6cm, respectively. Formation of agglomerations was observed at 2 and 6 cm distances, which makes the inter-electrode distance of 4cm is the optimum to prepare such nanostructures. The roughness of the prepared nanostructures was varied with the inter-electrode distance and the higher surface area was obtained at 4cm distance. The average diameter of the grains formed from Si_3N_4 nanoparticles was 99.22nm.

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