



Structural, compositional and electrical characterization of Si-rich SiO_x layers suitable for application in light sensors



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ABSTRACT

Metal-Oxide-Silicon (MOS) structures containing silicon nanoparticles (SiNPs) in three different gate dielectrics, single SiO_x layer (c-Si/SiNPs-SiO_x), two-region (c-Si/thermal SiO_x/SiNPs-SiO_x) or three-region (c-Si/thermal SiO₂/SiNPs-SiO_x/SiO₂) oxides, were prepared on *n*-type (100) c-Si wafers. The silicon nanoparticles were grown by a high temperature furnace annealing of sub-stoichiometric SiO_x films (*x*=1.15) prepared by thermal vacuum evaporation technique. Annealing in N₂ at 700 or 1000 °C leads to formation of amorphous or crystalline SiNPs in a SiO_x amorphous matrix with *x*=1.8 or 2.0, respectively. The three-region gate dielectric (thermal SiO₂/SiNPs-SiO₂/SiO₂) was prepared by a two-step annealing of c-Si/thermal SiO₂/SiO_x structures at 1000 °C. The first annealing step was carried out in an oxidizing atmosphere while the second one was performed in N₂. Cross-sectional Transmission Electron Microscopy and X-ray Photoelectron Spectroscopy have proven both the nanoparticle growth and the formation of a three region gate dielectric. Annealed MOS structures with semitransparent aluminum top electrodes were characterized electrically by current/capacitance–voltage measurements in dark and under light illumination. A strong variation of the current at negative gate voltages on the light intensity has been observed in the control and annealed at 700 °C c-Si/SiNPs-SiO_x/Al structures. The obtained results indicate that MOS structures with SiO_{1.15} gate dielectric have potential for application in light sensors in the NIR–Visible Light–UV range.

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1. Introduction

Silicon oxide layers with Si nanoclusters have a potential for application in a wide variety of semiconductor devices,

such as non-volatile memories [1,2], Si based light emitters [3,4], solar cells [5] and Si nanoparticle-based sensors [6]. A standard method to obtain Si nanoclusters is high temperature furnace annealing of sub-stoichiometric SiO_x layers. The SiO_x films are usually deposited by Chemical Vapor Deposition (CVD) [1], plasma-enhanced CVD (PECVD) [7] or Physical Vapor Deposition (PVD) [8,9] techniques. The high temperature annealing promotes silicon aggregation and at temperatures of about 700 °C amorphous Si nanoparticles are formed [10], while at *T* > 900 °C Si nanocrystals (Si NCs) are grown [11].

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Recently, Si based photodetector using Si nanocrystal/SiO₂ superlattice has been reported in Ref. 12 with sensitivity in a wide wavelength range from UV to near IR. SiO₂ layers containing Si NCs have also been used in structures suitable for gamma [13,14] and UV radiation dosimeters [15,16]. The optimal use of SiO_x layers in light sensors requires good knowledge of their properties. Here, we focus on the effect of high temperature annealing under different temperatures and atmospheres on the Si nanoparticle formation and spatial distribution in annealed SiO_x layers with $x=1.15$. The changes of the electrical characteristics of MOS structures with such layers as gate dielectrics are also investigated.

2. Experimental details

Three types of Metal-Oxide-Silicon (MOS) structures, with single layer, two-region and three-region gate dielectrics containing Si nanoparticles were prepared on *n*-type (100) *c*-Si wafers with resistivity of 4–6 (Ω cm). Prior to the gate oxide formation the substrates were cleaned in piranha solution (a mixture of H₂SO₄ and H₂O₂) followed by oxide etching with 10% HF and rinsing in deionized water. In the *first group* of samples (*c*-Si/SiNPs-SiO_x) the dielectric was formed by deposition of an ~ 80 nm thick SiO_x ($x=1.15$) layer which was further furnace annealed in a nitrogen (N₂) atmosphere at 700 or 1000 °C for 60 min to grow amorphous or crystalline Si NPs, respectively. The SiO_x layers in all groups of samples were prepared by thermal evaporation of silicon monoxide in vacuum [9,10]. In the *second* and *third group* of structures a SiO₂ layer with thickness of ~ 25 nm was thermally grown at 1000 °C for 5 min in dry oxygen. The oxidation process was followed by a 30 min nitrogen annealing at the same temperature. Then, a SiO_x layer was deposited on the thermal oxide. The samples of the second group (*c*-Si/thermal SiO₂/SiNPs-SiO_x) were further furnace annealed for 60 min in N₂ at 700 or 1000 °C. The third group of samples (*c*-Si/thermal SiO₂/SiNPs-SiO_x/SiO₂) was obtained by annealing at 1000 °C in two steps: first, in an oxidizing atmosphere (90%N₂+10% O₂) for 10 min and then in N₂ for 50 min. Since the total annealing time for all the samples was 60 min one can expect approximately equal Si nanoparticle size in all structures annealed at the same temperature. Our previous results have proven that 60 min annealing of SiO_{1.15} in N₂ at 700 °C causes phase separation and formation of amorphous silicon nanoparticles in a SiO_x matrix with $x=1.8$ – 1.9 [9,16], while annealing at 1000 °C leads to growth of 5–6 nm Si nanocrystals in a SiO₂ matrix [9,17]. Control samples were also produced by annealing at 250 °C for 30 min in an argon atmosphere. Such annealing ensures good stability of the suboxide layer at standard room atmosphere; no nanoparticle formation takes place in this process. After the annealing top semitransparent Al electrodes (referred from now on as control gates) with area of $125 \times 125 \mu\text{m}^2$ were deposited through a mask on the dielectric surface. Al electrode was also deposited on the Si wafer back side.

The effect of the annealing atmosphere was studied by cross-sectional Transmission Electron Microscopy (XTEM) and X-ray Photoelectron Spectroscopy (XPS). TEM images

were obtained by JEOL JEM-2100F electron microscope at 200 kV. XPS spectra were measured by SPECS high resolution spectrometer using Al K α X-ray source (1486.6 eV). The ellipsometric parameters Ψ and Δ and transmission spectra were measured in the 245–1000 nm wavelength range by M2000U J.A. Woollam ellipsometer. Electrical characterization of the fabricated MOS structures was carried out by current–voltage (*I*–*V*) and capacitance–voltage (*C*–*V*) measurements at room temperature in dark environment or under light illumination using Keithley 4200 Semiconductor Characterization System connected to a shielded probe station.

Groups of nine conventional commercial light emitting diodes (LEDs) of the same color were used as IR, red, green, blue, UV and visible light sources. In addition, red, green and blue lasers with wavelengths of 650, 532 and 405 nm were also used. The light was transmitted from the source to the sample using an optical fiber with a lens at the sample side. The size of the light spot was ~ 1 cm, which resulted in uniform illumination over a much larger area than that of the control gate. Micromanipulator positioned against the optical fiber lens was used to make contact with the control gate. Because of the measurement configuration and since the diameter of the needle tip was less than 30 μm , the effect of shadowing and light scattering was minimal. However, care has been taken to measure the light intensity under conditions very close to the ones used in the electrical characterization. The optical power delivered to the sample position was measured by Thorlabs S120C Standard Photodiode Power Sensor connected to PM100USB Optical Power and Energy Meter. The obtained results are shown in Table 1. The optical power of each source was measured at the driving current given in column 3 used in the experiments performed. The measured optical powers of the red, green and blue lasers were 38.2, 16 and 16.5 W/m².

3. Results and discussion

Fig. 1 shows a transmission spectrum of Al film deposited simultaneously on Corning glass substrate with the MOS structures metallization. The transmission spectrum of the glass substrate is also shown. The best fit to the aluminum transmission spectrum (curve 2) and the ellipsometric parameters Ψ and Δ (not shown here) gave a value of 10.7 nm for the Al film thickness. The obtained value is close to the one measured by quartz microbalance during metallization. The maximum transmission of the

Table 1
Driving currents and the corresponding optical powers of the LED sources.

Color	Wavelength (nm)	Current (mA)	Optical power density (W/m ²)
IR	940	36.2	0.24
Red	635	30.1	0.1
Green	530	21.4	0.24
Blue	470	17.9	0.17
UV	400	18.2	0.18
Visible light		20.15	0.2

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