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Characterization of in-depth cavity distribution after thermal annealing of helium-implanted silicon and gallium nitride

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ABSTRACT

Single-crystalline silicon wafers covered with sacrificial oxide layer and epitaxially grown gallium nitride layers were implanted with high-fluence helium ions ($2–6 \times 10^{16}$ cm^{-2}) at energies of 20–30 keV. Thermal annealings at 650–1000 °C, 1 h were performed on the Si samples and rapid thermal annealings at 600–1000 °C, 120 s under N_2 were performed on the GaN samples. The as-implanted samples and the near-surface cavity distributions of the annealed samples were investigated with variable angle spectroscopic ellipsometry. In-depth defect profiles and cavity profiles can be best described with multiple independent effective medium sublayers of varying ratio of single-crystal/void. The number of sublayers was chosen to maximize the fit quality without a high parameter cross-correlation. The dependence of the implantation fluence, oxide layer thickness and annealing temperature on the cavity distribution was separately investigated. The ellipsometric fitted distributions were compared and cross-checked with analyses of transmission electron micrographs where the average surface cavity was determined sublayer by sublayer. The in-depth profiles were also compared with simulations of He and vacancy distributions.

1. Introduction

High-dose helium implantation followed by thermal annealing leads to extended defect formations, such as dislocations and cavities in single-crystalline silicon (c-Si) and gallium nitride. Cavities in these materials can be used for different applications, such as gettering of impurities during device processing, smart cut process or the diffusion control of dopants for ultrashallow junctions [1–5]. A further interest of He implantation induced defects in GaN concerns the formation of resistive guard rings in a Schottky diode process [6]. Transmission electron microscopy (TEM) is an established tool to investigate these structures, but unfortunately it is very time consuming and difficult to determine at a same time a depth distribution profile of the cavities and to observe defects of a few nm to thousands of nm that are encountered in the same area. These structures can be studied by spectroscopic ellipsometry (SE) and, with appropriate multilayered models, the in-depth profiles of the implantation caused amorphization of Si and the cavity formation after thermal annealing can be evaluated [7–10]. SE has the advantage over TEM that it is fast, non destructive and so can be used as a feedback control during industrial process. In this study a large number of Si wafers and GaN layers subjected to different implantation and annealing conditions are investigated. The formation of cavities in Si is investigated as a function of the implantation dose, the annealing temperature and the thickness of a sacrificial oxide layer. Furthermore the well established multilayered model is utilized to evaluate the damaged GaN as well.

2. Experimental details

The sample preparations, the implantations and the annealings, as well as the TEM observations were made at GREMAN institute. The ellipsometric measurements and evaluations were made at MTA TTK MFA. Two sets of samples were prepared and analyzed: silicon wafers with sacrificial oxide and GaN on sapphire substrates.

Single crystalline silicon *p*-type Czochralski (111) substrates, covered with a sacrificial oxide layer (1300, 1500 and 1700 Å), were implanted at 7° tilt with high helium fluences ($2–6 \times 10^{16}$ cm^{-2}) at an energy of 20 keV. The SiO_2 layers were removed by chemical etching in a 10% hydrofluoric acid solution. The samples were thermally annealed with conventional furnace annealing (FA) under N_2 atmosphere at 650, 800 and 1000 °C for 1 h. These samples will be referred to as SiO_2/Si samples.

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GaN on sapphire samples consist of a stack of "n$^-$ GaN (9 μm)/n$^+$ GaN (3 μm)/AlGaN–GaN buffer (3.2 μm)" epitaxially grown with metalorganic chemical vapor deposition on sapphire. These samples were implanted with 30 keV He ions at a fluence of 6×10^{16} cm^{-2}, afterwards they were subjected to [600–1000]°C rapid thermal annealing (RTA) for 2 min while covered with a 200 nm Tetraethyl orthosilicate cap-layer. These samples will be referred to as GaN/sapphire samples. Additionally, a thin layer GaN grown on Si substrate was also prepared (GaN/Si). This GaN/Si sample served to compare its optical response to that of the non-implanted sample of the GaN/sapphire type samples. Table 1 summarizes the implantation and annealing conditions for all the investigated samples.

The SE measurements were performed on the non-implanted, the as-implanted and the annealed samples using a Woollam M-2000DI variable angle spectroscopic ellipsometer. It is a rotating compensator ellipsometer with a multichannel detection system. This setup enables the measurement of the ellipsometric angles with an accuracy of 5×10^{-2} for both Ψ and Δ. The measurements were performed at an angle of incidence between 70° and 78° for the SiO_2/Si type samples and between 60° and 70° for the GaN thin layer type samples, in the wavelength range from 193 to 1690 nm, with a spectral resolution of 1 nm. These incident angles were chosen in order to correspond to the Brewster angle for the Si or the GaN for some wavelength within the spectral range. The recorded ellipsometric spectra were evaluated with CompleteEASE v4.72 and with WVASE v3.386 data acquisition and analysis software. These software use regression analysis to fit the free parameters of the optical models by the minimization of a well defined merit of fit, in our case the χ^2, defined the following way:

$$\chi^2 = \frac{1}{n-P-1}\sum_{j=1}^{n}\left\{\left(\frac{\Delta_j^{meas}-\Delta_j^{calc}}{\sigma_{\Psi j}^{meas}}\right)^2+\left(\frac{\Psi_j^{meas}-\Psi_j^{calc}}{\sigma_{\Delta j}^{meas}}\right)\right\},$$

where Ψ and Δ are the measured ('*meas*') and calculated ('*calc*') ellipsometric angles, n is the number of independently measured values, P is the number of unknown model parameters and σ is the measurement error serving for the weighting of the difference of the measured and calculated values in the numerators.

Crystalline structures of the implanted samples were characterized using TEM and scanning TEM (STEM) to investigate the material amorphization or the distribution of cavities within the different materials (Si/GaN) after annealing. TEM lamella were prepared and observed with an "FEI Strata 400" dual-beam system (scanning electron microscopy and focused ion beam) equipped with a flip stage for lamella transfer on TEM grid and with a STEM detector for observations. A JEOL 2100 F was used in classical observation modes for TEM observations.

Table 1
Implantation and annealing conditions for the investigated sample types.

Sample type	SiO_2/Si	GaN/sapphire	GaN/Si
SiO_2 or n$^-$/n$^+$-GaN/buffer thickness	130 nm 150 nm 170 nm	9/3/3.2 μm	5/2/5 μm
Implantation energy	20 keV	30 keV	
Implantation fluence	Non-implanted 2×10^{16} cm^{-2} 4×10^{16} cm^{-2} 6×10^{16} cm^{-2}	Non-implanted 6×10^{16} cm^{-2}	Non-implanted
Annealing temperature	as-Implanted 650 °C 800 °C 1000 °C	as-Implanted 600 °C 700 °C 800 °C 900 °C 1000 °C	
Annealing time	60 min FA	2 min RTA	
Number of samples	37	7	1

3. Modelling and evaluations

The ellipsometric models describing the two different kinds of annealed samples as well as the as-implanted counterpart followed a similar pattern. A surface native oxide layer, an amorphous or cavity layer and a semi infinite substrate layer were used for the SiO_2/Si samples, while a simpler model, a defected layer and a semi infinite bulk layer was used for the GaN/sapphire samples. To account for the optical response of the intermediate layer (partially amorphous, or defected layer) several independent sublayers with varying fraction of component content was used with the help of the Bruggeman effective medium approximation (EMA): A mixture of c-Si and amorphous Si (a-Si) for the amorphous region of the as-implanted Si samples (see inset in Fig. 1b) and a mixture of c-Si and void for the cavity region of the annealed Si samples, as it was previously demonstrated to be a very good model choice [7,8]. Scattering effects are negligible because the typical size of the cavities is much less than the wavelength of the probing light (see TEM of Fig. 2). The low depolarization values (less than 4% at the whole spectral region) also evidence that there is no scattering effect, confirming the validity of EMA based models. The dielectric function of the c-Si and SiO_2 were taken from reference measurements from the literature. The dielectric function of the a-Si component was described with a single Tauc–Lorentz oscillator model using 4 independent fit parameters. As for the GaN/sapphire samples (see inset in Fig. 8), the defected regions of the implanted and annealed GaN thin layers were considered to be a composition of reference GaN measured before implantation (non-implanted GaN), of reference implanted GaN

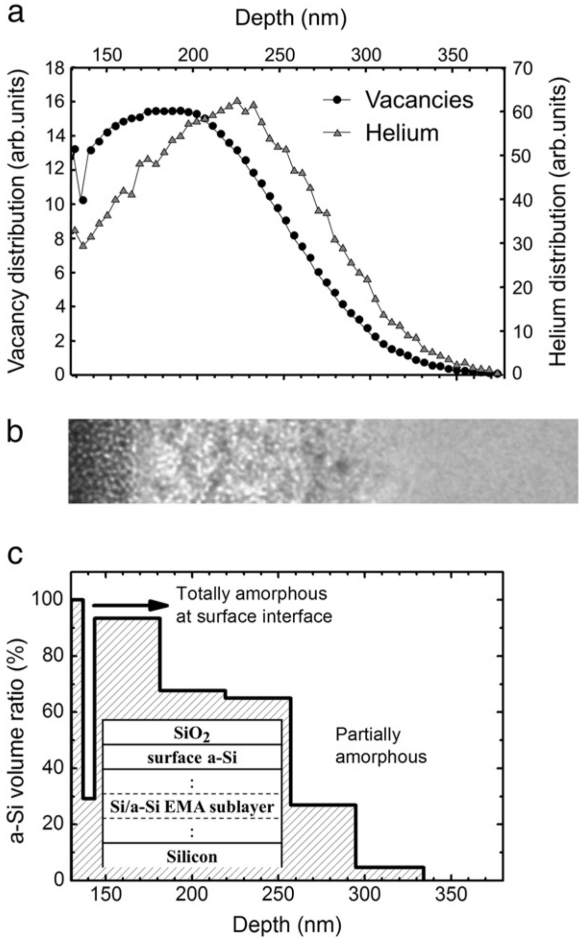

Fig. 1. a) He and vacancy depth distribution from simulations for 20 keV, 4×10^{16} cm^{-2} implantation through 130 nm sacrificial oxide layer, b) cross-sectional TEM image and c) amorphous volume fraction depth distribution from SE evaluation and ellipsometric model inset.

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