



Nitrogen ion induced nitridation of Si(111) surface: Energy and fluence dependence



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HIGHLIGHTS

- A systematic study for the formation of silicon nitride on Si(111).
- Investigation of optimal energy and fluence for energetic N_2^+ ions.
- Silicon nitride formation at room temperature on Si(111).

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ABSTRACT

We present the surface modification of Si(111) into silicon nitride by exposure to energetic N_2^+ ions. *In-situ* UHV experiments have been performed to optimize the energy and fluence of the N_2^+ ions to form silicon nitride at room temperature (RT) and characterized *in-situ* by X-ray photoelectron spectroscopy. We have used N_2^+ ion beams in the energy range of 0.2–5.0 keV of different fluence to induce surface reactions, which lead to the formation of Si_xN_y on the Si(111) surface. The XPS core level spectra of Si(2p) and N(1s) have been deconvoluted into different oxidation states to extract qualitative information, while survey scans have been used for quantifying of the silicon nitride formation, valence band spectra show that as the N_2^+ ion fluence increases, there is an increase in the band gap. The secondary electron emission spectra region of photoemission is used to evaluate the change in the work function during the nitridation process. The results show that surface nitridation initially increases rapidly with ion fluence and then saturates.

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1. Introduction

The large lattice and thermal expansion mismatch between Si and GaN results in highly defected GaN films when grown on Si(111) substrates. To improve the quality of GaN films, different approaches such as, use of buffer layers, surfactants, epitaxial lateral overlayer growth, etc. have been employed [1–4]. The surface modification of Si into silicon nitride is an alternate solution, because of its lattice parameter being double of that of GaN and its anti-surfactant nature process compatibility to MBE growth [5,6]. Several approaches to form high quality silicon nitride films, such as CVD, PECVD, reactive sputtering etc., have been adopted of-late to improve the film quality [7–9]. All these processes involve high temperature growth, which frequently induce dopant diffusion and also incorporation of bonded hydrogen, leading to limitations in microelectronics fabrication. For this reason N_2^+ ion

bombardment on Si surface is investigated as a potential method to grow a thin silicon nitride layers at room temperature without changing the desirable bulk properties of the Si substrate. In literature there are several reports on the nitridation of various silicon surfaces by using N_2^+ ion bombardment; Taylor et al. [10] studied the nitridation of Si(100) by using N_2^+ ions in the kinetic energy range of 30 eV to 3 keV and proposed charge neutralization and collisional dissociation of N_2^+ ions as required steps for nitridation. Back et al. [11] nitrided the Si(100) surface forming silicon nitride films at RT containing patches of ordered micro-crystallites which convert into an ordered layer after annealing. Pan et al. [12,13] have reported a AES analysis of silicon nitride formation on Si(100) by 2–10 keV N_2^+ ions, Kim et al. [14] probe the effect of ion beam and annealing while Park et al. [15] and Palacio et al. [16] have shown an angle resolved XPS study of the nitridation of the Si(100) surface.

Though different mechanisms for the formation of silicon nitride are proposed in the literature, yet there is lack of systematic parametric dependence studies to arrive at a consensus for the Si(111) surface. We have recently reported on several surface

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modification studies on GaAs (001) [17] and Si [18–20] surfaces, to make them compatible for GaN growth. The need of systematic nitridation of Si to form silicon nitride at room temperature which can be used a template for GaN growth, has prompted us to undertake this work. Surface sensitive X-ray Photoelectron Spectroscopy (XPS) is employed by monitoring the Si(2p) and N(1s) core level chemical shifts and deconvolution into their components to understand the different bonding configurations. In addition, the valence band region was examined to elucidate the changes in the electronic structure, while secondary electron emission spectra have been acquired to analyze the work function dependence on N_2^+ ion energy. We have investigated the optimal energy and fluence of energetic N_2^+ ions for the formation of stoichiometric silicon nitride. The energetic ion bombardment creates disorder in the material in the form of point defects upto its projected range. The lattice disorder depends on the incident ion energy, fluence and the sample temperature and especially for low energetic ions this disorder can be annealed out for further applications. Our experiments, we have performed at room temperature thus, we believe to have some damage on the surface depending on ion energy and fluence. However, to have quantitative values for sample damage (which is not the motivation of this work) Rutherford backscattering spectrometry/channeling (RBS/C) measurements has to be done, which can be consider for further investigations.

2. Experimental

The experiments were carried out in an ultra high vacuum X-ray Photoelectron Spectroscopy system (Perkin Elmer model PHI-1257), working at the base pressure of better than 5×10^{-10} torr, and rising up to 2×10^{-7} torr during N_2^+ ion exposure. The chamber is equipped with a high precision sample manipulator, high resolution hemispherical electron energy analyzer and a differentially pumped N_2^+ ion source with precise control of energy (0–5 keV), fluence and raster area on the sample. The samples used were p-type boron doped silicon wafers of size $10 \text{ mm} \times 10 \text{ mm}$ with (111) surface orientation. The samples were externally cleaned by the Shiraki process [21], and then in UHV degassed at $600 \text{ }^\circ\text{C}$ for 12 h followed by repeated flashing to $1200 \text{ }^\circ\text{C}$ by electron beam heating and cooling to RT at a slow rate of $5 \text{ }^\circ\text{C s}^{-1}$. The atomic cleanliness of

the samples by this process was ascertained by XPS, AES and LEED [19,20]. The experiment involves a controlled bombardment of 0 eV to 5 keV N_2^+ ions with the sample current of 1.5×10^{-6} A. The ion fluence is calculated from the exposure time, spot size and raster area. First part different samples of clean Si(111) surface have been exposed to a constant fluence for different N_2^+ ion energy beam and then for each beam energy, different samples of clean Si(111) surface have been exposed to different ion fluence, while the surface chemistry is monitored by *in-situ* XPS.

3. Results and discussions

In the first set of experiments, we have exposed the clean Si(111) surface to different energies of N_2^+ ions ranging from 0.1 keV to 5.0 keV for a constant fluence of 1×10^{14} ions cm^{-2} . The results presented in Fig. 1(ii), plots the ratio of reacted nitrogen atoms to the reacted Si atoms determined from XPS core-level scans estimated by

$$R_{N-Si} = [I_N/0.42]/[(I_T - I_{Si-N})/0.27]$$

where I_N , I_T and I_{Si-N} are the area under the N(1s) peak, total area of Si(2p) peak and area under the Si–N (reacted Si into silicon nitride) related contribution, after Gaussian deconvolution. ($I_T - I_{Si-N}$) represents the amount of un-reacted silicon. The numerical values, 0.42 and 0.27 are the relative atomic sensitivity factors for the N(1s) and Si(2p) core level spectra. Fig. 1 shows that as the N_2^+ ion energy increases, the ratio in the uptake curve increases upto 1 keV and then decreases for higher energy.

To get qualitative values for the formation of silicon nitride we have deconvoluted the Si(2p) core level spectra into their components for the [Fig. 1(i)] and are shown in Fig 1(i), where (a), (b), (c), (d) and (e) correspond to clean Si(111), 0.5 keV, 1.0 keV, 3 keV and 5 keV nitridation, respectively. Curve (a) shows the Si(2p) core level spectra for clean Si with position at 99.9 eV and FWHM of 1.4 eV. We have observed a shift of 0.5 eV in the Si(2p) core level spectra after 0.5 keV N_2^+ ion bombardment with FWHM of 2.4 eV. As this surface is bombarded by 1 keV N_2^+ ions the Si(2p) peak shifts to 101.5 eV with an increase in the FWHM to 3.1 eV. For the higher energy (>1 keV) N_2^+ ion bombardment, we have observed the Si(2p)

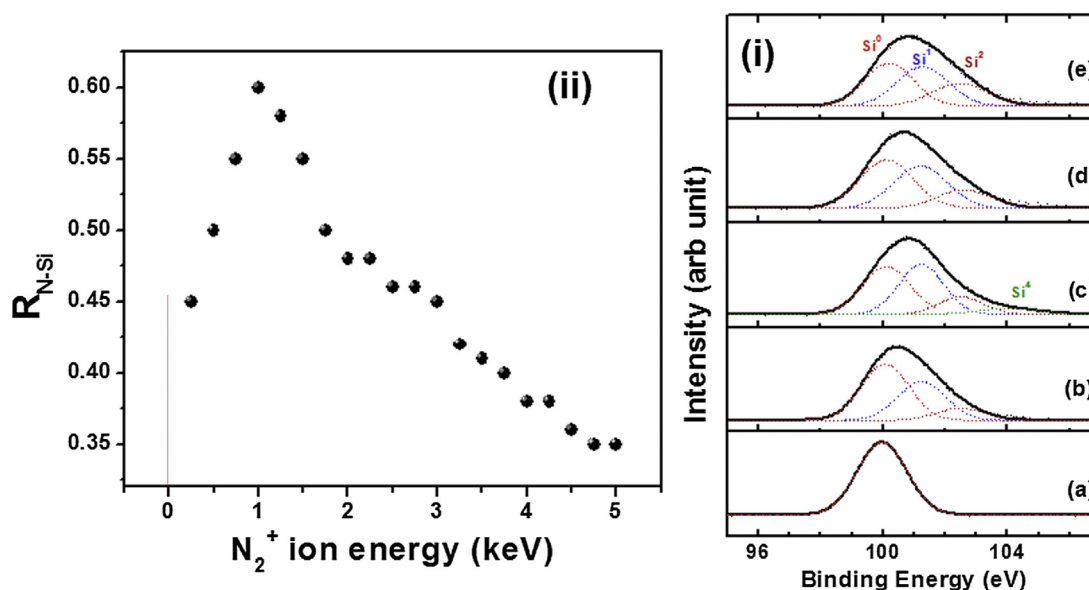


Fig. 1. (i) Deconvoluted Si(2p) core level spectra, where (a), (b), (c), (d) and (e) correspond to the clean Si, nitridation by 0.5 keV, 1 keV, 3 keV and 5 keV, respectively. (ii) plots the ratio (x) with the N_2^+ ion energy, when Si(111) surface is exposed to different energy N_2^+ ions with fixed fluence of 1.0×10^{14} ions cm^{-2} .

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