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Optical properties and surface damage studies of crystalline silicon caused by swift iron ions

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ABSTRACT

p-Type silicon samples irradiated with 70 MeV ^{56}Fe^{5+} ions for various fluences varying between 5×10^{12} and 4×10^{14} ions cm^{-2} have been studied using spectroscopic ellipsometry and Fourier transform infrared spectroscopy. The microstructure of the irradiated samples was modeled from ellipsometric data, using a multilayer optical model and Bruggeman effective medium approximation. The values of pseudodielectric function, absorption coefficient and Penn gap energy were determined with respect to ion fluence. The effective medium analysis suggests that the superficial silicon layer can be explained as a mixture of crystalline and damaged silicon. The thickness of the damaged layer and percentage of voids present in the layer were found to increase with increase in the ion fluence. The effect of disorder on the interband optical spectra, especially on the critical point E_1 at 3.4 eV was found to vary with ion fluence. A red shift in the critical point E_1 with increasing ion fluence was observed. FTIR study showed of silicon samples irradiated with 70 MeV ^{56}Fe^{5+} ions produced the oscillations in the spectral region 1000–400 cm^{-1}. As irradiated sample showed more pronounced fringes, while contrast of the fringes and amplitude both were found to decrease with increase in depth.

1. Introduction

The irradiation of materials with swift heavy ions produces electronic excitation of the atoms in the material with negligible contribution from nuclear energy loss and the effective electronic energy loss that brings out interesting change in the materials properties [1–4]. In the past, several study on silicon ion irradiation with swift heavy ions with different species i.e. Au, Ag, Si, Xe, Kr etc. have been reported by number of researchers [5–8]. To the best of our knowledge, the irradiation of silicon with swift transition metal ions such as Fe, Co, Ni is much less investigated [9]. Generally, swift heavy ion irradiation of silicon with any kind of species in the electronic energy loss regime produces the point defects and defect clusters. In addition to the defects mentioned above, it has been also observed that the swift heavy ion create latent tracks in silicon due to high electronic energy loss [10–13], however the reports on experimental evidence of the formation of latent tracks in silicon is very limited [13]. Damage induced by 90 MeV silicon ions with various fluences varying between 1.25×10^{14} and 5×10^{14} ions cm^{-2} in silicon have been studied using grazing angle X-ray diffraction, UV–Vis spectroscopy and life time of minority carriers measurements [5]. AFM studies of p-silicon showed the formation of craters surrounded with hillocks after irradiation with 100 MeV gold ions with various fluences ranging between 1×10^{10} and 1×10^{12} ions cm^{-2} [6]. Atomic force microscopy studies of crystalline silicon irradiated with 200 MeV Ag ions showed increase in the surface roughness with respect to ion fluence [7]. The effects 5.2 GeV Kr ions in semiconductors with different fluences varying between 1×10^{8} and 1×10^{14} ions cm^{-2} have been investigated [8]. Optical studies of crystalline silicon irradiated with nickel ions with different fluences varying between 1×10^{13} and 1×10^{15} ions cm^{-2} with energy varying between 60 and 75 MeV have shown the change in refractive index, extinction coefficient and optical absorption in the fundamental absorption and inter band transition regions [9]. In our earlier work, we have reported the electron spin resonance, high resolution X-ray diffraction and atomic force microscopic studies of crystalline silicon samples irradiated with swift iron ions with different fluences varying between 1×10^{12} and 5×10^{12} ions cm^{-2} [14–17]. In this paper, spectroscopic ellipsometry and Fourier transforms infrared measurements have been used to investigate the optical properties of crystalline silicon samples after irradiation with swift (70 MeV) iron ions with various fluences varying between 5×10^{12} and 4×10^{14} ions cm^{-2}. The result of the present study will provide the basic understanding, how the void fraction and damaged layer

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induced by swift iron ions in silicon affects the complex dielectric function and their critical points.

2. Experimental details

In the present work, we have used p-type, $\langle 1\,1\,1\rangle$ oriented, single crystalline silicon wafers with sheet resistivity of 0.05 Ω-cm, that was cut into pieces typically 1 cm × 1 cm. samples. These samples were irradiated at room temperature with 70 MeV $^{56}Fe^{5+}$ ions with different fluences varying between 5×10^{12} and 4×10^{14} ions cm^{-2} using the 15 UD Pelletron Facility at the Inter University Accelerator Centre, New Delhi. According to SRIM calculation, such experimental conditions lead to electronic energy loss 6.60 keV/nm, nuclear energy loss 0.0155 keV/nm and projected range of 14.87 μm [18]. The samples were mounted on a copper target ladder with silver paste and beam was magnetically scanned over 8 mm × 8 mm area on sample surface for uniform irradiation. The vacuum inside the irradiation chamber was maintained at about 10^{-6} mbar. Spectroscopic ellipsometry measurements were performed at room temperature using ellipsometer with rotating polarizer [Model M-2000™, J.A. Woollam Co.]. The data was acquired at fixed incident angle of 75° in the energy range between 1.2 and 5.0 eV. The wavelength range used in the measurement was between 240 and 1000 nm. This wavelength range covers the ultraviolet to near infrared regions. In order to study the depth profile, one of the sample irradiated for the fluence of 2×10^{14} ions cm^{-2} was etched in HF:HNO$_3$:H$_2$O solution. Analytical grade hydrofluoric acid, nitric acid and de ionized water were used for all etch in the volume ratio of 8 ml:1 ml:1 ml. De-ionized water was used as diluents in place of acetic acid. The chemical reaction with silicon during the etching processes is given in Eq. (1) [19].

$$3\mathrm{Si} + 4\mathrm{HNO_3} + 18\mathrm{HF} \rightleftharpoons 3\mathrm{H_2SiF_6} + 4\mathrm{NO} + 8\mathrm{H_2O} \tag{1}$$

The etching rate (r) was estimated from the etched silicon layer Δd and the immersion time (t_{etch}) using the following relation;

$$r = \frac{\Delta d}{t_{\mathrm{etch}}} = \frac{\Delta m}{t_{\mathrm{etch}} A \rho\ (\mathrm{Si})} \tag{2}$$

where Δm is mass loss obtained by differential weight, A is area = 1 cm^2 and ρ (Si) is the density of silicon = 2.33 g cm^{-3}. The etching rate in our experiment was found to be about 0.33 μm per sec. After every etch, mid-infrared Fourier transform reflectance spectra were recorded using Fourier transform infrared spectrometer (*JASCO*-610) in the spectral region 1400–400 cm^{-1}.

3. Results and discussion

3.1. Spectroscopic ellipsometric studies

Ellipsometry is a optical characterization technique based on the measurement of the polarization transformation that occurs after reflection or transmission of polarized beam by a given sample. Ellipsometry parameters tan Ψ and cos Δ for nonirradiated and samples irradiated with 70 MeV $^{56}Fe^{5+}$ ions with various ion fluences were recorded. The values of Ψ and Δ are related to the ratio of the Fresnel reflection coefficient R_p and R_s for p direction lying in the plane of incident) and s (direction lying perpendicular to the p-direction) directions of polarized light, respectively. The ratio of the complex reflection coefficients R_p and R_s can be written as:

$$\rho = \frac{R_p}{R_s} = \tan(\psi)e^{i\Delta} \tag{3}$$

where tan(Ψ) is the attenuation of the relative amplitudes of p and s components after being reflected on each surface, while Δ is the phase difference change through this process. The imaginary and real parts of the pseudo dielectric functions were evaluated from the ellipsometric parameters. Evaluation of the data was carried by using multilayer optical model and Bruggeman effective medium approximation [20–22]. In order to fit the parameters, three layered model consisting of the substrate; the damaged layer, and silicon oxide layer was used. The best fit was obtained when the damaged layer was considered as a mixture of silicon and void. Linear regression method was used to determine the thickness of the damaged layer and the volume fraction of voids and the thickness of silicon oxide layer [23]. The void fraction and the thickness of the damaged layer for various ion fluences are given in Table 1. It is evident from the Table 1 that the percentage of voids and thickness of the damaged layer increase with increase in the ion fluence. The pseudo dielectric functions were calculated from the ellipsometric parameters using the relation;

$$< \varepsilon(E) >= \varepsilon\left[\sin^2\phi + \left[\frac{1-\rho}{1+\rho}\right]^2 \sin^2\phi\tan^2\phi\right] \tag{4}$$

where ϕ is the angle of incidence and ρ is the complex reflectance ratio defined as in Eq. (3). The imaginary part of the pseudo dielectric function $\varepsilon_2(E)$ of silicon, after correction for silicon oxide layer, for non-irradiated sample and samples irradiated with various fluences in the energy range between 3.0 and 5.0 eV are shown in Fig. 1. There is no change in the position and value of E_1 peak as compared to non irradiated silicon up to a fluence of 2×10^{14} ions cm^{-2}. However, when the samples irradiated with the fluences of $\geqslant 2 \times 10^{14}$ ions cm^{-2} a red shift is observed in the E_1 peak and the value of the dielectric function decreases with increase in the ion fluence, which may be due to increase in strain in the irradiated layers with ion fluence. This result also suggests the weakening of covalent bonds in silicon at higher fluences.

The imaginary part of the pseudo dielectric function of one of the silicon sample irradiated with Fe^{5+} ions for the fluence of 1×10^{13} ions cm^{-2} solid circles (as measured), open circles (fitted) and solid squares (corrected for silicon oxide layer), after the correction of the presence of silicon oxide layer is shown in Fig. 2. The imaginary part of the pseudo dielectric function of silicon has two critical points E_1 and E_2. The E_2 peak must be greater than E_1 peak. However, in our experiment, we have observed the E_1 peak greater than E_2 peak in the measured ellipsometry spectra. This result indicated the presence of a thin silicon oxide layer on the surface of silicon. The thickness of silicon oxide layer estimated using the Linear regression method were found to be 5.66, 3.50, 2.82, 2.00 and 1.03 nm for the ion fluence 5×10^{12}, 1×10^{13}, 2×10^{14}, 3×10^{14} and 4×10^{14} ions cm^{-2} respectively . The decrease in thickness of the silicon oxide layer present on the surface of silicon indicated the desorption's of oxygen atoms from silicon surface. The oxygen atoms adsorbed in silicon species into Si–Si bonds to form Si–O–Si bridging configurations [24]. Linear energy transfer of 70 MeV $^{56}Fe^{7+}$ ions in silicon at the surface estimated from the SRIM code was found to be ~6.60 keV/nm [18], which caused the instability in the lattice structure of Si–O–Si species and its temperature increased. As the ion fluence increases, oxygen atoms adsorb in silicon surface into Si–Si bonds to form Si–O–Si species desorbed through the surface and lowering the oxygen content in the sample.

Penn gap energy was calculated using the following relation [25]

$$E_g = M_1/M_r \tag{5}$$

where M_r, the optical moment of $\varepsilon_2(E)$ is given by,

$$M_r = \frac{2}{\pi}\int_{E_{g'}}^{\infty} E^r \varepsilon_2(E)\,dE \tag{6}$$