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Energy-separated sequential irradiation for ripple pattern tailoring on silicon surfaces

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ABSTRACT

Nanoscale ripples on semiconductor surfaces have potential application in biosensing and opto-electronics, but suffer from uncontrolled surface-amorphization when prepared by conventional ion-irradiation methods. A two-step, energy-separated sequential-irradiation enables simultaneous control of surface-amorphization and ripple-dimensions on Si(1 0 0). The evolution of ripples using 100 keV Ar^+ bombardment and further tuning of the patterns using a sequential-irradiation by 60 keV Ar^+ at different fluences are demonstrated. The advantage of this approach as opposed to increased fluence at the same energy is clarified by atomic force microscopy and Rutherford backscattering spectroscopy investigations. The explanation of our findings is presented through *DAMAGE* simulation.

1. Introduction

Nanoscale surface engineering of solids has attracted significant interest recently because of its potential applications in advanced photonics, micro electro-mechanical systems, photovoltaics, and surface-wetting tailoring [1–6]. Surface patterns are also highly desirable for lab-on-a-chip bio-applications for providing specificity towards cell proliferation and differentiation. Well textured micro-grooves on biomedical materials, have shown improvement in the integration of surrounding tissue [7]. Among the various surface patterning techniques, ion-beam irradiation of the solid surface is considered a very effective approach to the generation of self-organized nanostructures and ripple patterns [8–14]. Apart that, ion beams of higher energies (for example swift heavy ion beams), can be used as/in phase transformations [15] and surface restructuring [16,17].

The passage of an energetic ion through the crystalline lattice transfers significant amount of energy to a target atoms to displace them from the lattice sites during binary collisions. During this interaction, the ion suffers Coulomb scattering with an atom in a solid, which generates a sequence of displacement events until the recoil atom retains sufficient energy to displace other atoms [18]. At sufficiently high doses, point defects, defect complexes, or locally amorphous regions can accumulate as successive cascades formed in the target material until highly damaged crystalline target becomes unstable and transforms into amorphous. Further, owing to the low energy ions under certain conditions, sputtering can roughen the surface, resulting in a pronounced topography, which generates in some cases well-ordered patterns such as ripples or nanodots. The first analytical approach towards understanding the dynamics of surface patterns was presented by Bradley and Harper [19], who emphasized that the two competing processes of sputtering induced roughening and diffusion produce self organized nanoscale patterns on the exposed surface. Later on, multiple of mechanisms of ion beam induced amorphization of Si surfaces were reported [20–22]. Generally amorphization occurs locally in dense regions of individual collision cascades. So with increasing ion dose, completely amorphous zones produced by individual ions accumulate and amorphization of the entire near surface layer takes place with further irradiation. Subsequently, a near surface amorphous layer and hence a buried crystal–amorphous interface can be produced in a crystalline sample with each impinging ion and an increasing number of collision cascades [23,24]. The thickness of the amorphous layer corresponds to the energy and mass of the incoming ion and the mean

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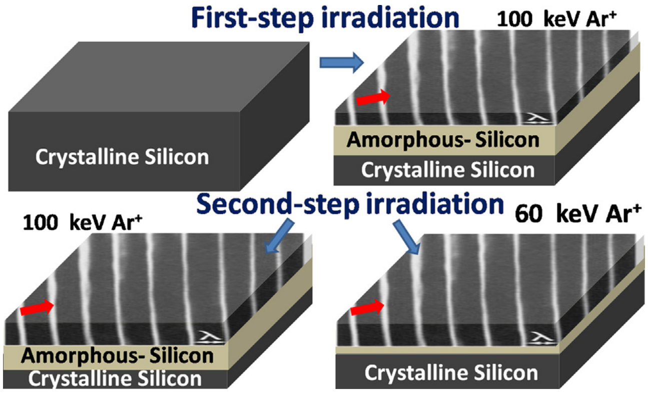

Fig. 1. Schematic diagram of the experimental plan. In the first step irradiation, ripple patterns are grown on a Si(1 0 0) surface using a 100 keV Ar^+ ion beam at a fluence of 1×10^{18} ions/cm^2. In a second step, two sets of samples which differ significantly in amorphous layer thickness are prepared using 100 keV and 60 keV Ar^+ ions. Red arrows indicate the projection on the surface of the ion beam direction. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

free path of collisions [25,26]. These amorphization–crystallization phenomena can be used in nano-sculpturing of solid surfaces such as ripple patterning, nano-dots, nano-pits formation. Variation of ion beam parameters such as energy, incident angle, flux and fluence are used to tune the dimensions of the ripples. However, a simultaneous control over the thickness of amorphous layer is lacking behind to obtain direct access to the underlined crystalline nano-ripples for potential applications.

Recently, on the basis of systematic theoretical [25] and experimental work [11,13] on low energy ion irradiation of Si surfaces, we have emphasized the importance of stress induced solid flow and the amorphous/crystalline (a/c) interface in the amorphous layer, which can be a sufficient cause for the evolution of surface ripple patterns. Taking the idea of the major role of the a/c interface in surface patterning, we hypothesized that if a lower energy beam having lower penetration range is used to bombard an already-grown rippled surface, one might expect growth of ripples with decreasing amorphous layer thickness due to sputtering from the surface. To validate this hypothesis, we have carried out focused experiment using energy-separated ion beam irradiation of Si surfaces. In this work, a sequential two step-irradiation approach is described for tuning the dimensions of ripples with simultaneous control over the thickness of the amorphous layer on the silicon sample, which can be generalized over different samples/matrices.

2. Experimental procedure

A simplified schematic of the experimental approach for simultaneously tuning the ripple dimensions and controlling the thickness of the amorphous surface layer is shown in Fig. 1. In the first-step, ripple patterns are prepared on the Si(1 0 0) surface using 100 keV Ar^+ ions at a fluence of 1×10^{18} ions/cm^2, and in the second-step 60 keV Ar^+ ions with fluence from 5×10^{16} ions/cm^2 to 7×10^{17} ions/cm^2 are used. Irradiation of samples was carried out at a constant current of 15 μA using the low energy ion beam facility at the Inter-University Accelerator Centre (New Delhi). During the irradiation, the pressure in the vacuum chamber was maintained at approximately 10^{-7} mbar. The Ar^+ ion beam was incident at an angle of 60° with respect to the surface normal (with the same direction of irradiation during the second-step irradiation). In order to further clarify the effect of the energy-separated sequential irradiation processes, samples were also prepared using, as the second step, 100 keV Ar^+ ion irradiation under the same conditions. The surface morphology of the processed samples was characterized using atomic force microscopy (AFM) in tapping mode with SiN tips having a nominal tip radius of <10 nm under ambient conditions. To investigate the variation in thickness of the amorphous layer Rutherford backscattering spectroscopy in channelling mode (RBS-C) was performed using a 2 MeV He^+ ion beam from a 1.7 MV 5SDH-2 Pelletron (Tandem) accelerator. The thickness of the near-surface amorphous layer was calculated from the RBS-C data using the *DAMAGE* code [27]. Also, the concentration of Ar, which is incorporated in near surface amorphous layer, is calculated using simulation of Rutherford backscattering analysis (*SIMNRA*) code.

3. Results and discussion

Fig. 2 shows AFM images (a) of pristine Si, and (b) after first-step irradiation with 100 keV Ar^+ at a fluence of 1×10^{18} ions/cm^2. It is evident that after the first-step irradiation clear ripple patterns are formed on the Si surface. The measured value of the root mean square (RMS) roughness for the pristine sample was 0.14 nm, which is an essential factor for the origination and growth of ripple patterns. After irradiation, ripple patterns are observed which are aligned perpendicular to the ion beam direction (the arrow in the figure indicates the direction of the ion beam), in agreement with the proposed models of surface patterning [19,25]. In Fig. 2(b), a 2D-fast Fourier transform (FFT) spectrum of the corresponding AFM image is also shown in the inset (lower left), which clarifies the alignment of the ripple patterns. For the corresponding sample, the average values of amplitude and wavelength of the ripples (measured from nano-scope software) are found to be 28 nm and 1.05 μm, respectively.

To investigate how the pre-pattern surface derives its surface morphology with energy-separated ion beams, two sets of samples were prepared using Ar^+ ion energies of 100 keV and 60 keV on the pre-rippled surface. Note that a shadow effect, in which higher amplitude ripples block the ions to reach to the valleys sections of ripples, can perturb the whole dynamics of ripple growth [28,29]. To avoid the contribution of this shadowing effect in the second-step irradiation, we have calculated the upper limit of incident angle in terms of the amplitude (R) to wavelength (λ) ratio through the following relationship [28];

$$\frac{2\pi R}{\lambda} \leq \tan\left(\frac{\pi}{2} - \theta\right) \tag{1}$$

Since, second-step irradiation was also carried out at an angle of 60°, the ratio R/λ should remain below 0.091 in the observed ripples after first-step irradiation to avoid the shadowing effect. The R/λ value after first-step irradiation is estimated to be 0.038, which neglects the role of shadowing in the growth evolution of ripples in second-step irradiation. Fig. 2(c) and (d) shows the variation in surface ripples after second-step sequential irradiation by 100 keV Ar^+ ions and 60 keV Ar^+ ions. From these AFM images of surface morphology it can be seen that the alignment of ripples remains perpendicular to the ion beam direction, similar to as observed in first-step irradiation.

Further, the growth of ripple dimensions (amplitude and wavelength) is observed with increasing ion fluence. The average values of wavelength and amplitude of ripples for both sets of samples are calculated and plotted in Fig. 3(a) and (b), respectively. Fig. 3(a) shows that for 100 keV second-step irradiation, the values of wavelength and amplitude vary from 1.08 μm to 1.11 μm, and 28 nm to 73 nm, respectively. For the used fluence variation, the percentage increment in wavelength and amplitude of ripples are found to be 2.8% and 160%, respectively. A similar kind of variation in ripple dimensions is observed with increasing fluence after second-stage irradiation at 60 keV as shown in Fig. 3(b). The variation in wavelength and amplitude are found to be from 1.05 μm to 1.12 μm,