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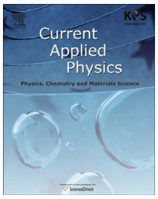

Tuning of ripple patterns and wetting dynamics of Si (100) surface using ion beam irradiation

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ABSTRACT

Ripple patterns on Si (100) surface have been fabricated using 200 keV Ar^+ oblique ion beam irradiation. Dynamical evolution of patterns is studied for the fluences ranging from 3×10^{17} ions/cm^2 to 3×10^{18} ions/cm^2. AFM study reveals that the exponential growth of roughness with stable wavelength of ripples up to higher fluence values is lying in the linear regime of Continuum models. Stylus Profilometer measurement was carried out to emphasize the role of sputtering induced surface etching in ripple formation. Rutherford Backscattering Spectroscopy shows the incorporation of Ar in the near surface region. Observed growth of ripples is discussed in the framework of existing models of surface patterning. Role of ion beam sputtering induced surface etching is emphasized in formation of ripples. In addition, the wetting study is performed to demonstrate the possibility of engineering the hydrophilicity of ripple patterned Si (100) surface.

1. Introduction

In last couple of decades, the formation of self-organized nanostructures under the effect of ion-beam sputtering has turned into one of the highly active research areas of surface science. The self organized surface nanostructures have many technological applications for the development of future optoelectronic, electronic, and magnetic devices [1,2]. Although, there are several methods to grow surface nanostructures like photolithography [3], sub-lithography [4], scanning probe tip [5], ion beam sputtering [6,7] etc. Among these, low energy ion beam sputtering of solid surfaces is a very elegant and one-step bottom-up approach. By varying the sputtering parameters in a controlled manner, one can evolve surface topography into well ordered nanostructures like one-dimensional ripples, regular arrays of dots and pits etc [6,8–10]. Taking the hint that roughness and wetting properties of surfaces are interrelated, these ripples can also be used to tune the wetting dynamics of the solid surfaces. Functionalized surfaces with controlled wetting properties have already attracted huge attention of the research community due to their broad range of potential applications, including micro-fluidic devices [11], microelectronics [12] and self cleaning surfaces [13]. It is well known that the spreading of liquid droplet on solid surfaces is strongly influenced by chemical heterogeneities and the roughness [14].

The behavior of ripples formation on solid surfaces subjected to energetic ion bombardment has been examined experimentally as well as theoretically by several researchers. The most known effect during ion bombardment is the direct transfer of energy and momentum to the target atoms under collision cascade processes within a finite penetration depth, which results in the sputtering and diffusion of atoms. In this direction, Bradley et al. [15] proposed first theoretical approach to study the surface morphological changes on the basis of two competing processes; spatial surface roughness developed due to curvature dependent sputtering and the smoothening process by the surface diffusion. However, Madi et al. [16] and Norris et al. [17] showed that the curvature dependent sputtering is dominated by mass redistribution under ion bombardment. In the previous work we have shown that the mass re-arrangement at amorphous/crystalline (a/c) interface due to stress induced solid flow plays a crucial role in the evolution of ripples under ion bombardment [18–20].

In this work, the dynamics of ripple patterns on n-Si (100) using the irradiation of 200 keV Ar^+ ion beam has been investigated. For explicit investigation of the role of sputtering induced surface etching, we have used Stylus profilometer for the first time. Further, the wetting dynamics or hydrophilic property of rippled patterned substrates is also presented.

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2. Experimental

In the present work, n-Si (100) samples were irradiated using 200 KeV Ar^+ beam in low energy ion beam facility (LEIBF) at IUAC, New Delhi. Half part of the samples was masked with an aperture inhibiting ion penetration completely to enable the comparison of irradiated and un-irradiated portion. Ion beam irradiation was carried out at an angle of 60° with respect to surface normal in a high vacuum chamber of base pressure of 8×10^{-7} mbar. Fluence was varied from 3×10^{17} ions/cm^2 to 3×10^{18} ions/cm^2. During the experiment, the source continuously delivered the stable ion current density of 18 μA/cm^2, which had been electromagnetically scanned uniformly over the samples' surfaces. To quantify the surface etching depth, *ex-situ* measurements were performed using Stylus Profilometer. After irradiation, the surface morphologies of samples were investigated in tapping mode using Nano Scope IIIa atomic force microscope (AFM) having SiN tips with a nominal tip radius of <10 nm under ambient conditions. Rutherford backscattering (RBS) measurement was carried out using 2 MeV He^+ ion beam from 1.7 MV 5SDH-2 Pelletron (Tandem) accelerator, to investigate the thickness of amorphous layer of irradiated samples. Field Emission Scanning Electron Microscopy (FE-SEM) was performed in resolution mode using MIRA II LMH (TESCAN) for 15 keV and 25 keV electron beam. The wetting properties of water droplet on samples were performed using KRŰSS DSA-10 contact angle measuring system.

3. Results and discussion

Fig. 1 shows the AFM image of pristine Si (100) sample. The observed rms roughness value of the sample is 0.17 nm. After the irradiation by 200 keV Ar^+ ion beam, AFM images (20 μm × 20 μm) of samples are shown in Fig. 2, for fluences of (a) 3×10^{17} (b) 5×10^{17} (c) 7×10^{17} (d) 9×10^{17} (e)1.5×10^{18} and (f) 3×10^{18} ions/cm^2, respectively. Arrow in the figure corresponds to the projection of ion beam on the sample's surface which indicates that the orientation of ripples is perpendicular to the ion beam direction. From these AFM images the calculated values of RMS roughness and wavelength of ripples are shown in Fig. 3, which shows the exponential growth of RMS roughness (σ) with ion beam fluence (φ) as $\sigma \propto \exp(\phi/\phi_o)$, where $\phi_o = 7.59 \times 10^{17}$ is a fitting parameter, while the magnitude of wavelength remains stable around ~1.6 μm. This exponential growth of roughness with more or less variation in wavelength indicates that the growth of ripples is in linear regime of continuum models [15].

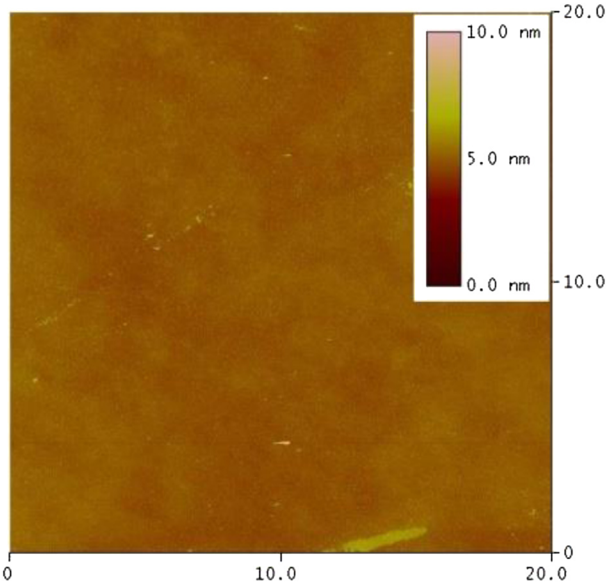

Fig. 1. AFM image (20 μm × 20 μm) for pristine Si (100) sample.

Fig. 4 shows the FE-SEM images for the ripple patterned Si (100) after 200 keV Ar^+ ion beam irradiation at the fluence of 3×10^{18} ions/cm^2. Fig. 4(a) shows oblique view of formation of ripple patterns on the surface. The observed cross-sectional view in Fig. 4(b) shows that the average wavelength and amplitude of ripples are 1.58 μm and 360 nm, respectively. Observed wavelength and amplitude of ripples are in good agreement with the AFM results.

Fig. 5 shows the experimentally observed RBS spectra for pristine Si (100) and irradiated samples for the fluence values of 3×10^{17} ions/cm^2, 7×10^{17} ions/cm^2 and 3×10^{18} ions/cm^2, respectively. RBS spectra of irradiated samples demonstrate the presence of Ar in the near surface region of Si. Using the SIMNRA [21] software the average thickness (h_o) of Ar incorporated amorphous layer for the irradiated samples was estimated to be ~190 nm. It has been observed that the average depth of Ar distribution in the near surface region of Si (100) is nearly same for the irradiation fluence values of 3×10^{17} ions/cm^2, 7×10^{17} ions/cm^2 and 3×10^{18} ions/cm^2.

Fig. 6 shows the surface etching depth of irradiated samples for different fluences using Stylus Profilometer. Left side section of the dotted line corresponds to the un-irradiated part of samples whereas the right side section for the rippled surfaces. From these measurements the values of etching depth are calculated as 440 nm, 1150 nm and 2350 nm for the fluences of 5×10^{17} ions/cm^2, 1.5×10^{18} ions/cm^2 and 3×10^{18} ions/cm^2, respectively. Also the surface etching depth were also calculated using the following formulism:

$$\text{Etching depth} = (\text{sputtering yield} \times \text{area of irradiation} \\ \times \text{Fluence})/\text{Atomic density of silicon}$$

Using SRIM-2008 we found that the sputtering yield of Si for 200 keV Ar is 4.98 atoms/ion. Area of irradiation is chosen 1 cm^2 for each irradiation fluences. The atomic density of silicon used as 4.97×10^{22} atoms/cm^3. The calculated and experimental observed sputtering induced etching depths of rippled surface for different fluence values are shown in Fig. 7.

Fig. 8 shows the contact angle measurement for the pristine as well as irradiated samples for different fluence values. Fig. 8(a) demonstrates that the contact angle (δ) is 82° for the pristine Si (100) sample. The observed contact angle demonstrate the hydrophilic property of pristine sample. After irradiation contact angle has been decreased from 76° to 50° with increasing the fluence from 3×10^{17} ions/cm^2 to 3×10^{18} ions/cm^2.

Variation of contact angle as a function of fluence and roughness is shown in Fig. 9. The contact angle of water droplet on pristine Si surface is 82°, therefore the pristine surface is hydrophilic in nature. Ion beam irradiation leads to the formation of ripples which changes the roughness of the surface. With increase in fluence from 5×10^{17} ions/cm^2 to 3×10^{18} ions/cm^2, the roughness of surface is drastically increased from 2.55 nm to 85.63 nm, whereas contact angle of surface is gradually decreased from 76° to 50° respectively. Thomas Young [22] showed that contact angle of liquid simply depends on the surface tension of interface of liquid–vapor, solid–vapor, and solid–liquid. In general, any surface is not ideally flat but it has some roughness. Wenzel [23] introduced the contribution of surface roughness in contact angle study of solid surfaces as

$$\cos \delta_R = r \cos \delta_F \tag{1}$$