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Observation of smoothing and self-affine fractal roughness on MeV ion-irradiated Si surfaces

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Abstract

Scanning tunneling microscopy (STM) was used to quantitatively investigate the fractal nature of MeV ion irradiated silicon surfaces. Si(1 0 0) surfaces (with native oxide) were irradiated at room temperature using 2 MeV Si^+ ions with a fluence of 4×10^{15} ions/cm^2. One half of the sample was masked during irradiation. Root-mean-square roughness of both the irradiated and the pristine halves of the sample has been measured as a function of STM scan size. Ion beam induced smoothing has been observed at length scales below ~50 nm. The roughness exponent of the smoothed surface is $\alpha = 0.53 \pm 0.03$. When the oxide is removed from the ion-bombarded surface by thermal flushing, the roughness exponent increases to 0.81 ± 0.04 with further smoothing below the length scale ~50 nm and roughening at higher length scales upto ~300 nm. The values of the roughness exponents indicate the self-affine nature of these surfaces.

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1. Introduction

Ion–solid interactions alter the topography of solid surfaces via competing surface roughening and smoothing processes. These competing processes are responsible for the creation of surface features like quasiperiodic ripples [1–4] and self-affine fractal topographies [4–6]. These have been observed in the ion mass-energy regime where sputtering is dominant and ion incidence is tilted to the surface normal. There is a large number of studies where ripple formation was observed [2,7]. However, only a couple of studies reported the scaling behaviour of the increase of surface roughness in ion bombardment [4–6]. Surface smoothing in ion bombardment has also been observed. However, no scaling studies have been reported for surface smoothing. Scaling studies can be performed by measuring surface roughness at various length scales. Surface root-mean-square roughness σ is defined as $\sigma = \langle [h(x,y) - h]^2 \rangle^{1/2}$, where $h(x,y)$ is the surface height at a point (x,y) on the surface and h is the average height. If the horizontal sampling length on the surface is L and $\sigma \propto L^{\alpha}$, where $0 < \alpha < 1$, the surface is termed self-affine [6]. The value of the scaling exponent α indicates how roughness changes with length scales. However, it does not tell whether the surface is

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roughened or smoothed up on any kind of surface treatment.

There are reasons to expect different scaling exponents for roughening and smoothing processes as surface diffusion plays a significant role. In the case of nonequilibrium film growth by deposition, theoretical models predict a roughness exponent $\alpha \approx 0.35$ when surface mobility of deposited atoms are not taken into account and $\alpha \approx 0.66$ when surface diffusion is allowed [8–10]. For ion-bombarded surfaces $\alpha \approx 0.38$ [11] has been predicted when surface diffusion is not dominant. However, there is no theoretical prediction of scaling exponents for ion-bombarded surfaces where surface diffusion is dominant. Surface smoothing indicates the role of an effective surface diffusion.

Here we report the observation of self-affine surface topographies of the ion-bombardment induced smoothed surface at nanometer length scales and the determination of the roughness scaling exponent. Given the importance of ion beam in semiconductor industry and with the growing importance of nanostructures, it is desirable to explore the effect of ion irradiation in the nanometer length scales. Our observation of scale dependent smoothing with enhanced smoothing at smaller length scales has direct bearing on ion beam processing of nanostructures. In surface studies and thin film growth, thermal treatment of the solid substrate is almost inevitable. Here we also report on the evolution of the surface roughness in thermal treatment of ion-bombarded surfaces and determine the scaling exponent. This is important from the viewpoint of growth of self-assembled nanostructures on surfaces.

2. Experimental

Si(1 0 0) substrates with native oxide were irradiated with 2.0 MeV Si^+ ions in the ion implantation beamline of our 3 MV tandem Pelletron accelerator [12,13]. The ion beam was incident along the surface normal ($\theta \approx 0$) and rastered on the sample in order to obtain a uniformly irradiated area. One half of the sample was masked and hence unirradiated. An ion beam flux of $\approx 1 \times 10^{12}$ $cm^{-2}\,s^{-1}$ was used with fluences in the range 10^{15}–10^{16} ions/cm^2. The pressure in the irradiation chamber was $\sim 10^{-7}$ mbar. After the irradiation the sample was taken out and inserted into an ultrahigh vacuum (UHV) chamber (pressure: 3×10^{-10} mbar) containing an Omicron variable temperature scanning tunneling microscope (STM). STM measurements were performed at room temperature. STM height calibration was done by measuring atomic step heights of clean Si(1 1 1) and Si(1 0 0) surfaces. The roughness measurements were made on both the pristine as well as irradiated halves of the sample with thin (~1.5 nm) native oxide layer. Afterwards this oxide layer was removed by heating the sample at 1200 °C under UHV condition for 2–3 min. On the clean pristine half of the sample we observed terraces and surface atomic steps as usually observed on the atomically clean Si(1 0 0) surfaces. Roughness measurements were made on irradiated half of the sample before and after removal of the oxide. To determine the roughness exponent from STM images we followed the procedure described in [6]. A large number of scans, each of size L, were recorded on the surface at random locations. The σ values for the rms roughness given by the instrument for the individual scans were then averaged. Each σ value was computed after the instrument plane fitting and subtraction procedure had been carried out. This procedure was repeated for many different sizes and a set of average σ versus L values was obtained (each $\bar{\sigma}$ is the average of six to fifteen measurements).

3. Results and discussions

The log–log plots of average surface roughness $\bar{\sigma}$ versus scan size L are shown in Fig. 1 for pristine (P), ion-bombarded (IB) and ion-bombarded-thermally-treated (TIB) surfaces. Above a length scale of ~50 nm the pristine and the ion-bombarded surfaces have practically the same roughness. That is, ion-bombardment has hardly any effect at these length scales. However, for length scales below ~50 nm, the pristine surface, although has a smaller roughness, has no linear variation in the log $\bar{\sigma}$ versus $\log L$ plot. On the

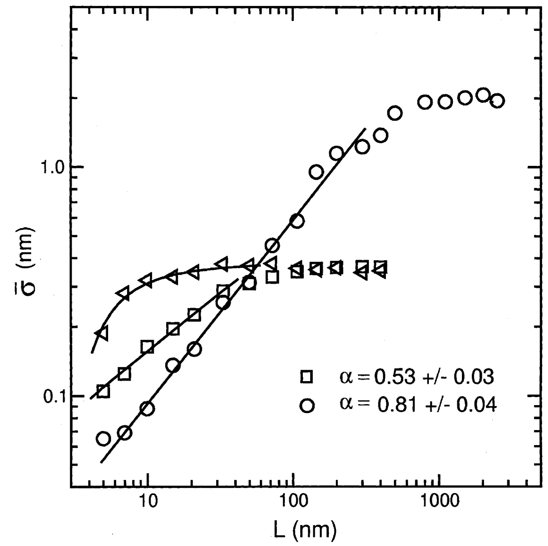

Fig. 1. Average root-mean-square roughness versus scan size on pristine (◁, P), ion-bombarded (□, IB) and ion-bombarded and thermally treated (○, TIB) surfaces. For P and IB samples Si surfaces have native oxide. There is no oxide on the TIB sample as thermal flush removes the oxide. Each data point represents an average of 6–15 scans recorded at random locations on the surface. Least-squares fit to the linear portion of the data gives the roughness exponents for the IB and the TIB samples.

other hand the ion-bombarded surface shows considerable smoothing with a linear $\log\bar{\sigma}$ versus $\log L$ variation with the roughness exponent $\alpha = 0.53 \pm 0.03$, indicating the self-affine nature of the ion-beam smoothed surface. The estimation of the error bar takes into account the effect of inclusion of an additional data point around the turning point towards saturation.

The annealed sample shows very contrasting behaviour for the pristine and the ion-bombarded halves of the sample. The pristine part shows the usual behaviour of clean surface with large terraces and steps of monatomic height. However, the ion-bombarded part shows quite striking surface roughness behaviour. At length scales below ~50 nm thermal treatment causes further smoothing of the ion-bombardment induced smoothed surface. However, at length scales above ~50 nm the surface roughness increases up to about a lateral dimension of ~300 nm following a linear relationship in the $\log\bar{\sigma}$ versus $\log L$ plot with a scaling exponent $\alpha = 0.81 \pm 0.04$, as seen in Fig. 1. A striking feature seen from Fig. 1 is that all the three curves have a cross-over at ~50 nm. A common feature is that in ion-bombardment and in the following thermal treatment the degree of smoothing depends on the initial roughness of the surface. The surface textures for the IB and TIB samples are shown in the STM images in Fig. 2.

In order to show the relative strength of the roughening and smoothing, Carter and Vishnyakov [2] extended the theoretical treatment given by

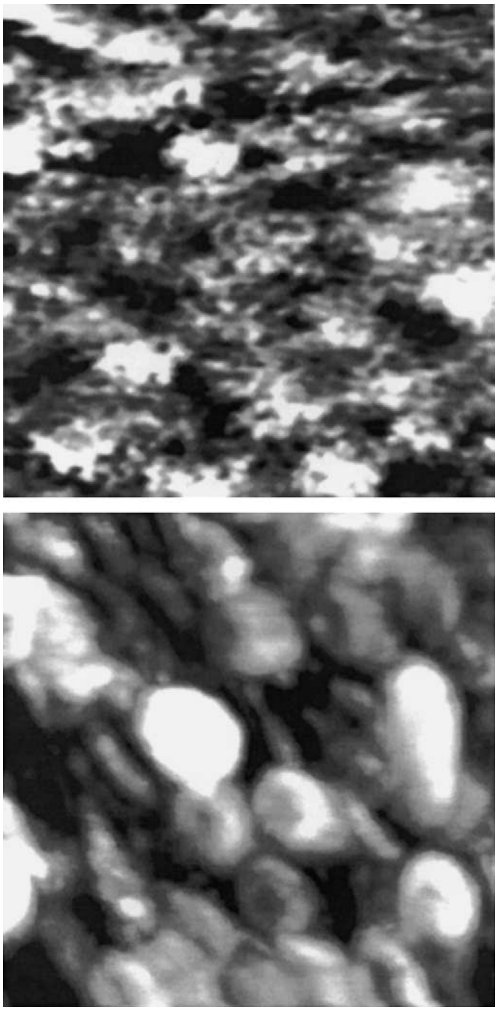

Fig. 2. STM images recorded on the IB (top) and the TIB (bottom) samples. For the IB sample the scan size is 300×300 nm^2 and the vertical scale (black to white) 2.2 nm. The corresponding values for the TIB sample are 700×700 nm^2 and 7.0 nm.

Bradley and Harper [14] and derived an expression containing the magnitudes of the roughening (sputtering) and the smoothing due to recoiled atoms. Their theory qualitatively explains the domination of smoothing over roughening at normal and near-normal incidence of the ion beam. However, their work does not deal with the scaling behaviour of surface smoothing. At present there is no existing theory that explains our observed roughness exponent. Now we discuss the surface roughness of the TIB sample. It is well known that the native oxide on a Si surface which is practically SiO_2, can be removed by flushing the sample at ~1200 °C under ultrahigh vacuum condition. This is a standard procedure for obtaining atomically clean Si surfaces, which have smooth terraces and atomic steps. This is also seen for the pristine half of our sample when flushed at 1200 °C. Ion-bombardment of the surface with native oxide leads to a different surface topography. Ion beam causes sputtering of atoms from the sample surface. A TRIM simulation [15] showing O and Si sputtering yield from a SiO_2(15 Å)/Si sample is shown in Fig. 3. We notice a large number of O and Si atoms reaching the surface with energies below the surface binding energy (~3.8 eV). These atoms cannot escape the surface. However, they can translate parallel to the surface providing an effective diffusion. This would lead to surface smoothing upon ion irradiation at short length scales.

In order to understand the behaviour of the ion-bombarded surface followed by a thermal flush at 1200 °C (TIB sample), let us concentrate on how the SiO_2/Si interface is roughened by ion bombardment.

The roughness of this surface at large length scales is typically 0.3 nm (Fig. 1), which is also the roughness of the SiO_2/Si interface before removing the oxide as known from X-ray reflectivity experiments. Ion bombardment makes this interface rough in addition to sputtering more O than Si (Fig. 3). Ion beam-induced atomic displacements across the SiO_2/Si interface are shown in the simulated results in Fig. 4. Sputtering and atomic displacements together produce an O deficiency in the oxide layer as well as displace O into the Si substrate across the interface, thus effectively producing a sub oxide SiO_x over a larger thickness. Removal of O or SiO_x by thermal flush from this

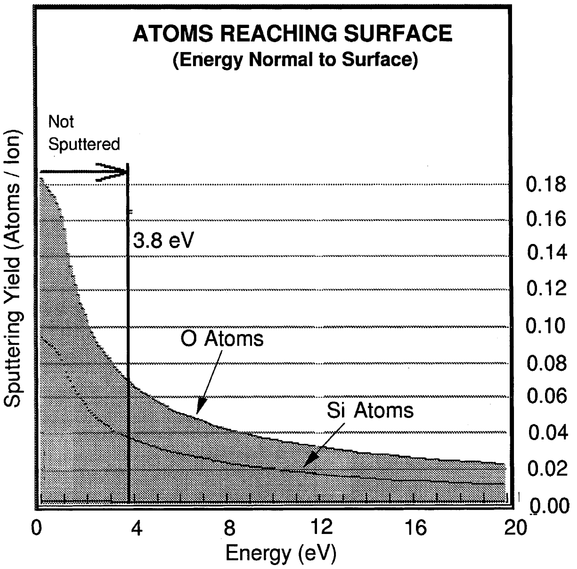

Fig. 3. Sputtering yield calculated by TRIM simulation. O sputtering is much more dominant than Si.

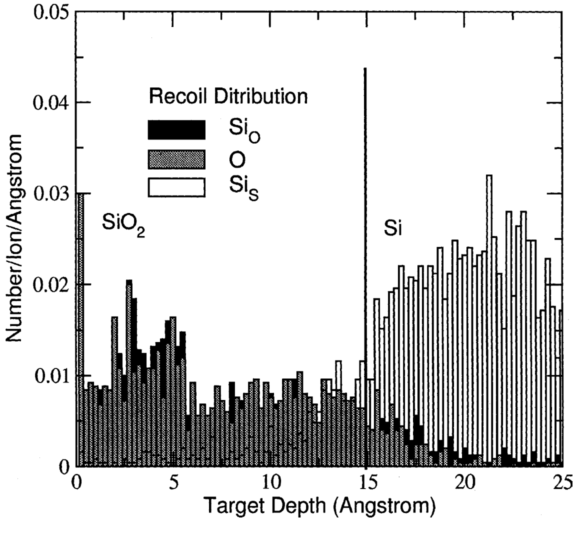

Fig. 4. Atomic displacements across a SiO_2/Si interface obtained form a TRIM simulation. Penetration of O into Si is observed. This apparently causes an increased surface roughness at larger length scales when SiO_x is removed by thermal flushing. Demarcation of the interface is shown by the vertical line at 15 Å. Si_O and Si_S indicate Si from the oxide and the substrate, respectively.

wider interface region apparently causes roughness enhancement at larger length scales for the TIB sample compared to the IB sample, as seen in Fig. 1. At smaller length scales surface smoothing in the IB sample compared to the pristing sample is due to ion beam induced ballistic atomic transport parallel to the surface. Further smoothing in the TIB sample compared to the IB sample at shorter length scales appear to be due to additional thermal diffusion.

In the present work we have explored a limited range of ion fluence (10^{15}–10^{16} ions/cm^2). (This was done to ensure amorphization of Si for future amorphization–recrystallization studies in mind). It would be desirable to explore the effect of lower fluences, as there would be a gradual transition from the pristine material to the self-affine behaviour observed here and identify the fluence at which the steady state is reached. We have also used a fixed anneal treatment (1200 °C for 2–3 min). This is what is used for removing the native oxide from Si surfaces to obtain an atomically clean surface under ultrahigh vacuum conditions. The choice of the annealing condition was dictated by our objective of removing oxide from the irradiated surface to study the exposed oxide/Si interface. Thus the roughness on the IB sample is that on the oxide surface, while the roughness on the TIB sample is that of clean Si surface after the removal of oxide. In order to explore the annealing effect on the irradiated oxide surface, annealing has to be carried out at temperatures below the oxide desorption temperature.

4. Conclusions

We have performed scaling studies by STM measurements on pristine, ion-bombarded (IB) and ion-bombarded-thermally-treated (TIB) Si surfaces with native oxide. At length scales below ~50 nm both IB and TIB samples show surface smoothing compared to the pristine surface. The IB and TIB sample surfaces are self-affine below ~50 and ~300 nm length scales, respectively, with corresponding roughness exponents $\alpha = 0.53 \pm 0.03$ and 0.81 ± 0.04. In order to understand these exponents more theoretical studies are required.

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