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ION-SPUTTER INDUCED RIPPLING OF Si(111)

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ABSTRACT

The morphology of ion sputtered Si(111) surfaces at glancing angles and elevated temperatures was studied by AFM. Under particular ion beam and sample conditions, the morphology of the surfaces was found to be rippled, with roughly sinusoidal-shaped bumps. This observation is in agreement with predictions of Bradley and Harper [1], and is similar to experiments performed by Chason et al. [2] on germanium. Observation of the temporal evolution by stopping growth and performing microscopy suggests that a steady-state rippled morphology develops by coalescence of oblong islands. The wavelength of the ripples can be controlled up to about 600 nm, with amplitudes up to 60 nm. This observation creates an opportunity to make nanostructured surfaces for the study of crystal surface dynamics.

INTRODUCTION

Ion-sputter induced rippling (SIR) of crystalline surfaces is caused by creating a competition between anisotropic roughening due to erosion by an off-normal ion beam and surface relaxation (annealing) due to surface diffusion. Because erosion operates at essentially all length scales, and surface diffusion only operates over short length scales, sputtering a high temperature surface causes features of a particular intermediate size to grow faster than features of any other size. Under some conditions this intermediate sized feature manifests itself in the form of uniform sinusoids.

Sputter induced rippling has been observed on metal surfaces [3] and also, more recently, on germanium [2]. Here we report sputter induced rippling of Si(111). To the best of our knowledge, this is the first report of the phenomenon on silicon surfaces. Additionally, the amplitude of the ripples produced are an order of magnitude higher than that those formed on Ge. We show here that there is a rather narrow window of operating parameters in which the effect can be seen, but under the right conditions uniform enough ripples can be created such that optical diffraction gratings are formed on the surface.

The theory of SIR was first discussed by Bradley and Harper (BH) [1], who derived an expression for the erosion rate of a surface as function of the ion flux and ion beam orientation relative to the overall surface normal using a calculation of sputter yield due to Sigmund [4]. Recent developments to the theory have been concerned with the effect of noise on the large length scale and long time scale evolution of the surface morphology [5]. For our purposes, a summary of the predictions Bradley and Harper's theory is sufficient. Let ψ be the angle of the ion beam from the surface normal. BH predicts a critical angle ψ_{crit} above which the wavevector of a forming ripple will be perpendicular to the beam. That is, under glancing angle conditions a ripple will grow with its valleys parallel to the ion beam. For the temperature T, the fastest growing wavelength λ_c is given by

$$\lambda_c = 2\pi \left(\frac{fk_B T}{2nD_S C} Y_0(\psi)|\Gamma(\psi)| \right) . \tag{1}$$

Here, f is the ion beam flux, n is the surface atomic density, D_S is the surface diffusivity, C is the adatom concentration, $Y_0(\psi)$ is the sputter yield of the ion beam at angle ψ to a flat surface (i.e., with no curvature), and $\Gamma(\psi)$ is a dimensionless variable primarily dependent on the volume in which an incident ion's energy is dispersed.

Features with wavelength λ_c and initial amplitude $h_0(\lambda_c)$ will grow exponentially in amplitude according to

$$\left| h(\lambda_c, T) \right|^2 = \left| h_0(\lambda_c) \right|^2 \exp(R_{\lambda_C} t) , \tag{2}$$

where

$$R_{\lambda_C} = -\frac{fa}{\rho} Y_0(\psi) \left(\frac{2\pi}{\lambda_c} \right)^2 \Gamma - \left(\frac{D_S C \gamma \Omega^2}{k_B T} \right) \left(\frac{2\pi}{\lambda_c^2} \right)^2 . \tag{3}$$

γ is the surface energy, ρ is the atomic density, Ω is the atomic volume, and a is the depth to which ion energy is dispersed in the material.

EXPERIMENT

The number of experimental variables involved in SIR are many, including (1) ion beam energy, (2) ion beam orientation to the overall surface normal, (3) azimuthal orientation of the ion beam to the crystallography of the substrate, (4) ion beam flux, (5) ion beam species, (6) substrate temperature, (7) substrate purity. To simplify the situation, the characteristics of the ion beam were fixed to be Ar, with energy of 500 eV, flux of 10 mA/cm^2. The beam was oriented along the $[1\bar{1}0]$ direction of the Si(111) target. Si(111) (n-type/phosphorus-doped 3-10 Ω-cm) wafers were mounted on a stainless steel heating block warmed by W filaments with a thin molten indium layer acting to "glue" the wafer to the heating block (as well as create a thermal contact between the wafer and the heater). A thermocouple was mounted to the heating block and calibrated to the wafer temperature using optical pyrometry. The geometry of the experiment is shown in Figure 1.

We now consider the experimental conditions under which rippling might hope to be observed using Bradley and Harper's theory. For most materials, $D_S C$ has Arrhenius form, and for Si(111), the effective activation energy is thought to be approximately 2.3 eV, although there is no definitive measurement of this value. The dimensions of the volume in which ion energy is dispersed was found using the simulation package TRIM-95. Using specific values relevant to Si(111), Table 1 was constructed, which shows the fastest growing wavelength, and the time for this wavelength to grow in amplitude 100-fold.
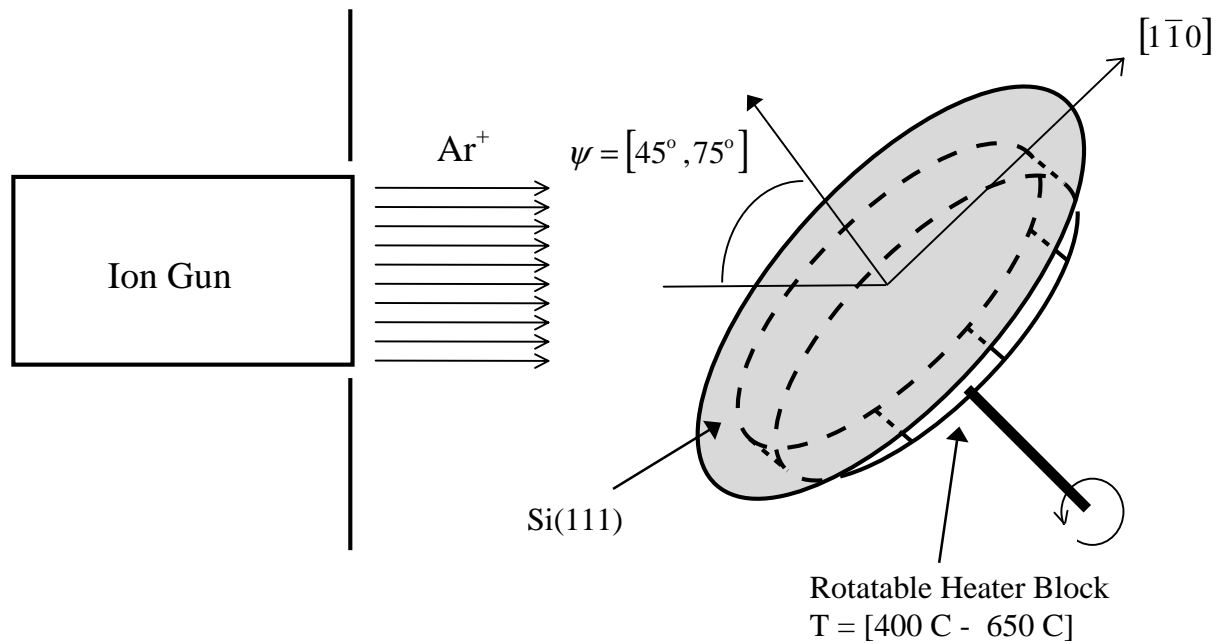
Figure 1. Experimental geometry. The sample-gun distance is approximately 10".

Table 1 contains information about the temperature and incident angle range in which one might hope to see rippling. The primary pragmatic issue is that sputtering time is limited to between a couple of seconds (instrument response) and about 11 hours (filament lifetime). For rippling on Si(111), this restricts the temperature range to 450° C - 650° C and angles of 45 to 75 degrees from normal.

Table 1. Predicted ripple wavelength (2nd column of pair, in nm) and time for 100-fold amplitude growth (1st column of pair, in hrs) vs. substrate temperature (columns, $^{\circ}$C) and angle from target normal (rows, in degrees). The tractable parameter range is highlighted.

	400		450		500		550		600		650	
45	0.0007	5	0.01	18	0.10	56	0.79	155	4.76	382	23.60	850
50	0.0010	5	0.01	19	0.15	61	1.13	170	6.80	417	33.70	929
55	0.0016	6	0.02	22	0.23	69	1.77	190	10.71	467	53.09	1041
60	0.0028	7	0.04	25	0.42	80	3.19	220	19.28	541	95.54	1205
65	0.0061	8	0.09	30	0.90	97	6.92	267	41.83	657	207.30	1463
70	0.0175	10	0.25	39	2.58	126	19.76	347	119.39	854	591.68	1901
75	0.0769	15	1.11	57	11.33	182	86.72	503	523.84	1236	2596.06	2752
80	0.7246	26	10.47	100	106.70	318	816.62	881	4932.91	2165	24446.47	4820

RESULTS

Figure 2 shows a 75 x 75 µm AFM scan of a Si(111) surface sputtered at $\approx 70^{o}$ incidence, approx. 600 C for 4.5 hrs. Ripples with wavelength 580 nm, amplitude 20 nm are clearly observable. This surface forms an optical diffraction grating observable to the naked eye. In general, the ripples extend long distances. One common feature, however, is for the ripples to worm themselves over by one or more wavelengths.
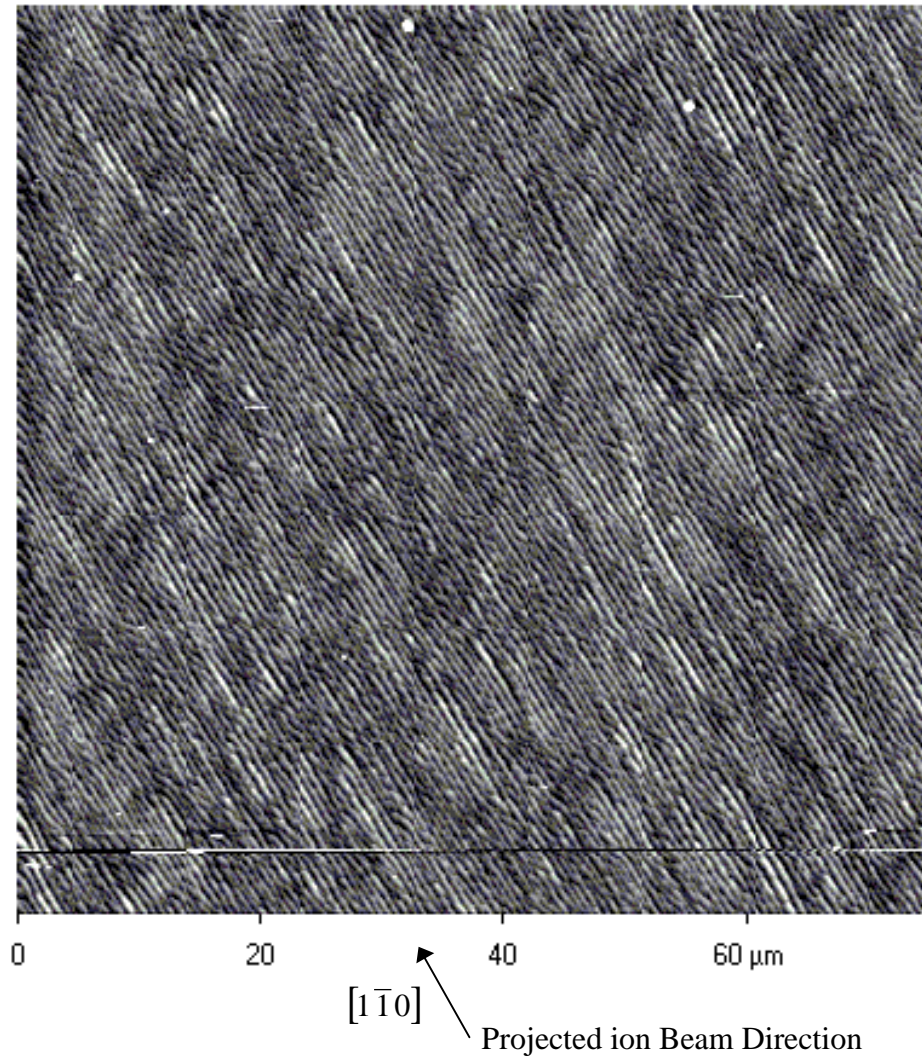

$[1\bar{1}0]$ Projected ion Beam Direction

Figure 2. Rippled Si(111) surface. Ripple wavelength = 580 nm; ripple amplitude = 20 nm.

At low temperatures under the same sputtering conditions as the sample shown in Figure 2, the surface roughens in a less organized manner. This is shown if Figure 3, which is a topograph of a sample sputtered near room temperature. It is speculated that this surface may have

amorphized. At temperatures above 650 ^{o}C, rippling was not observed in any sample. Instead, the surface remained as smooth, with roughness less than 5 nm.

Figure 4 shows another sample sputtered at approx. 60° incidence, 650 ^{o}C, 5.5 hr. Although there is clearly a directionality to the surface morphology, very long ripples are not formed. Nonetheless, there still is a characteristic wavelength. In Figure 4, this wavelength is approximately 400 nm showing that, in principle, wavelength of a particular size can be tuned. The amplitude in Figure 4 is actually slightly greater than the sample in Figure 2, being of order 60 nm.

SEM of surfaces sputtered for short times (< 4 hrs) suggests that in the early stages of the ripple formation, elongated bumps with width approximately equal to the ripple wavelength are formed. It is hypothesized that as these bumps grow in amplitude, they begin to impinge on each other coalesce into ripples.
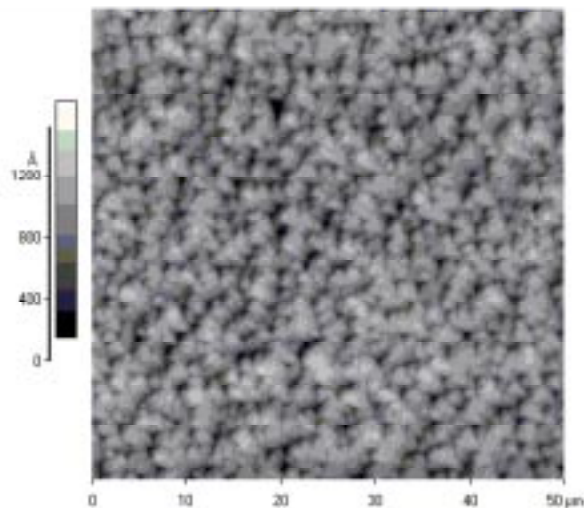

Figure 3. Roughened Si(111) surface, sputtered under identical conditions as the sample in Figure 2, except at a temperature of 50 ^{o}C.
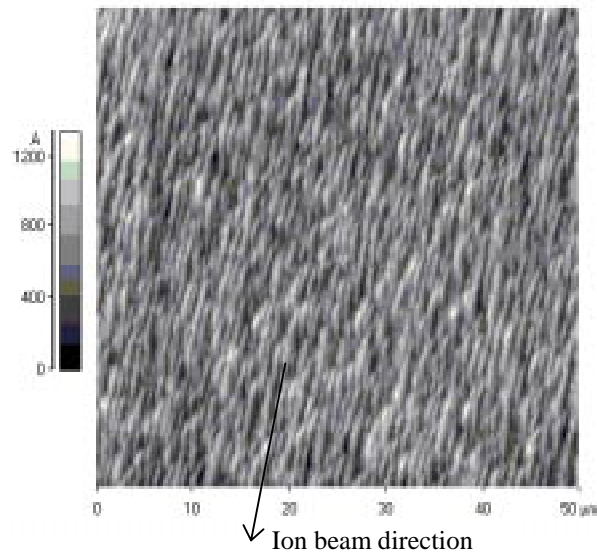
Ion beam direction

Figure 4.   Semi-rippled Si(111) sputtered at 60° incidence, 650 ^{o}C, 5.5 hr.

The variation of wavelength with temperature/ion beam angle does not fit exactly the values predicted in Table 1. This may be due to uncertainties in the relevant activation/formation energies for surface diffusion, as well as errors in measuring ion flux, etc. However, the data presented in Table 1 does exhibit the some characteristics as seen in the experimental data. The important ones are (1) higher wavelengths are seen at more grazing incidence, (2) to see rippling sputtering must be done for a number of hours, (3) neither at too low temperatures nor at too high temperature is rippling observed. These observation suggest that more experimental data, in particular time evolution information, might be used to fit Bradley and Harper's theory and extract the formation and migration energies for atom motion on Si(111). Larger length scale and longer time scale data might also be used to test the newer ideas of ion induced rippling given by Cuerno and Barabási [5].

CONCLUSIONS

The morphology Si(111) surfaces sputtered at glancing angles and elevated temperatures was studied by AFM. Within a particular range of angles and temperatures, the morphology of the surfaces was found to be rippled, with rough sinusoidal-shaped bumps. The observation of rippling on Si(111) is similar to that seen in experiments performed by Chason et al. [2] on germanium. The range of angles and temperatures in which rippling on Si(111) is observed is consistent with predictions of Bradley and Harper [1]. The wavelength of the ripples can be varied up to about 600 nm, with amplitudes up to 60 nm.

ACKNOWLEDGMENTS

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