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# Measurement of microscopic surface deformation due to low energy ion bombardment on Si(111)

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ABSTRACT

A low energy electron diffraction spot profile analysis of the Si(111) surface, after argon ion bombardment at an elevated temperature, finds a continuous, low amplitude distribution of surface height, in addition to the atomic-step-and-terrace structure. With an amplitude of tenths of an Angstrom or less in height, correlated laterally over tens of Angstroms, this microscopic surface deformation is measured versus ion dose and for various sample temperatures during bombardment and annealing. For 230 eV argon ion doses increasing in the range of $10^{15} - 10^{16}$ cm$^{-2}$ with the sample held at 580 K and 800 K, the amplitude of surface deformation, measured as the standard deviation $w_z$ of the continuous height distribution, increases steadily in the range 0.06 − 0.10 Å. For higher ion doses in the range of $3 \times 10^{16} - 1 \times 10^{17}$ cm$^{-2}$, saturation-like behavior with $w_z \approx 0.14 - 0.15$ Å is measured at 800 K. After an ion dose of $10^{17}$ cm$^{-2}$, the surface deformation shows a lateral correlation length of 20 Å, implying an average lateral feature size of ≈40 Å, slightly less than the average terrace width of ≈60 Å. On the other hand, after the same ion dose at 300 K followed by annealing at 800 K, the surface deformation has a similar correlation length but a much smaller height amplitude of $w_z = 0.06$ Å. It is notable that $w_z$ depends quite differently on sample preparation conditions overall, compared to the average terrace width and to the degree of $(7 \times 7)$ order indicated by diffraction intensities, both of which are found to evolve much more slowly with ion dose in conditions for which the height amplitude increased in the range of 0.06 − 0.14 Å. The surface height deformation due to strain around buried, subsurface defects is estimated as a function of defect concentration using a simple, order-of-magnitude theoretical model, in which a distribution of subsurface defects is modeled as small inclusions in a continuous elastic medium. This approximate model is consistent with the measured growth of the deformation amplitude $w_z$, for a range of possible parameter choices for small defect clusters located in a shallow layer below the surface. The surface deformation is partially removed by annealing above 870 K.



## 1. Introduction

The interaction of low-energy ions with surfaces is fundamental to various techniques for materials processing, from sputter-cleaning samples in ultrahigh vacuum and ion-beam etching, to ion-implantation and defect engineering [1], as well as for ion beam-assisted thin-film growth, surface pattern and ripple formation [2,3] and blistering phenomena [4].

In addition to causing surface defects, ion bombardment of silicon creates sub-surface defects which have been studied in the past using a number of techniques, for a range of ion bombardment conditions and sample temperatures. [5–9] The strain field in the subsurface region has been measured quantitatively (although laterally averaged) by XRD after low energy argon ion bombardment [10–12], as well as after nitrogen [13], hydrogen [14] and boron [15] implantation. In the latter work, cross-sectional TEM images revealed the non-uniformity of the strained layer.

Furthermore, the surface morphology of Cu(110) has been characterized by reflectance difference spectroscopy (RDS), spot profile analysis low energy electron diffraction (SPALEED), and scanning tunneling microscopy (STM) [16,17]. After pulsed laser irradiation, evidence of both surface defects and subsurface dislocations lines was found, while after 900 eV Ar ion bombardment instead, the dependence of the SPALEED diffraction profile widths on increasing annealing temperature was ascribed to annealing of first surface defects, and then subsurface point defects, consistent with RDS measurements. X-ray diffuse scattering measurements of homoepitaxial Ag(001) films grown at low temperature by Kim et al [18] indicated the presence of large vacancy clusters with local dilatation volume of 750 Å$^3$. Local surface deformations of ≈1–2 Å in height have been observed by STM by Springholz et al above buried misfit dislocations in heteroepitaxy of EuTe on PbTe(111) [19], and above buried PbSe quantum dots in PbTe [20].

The autocorrelation function of "mottled" STM images of a III–V alloy, with a surface height amplitude of ≈0.1 Å and lateral correlation

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length of ≈ 10 Å, has been modeled in terms of strain effects due to compositional fluctuations [21]. A shallow surface depression of similar dimensions was observed after 8 keV Kr ion bombardment of PbS(001) by STM, and interpreted in terms of lattice distortion [22]. After 50 eV Ar ion bombardment of graphite, dome-like features of similar lateral width but with height ≈1 Å (retaining regular lattice periodicity) were observed by STM and attributed to lattice deformation around ions embedded between the first two atomic planes. [23] The properties of Ar ion-induced vacancy defects in graphene on Pt(111) have also been studied by STM. [24] Argon-filled nanocavities buried deeper beneath Cu(001) and (110) surfaces have been studied recently by analysis of STM and STS data [25].

However, a spot profile analysis of low energy electron diffraction (SPALEED) data provides characterization of the surface morphology complementary to scanning probe techniques: a direct, statistical measurement of the variation in surface height across the surface, including the step-and-terrace distribution and the distribution of elastic deformation in surface height, as well as static Debye–Waller factors for other atomic disorder on the surface are obtained [26–28]. In our previous work, a surface deformation after ion bombardment was detected on GaAs(110), which we attributed to strain around disordered, subsurface defects [29]. After argon-ion sputtering at 230 eV at room temperature and annealing to temperatures less than 740 K, LEED spot profile measurements determined the standard deviation of the surface-height deformation to be in the range of 0.1–0.2 Å with lateral correlation length of about 50 Å, [30] and showed slower annealing kinetics than that of the atomic step density on the surface.

In heteroepitaxial growth systems, surface deformations have been measured by LEED in the past [28,31–33]. For Ge thin films on Si(111), Horn-von Hoegen et al. [28] found a surface undulation with amplitude 0.5 Å and lateral spacing 10 nm due to the periodic, underlying dislocation array at the buried interface. This regular surface deformation has been studied by scanning tunneling microscopy as well [34]. This effect has also been measured in epitaxial Bi(111) films on Si(001). [35] In ultrathin Pb films deposited on Si(111) at 25 K, SPALEED measurements indicated deviations from exact epitaxial positions, which could be modeled as a non-periodic array of domains with a height variation of ≈0.2 Å, joined by inclined regions in between. [33] More recently, analysis of SPALEED and STM measurements of the growth of Pb on the Pb/Si(111)-$\alpha\sqrt{3}\tilde{n}\sqrt{3}$ phase was used to probe the structure of the metal/semiconductor buried interface [36].

In this paper, the application of LEED spot-profile-analysis to characterize the surface-height deformation after ion bombardment will be studied, using the Si(111) surface as a model system. Experimental details of the surface preparation are summarized in Section 2, and the kinematical LEED analysis used to characterize the deformation-height distribution independently of other surface defects is outlined in Section 3.

The height amplitude (i.e. contribution to the interface width) $w_z$ of surface deformation is studied in Section 4, after a range of ion bombardment and annealing conditions for which the effect is seen very clearly in the LEED spot profiles. For this purpose, moderately elevated temperatures are used to reduce short-range atomic disorder on the surface, and ion doses in the range of $10^{15}$–$10^{17}$ cm$^{-2}$ are used to give a relatively large population of subsurface defects. These ion doses are similar to those used in a submonolayer to multilayer kinetic roughening study by Chan and Wang, [37] although smaller ion doses were used in the XRD strain measurements of Ghose et al. [10] and much higher doses are also of interest for ripple and pattern formation on the Si(111) surface [38].

The (7 × 7) LEED intensity is measured as an indication of surface order and the average terrace width on the surface is also determined.

The growth of the amplitude $w_z$ of the continuous height distribution with ion dose is then compared with a simple theoretical model of the surface distortion in which the solid is treated as a semi-infinite elastic continuum with a distribution of small inclusions representing defects in the subsurface region.

## 2. Experimental setup and sample preparation

The experiments were performed in an ultrahigh vacuum chamber with a base pressure below $10^{-10}$ Torr and equipped with surface analysis instruments for low energy electron diffraction spot profile analysis (SPA-LEED) [39] and Auger electron spectroscopy (AES), as described previously [40].

Samples were taken from a Si(111) wafer, cleaved to a size of about 12 × 12 mm$^2$ and mounted in a molybdenum holder as described previously, [40] on a backing plate heated from behind. The sample temperature was monitored by a thermocouple spot-welded to the Mo backing plate and controlled automatically. The measurement accuracy of the sample temperature is about ±25 K, after a calibration of the steady-state temperature difference between the sample and holder [29].

One sample was prepared in an ultrahigh vacuum by a conventional, high temperature heating at 1450–1500 K for 20 s, followed by slow cooling below 1200 K [41]. The backing plate was heated through the lower temperature range for sample temperatures up to 1050 K by thermal radiation from a tungsten wire heater in alumina tubes, with electron bombardment heating of the backing plate from a second tungsten filament used to quickly heat the sample through the upper temperature range, monitored by a C-type thermocouple.

Another sample was prepared in an ultrahigh vacuum previously by argon ion sputtering at 230 eV and annealing at up to 1050 K using the radiative heating alone and monitored by a K-type thermocouple. This sample provides some points of comparison as discussed later.

The ion beam was defocused to give a more uniform incident ion flux over the central region of the sample monitored by the LEED beam which has a width of ≈1 mm. The current density profile of the ion beam was measured to have a FWHM of ≈5 mm at the sample. The absolute ion dose was calibrated to an accuracy of ≈30%.

Auger spectroscopy detects no contamination by C, O, (nor Ni) above the noise level of ≈0.5 % on either sample, although a low density of carbon-related defects in the sub-surface region may still be present, as has been observed after ion bombardment elsewhere [8,42]. After ion bombardment at elevated, but lower temperatures, a small concentration of embedded argon near the surface is detected by Auger spectroscopy. At 800 K an argon Auger signal with peak ratio Ar(215 eV)/Si(92 eV) of 1 % is found, increasing to 4% for bombardment at 580 K. However, the Auger signal preferentially samples the first few atomic layers.

The overall argon concentration in the near-surface region is also estimated based on rough measurements of the amount released using a quadrupole mass spectrometer (Spectramass Selectorr 100) to measure the partial pressure of argon in the chamber with ion pumps on, as the sample temperature is increased to 1500 K. The areal concentration of embedded argon is estimated by assuming it follows the shape of the ion beam profile on the surface described above. The subsurface argon concentration is thus found to increase in the sequence ≈1.2 → 4 → 5 × $10^{14}$ cm$^{-2}$ for ion doses of 0.2 → 1.5 → 7.5 × $10^{16}$ cm$^{-2}$, respectively, at 800 K. In addition, an ion dose of 1 × $10^{17}$ cm$^{-2}$ at 300 K followed by annealing for 25 minutes at 800 K gave an argon concentration of ≈5 × $10^{14}$ cm$^{-2}$ as well.

## 3. LEED spot profile analysis

All LEED measurements were made with the sample held at 350 K between stages of ion bombardment or annealing. Profiles of the (0,0) diffraction spot were made using the SPALEED instrument in its conventional configuration with near-normal incidence [39].

The LEED diffraction spot profile at a crystalline surface may be described in the kinematical approximation by a structure factor $S\left(\vec{k}\right)$ defined as the squared modulus of the 2-d Fourier transform of the scattering amplitude for a single 2-d unit cell (labeled $i$) on