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Full Length Article

Effects of 1D disorder on the reciprocal space of rare-earth silicide/Si (1 1 1) epilayers

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ABSTRACT

Sub-monolayer epitaxial self-assembled silicides of Er and Gd on Si(1 1 1) exhibit unique structural and morphological features that play a crucial role in their magnetic behavior. The effects of disorder occurring between the characteristic quasi-1D surface reconstructions of such systems were analyzed by low energy (LEED) and reflection high energy (RHEED) electron diffraction. The observed Rare-Earth Metal (REM)-Si antiphase domains caused appearance of diffuse intensity planes in the surface reciprocal space, visualized as straight (arched) streaks in LEED (RHEED) diffraction patterns. Random registry shifts between adjacent adsorbate atomic chains caused extinction of the half-order satellites in diffraction patterns of a surface reconstruction identified by scanning tunneling microscopy (STM) as Si(1 1 1)-(3 × 2)-Gd.

1. Introduction

Since the beginning of the previous century, electron diffraction techniques have been a useful tool for determination of the surface structure of crystals. Even with the advent of direct and more local surface characterization techniques such as STM, electron diffraction methods are still reliable complementary tools for analysis of symmetry and periodicity on crystal surfaces [1–7]. RHEED and LEED are standard methods for identification of multiple surface structures and orientation relations between them, as well as for monitoring the evolution and the degree of crystallinity of surfaces during epitaxial growth processes [7–15]. Both techniques can be used for detection of defects and disorder, *via* the effect of the latter on intensities of the selected Bragg reflections [16–21].

Non-vanishing spherically symmetrical distribution of diffracted intensity, *aka* diffuse scattering, is a common effect that arises from surface disorder and causes an increase in the background intensity of diffraction patterns. Diffuse intensity planes (DIPs) are parallel, regularly spaced arrays of reciprocal planes, arising as a consequence of out-of-phase disorder between linear and parallel atomic chains in the real space. These planes are perpendicular to the chains, with the interplanar periodicity reciprocal of the real space interatomic distance along the chains [17,18,21,22].

As the incident electron beam in LEED geometry is normal to the surface, LEED provides a top view of the reciprocal space, where intersections of the DIPs with the Ewald sphere always appear as parallel straight lines. In RHEED geometry, the grazing incident electron beam is practically parallel to the surface, providing a side view projection of the reciprocal lattice onto RHEED screen. Consequently, the curvature of the intersection lines between the DIPs and the Ewald sphere depends on the angle α between the electron beam azimuth and the atomic chains, and on the distance L between the sample and the screen. Straight vertical streaks (normal to the shadow edge in RHEED patterns) can only appear on the screen when the beam azimuth is perpendicular to the atomic chains. Rows parallel to the incident beam generate DIPs parallel to the screen, resulting in an arc outlining the top part of the Ewald sphere, but effectively causing only an increase of the background intensity. For any angle $0 < \alpha < \pi \wedge \alpha \neq \pi/2$, DIPs intersections with the Ewald sphere are projected onto RHEED screen as elliptical segments, according to the equation by *Deleschuze* et al. [17]:

$$[x/\cos(\alpha) - L\sin(\alpha)]^2 + y^2 - L^2\sin^2(\alpha) = 0 \quad (1)$$

DIPs have been mostly reported to appear due to adsorbed and chemisorbed species on single crystal surfaces, with 1D disorder boundaries (e.g. S on Ni, CO on W, O on Ta and Cr) [16,17,23,24]. Diffuse scattering has been attributed to the presence of long-range disorder between high densities of quasi-1D reconstructed Anti Phase Domains (APDs), as in the case of epitaxial growth of GaAs(0 0 1) surfaces [4,18,21,25]. Evidence of diffuse scattering in LEED and RHEED patterns were also reported to take place upon metal adsorption on Si

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surfaces and silicide formation [22,26–35].

Nanostructure shape and ordered arrangement in the array play a critical role in controlling the collective magnetic response of magnetic nanoparticles [36,37]. Self-assembled rare-earth silicide nanostructures obtained *via* epitaxial growth of sub-monolayer amounts of metal on Si surfaces exhibit unique anisotropic features that can strongly influence their electronic and magnetic properties [38,39]. Such REM/Si epilayers are characterized by quasi-1D reconstructions consisting of arrays of parallel atomic chains. Diffuse scattering from self-assembled atomic chains of REM atoms adsorbed onto Si(1 1 1) surfaces has been documented [40–44], however to date, the effects of linear disorder on the reciprocal space of REM-Si surfaces have not yet been thoroughly investigated.

In this work, the effects on electron diffraction patterns caused by one dimensional disorder on vicinal Si(1 1 1) surfaces exposed to sub-monolayer amounts of Gd and Er, are analyzed. On Er/Si(1 1 1) surfaces, STM revealed (2 × 1) reconstructed Er disilicide islands, elongated along $[1\bar{1}0]$ and two other equivalent in-plane directions, and Gd-induced (3 × 2) quasi-1D surface reconstructions materialized on Gd/Si (1 1 1) surfaces. Long-range linear disorder between the superstructures accounted for the diffuse streaking in LEED and RHEED patterns, whereas stochastic registry shifts along Gd atomic chains also affected the intensity of the half-order diffracted beams. A simple model for extinction of the half-order satellites due to antiphase relations between the adjacent domains is presented.

2. Material and methods

The experiments were conducted in an ultra-high vacuum (UHV) variable-temperature scanning tunneling microscope (VT-STM) by Omicron Nanotechnology GmbH, equipped with low- (LEED) and reflection high- (RHEED) energy electron diffraction (20 kV), and capable of operation up to 1250 °C by direct-current heating. Si(1 1 1) wafers, with a 4°-miscut towards the $\langle 11\bar{2}\rangle$ direction, were cut into 10 mm × 1 mm strips, chemically degreased and cleaned *ex-vacuo*, and introduced into the UHV system. We routinely employ such regularly stepped vicinal surfaces as a template for epitaxial step-decoration growth [45–48], however this is not the focus of this paper. In UHV (base pressure $\approx 1 \times 10^{-10}$ mbar), after thorough degassing, the oxide was evaporated by repeated flashes at 1150–1200 °C, and the clean Si (1 1 1) surface slowly cooled until well-ordered (7 × 7) reconstruction became apparent in diffraction and STM images. Er was e-beam evaporated from a pure Er wire onto a Si(1 1 1) substrate held in the VT-STM stage at 400 °C (RD). Gd was evaporated from a tungsten crucible filled with pure Gd pellets onto the Si substrate at room temperature (RT). The covered substrate underwent a series of consecutive 2 h annealing treatments at 500 °C, 550 °C, and 600 °C (SPE) under continuous STM imaging. The principal crystallographic $\langle 1\bar{1}0\rangle$ and $\langle 11\bar{2}\rangle$ in-plane directions marked in Figs. 1(a), 2(a) and 3(a) hold for every other STM image in this paper. The initial metal coverage ($0.8 < \theta < 0.9$ eq. ML's) was estimated from image analysis of unreacted metal clusters immediately following the deposition, and assuming spherical cluster shape, as described in our recent works [45–47]. LEED patterns were acquired at beam energies between 40 eV and 125 eV, and RHEED patterns at 20 kV beam energy along the $\langle 1\bar{1}0\rangle$ and $\langle 11\bar{2}\rangle$ azimuths. STM images were acquired in a constant-current mode using tunneling conditions of $0.1\text{nA} < I < 0.3\text{nA}$ and $-3.5\,\text{V} < V < +3.5\,\text{V}$ sample bias, with scanning direction rotated by a 45° with respect to the sample long edges. This rotation is the reason for a diagonal appearance of the step edges in the figures. WSXM freeware was used for post-acquisition image processing.

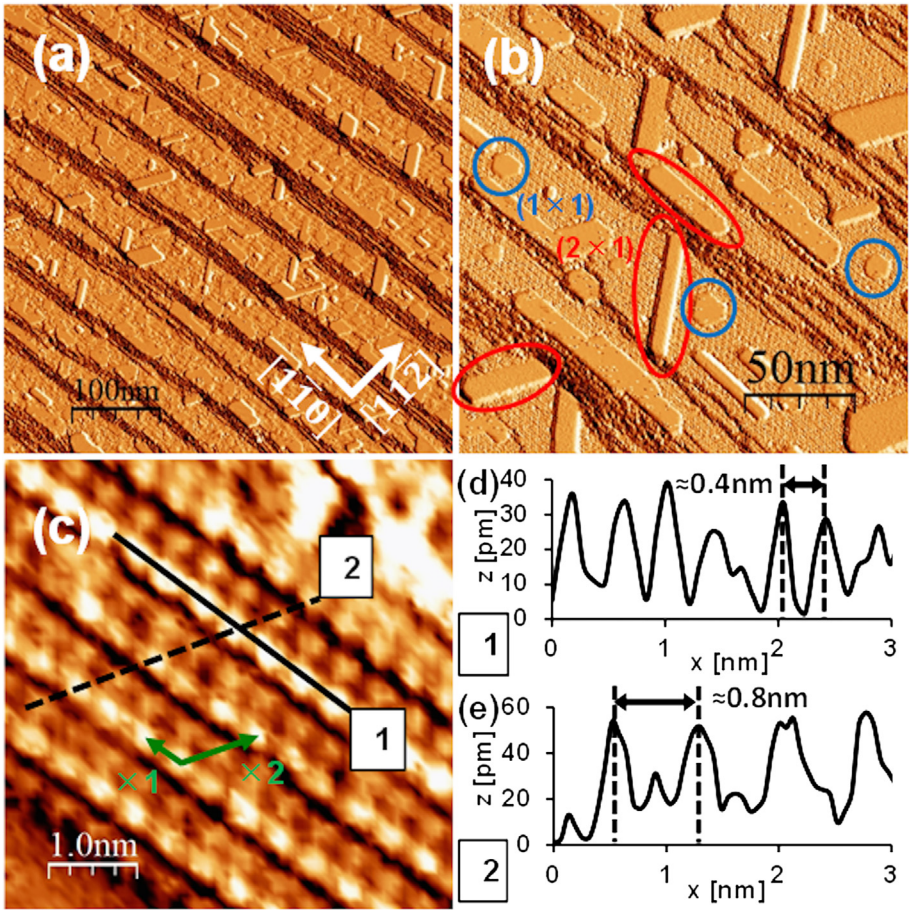

Fig. 1. (a) 500 nm × 500 nm, (b) 200 nm × 200 nm STM current-images acquired after reactive deposition at 400 °C of a sub-monolayer Er on a vicinal Si (1 1 1) surface. Examples of 2 × 1 and 1 × 1 reconstructed islands are outlined in red and blue respectively. (c) High resolution 5 nm × 5 nm STM image showing the 2 × 1 reconstruction on the top facet of a rod-shaped island; green arrows outline the 2 × 1 unit mesh. The scans were obtained with a -1 V bias. Line profile plots obtained along the (d) ×1 (full line in (c)) and (e) ×2 (dashed line in (c)) periodicity of the 2 × 1 reconstructed surface. The mean interatomic distances were equal to and double of a single Si surface lattice constant ($a = 0.384$ nm) in (d) and (e) respectively. In-plane directions shown in (a) are valid for all the STM images presented in the paper. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)