STM and LEED studies of the atomically ordered terraced Si(557) surfaces

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We report results of LEED, STM and photoemission studies of atomic and electronic structure of atomically ordered terraced Si(hhm) surfaces. LEED and STM data demonstrate a possibility to fabricate atomically accurate terraced structures based on Si(557) with different periodicities depending on thermal treatment procedure. Atomically resolved STM images reveal (7×7) terrace ordering and triple step structure. Comparative photoemission studies of the valence band and Si $2p$ core level electronic structure have been done on clean stepped surfaces with different terrace widths (namely, Si(557), Si(556)) and flat Si(111)-(7×7).

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In the last decades there has been a growing interest to such kind of low-dimensional objects as quantum wires, dots, stripes and so on. Stepped surfaces with atomically accurate hill and valley structures are good candidates for fabrication of these nanoobjects and their arrays, for example by deposition of submonolayer coverages of metals with unique transport and magnetic properties. Application of stepped Si(hhm) surfaces obtained by deflection from Si(111) plane looks very promising for the following reasons: 1) high stability of well-studied Si(111)-(7×7) reconstruction that can determine and stabilize a terrace width, 2) possibility to investigate electronic structure, magnetic and transport properties of fabricated metallic nanostructures on the isolating substrate (for example, at low temperatures). The principal possibility of fabrication of atomically accurate regular low-dimensional stepped structures based on Si(557) vicinal surface was demonstrated by scanning tunneling microscopy (STM), for example, in Refs.[1,2]. In the recent years, a number of experimental studies of electronic structure and transport properties of organized Au atomic wires on well-ordered Si(557) have been published [3–11]. These studies exhibited a variety of interesting properties of quasi-one-dimensional Au chains on the stepped silicon surface. However, there are a few works related to the studies of atomic and electronic structure of clean stepped silicon surface. Besides, there is some divergence in published data concerning possible dependence of stepped silicon surface structures on surface preparation procedure. Note also that the previously published STM data [1,2] could not give exact information on step atomic structure that became later an object of extensive spot profile analysis of low energy electron diffraction (SPA-LEED) investigations [12] which revealed triple steps with (113) oriented facets. The current experiments were mainly related to investigation of the atomic and electronic structures of clean well ordered terraced surfaces (namely, Si(557), Si(556)) by LEED, STM and photoelectron spectroscopy. We tried also to obtain information about the influence of the surface preparation procedure on atomic arrangement on the terraces and steps facets for regular step arrays.

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The STM experiments on Si(557) surfaces were performed in ultra high vacuum (UHV) electron spectrometer LAS-3000 (RIBER) equipped with Auger electron spectroscopy (AES), LEED and STM techniques. Base pressure in spectrometer chamber was below $1\cdot10^{-10}$ Torr. The investigations of Si(557) atomic structure at room temperature (STM GPI-300) were done using electrochemically etched tungsten tips. The etched in 2M-NaOH polycrystalline tungsten tips were cleaned and sharpened in UHV STM chamber by flash heating (800–900°C) and Ar^+ ion sputtering at argon pressure $p = 5\cdot10^{-5}$ Torr and ion energy $E = 600$ eV.

Photoemission (PE) experiments on Si(557) and Si(556) surfaces were performed in Russian German Beam line of synchrotron center BESSY-II (Berlin) with CLAM-4 electron spectrometer. Base pressure in spectrometer chamber was about $1\cdot10^{-10}$ Torr. Photoemission spectra of valence band and Si $2p$ core level as well as LEED patterns were taken at the sample temperature of 100 K.

Si(hhm) surfaces studied in this work consist of (111) oriented terraces and steps along $[1\bar{1}0]$ direction. Si(557) samples were obtained by 9.45° deflection, Si(556) — by 5.05° deflection from (111) in $[\bar{1}12]$ direction. In the investigations n-type Si(557) and Si(556) wafers (3 mm width, 8 mm length, P-doped, $25\,\Omega\cdot$cm at 300 K) were used. Clean Si surfaces were obtained by resistive heating in UHV chamber with direct current both parallel and perpendicular to steps. In our experiments we could not reveal any clear dependence of terrace atomic structure (and terrace width) on current direction. The most important factor determining the ordered terraced surface structure in our experiments was the thermal treatment procedure but not the current direction. We used the following procedure to obtain well-ordered atomic structures: 1) outgasing of the sample and sample holder at 600°C for 10–15 h (typically, overnight), 2) flash heating at 1250°C for 10 s, 3) slow cooling to 1050°C for approximately 2 min, 4) fast cooling to 850°C for 2–3 s, 5) annealing at 850°C and then cooling to room temperature in several seconds. The annealing time at 850°C was varied to obtain ordered atomically accurate

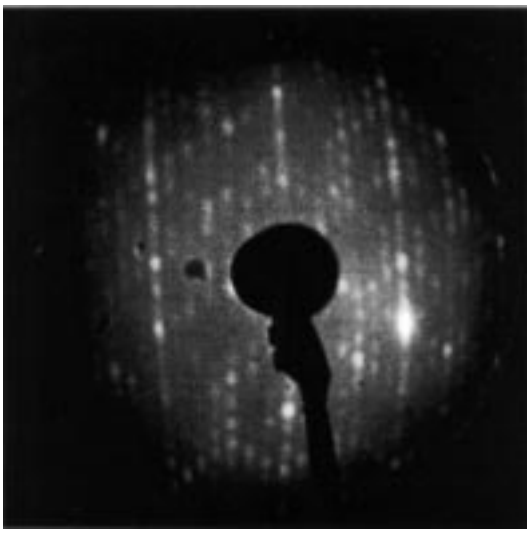

Figure 1. Typical LEED pattern of Si(hhm) surface prepared by thermal treatment procedure described in the text — LEED pattern of Si(556) taken at 100 K (20 s annealing at 850°C, electron energy $E_p = 87$ eV).

structures on Si(557). Pressure during flash heating at 1250°C and annealing at 850°C did not exceed $4 \cdot 10^{-10}$ and $1.5 \cdot 10^{-10}$ Torr, respectively. In our experiments the time of annealing at 850°C played a crucial role in formation of regular periodic structures with desired periodicity.

The best STM and LEED data for well-ordered Si(557)-(7×7) surfaces were obtained at two periods of annealing at 850°C (slightly below (1×1) → (7×7) phase transition) equal to ∼ 20 s and ∼ 20 min. LEED patterns from the surface prepared by both procedures demonstrated (7×7) superstructural spots as well as vertical stripes and elongated spots corresponding to the presence of step arrays on the surfaces. However, the (7×7) spots were more distinct in the LEED pattern measured from Si(556) and Si(557) surface annealed at 850°C for 20 min. As an example, Fig. 1 shows the LEED pattern of Si(556) surfaces annealed at 850°C for 20 s. Note, that the LEED patterns of Si(557) surfaces after longer annealing at 850°C were similar to the ones taken from Si(556) surface annealed at 850°C for 20 s (Fig. 1).

STM investigations showed that the observed slight difference in the LEED patterns of Si(557) surfaces prepared by two procedures can be related to different periodicities of the obtained terraced structures. Fig. 2,*a*, *b* demonstrate two STM images of Si(557) surfaces with different terrace widths that correspond to samples annealed at 850°C for 20 s (Fig. 2, *a*) and 20 min (Fig. 2, *b*). One can see from the images that the periodicity of the stepped structure on Si(557) surface is approximately two times larger at annealing time of 20 min (Fig. 2, *b*). It may be supposed from the STM data that the surface ordering with doubled (7×7) terraces is energetically favourable relative to the Si(557) surface ordering with one (7×7) unit cell per terrace. Interestingly enough that the step bunching in our experiments was observed at current direction both parallel and perpendicular to the steps.

STM images with higher resolution (Fig. 3, *a*–*c*) also demonstrate difference in periodicity of hill and valley structures, (7×7) ordering within terraces, and triple step structure for surfaces obtained at both thermal treatment procedures. The triple step structure is more apparent in 3D presentation of the measured STM images. As an example, Fig. 3, *b* and *c* show 3D-view of the STM images of Si(557) corresponding to tunneling from empty (*b*) and filled (*c*) surface states. These images clearly demonstrate triple steps and (7×7) cells on terraces of different widths. Our STM images reveal different step facet orientations for samples fabricated by two different thermal treatment procedures. The facet orientations in the cases of 5.7 and 11.4 nm periodic structures let us suppose about the presence of

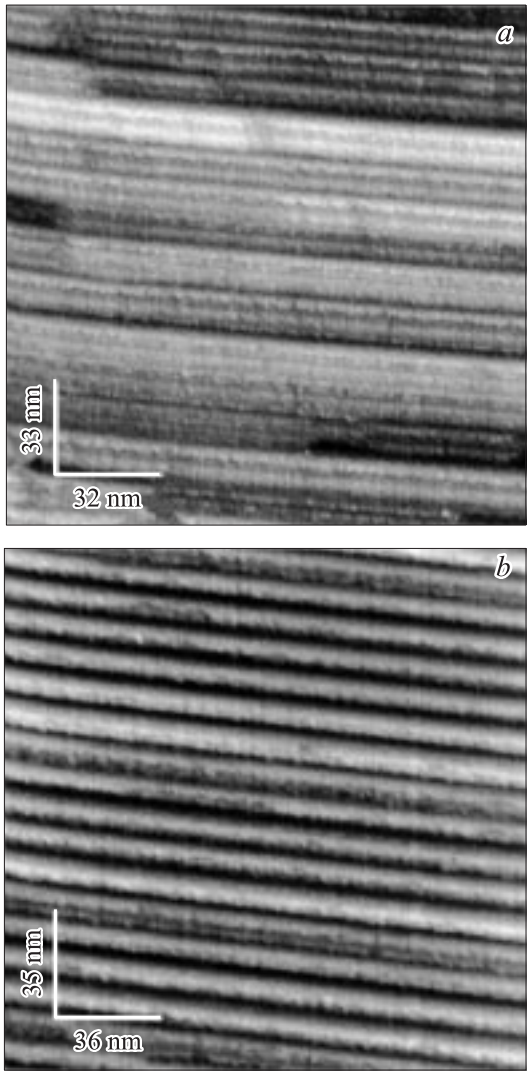

Figure 2. STM images of Si(557)-(7×7) with different terrace widths: *a* — 163×163 nm^2, tunneling voltage $U_t = -600$ mV, tunneling current $I_t = 0.12$ nA; *b* — 178×178 nm^2, tunneling voltage $U_t = -800$ mV, tunneling current $I_t = 0.08$ nA.

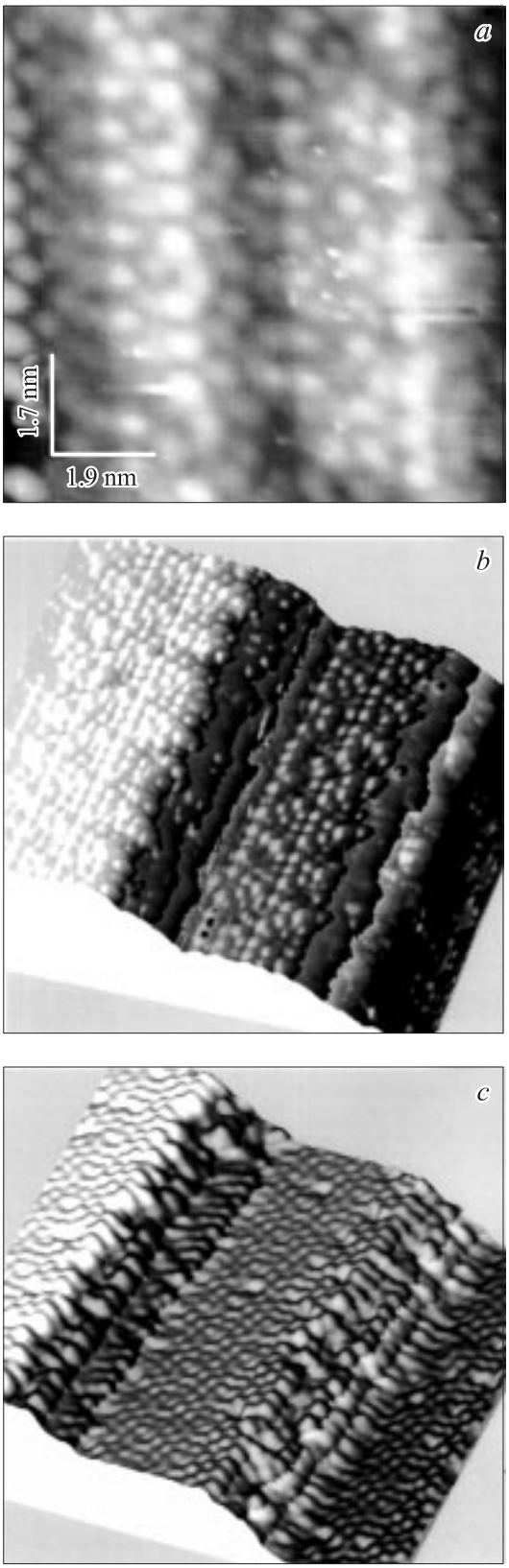

Figure 3. Top- (a) and 3D-view (b, c) of the atomically resolved Si(557) STM images demonstrating (7×7) terraces and triple steps: a — $11\times11\,\text{nm}^2$, $I_t = 0.09\,\text{nA}$, $U_t = 400\,\text{mV}$, filled surface states; b — $20\times20\,\text{nm}^2$, $I_t = 0.07\,\text{nA}$, $U_t = -600\,\text{mV}$, empty surface states; c — $25\times24\,\text{nm}^2$, $I_t = 0.08\,\text{nA}$, $U_t = 500\,\text{mV}$, filled surface states.

both (112) and (113) facet planes proposed in Refs. [1,12]. The difference in the step facet orientation can also be seen from the step widths in 3D-images of Figs 3, b and 3, c. We suppose that predominance of one or another step face can be determined by a periodicity of hill and valley structure — step bunching and terrace widening lead to changing of the step facet plane.

Basing on STM and LEED data obtained in RIBER and STM GPI-300 chamber, during photoemission experiments we used the procedure with flash heating to 1250°C, slow cooling to 1050°C, fast cooling to 850°C and 20 s annealing at 850°C for the clean surface preparation. According to our STM data, these procedures should provide terraced structures with periodicities of 5.7 and 11.4 nm on Si(557) and Si(556) surfaces, respectively. We believe that LEED data obtained on Si(556)-(7×7) surface which were similar to Si(557)-(7×7) with double terrace width support this suggestion.

Valence band (VB) of PE spectra taken from Si(111), Si(556) and Si(557) surfaces presented in Fig. 4, a demonstrate common features S1–S3 due to the presence of Si(111) oriented terraces on stepped surfaces. PE spectra taken from Si(556) with wider terraces show closer resemblance to spectra of Si(111). The VB spectra from stepped surface with narrower terraces (Si(557)) do not reveal as sharp surface state features as Si(111) VB spectra. One can see that smashing of VB peaks due to breaking periodicity on stepped structures is more distinct for features appearing in photoemission spectra at 0.15 and 1.8 eV binding energy relative to Fermi level.

Core level Si $2p$ spectra of Si(hhm) and Si(111) surfaces were measured at photon energies $h\nu = 110$ and 130 eV corresponding to dominant electron emission from bulk and surface states, respectively. PE spectra of Si(111) taken in surface-sensitive regime demonstrate clearly seen features corresponding to fine Si $2p$ structure. This fine structure is related to different atom positions on (7×7) reconstructed surface (namely, to adatoms, dimer atoms, atoms bound to adatoms, rest atoms). The spectrum of Si $2p$ core level taken from Si(111) (Fig. 4, c), possibly, is not so fine in details but rather similar to high resolution Si $2p$ spectra published in Ref. [13]. The fine structure of Si $2p$ core level spectrum in our experiments is not so clear for Si(556) stepped surface and it is almost absent for Si(557) PE spectra. Besides, one can see a shift of both bulk (Fig. 4, b) and surface-sensitive (Fig. 4, c) core level spectra of Si(557). As it can be seen, both Si $2p$ spectra measured from Si(557) are approximately 0.05 eV shifted to lower binding energies relative to spectra taken from Si(111). However, to determine fine Si(hhm) core level structure and step atom features more detailed analysis of spectral features with applying multi-peak synthesis is required.

In conclusion, we have studied atomic and electronic structures of clean ordered vicinal Si(hhm) surfaces prepared by direct current annealing with special thermal treatment procedure. The periodicity of the stepped

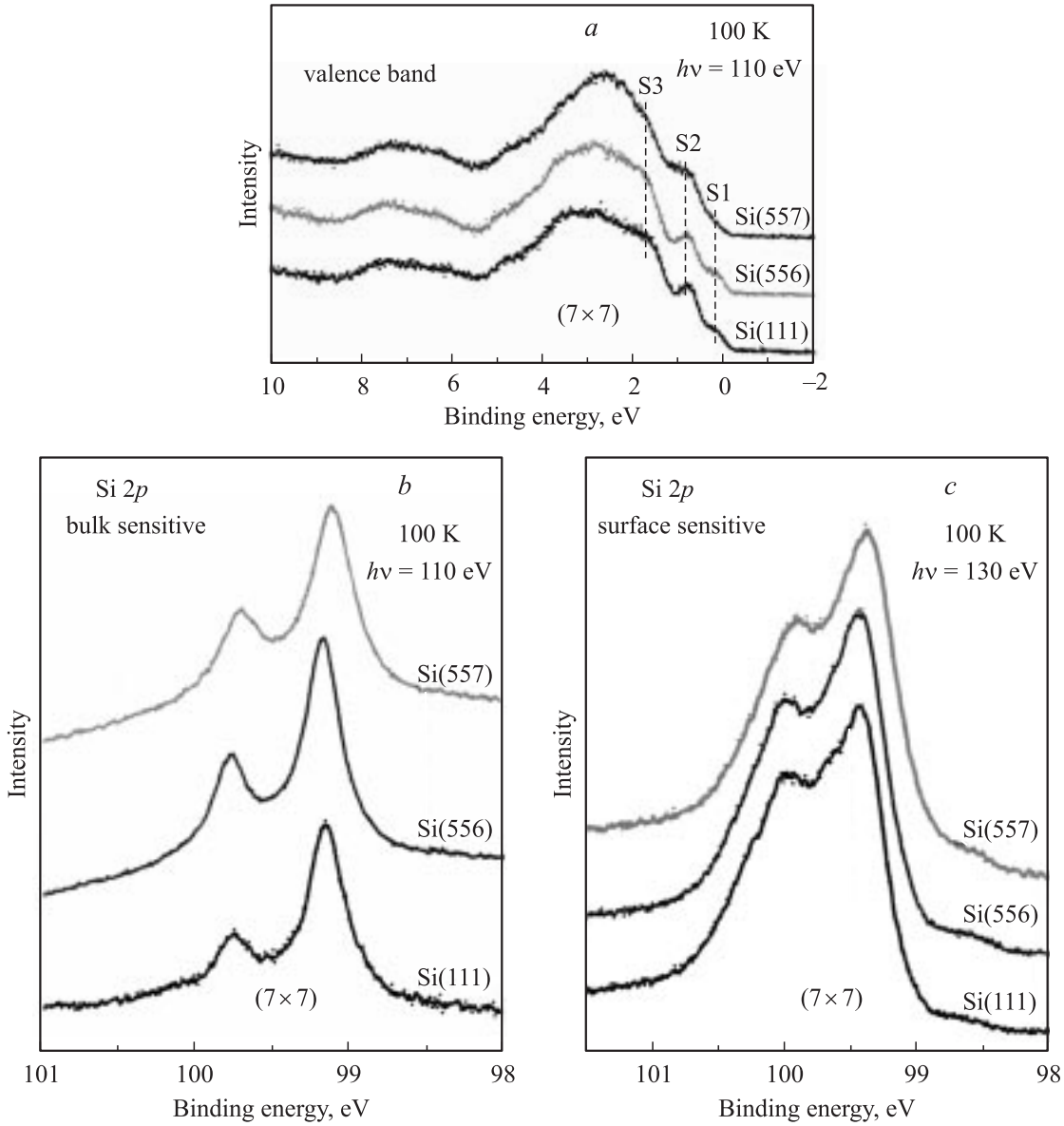

Figure 4. Si(111), Si(556) and Si(557) normal emission valence band (*a*) and Si 2*p* core level photoemission spectra measured in bulk- (*b*) and surface- (*c*) sensitive modes.

structures based on Si(557) is determined by the annealing procedure that may induce formation of regular atomically ordered arrays with different periodicity on Si(557) surface. The terrace and step atomic structures were resolved by room temperature STM. Atomically resolved STM images of Si(557) demonstrate triple steps with different facet orientations for surfaces with different terrace widths. High resolution valence band and Si 2*p* core level spectra were measured on clean stepped surfaces Si(557), Si(556) and compared with spectra taken from clean Si(111).

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