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Low temperature formation of Si(1 1 1)-$(2n+1) \times (2n+1)$ surface reconstructions

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Abstract

Scanning tunneling microscopy was used to study the surface morphology changes of the H-terminated Si(1 1 1) surface during Ag deposition at elevated temperatures. Flat H-terminated Si(1 1 1) surfaces were prepared by NH_4F etching. Domains of various dimer-adatom-stacking-fault superstructures, such as 3×3, 5×5, 7×7, and 9×9 were observed on the Si(1 1 1) surface after *hot* deposition of 1 ML Ag at 550 °C. This phenomenon was compared with hydrogen thermal desorption experiments, which did not show the formation of metastable surface superstructures at temperatures near 550 °C. All metastable superstructures obtained after hot Ag deposition at 550 °C were found to convert into the 7×7 reconstruction after annealing at 600 °C.

Keywords: Silicon; Surface relaxation and reconstruction; Surface structure, morphology, roughness, and topography; Silver; Scanning tunneling microscopy

1. Introduction

Since the first discovery of the 7×7 reconstruction of the bare Si(1 1 1) surface [1] the analysis and determination of surface reconstructions on Si surfaces have been one of the central issues in surface science.

The 7×7 reconstruction is known to be the most stable one on the bare Si(1 1 1) surface at low temperatures [2,3]. The dimer-adatom-stacking-fault (DAS) model [4] proposed for the Si(1 1 1)-$(2n+1) \times (2n+1)$ ($n = 1$–5) structures was proven by several surface analysis methods, such as scanning tunneling microscopy (STM) [5] and low energy electron diffraction (LEED) [1,6]. Normally the 7×7 reconstruction is obtained by thermal annealing of a Si(1 1 1) substrate above 1000 °C under ultra-high-vacuum (UHV) conditions and subsequent cooling to ambient temperature. By slow cooling, the high temperature 1×1 structure is transformed at approximately 860 °C into the thermodynamically more stable 7×7 low temperature surface structure of Si(1 1 1) [3,6].

Besides the equilibrium 7×7 reconstruction a variety of other surface reconstructions was observed on the Si(1 1 1) surface [7–9]. Cleaving of Si(1 1 1) single crystals in UHV results in the formation of a metastable 2×1 surface, which forms due to kinetic limitations at room temperature. Thermal annealing of the 2×1 metastable

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structure below 400 °C leads to the formation of the equilibrium 7×7 reconstruction [7]. Also intermediate 5×5 reconstructions were observed during the (2×1) to (7×7) transition [8]. Some other metastable Si(1 1 1) surface structures can be obtained by a combination of laser and thermal annealing of the 7×7 surface. After Q-switched laser annealing of the equilibrium 7×7 reconstruction the non-DAS structures c-(2×4) and 2×2 were obtained whereas subsequent thermal annealing of these surfaces leads to different DAS structures, such as 5×5, 7×7, and 9×9 [9].

Electrons of moderate energies (90–2000 eV) previously believed not to influence the surface can actually induce structure modifications on the Si surface [10]. The thermodynamically equilibrium Si(1 1 1)-(7×7) surface can be achieved by synchrotron radiation illumination at temperatures significantly lower than that necessary for standard thermal annealing procedure [11].

The formation of different surface superstructures was observed during thermal H-desorption of a hydrogen-terminated Si(1 1 1) surface prepared by hydrogen exposure of the Si(1 1 1)-(7×7) structure in an UHV environment at elevated temperature (dry processed surface) [12,13]. After thermal desorption of hydrogen from this dry processed surface and annealing at around 500 °C [11] the Si(1 1 1)-(7×7) superstructure was observed by STM, which is below 860 °C for the (7×7) to (1×1) transition [14]. Also non-DAS surface reconstructions, such as 2×2, 2×4, and $\sqrt{3} \times \sqrt{3}$ were observed after 500 °C annealing of dry processed H/Si(1 1 1) surfaces [13].

Recently the H/Si(1 1 1) surfaces prepared by chemical etching in HF and NH_4F aqueous solutions (*wet processed surface*) were extensively studied [15]. The UHV H-desorption experiments on the H/Si(1 1 1)-(1×1) surface showed that after annealing at temperatures of 450–500 °C different metastable structures can be obtained as non-DAS c-(2×4), 2×2, and $\sqrt{3} \times \sqrt{3}$ superstructures as well as the DAS-family of 5×5, 7×7, 9×9 and 11×11 reconstructions [16]. By further annealing at 700 °C all meta-stable structures transform into the stable Si(1 1 1)-(7×7) reconstruction [16]. There are reports that 7×7 reconstructions acquired by H-desorption from the wet processed H/Si(1 1 1) were also observed by RHEED at 550 °C [17].

Metal induced surface reconstructions of the Si(1 1 1) surface, where the deposition of metals supports the formation of different superstructures, are also known [18–21]. In case of Ag deposition on the Si(1 1 1) surface several reconstructions were reported to appear depending on the deposition (or annealing) temperature and on the coverage of Ag, i.e. $\sqrt{3} \times \sqrt{3}$, 6×1, 3×1, and 5×2 [19]. Thermal desorption of Ag from the Ag/Si(1 1 1)-$(\sqrt{3} \times \sqrt{3})$ surface, which starts around 550 °C, results in the recovery of the 7×7 structure at temperatures below 860 °C.

In this paper the role of Ag deposition at elevated temperatures (''*hot*'' Ag deposition) on the formation of Si(1 1 1)-$(2n+1) \times (2n+1)$ reconstructions will be discussed and compared with the results of the reconstruction formation on a H/Si(1 1 1) surface after H-desorption by thermal annealing. Also the influence of Ag deposited at lower temperature $T = 250$ °C (''*cold*'' Ag deposition) on the ''wet prepared'' H/Si(1 1 1) surface is compared with the hot Ag deposition at 550 °C on H/Si(1 1 1).

2. Experimental

Thin film deposition by molecular beam epitaxy and in situ characterization by STM were carried out using an Omicron Surface Science UHV System with a base pressure of 8×10^{-11} mbar. The substrates were of rectangular shape ($10 \times 3 \times 0.4$ mm^3) cut from a B-doped p-type Si(1 1 1) wafer (4.5 Ω cm) within an out-of-plane misorientation angle of $\pm 0.5°$. The sample cleaning procedure and hydrogen (H) termination was carried out in accordance to the procedure described elsewhere [22]. This preparation procedure is known to ensure an ideal mono-hydride terminated Si(1 1 1) surface [23]. After the preparation process the samples were brought into a load-lock chamber, pumped down by an oil-free prepump and a turbomolecular pump within 30 min. The samples were introduced into the growth/analysis chamber after the pressure in the load-lock reached 1×10^{-7} mbar.

The H/Si(1 1 1) samples were heated using a radiative heater placed behind the sample holder [1] while keeping the pressure below 5×10^{-9} mbar. The temperature was monitored by a Chromel–Alumel thermocouple located between the heater and the sample holder. The absolute accuracy of temperature measurements was ±20 °C. After each heating process the samples were slowly cooled down to room temperature (cooling speed was less than 0.5 °C/s).

Silver (Ag) was deposited from a tungsten crucible heated by electron bombardment with a deposition rate of 1 monolayer (ML)/min.

All STM measurements were carried out at room temperature using electrochemically etched polycrystalline tungsten tips cleaned in UHV by Ar^+ sputtering. The STM images presented were performed in the constant-current-mode. For all images the values of tunneling current and sample bias are given in the figure captions.

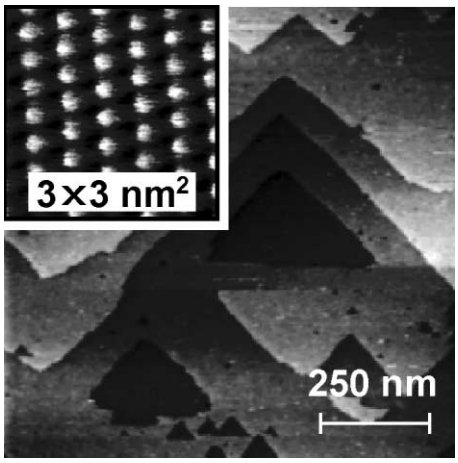

Fig. 1. STM image of large area triangle-shaped wet processed H/Si(1 1 1) terraces. The image was acquired with a tunneling current (I_T) of 0.12 nA and a tunneling voltage (U_T) + 0.24 V sample bias. The inset shows that the mono-hydride termination leads to a H/Si(1 1 1)-(1 × 1) surface ($I_T = 0.12$ nA; $U_T = +2.08$ V).

3. Results and discussion

After the introduction of H/Si(1 1 1) samples into the UHV the quality of the surface was checked by STM. Fig. 1 shows the STM image of a wet processed Si(1 1 1) surface. The typically triangle-shaped terraces are atomically flat and extended over hundreds of nanometers with no evidence of significant contamination. The height of the steps in Fig. 1 are 3.1 Å ± 0.1 Å. An atomically resolved image of the H/Si(1 1 1) surface shown in the inset of Fig. 1 confirms the mono-hydride termination of the terraces.

Fig. 2(a) shows a STM image of the H/Si(1 1 1)-(1 × 1) surface after annealing at 150 °C for 10 min. The 1 × 1 atomic arrangement of the hydrogen atoms is clearly seen and no significant change of the surface morphology after the annealing process has been observed. The step height of the terraces of 3.1 Å ± 0.1 Å is the same as before annealing (see also Fig. 1).

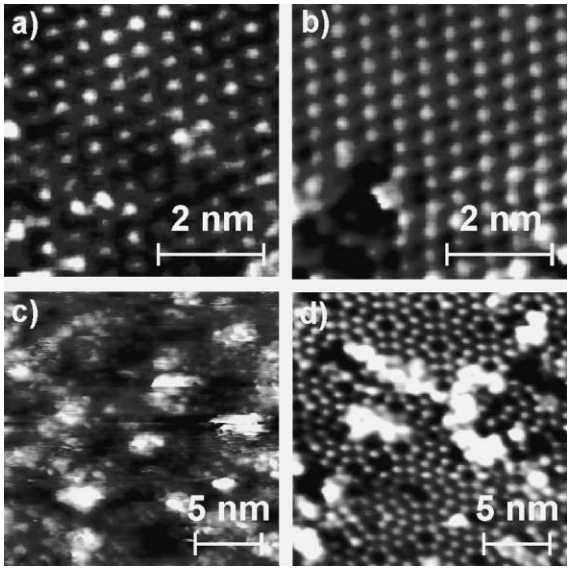

Fig. 2. STM image of H/Si(1 1 1)-(1 × 1) surface (a) after annealing at 150 °C for 10 min, (b) after annealing at 300 °C for 15 min, (c) after annealing at 550 °C for 70 min, and (d) converted into the Si(1 1 1) 7 × 7 reconstruction after annealing at 650 °C for 60 min ($I_T = 0.12$ nA; $U_T = +2.08$ V).

After 15 min of additional annealing at 300 °C of the same sample the terrace shape and also the

[1] This heating procedure differs from direct resistive heating of the sample, which may give rise to reconstruction or electromigration effects (see: [24]).

step height did not change, but a larger number of holes of different shape compared with the initial triangular-shaped etch-pits and irregular-shaped clusters appears on the surface (Fig. 2(b)). The depth of the pits and the height of the clusters are the same as the ones on the H/Si(1 1 1) surface (around 3 Å) before annealing. The clusters were atomically resolved and exhibit again a 1×1 structure (not shown). It can be seen in Fig. 2(b) that the 1×1 structure is still well arranged without any perturbation even if there exist irregular-shaped pits and clusters. New pits might be formed by desorption of dihydride and trihydride from the surface followed by the H desorption around these defects.

Fig. 2(c) shows the topography of the H/Si(1 1 1) surface after annealing at 550 °C for 70 min. The shapes of the steps and the terraces are preserved (not shown) but the atomic arrangement changed remarkably and it seems that there is no long range order as observed on the H/Si(1 1 1)-(1×1) surface. The clusters with a 1×1 structure disappear from which one can conclude that the clusters were neither contaminations (hydrocarbonates, carbides or metals) nor clusters of silicon oxide which can be normally desorbed from the surface only at temperatures significantly higher than 550 °C. Thus they consist of silicon atoms which is consistent with the results of Komeda et al. [16]. The structure becomes irregular and the surface looks very rough, but the rms roughness was almost the same as for the annealed surfaces at 150 and 300 °C shown in Fig. 2(a) and (b), respectively.

After 1 h of additional annealing at 650 °C the surface was converted into an almost perfect 7×7 reconstruction (see Fig. 2(d)). The transformation temperature (annealing temperature) is significantly higher than in the case of hot Ag deposition as will be discussed in the next section. One can observe relatively high clusters (maximum height 3.0 nm) on top of a well defined 7×7 reconstructed surface, which are supposed to consist of silicon atoms. These clusters are spread uniformly over the Si(1 1 1)-(7×7) surface. Although there exist some defects in the 7×7 structure, the images show the same structure as obtained by the methods discussed in the introduction [3,6,14,16]. The silicon clusters present might be the reason why no clear 7×7 diffraction patterns of this sample have been resolved using LEED and RHEED techniques.

In the following part the influence of the hot ($T = 550$ °C) and cold ($T = 250$ °C) Ag deposition on the surface structure of the Si(1 1 1) surface will be discussed.

The wet prepared H/Si(1 1 1)-(1×1) substrate was heated up to 550 °C and annealed for 40 min. Annealing at this temperature should lead to a complete desorption of the hydrogen from the H–Si(1 1 1) surface. Then 1 ML of Ag was deposited at 550 °C (further on this will be called hot Ag deposition). After deposition the temperature was maintained at 550 °C for 30 min and then the sample was cooled down to room temperature. Fig. 3 shows the STM image of a Si(1 1 1) surface after hydrogen desorption followed by the hot deposition of Ag.

Fig. 3 demonstrates the coexistence of different DAS structures with the periodicity of $(2n + 1) \times (2n + 1)$ [4] that can be found on the Si(1 1 1) surface such as Si(1 1 1) 3×3, 5×5, 7×7, and 9×9 reconstructions (magnifications are shown in Fig. 4(a)–(d)). An Ag/Si(1 1 1)-(3×1)

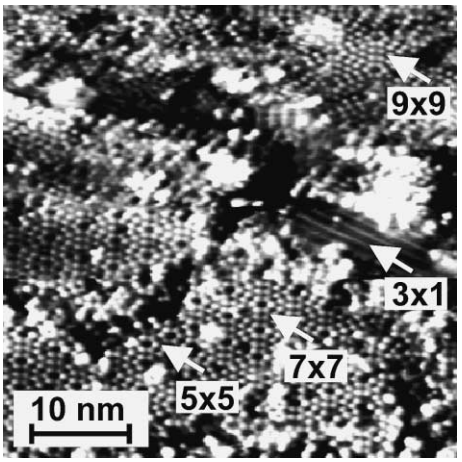

Fig. 3. STM image of a Si(1 1 1) surface after hydrogen desorption from wet prepared H/Si(1 1 1)-(1×1) sample at 550 °C followed by the deposition of 1 ML Ag at the same temperature ($I_T = 0.15$ nA; $U_T = +2.08$ V). The present Ag/Si(1 1 1)-(3×1) and Si(1 1 1)-$((2n + 1) \times (2n + 1))$ reconstructions are marked with arrows.

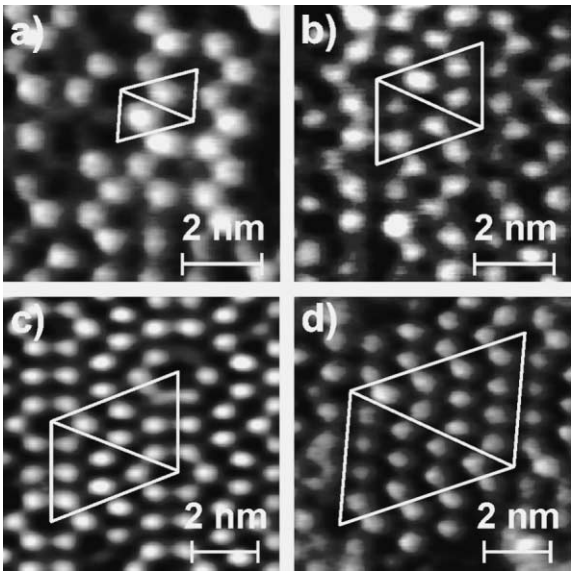

Fig. 4. Magnifications of observed Si(1 1 1)-($(2n+1)\times(2n+1)$) reconstructions after deposition of 1 ML Ag at 550 °C on the Si(1 1 1) surface (hot Ag deposition): (a) Si(1 1 1)-(3 × 3), (b) Si(1 1 1)-(5 × 5), (c) Si(1 1 1)-(7 × 7), and (d) Si(1 1 1)-(9 × 9) ($I_T = 0.12$ nA; $U_T = +2.08$ V).

reconstruction [19] is also present on the surface. No areas of Ag/Si(1 1 1)-($\sqrt{3}\times\sqrt{3}$) were observed on the sample may be due to the low coverage, i.e., 1 ML of Ag. For another sample, where 3 ML of Ag were deposited under the same condition, areas of Ag/Si(1 1 1)-($\sqrt{3}\times\sqrt{3}$) were also found.

The formation of the Ag/Si(1 1 1)-($\sqrt{3}\times\sqrt{3}$) phase is known to involve a substantial redistribution within the top Si(1 1 1) substrate layer (i.e. surface Si mass transport) [25]. Si transport as evidenced by a Ag–Si interaction [26–28] can be attributed to the enhancement of the surface migration of Si atoms under Ag irradiation at elevated temperature. During the Ag evaporation onto the Si(1 1 1) substrate at 550 °C Si atoms may be more mobile than without Ag atoms flux.

The disordered high temperature "1 × 1" structure on the Si(1 1 1) surface is considered to be energetically closer to the $\sqrt{3}\times\sqrt{3}$ reconstruction, because this disordered structure contains Si adtoms on the surface as suggested by Yang et al. [29]. According to theoretical total energy calculations the difference of total energy between ideal 1 × 1 and 7 × 7 structures [30] and between ideal 1 × 1 and $\sqrt{3}\times\sqrt{3}$ structure [31] are 0.4 and 0.3 eV per 1 × 1 unit cell, respectively. The Ag on the Si(1 1 1)-(1 × 1) surface may induce the Si adatom formation which supports the formation of the DAS structures.

It is known that the Ag/Si(1 1 1)-($\sqrt{3}\times\sqrt{3}$) phase can be formed by Ag deposition on the 7 × 7 surface at temperatures in the range of 220–550 °C [32,33]. Around 550 °C Ag starts to desorb from the Ag/Si(1 1 1)-($\sqrt{3}\times\sqrt{3}$) surface. As Ag desorbs from the surface, besides the $\sqrt{3}\times\sqrt{3}$ structure generally also 3 × 1 and 6 × 1 phases appear depending on how much Ag is removed from the surface [19]. That means that the observed 3 × 1 reconstruction (Fig. 3) gives clear evidence of the existence of Ag on the sample surface. During the formation of 3 × 1 reconstruction areas the Si atoms are displaced by Ag and this may also be the source of the Si adatoms.

After annealing this sample with a nominal coverage of 1 ML Ag on the surface at a temperature of 600 °C for 60 min Ag was desorbed and all the observed superstructures were converted into the almost perfect 7 × 7 reconstruction. The images were obtained in *pseudo* constant-height STM mode (high scan speed, feedback loop set to minimum) with a sample bias −2.51 V showing the difference of surface electronic states between the stacking faulted (SF) regions and stacking unfaulted (UF) regions in the 7 × 7 unit cell (not shown here). This proves that the obtained images show real 7 × 7 reconstruction. The annealing temperature of 600 °C is far below 860 °C for the (1 × 1) to (7 × 7) transition and is also below the temperature of the 7 × 7 structure formation observed for thermal hydrogen desorption and annealing experiments at 700 °C on the wet-processed H/Si(1 1 1) surface [16]. The possibility of the 7 × 7 structure formation at temperatures lower than 700 °C was also shown by RHEED in the thermal H-desorption experiments on the wet processed H/Si(1 1 1) surface [17] which is consistent with direct STM imaging shown in this paper.

After deposition of nominally 3 ML Ag onto a wet processed H/Si(1 1 1) surface at a temperature of only $T = 250$ °C (cold Ag deposition), the sample was annealed at 550 °C for 40 min as well as at 700 °C for 80 min. After each annealing

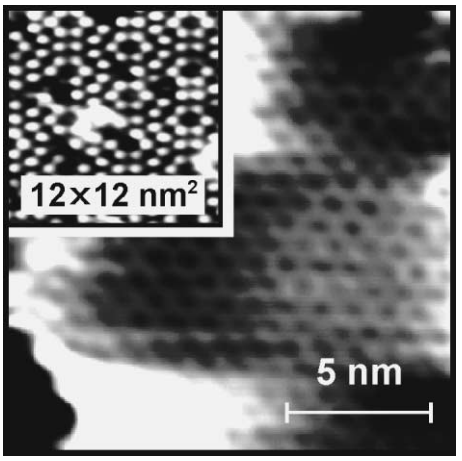

Fig. 5. STM image of the Si(1 1 1) surface (Ag/Si(1 1 1)-($\sqrt{3} \times \sqrt{3}$)) after the deposition of 3 ML Ag at $T = 250°$ C (cold Ag deposition) and an annealing step at 550 °C for 40 min ($I_T = 0.35$ nA; $U_T = +2.08$ V). The inset shows the same surface after additional annealing at $T = 700$ °C (for 80 min) showing a clear Si(1 1 1) 7×7 reconstruction ($I_T = 0.12$ nA; $U_T = +2.51$ V).

experiment the sample was cooled down to room temperature and the surface topography was checked by STM. At a temperature of around 550 °C after the desorption of hydrogen from the H/Si(1 1 1) surface the Ag forms a Ag/Si(1 1 1)-($\sqrt{3} \times \sqrt{3}$) reconstruction as shown in Fig. 5. [2] After additional annealing at a temperature of approximately 700 °C for 80 min Ag desorbs from the Ag/Si(1 1 1)-($\sqrt{3} \times \sqrt{3}$) surface and a transition towards the 7×7 reconstruction occurs. After performing this annealing procedure with a total annealing time of 120 min a clear 7×7 reconstruction could be seen (inset Fig. 5). The DAS structure was confirmed by resolving SF and UF regions of the 7×7 unit cell by STM with a sample bias -2.51 V (not shown here). No evidence of other DAS structures such as 3×3, 5×5, and 9×9 has been found. These measurements imply that the hot Ag deposition promotes the formation of meta-stable Si(1 1 1)-$(2n + 1) \times (2n + 1)$ reconstructions.

[2] In this case we deposited nominally 3 ML of Ag, therefore only some part of deposited Ag participates in the formation of the Ag/Si(1 1 1)-($\sqrt{3} \times \sqrt{3}$) phase and the remaining Ag would be clustered on the Ag/Si(1 1 1)-($\sqrt{3} \times \sqrt{3}$) surface.

4. Conclusion

In conclusion, surface morphology changes of the wet processed H-terminated Si(1 1 1) surface during Ag deposition at elevated temperatures were studied using STM. Various DAS superstructures as 3×3, 5×5, 7×7, and 9×9 were observed on the Si(1 1 1) surface after the deposition of 1 ML Ag at a substrate temperature of 550 °C. All the metastable structures convert into the 7×7 reconstruction after additional annealing at 600 °C. Thermal hydrogen desorption experiments of the pure H-terminated Si(1 1 1) surface did not show a significant formation of DAS structures at temperatures near 550 °C. After Ag deposition at a substrate temperature of $T = 250$ °C followed by annealing at 550 °C also no metastable DAS structures were found on the surface. Therefore, the Ag deposition at 550 °C (hot Ag deposition) promotes the formation of metastable Si(1 1 1)-$(2n + 1) \times (2n + 1)$ reconstructions.

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