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Real-time low-energy electron microscopy study of Ga adsorption and facet array formation on Si(113)*

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The evolution of Ga adsorption on a Si(113) surface at sample temperatures between 460°C and 500°C has been investigated using in-situ low-energy electron microscopy and low-energy electron diffraction. The formation of surface facets in the $[1\bar{1}0]$ direction was observed. From series of low-energy electron diffraction images at different electron energies, reciprocal space maps in different $k_\perp$-$k_\parallel$ planes were obtained. This allowed to determine both facet angles to be 9.8° ± 1.0° and thus the facet orientations to be (112) and (115). With low-energy electron microscopy, a well ordered array of nanofacets with about 40 nm periodicity in the $[33\bar{2}]$ direction was observed.

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I. INTRODUCTION

High-index Si and Ge surfaces often have a particular tendency to facetting after adsorption of group III and group V materials [1, 2]. Intentional and regular facet formation could be an appealing approach to realize self-assembled nanostructures, like nanowires or nano-dots. Si(113) is one of the thermally stable high-index surfaces of Si. The formation of rotational domains during growth is prevented by the lack of rotational symmetry. Self-organized growth of Ge nanowires on Si(113) was already observed [3–5]. Ga is a well known surfactant material, the adsorption of which leads to the formation of nanopatterns on Si(111) [6–8].

It has been shown recently [9] that Ga adsorption on Si(113) can induce surface facetting. Also the orientation of the facets has been determined using scanning tunneling microscopy and reflection high-energy electron diffraction [9]. In this contribution we present an in-situ low-energy electron microscopy (LEEM) and low-energy electron diffraction (LEED) study, providing a more detailed insight into the evolution of the surface reconstruction during Ga adsorption. Moreover, we will show that a periodic arrangement of nanofacets can be achieved.

II. EXPERIMENTAL

The sample preparation and the in-situ measurements were performed in an ultra-high vacuum system equipped with a spectroscopic photoemission and low-energy electron microscope (SPELEEM) [10] at the undulator beamline 1.2 [11] at ELETTRA, Italy.

Heating of the Si(113) substrates was carried out by electron beam bombardment from the back side of the substrates, while the temperature was monitored with a thermocouple attached to the sample holder. A clean Si(113)-(3×2) reconstruction of the surface was achieved after several short flashes up to 1200°C, after degassing the Si(113) substrates for at least 12 hours at 600°C.

Ga was evaporated from an electron beam effusion cell at different sample temperatures, whereas the deposition rate was kept fixed at about 0.4 ML/min. The deposition was monitored in-situ with either LEED or LEEM using the (00) beam to obtain bright-field LEEM images. The Ga deposits were determined from a flux control monitor attached to the evaporator, which was calibrated in a deposition experiment performed at 460°C. A value of 1 ML Ga was assigned to the complete evolution of the (2×2) reconstruction. For measurements after Ga deposition, the substrate temperature was slightly reduced to prevent ripening effects.

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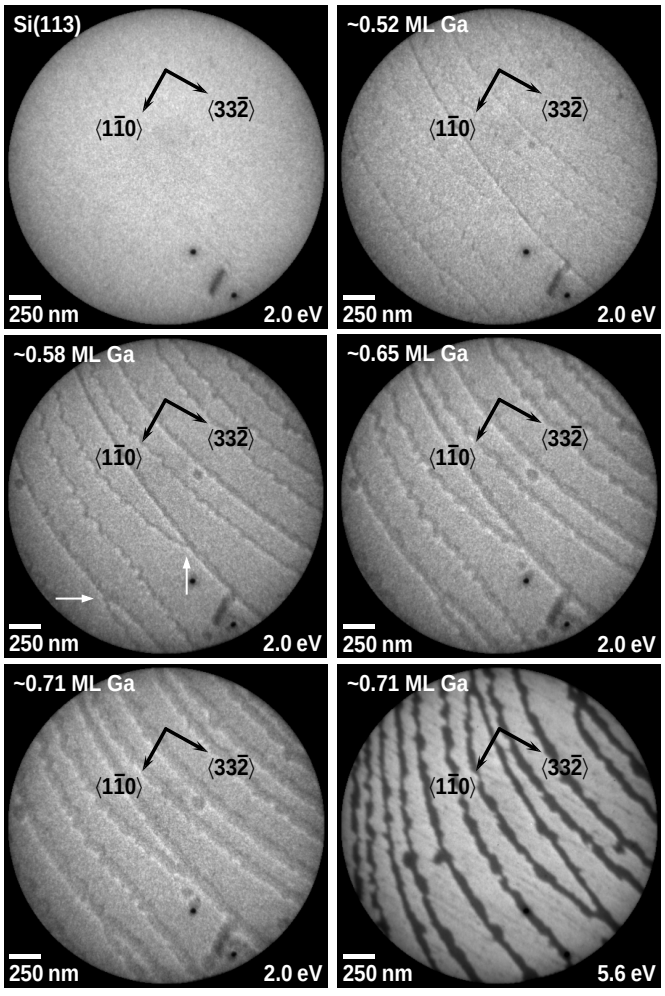

FIG. 1: Bright-field LEEM images of Si(113) during Ga deposition at 490°C. Step edges run from the top left to the bottom right. The electron energy is denoted at the bottom right of the images.

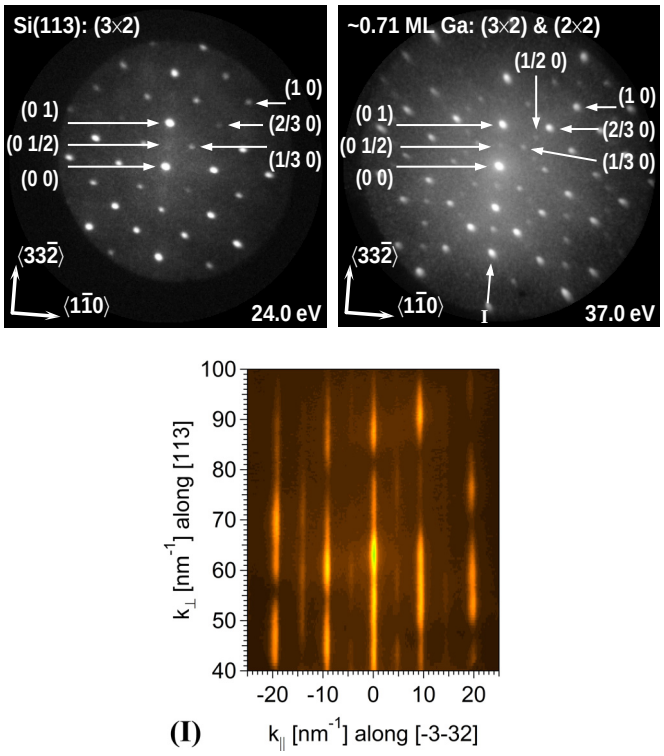

FIG. 2: Top row: LEED images of Si(113) before and after Ga deposition at 490°C. Bottom row: Reciprocal space map with $k_\parallel$ in the [33$\bar{2}$] direction through the (00) spot.

III. RESULTS AND DISCUSSION

A. Submonolayer Ga deposition

Figure 1 shows a series of bright-field LEEM images of the clean Si(113)-(3×2) surface and for subsequent Ga adsorption at a sample temperature of 490°C. In the LEEM image of the initial surface, step edges are faintly visible, running from the top left to the bottom right. The average terrace width amounts to approximately 250 nm. In several cases, step edges merge as indicated by the arrows in Fig. 1. This is indicative of step bunching, resulting in steps exhibiting heights of multiple monolayers.

During Ga deposition the LEEM images show a distinct change in contrast at the step edges. Hence we conclude that Ga-covered domains start to nucleate at the step edges. The change in contrast is explained by a change in surface reconstruction from the initial Si(113)-(3×2) structure to a (2×2) structure of these domains. Furthermore, with increasing Ga deposition the step edges appear in a more and more meandering shape. This is attributed to a change of the line energy of the step edges.

The above-mentioned change in surface reconstruction is also visible in LEED images taken before and after Ga deposition as shown in Fig. 2. Prior to Ga deposition, a (3×2) pattern is observed with weak and diffuse ($n\frac{1}{2}$) spots. After the Ga deposition, a (2×2) pattern can be found in addition to the (3×2) pattern of the Si(113) surface. A series of LEED images was taken at different electron energies to obtain reciprocal space maps in different $k_\perp$-$k_\parallel$ planes. Such a map with $k_\parallel$ oriented along the [33$\bar{2}$] direction through the (00) spot is shown in Fig. 2. Integer and half-order surface diffraction rods are clearly identified. Apart from these vertical rods no facet rods are visible. From similar maps, e.g. with $k_\parallel$ along [1$\bar{1}$0], the presence of facets on this surface can be excluded.

B. Ga saturation coverage

Figure 3 shows a series of bright-field LEEM images of the clean Si(113)-(3×2) surface and the Ga adsorption up to saturation coverage at a sample temperature of 460°C.

As laid out above, the Ga adsorption takes place at the step edges of the Si(113) surface, which run from the top left to the bottom right. With increasing Ga deposition loops are visible at the step edges (see image for about 0.52 ML Ga deposit). This may be explained by a local debunching of the steps due to Ga decoration. Also it is obvious that Ga (2×2) domains start to nucleate in the center of the terraces. Since this characteristics is observed only for wider terraces, this points to a kinetic limitation, with the Ga adatom diffusion length being in the range of about 100 nm. At around 1 ML Ga deposit the surface is completely covered with Ga, and the contrast of the bright-field LEEM images does not change

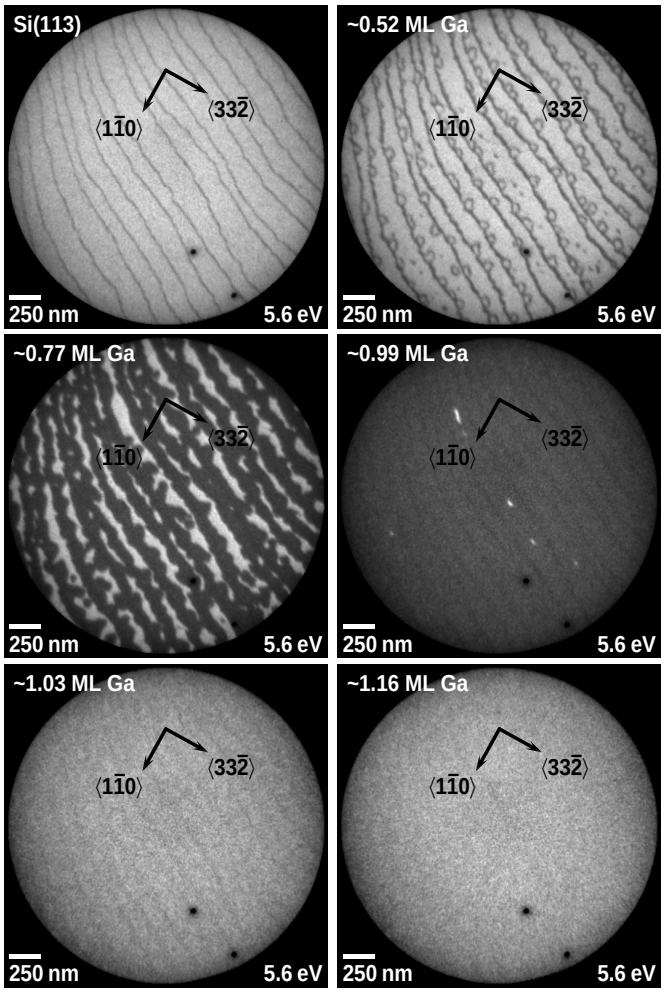

FIG. 3: Bright-field LEEM images of Si(113) during Ga deposition at 460°C. Step edges run from the top left to the bottom right.

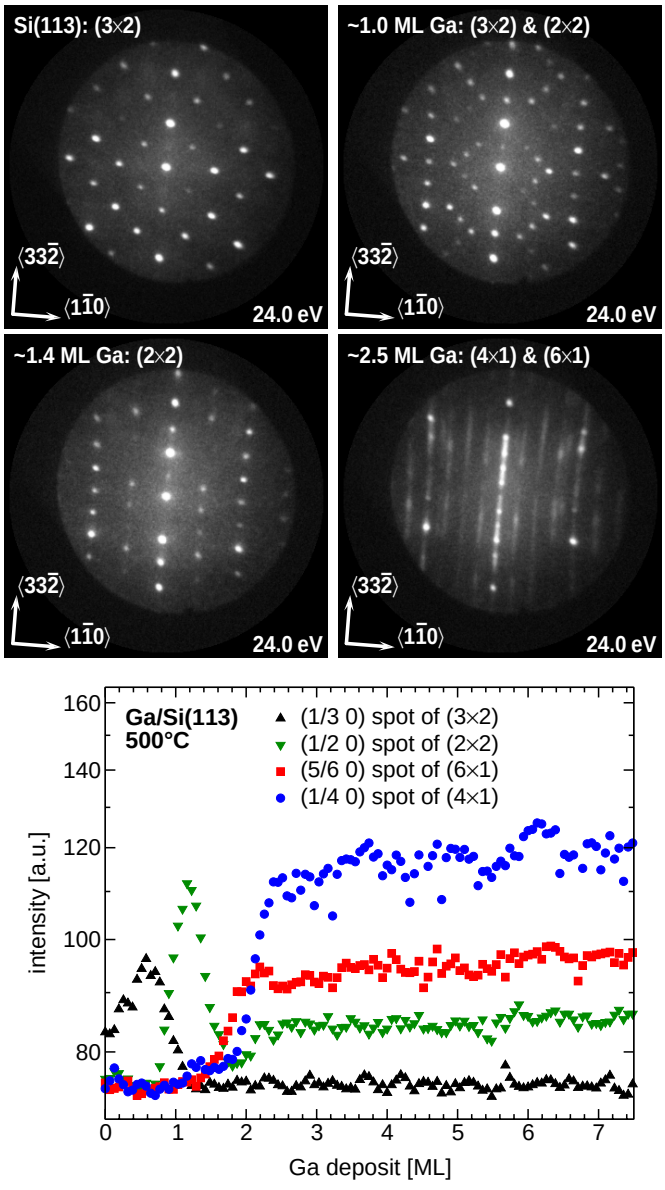

FIG. 4: LEED images of Si(113) and evolution of selected LEED spots during Ga deposition at 500°C.

upon further Ga deposition, except for the fading of contrast at step edges.

To obtain a complementary view of the evolution of the Si(113)-(3×2) surface reconstruction during Ga adsorption, in Fig. 4 a series of LEED images during Ga deposition at 500°C is given. The clean Si(113) surface at 500°C shows a (3×2) structure with faint ($n\frac{1}{2}$) spots, as the phase transition between the (3×2) and the (3×1) reconstruction takes place at 507°C [12]. At about 1 ML Ga deposit, a superposition of a (3×2) and (2×2) pattern is obtained, which is explained by the coexistence of bare Si(113)-(3×2) and Ga-covered (2×2) surface areas. In contrast to the situation at 460°C after the deposit of about 1 ML Ga (cf. Fig. 3), the surface is not completely covered by Ga, which points to a reduced sticking coefficient at 500°C. Upon further Ga deposition, only the (2×2) contribution to the LEED pattern remains (see image for about 1.4 ML). For even larger Ga deposits, a complex LEED pattern with 4-fold and 6-fold periodicity along [1$\bar{1}$0] evolves, as depicted in the image for about 2.5 ML Ga deposit in Fig. 4. As will be discussed below, this is due to the formation of (4×1) and (6×1) reconstructed facets. The evolution of the different surface reconstructions is illustrated by the viewgraph at the bottom of Fig. 4, where the intensities of the respective LEED spots are displayed as a function of Ga deposit.

In Fig. 5 a bright-field LEEM image, a LEED pattern and reciprocal space maps in different $k_\perp$-$k_\parallel$ planes are shown as obtained after deposition of about 9.8 ML Ga at 500°C. For these measurements the sample temperature was decreased to 350°C. Within all reciprocal space maps, no vertical but only inclined rods are observed. This points to a complete facetting of the whole surface.

For the reciprocal space map through the (00) spot, two types of facets are found, with opposite inclination with respect to the [113] direction. These two facet types are well separated when taking reciprocal space maps for non-vanishing $\vec{k}_\parallel$ components $k_\parallel^{1\bar{1}0}$ in [1$\bar{1}$0] direction, as shown for $k_\parallel^{1\bar{1}0} = \frac{1}{4}$ surface brillouin zones (SBZ) and $k_\parallel^{1\bar{1}0} = \frac{1}{6}$ SBZ, respectively. This proves that two different reconstructions with 4-fold and 6-fold periodicity along [1$\bar{1}$0] are associated with either facet type. Both facet types exhibit an inclination angle of 9.8° ± 1.0° with respect to the (113) surface, as directly

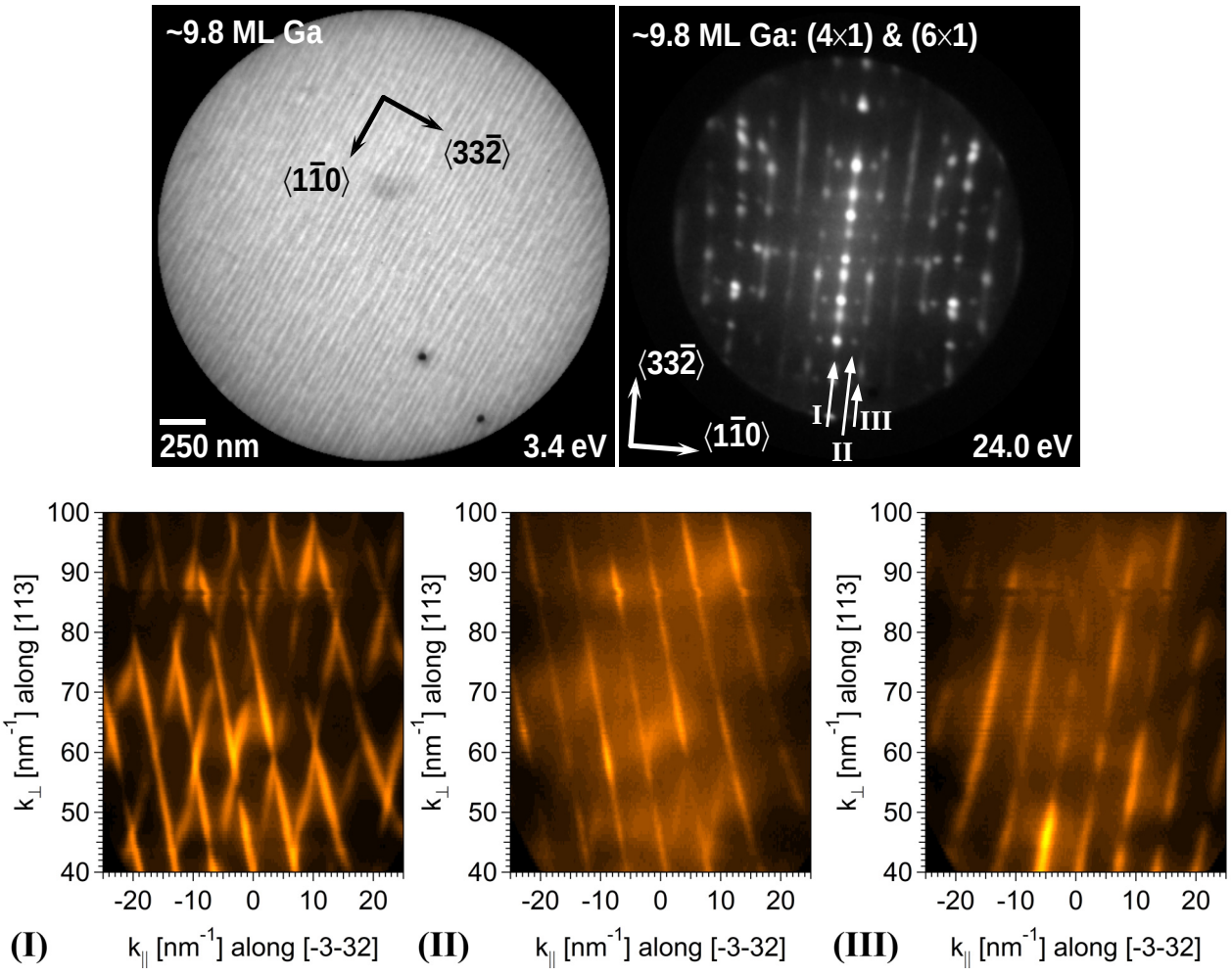

FIG. 5: Top row: Bright-field LEEM and LEED image of Si(113) after deposition of about 9.8 ML Ga at 500°C. Bottom row: Reciprocal space maps with $k_{\|}$ in the [33$\bar{2}$] direction through the (00) spot (I), the ($\frac{1}{6}n$) spots (II) and the ($\frac{1}{4}n$) spots (III). All measurements obtained at 350°C sample temperature.

determined from the reciprocal space maps. Hence, the facets can be identified to be (112) and (115), which are inclined by 10.0° and 9.4°, respectively. This is in agreement with a STM study by Suzuki et al. [9], who found (6×1) reconstructed (112) facets and (4×1) reconstructed (115) facets. These authors report a width of the facets of about 5 nm which leads to a periodicity along [33$\bar{2}$] of 10 nm. This, however, is distinct from our results. In the LEEM image displayed in Fig. 5, a bright and dark contrast pattern with stripes oriented along the [1$\bar{1}$0] direction is visible, which arises from the different surface orientations and reconstructions of the two facet types. This stripe pattern is well ordered and shows a periodicity of about 40 nm along [33$\bar{2}$] which is about four times larger than the value reported by Suzuki et al. [9]. As an explanation for this difference, we suggest that the periodicity length and the long range ordering depend on the preparation conditions. The very low deposition rates and long exposure times used here could allow for a promoted self-assembly of the nanostripe pattern. This process is going to be investigated in future experiments as it might offer a convenient way to tune the structural size of the nanopattern, e.g. for the subsequent selective growth of quantum wires.

IV. CONCLUSION

In conclusion, we have presented a combined real-space and reciprocal space analysis of the Ga adsorption on Si(113) using low-energy electron microscopy and low-energy electron diffraction. We observed the evolution of evenly spaced surface facets in [1$\bar{1}$0] direction with a periodicity of about 40 nm in [33$\bar{2}$] direction. From reciprocal space maps extracted from the LEED data, we determined both facet angles to be 9.8° ± 1.0° with respect to the [113] direction and thus identified the facet orientations as (112) and (115), respectively.

Acknowledgments

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