

## Experimental evidence for a stable GaAs surface near (113)

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GaAs surfaces vicinal to (113) with a continuous range of misorientation angles up to  $11.5^\circ$  in all azimuthal directions were created by grinding a spherical depression into (113) oriented samples. Thin homoepitaxial layers were grown onto these samples by molecular beam epitaxy (MBE), and the surfaces were *in situ* studied by low-energy electron diffraction (LEED) and scanning tunneling microscopy (STM). The surface quality in the depression was verified by reproducing LEED patterns of the (113) and (114) surfaces. A stable GaAs surface was found that is oriented from (113) by  $9^\circ \pm 2^\circ$  towards  $[1\bar{1}0]$ . STM and LEED images of this surface are presented.

In surface science and device fabrication it is often advantageous to use samples that are slightly misoriented from the nominal direction of a stable surface. On these vicinal surfaces a high density of steps is expected in a direction determined by the azimuthal direction of the miscut. The roughness and actual direction of the step edges and the possible occurrence of step bunching give an insight into the atomic structure of the steps. In addition, the influence of step edges on growth phenomena can be studied. For instance, growth on vicinal surfaces can be controlled to take place in step-flow growth mode rather than by island nucleation.<sup>1</sup> Thus, the resulting surface is smoother. This can be utilized to achieve high-quality interfaces in heteroepitaxial structures. Also, on regular arrays of steps self-organized low-dimensional structures such as quantum wires can be prepared.<sup>1,2</sup>

So far, only surfaces vicinal to low-index orientations have been examined. However, a particularly interesting subject of such a study is GaAs(113)A. This surface exhibits over a wide range of preparation conditions an  $(8 \times 1)$  reconstruction,<sup>3</sup> whose step structure is highly anisotropic:<sup>4</sup> While step edges along  $[3\bar{3}\bar{2}]$  are straight without any kinks for up to 2000 Å, step edges along  $[1\bar{1}0]$  are extremely rough. Compared to low-index surfaces, the surface roughness is fairly high and also anisotropic.<sup>5–8</sup> The reason for this anisotropic roughness on a mesoscopic scale is not yet known. The examination of surfaces vicinal to (113) could help on two issues: First, the very straight step edges along  $[3\bar{3}\bar{2}]$  could be utilized for the self-organized growth of quantum wires. Second, the origin of the mesoscopic surface roughness could be elucidated.

However, the experimental realization of such a study is not easy. *A priori* it is unclear what values of the misorientation and of the azimuthal angle would yield the best results. In addition, wafers with such orientations are not a standard product of semiconductor manufacturers and are thus very expensive. For these reasons we have developed a fairly low-cost technique to make a continuous range of vicinal surfaces accessible: A spherical depression is grinded into a nominally oriented sample. The quality of the created surfaces is verified by the observation of low-energy electron diffraction (LEED) patterns of known surfaces. Unexpectedly, in the

depression a stable surface of different orientation has been discovered. Although not directly in line with our original motivation, this finding is so important that it is the main topic of this paper.

Samples with a typical size of  $10 \times 10$  mm<sup>2</sup> were cut from GaAs(113)A wafers (*n* type, Si doped, carrier concentration  $2.5 \times 10^{18}$  cm<sup>-3</sup>, Laser Diode). The depression was created with a Precision Dimple Grinder (Gatan, model 656/3) whose original purpose is the thinning of transmission electron microscopy (TEM) samples. Consecutively, diamond polish with grain sizes of 1 μm and 0.25 μm was applied. Afterwards the samples were cleaned with ultrasound in distilled water, propanole, and acetone. Scanning electron microscopy (SEM) was used to check the condition of the surface and ensure that it was free of contaminations from the polish. At this point, scratches were observed whose width corresponded to the smaller grain size. Then the samples were introduced into a multichamber ultrahigh-vacuum system, which is described in detail elsewhere.<sup>9</sup> After oxide desorption samples were treated with several ion-bombardment and annealing (IBA) cycles. SEM experiments carried out after this step showed that IBA is an effective method to remove the scratches from the last polishing. Homoepitaxial layers 20–50 nm thick were grown by molecular beam epitaxy (MBE) at temperatures between 520 and 580 °C. The As<sub>2</sub>:Ga beam equivalent pressure ratio was 15. After growth, samples were cooled down to 480 °C while simultaneously the As pressure was reduced. At a base pressure of less than  $3 \times 10^{-9}$  mbar samples were transferred *in situ* to the analysis chambers. Surfaces were studied by LEED and by scanning tunneling microscopy (STM, Park Scientific Instruments, VP2). STM images were acquired in constant-current mode with tunneling currents between 0.1 and 0.2 nA and sample voltages between  $-2$  and  $-3$  V.

In Fig. 1 a schematic drawing of the depression is shown. The radius of curvature of the depression is 7.5 mm, its depth is 150 μm, and its radius is 3 mm. Thus, surfaces vicinal to (113) were created in all azimuthal directions with a continuous range of misorientation angles up to  $11.5^\circ$ . A misorientation in direction  $[3\bar{3}\bar{2}]$  yields the surfaces with the orientations  $(11n)$ . Regions with both the orientations (112) and

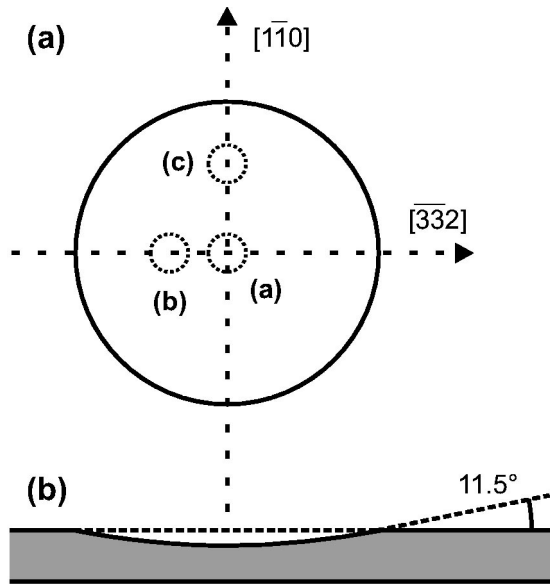


FIG. 1. Schematic drawing of the depression in a (113) oriented wafer. (a) Top view. The labels (a)–(c) mark the areas where the LEED images were acquired that are presented in the corresponding parts of Fig. 2. (b) Side view.

(114) should be present in the depression because the angles to (113) are  $10.0^\circ$  and  $5.8^\circ$ , respectively.

The first object of the experiments was to check if the surface quality in the depression is sufficiently good. For this purpose LEED images were taken when the electron beam was directed at different parts of the depression. This was achieved by moving the sample perpendicularly to the beam. In Fig. 2(a) such an image is presented that was acquired at the center of the depression. In this area the surface is oriented in the nominal [113] direction. Indeed, the  $(8 \times 1)$  pattern known<sup>4,10</sup> from GaAs(113) was observed. This image is essentially identical to ones that were taken on the sample surface next to the depression (not shown). For image (b) the electron beam was shifted in direction  $[33\bar{2}]$ . As expected, the pattern of the  $(114)A-c(2 \times 2)$  reconstruction<sup>11</sup> was found. The occurrence of double-crescent-shaped features (middle right-hand part of the image) is typical for this surface.<sup>12</sup> On the opposite side of the depression a LEED

pattern similar to that known from GaAs(112) was observed. However, as this surface is unstable and has facets,<sup>13,14</sup> the pattern is not so characteristic. Also, faceting is not necessarily an indication for a good surface quality. Altogether, the observation of LEED patterns of known, stable surfaces in the depression proves that the described series of grinding, IBA, and subsequent growth by MBE is an appropriate method to prepare vicinal surfaces of good quality. An interesting point about this method is that the grinding was carried out with a commercial device that was designed for a different purpose, and it may be available in many institutes. This is an advantage over other reports on spherical samples.<sup>15</sup>

When the electron beam was directed at locations along the line in the  $[1\bar{1}0]$  direction in Fig. 1(a), unexpectedly a hitherto unknown LEED pattern was observed. It is shown in Fig. 2(c). On the opposite side of the depression the same pattern was found but mirrored. This is due to the fact that the plane perpendicular to (113) along  $[33\bar{2}]$  is a mirror plane of the zinc-blende lattice. This LEED image is evidence that there is a stable GaAs surface that has been hitherto unknown. Compared to the images in directions  $[113]$  and  $[114]$ , the spots are sharper, and fewer are lost in the background. Thus, the surface quality is higher. This observation holds for all preparation conditions that were used and indicates that this surface is stable under a wide range of preparation conditions. LEED images acquired on several different samples were evaluated in order to determine the dimensions of the unit mesh. In real space, the lengths of the unit vectors are  $10.4 \pm 0.4 \text{ \AA}$  and  $18.4 \pm 0.6 \text{ \AA}$ , and the angle between them is  $82^\circ \pm 2^\circ$ .

In order to determine the orientation of this surface, the electron beam was directed at a dense series of areas along the line in direction  $[1\bar{1}0]$  in Fig. 1(a). Between two consecutive measurements the sample was moved by 0.2 mm. From this series of LEED images the projected positions of the surface (on both sides) and of the center of the depression were determined. However, since the diameter of the electron beam is ca. 1 mm, the result of this method cannot be very exact. The angle between the new surface and (113) was calculated to  $9^\circ \pm 2^\circ$ . The rather large error reflects the fact that adjacent images are almost identical. Besides this

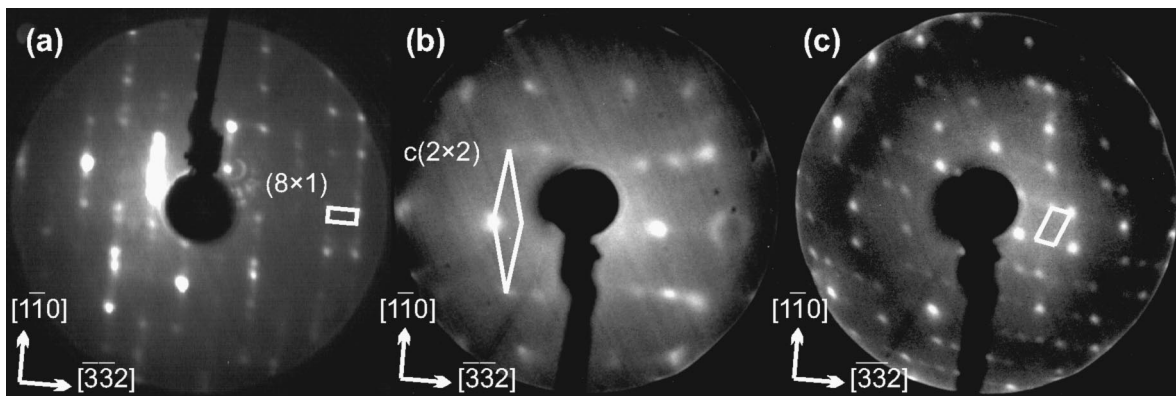


FIG. 2. LEED images acquired with the electron beam directed at different areas of the depression that are marked in Fig. 1(a). The indicated real space directions lie in the (113) plane. (a)  $(113)A-(8 \times 1)$ ;  $E = 50 \text{ eV}$ . (b)  $(114)A-c(2 \times 2)$ ;  $E = 43 \text{ eV}$ . (c) New surface;  $E = 93 \text{ eV}$ . ( $E$  is the electron energy).

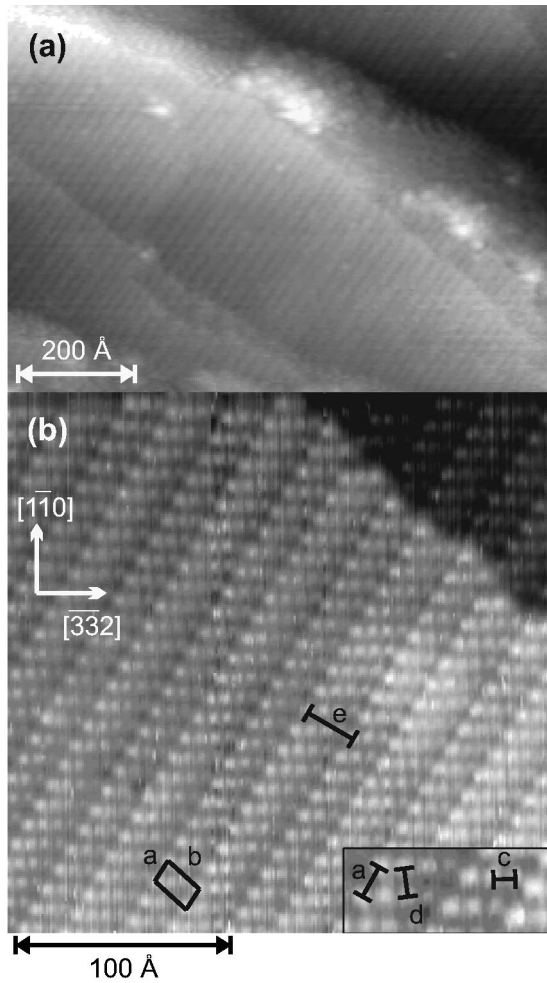


FIG. 3. (a) Overview STM image of the new surface.  $U_{\text{Sample}} = -2.5$  V,  $I = 0.1$  nA. (b) High-resolution STM image of the new surface, magnified part in inset. The lengths of the labeled lines are given in Table I.  $U_{\text{Sample}} = -3$  V,  $I = 0.2$  nA.

uncertainty in the  $[1\bar{1}0]$  direction, there is an additional one in the  $[33\bar{2}]$  direction. The area of the surface could in fact also lie not on but next to the line through the center that is marked in Fig. 1(a).

An overview STM image taken in the region of the surface is presented in Fig. 3(a). Several broad terraces are visible, which is the second piece of evidence that there is indeed a stable surface. In the lower part of the image the terraces are separated by single steps, and in the upper part there is a step bunch. On the terraces dark lines can be seen that run from the lower left to the upper right. The occurrence of step bunching indicates that this image was not acquired in the center of the region where the new surface is located. However, it also shows that the appearance of this surface is not limited to a very small angular range in the depression.

In Fig. 3(b) a smaller area is depicted with higher resolution. In the upper right-hand corner there is a single step down to a lower terrace. Both terraces are composed of microfacets whose edges run from the lower left to the upper right. These edges are visible in the overview image, too. Every microfacet is composed of series of either three or four humps roughly in the  $[33\bar{2}]$  direction. Each microfacet con-

TABLE I. Average values of the distances marked in Fig. 3(b) (in Å).

Labels in Fig. 3(b)				
$a$	$b$	$c$	$d$	$e$
$10.5 \pm 0.1$	$19.4 \pm 1.0$	$7.0 \pm 0.5$	$9.1 \pm 1.2$	$24.6 \pm 0.9$

tains only one type of series. The left-hand side of the microfacets is higher than the right-hand side, and the series of humps on adjacent microfacets are shifted with respect to each other in the  $[1\bar{1}0]$  direction. Since filled states are imaged, it is reasonable to assign the humps to As-related orbitals. However, without the knowledge of the bulk-truncated surface even a speculation about their nature (single atoms, dimers, or even trimers) seems not reasonable. Characteristic distances are marked in Fig. 3(b), and the corresponding values are summarized in Table I. The unit mesh  $a \times b$  that corresponds best to the data extracted from the LEED experiments is also indicated. Apparently, the series of four humps is not typical for the surface but is rather an element of disorder.

As in the LEED experiment, it is not possible to determine the exact location of the characterized area with respect to the center of the depression. Also, the STM tip can be moved in the surface plane over micrometer distances in only one direction. Thus, it is difficult to access in a well-defined way the regions of interest. Therefore, the exact orientation of the surface cannot be extracted from the STM data, either.

Although the exact orientation of the surface could not be determined from our experiments, comparison with other studies is enlightening. In fact there have been indications for a stable surface in this direction before. Platen and co-workers observed with LEED on the unstable GaAs(112)A surface facets with the orientation  $\{124\}$ .<sup>13,14</sup> Lee *et al.* examined the shape of three-dimensional InAs islands on GaAs(001) with reflection high-energy electron diffraction (RHEED) and reported bounding facets corresponding to  $\{136\}$  planes.<sup>16</sup> Hasegawa *et al.* characterized the same material system with STM and concluded that some of the sidewalls have the orientation  $\{125\}$ .<sup>17</sup> Series of humps are resolved on these sidewalls, but the facet area is too small for the microfacets to be visible that are characteristic for our STM images. The self-organized formation of facets of a certain orientation indicates that the corresponding surface is of low energy and stable. The mentioned planes lie all in an angular range of about  $5^\circ$ . Taking into account the experimental uncertainties, the facets observed in these studies could actually all be of the same orientation.

A careful analysis of the  $(8 \times 1)$  reconstruction on GaAs(113) hints to a further possibly stable surface. The main element of the reconstruction is a zigzag chain of As dimers along  $[33\bar{2}]$ , which occurs in the unit mesh in two different atomic layers.<sup>3</sup> In a recent report we showed that this structure with an additional zigzag chain on top still fulfills the electron counting rule and is thus presumably stable.<sup>4</sup> Hence, a continuous further stacking of zigzag chains seems a promising way to form a new stable surface. The orientation of the plane constructed this way is  $(3\ 7\ 15)$  and lies between the different facet orientations mentioned



TABLE II. Angles between the specified directions, projected into the plane spanned by  $[1\bar{1}0]$  and  $[113]$  (cf. text), and  $[113]$ .

[124]	Directions		
	[125]	[136]	[3 7 15]
8.9°	7.4°	12.0°	9.7°

before. However, the STM image of stacked dimer chains is expected to look different than the microfacets seen in Fig. 3.

A direct comparison with our data is possible only via the angle towards (113). From our series of LEED images only the angles that lie in the plane spanned by  $[1\bar{1}0]$  and  $[113]$  can be extracted. Thus, the aforementioned orientations were projected into this plane. The angles between these projected directions and  $[113]$  are presented in Table II. With the exception of [136] these values are all in good agreement with the experimentally determined angle. Therefore, the stable surface presented in this paper is probably the same that forms the facets in Refs. 13,14,16 and 17.

In conclusion, the technique presented here of creating a

wide range of vicinal surfaces by means of a spherical depression has been shown to be adequate to prepare surfaces of good quality. A stable GaAs surface was found in the depression. This surface is oriented from (113) by  $9^\circ \pm 2^\circ$  in direction  $[1\bar{1}0]$ . In the opposite direction a surface with mirrored structure was observed, as expected from crystallographic symmetry. STM images show microfacets with series of either three or four humps. This surface is probably responsible for the formation of facets in Refs. 13,14,16 and 17. A study on wafers oriented in this direction is under way in order to determine the exact orientation of this stable GaAs surface.<sup>18</sup>

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