Summary Abstract: Observation and properties of the Ge(111)-7×7 surface from Si(111)/Ge structures

H.-J. Gossmann, J. C. Bean, L. C. Feldman, E. G. McRae, and I. K. Robinson
AT&T Bell Laboratories, Murray Hill, New Jersey 07974

(Received 5 December 1984; accepted 13 January 1985)

We have observed a 7×7 reconstruction of the (111) surface of thin (< 1000 Å) films of Ge grown by molecular beam epitaxy (MBE) on Si(111)-7×7 substrates. The 7×7 reconstruction has long been known for Si[1] but has not been observed previously for any other clean crystal surface. The Ge-7×7 reconstruction differs radically from the Ge(111)-$c2\times8$ reconstruction that is observed after thermal annealing of massive crystals.[2–4] We report low energy electron diffraction (LEED) and Rutherford backscattering/channeling (RBS) measurements indicating a close resemblance between the 7×7 reconstruction of Ge(111) and Si(111) surfaces. X-ray measurements show that the Ge(111)-7×7 films are compressed laterally relative to a bulk Ge crystal, suggesting a correlation between surface strain and the occurrence of the 7×7 superstructure.

A 7×7 pattern was already reported[5] from thin films (thickness ≈65 Å) of Ge_xSi_{1-x} grown by MBE on Si(111)-7×7 substrates for $x = 1$, while $x = 0.5$ clearly showed a 5×5 LEED pattern. In that study it was not possible to establish whether the 7×7 originated from an epitaxial Ge-7×7 layer or was the result of islanding, leading to an exposed Si-7×7 region. Using thicker films we unambiguously establish here that the films are continuous and that the observed 7×7 structure indeed originates from a pure Ge film.

Ge films of varying thickness were grown by MBE on Si(111)-7×7 substrates at 550 °C as described previously.[6] The surface periodicity was monitored *in situ* by LEED after each Ge deposition. A 7×7 pattern was observed for film thicknesses up to 1000 Å. A $c2\times8$ pattern, like that usually observed with massive Ge crystals, was observed for a film thickness of 5000 Å, while an ill-defined superposition of 7×7 and $c2\times8$ patterns was seen for intermediate thicknesses.

For detailed surface and film examination the samples were transferred in air to different experimental stations.

RBS was used to investigate the possibility that Si present in the Ge overlayer film or at its surface might be the cause of the observed 7×7 superstructure. No surface preparation was done between growth in the MBE system and the RBS experiment. Making a worst-case assumption we obtained an upper limit of 1×10^{21} cm^{-3} Si atoms in the overlayer. (Subsequent secondary ion mass spectroscopy resulted in an upper limit of $\ll10^{19}$ cm^{-3}.) These results indicate that the films were pure and no segregation of Si to the surface had occurred.

For the AES and LEED investigations, samples (500 Å thickness) were mounted in a ultrahigh vacuum (UHV) chamber. AES, performed before any *in situ* preparation, indicated only the presence of Ge, and O and C contaminants, but no Si. After two sputter (4×10^{15} cm^{-2} Ar ions, 1 keV, grazing incidence, room temperature) and anneal (20 min at 670 K and 10 min at 800 K) cycles the sample surface was pure Ge as indicated by AES.[7] The absence of Si at levels above the AES limit of detection (0.01 monolayers) shows that Ge was present as a continuous film and that no segregation of Si to the surface or any intermixing had taken place, confirming the conclusions drawn from RBS.

A LEED pattern obtained after this treatment is shown in Fig. 1(a). The distribution of intensity among the (1/7)th order spots is practically identical to that for Si(111)-7×7 [Fig. 1(b)] for nearly the same electron energy. This similarity applies over the range of electron energies for which comparisons could be made (25–50 eV), and extends also to cases were more severe sputtering resulted in mixed 7×7 and $c2\times8$ patterns.

For ion beam surface crystallography[8] a sample (500 Å overlayer thickness) was inserted into a UHV chamber

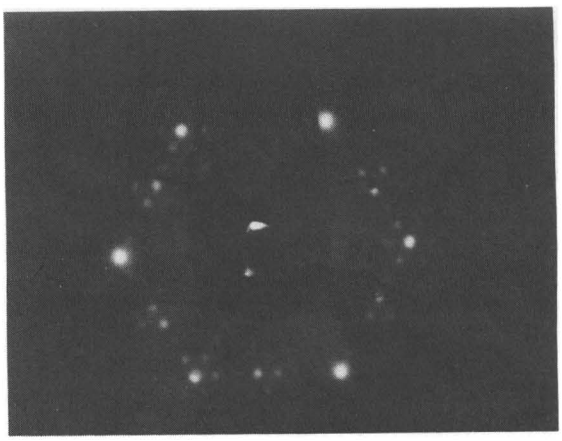

FIG. 1. LEED pattern in normal incidence (a) Ge(111)-7×7, electron energy 33 eV; (b) Si(111)-7×7, 35 eV.

 0734-2101/85/031633-02$01.00

equipped with AES and LEED and coupled to an accelerator. The preparation described above yielded a Ge(111)-7×7 surface that was both clean and pure as indicated by AES and RBS. Ge surface peak measurements (1.0 MeV He^+ ions) resulted in (3.13 ± 0.17) atoms/row in normal ⟨111⟩ incidence and (4.61 ± 0.20) atoms/row in off-normal ⟨11$\bar{1}$⟩ incidence (the expected value for a bulklike terminated surface would be 2.52 atoms/row). The number of monolayers displaced due to the Ge-7×7 reconstruction is thus larger in ⟨11$\bar{1}$⟩ incidence than in normal incidence, similar to the results found for Si(111)-7×7.[9]

The average lattice dimensions of the entire Ge film were determined by x-ray diffraction[6,10] immediately after removal of a sample from the MBE growth chamber. The 5000 Å thick film was found to have a lattice constant within 0.1% of 5.658 Å (bulk Ge), both parallel and perpendicular to the film. The 500 Å film had a lattice constant equal to bulk Ge normal to the film but a 0.8% smaller value (5.61 Å) in the lateral direction.

Our LEED and RBS results indicate a close structural similarity between Ge(111)-7×7 and Si(111)-7×7 surfaces. The dependence on film thickness and the x-ray observation of lateral contraction in the Ge films support the hypothesis that the 7×7 reconstruction is promoted over the $c2\times8$ one by lateral compressive stress which plays an important role in a particular model of the Si(111)-7×7 reconstruction.[11,12]

A detailed account of our results will appear elsewhere.[10]

We acknowledge the technical assistance of L. C. Hopkins, R. T. Lynch, R. A. Malic, and W. K. Waskiewicz.

[1]R. E. Schlier and H. E. Farnsworth, J. Chem. Phys. **30**, 917 (1959); J. J. Lander, in *Progress in Solid-State Chemistry*, edited by H. Reiss (Pergamon, Oxford, 1965), Vol. 2, p. 26.
[2]P. W. Palmberg and W. T. Peria, Surf. Sci. **6**, 57 (1967).
[3]D. J. Chadi and C. Chiang, Phys. Rev. B **23**, 1843 (1981).
[4]W. S. Yang and F. Jona, Phys. Rev. B **29**, 899 (1984).
[5]H.-J. Gossmann, J. C. Bean, L. C. Feldman, and W. M. Gibson, Surf. Sci. **138**, L175 (1984).
[6]J. C. Bean, L. C. Feldman, A. T. Fiory, S. Nakahara, and I. K. Robinson, J. Vac. Sci. Technol. A **2**, 436 (1984).
[7]While this treatment produced a pure Ge surface, annealing at higher temperatures (10 min at 950 K) did lead to segregation of Si to the surface.
[8]L. C. Feldman, J. W. Mayer, and S. T. Picraux, *Materials Analysis by Ion Channeling* (Academic, New York, 1982).
[9]R. J. Culbertson, L. C. Feldman, and P. J. Silverman, Phys. Rev. Lett. **45**, 2043 (1980).
[10]H.-J. Gossmann, J. C. Bean, L. C. Feldman, E. G. McRae, and I. K. Robinson (to be published).
[11]E. G. McRae, Phys. Rev. B **28**, 2305 (1983).
[12]E. G. McRae, Surf. Sci. **147**, 663 (1984).

Summary Abstract: Domain structure of the Si(111) 2×1 surface studied by reflection electron microscopy

P. A. Bennett, H. Ou, C. Elibol, and J. M. Cowley
Department of Physics, Arizona State University, Tempe, Arizona 85287

(Received 15 October 1984; accepted 6 November 1984)

The Si(111) cleaved surface produces a metastable, rectangular 2×1 superlattice structure, and the threefold symmetry of the substrate allows for three domains oriented at 120° apart.[1] Little is known in detail of the mechanism of orienting these domains, nor of the shape and energetics of their boundaries. From LEED studies it has been inferred that the 2×1 structure on cleaved silicon and germanium surfaces is oriented by the ledges of step arrays and suppressed in a 50 Å strip near step edges.[2–5] Also it has been reported that a single domain is preferred for (211) cleavage, while a two domain structure normally results for (110) cleavage.[6] The recent advent of UHV electron microscopy provides a unique tool to directly observe the domain structures.[7]

We have developed a UHV field emission based scanning electron microscope with diffraction capability whose construction is detailed in Ref. 8. For the present experiments we have used a well collimated 18 keV beam with a 1000 Å spot size and beam current of 10^{-10} A. RHEED patterns are displayed on a TV monitor which views a channelplate-phosphor-light pipe assembly. Dark field images are formed by isolating part of the diffraction pattern (external to the vacuum) and detecting this signal with a photomultiplier tube as the beam is rastered over the specimen. Conventional SEM images (secondary electron yield) are also available using a spiraltron detector mounted near the specimen. The surface is prepared by breaking a (110) oriented $\frac{1}{4}$ mm silicon wafer *in situ*, exposing a $\frac{1}{4}\times2$ mm area of (111) surface for study. Most surfaces so prepared are flat enough to allow reflection microscopy imaging, although considerable roughness is visible in the microscope images.

Microdiffraction patterns at 18 keV on the (112) azimuth and incident angle $\theta\sim3°$ are shown in Fig. 1 with the beam stopped at two locations on the surface. A given row of reflections normal to the incident beam constitutes one Laue arc as illustrated in panels (a–c). The glancing angle results in an oval spot on the specimen of area $0.1\times2\,\mu$ which is seen to include type A and C domains in panel d and type A and B in panel e. The dark field images using a type C superlattice reflection and an integer order reflection are shown in Fig. 2, panels (a) and (b), respectively. Superlattice domain contrast is visible in the nearly periodic fanned arcs appearing in the upper left of the image. The bright areas correspond to the presence of type C superlattice domains, while the dark areas correspond to a 1×1 structure. Microdiffraction patterns confined to these regions show, respectively, only type C diffraction spots or no superlattice reflections. Comparison

