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**DEVELOPMENT OF X-RAY FACILITIES  
FOR  
MATERIALS RESEARCH  
AT  
THE ADVANCED PHOTON SOURCE**

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**"Development of X-ray Facilities for Materials Research  
at the Advanced Photon Source"**

**M. J. Bedzyk**

**Abstract:**

The P.I. and his research team successfully used the funds from the DOE Instrumentation grant entitled "Development of X-ray Facilities for Materials Research at the Advanced Photon Source", to design, build, test, and commission a customized surface science x-ray scattering / spectroscopy chamber. This instrumentation, which is presently in use at an APS x-ray undulator beam line operated by the DuPont-Northwestern-Dow Collaborative Access Team, is used for x-ray measurements of surface, interface, thin film and nano- structures.

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## 1.0 Overview

In the four years of the contract DE - FG02 - 96ER45588, the PI's research team at Northwestern University has achieved the project goals, which were to support the construction of a versatile ultrahigh vacuum (UHV) x-ray endstation that would be used at the Advanced Photon Source (APS) x-ray beam lines operated by the DuPont-Northwestern-Dow Collaborative Access Team (DND-CAT) for studies of surface, interface and thin film structures. Capital funds from this grant were used to underwrite a large fraction of the cost of the x-ray diffractometer and surface science equipment. Other equipment for this project has been purchased with funds from other sources (the initial DOE instrumentation grant DE-FE-FG02-94ER45527, funds from the NSF-funded Materials Research Center at NU, and funds from the NSF/DOE-funded Institute of Environmental Catalysis at NU). The entire instrumentation package (chamber, diffractometer, detectors and post-monochromator optics) is used for various types of x-ray scattering and spectroscopy measurements of surface, interface, thin film, and nano- structures. The Senior Research Associates (Drs. Paul F. Lyman and Donald A. Walko) funded by this grant (DE-FG02-96ER45588) served to design, procure, build, test, commission, operate, and document the equipment associated with this instrumentation project. Publications from the PI's research group that came as a result of the instrumentation funded by this grant are listed in Section 3.0.

## 1.1 Synchrotron Radiation Instrumentation Development

For the past four years the PI's research team has utilized DOE funds for designing, building, and commissioning a customized x-ray scattering and spectroscopy facility [1] at the Advanced Photon Source. This facility, which is located at the DuPont-Northwestern-Dow Collaborative Access Team (DND-CAT) 5ID-C undulator station, is now being used for high-resolution studies of surface, interface, and thin-film structures. A floor plan layout is shown in Fig. 1. The x-ray source is a high-brightness 144 pole type-A x-ray undulator furnished by the APS. The 5ID beamline furnished by DND includes a L-N<sub>2</sub>

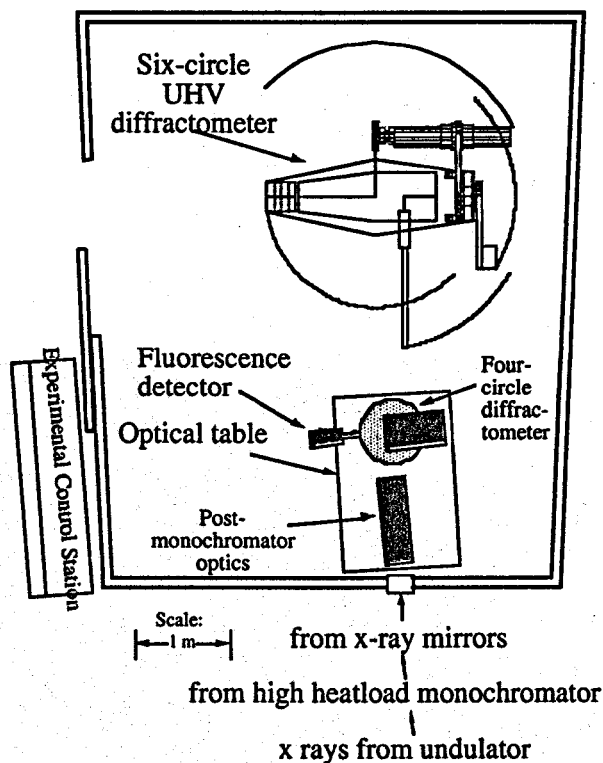


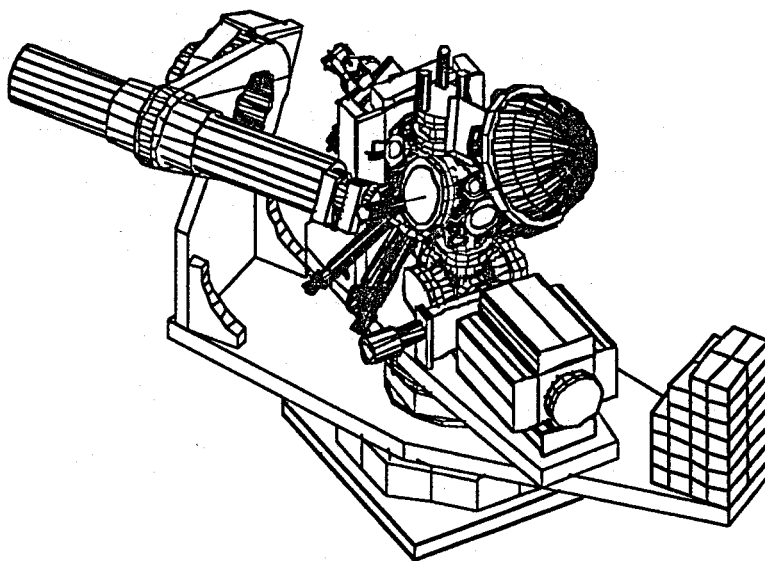
Fig. 1. The DND-CAT 5ID-C hutch floor plan layout.

cooled high heat-load Si(111) monochromator followed by a pair of x-ray mirrors that deflect and focus the monochromatic beam in the horizontal plane before it reaches the 5ID-C station. The approximately 6x5x3 cubic meter lead enclosure of 5ID-C is also furnished by DND-CAT. Inside and at the upstream end of this room is our kinematically controlled optical table that holds our postmonochromator optics with options for increasing the x-ray monochromaticity, angle collimation, microbeam focusing (with Kirkpatrick-Baez mirrors), and circular polarization. At the downstream end of this 2m long table sits a 4-circle diffractometer and a fluorescence detector for non-UHV experiments.

In the down-stream half of 5ID-C is our UHV surface chamber that is intimately coupled to a large diffractometer. [1] The vacuum chamber has standard surface science capabilities for MBE surface preparation (sample heating and cooling, Knudsen cells, an electron-beam evaporator, an oxygen atomic-

source, differentially pumped ion sputtering gun) and characterization (LEED, Auger electron spectroscopy and x-ray photoelectron spectroscopy with a double-pass cylindrical mirror analyzer (CMA)). The UHV chamber sits on a six-circle diffractometer built with "psi-circle" geometry (Fig.2). The diffractometer with vacuum chamber weighs 2 tons and sits on a heavy-duty high precision X-Z motorized table. The capabilities of the UHV endstation include grazing-incidence X-ray diffraction, crystal truncation rod scattering, X-ray reflectivity, X-ray standing waves (XSW), surface extended X-ray absorption fine structure (SEXAFS), X-ray holography, and X-ray photoelectron spectroscopy. For the spectroscopy experiments (XSW, SEXAFS, x-ray holography), the CMA or hemispherical electron analyzer, and/or a seven-element solid-state fluorescence detector system will be used.

All of the non-UHV components at 5ID-C have been tested and used for the past two years. The results of the open air XSW, XAFS and X-ray diffraction experiments performed as a part of the commissioning stage are presented in sections 2.1 -2.4 below. These experiments include single monolayer of  $\text{InAs}_x\text{Sb}_{1-x}$  buried in an  $\text{InSb}(111)$  matrix, the structure of a passivating sulfide layer on  $\text{Ge}(001)$ , polarization determination of thin ferroelectric  $\text{PbTiO}_3$  film, polarization switching in thin



*Fig. 2. Psi-circle diffractometer with UHV capabilities under operation at the 5ID-C beamline at the DND-CAT, APS.*

ferroelectric  $\text{PbZr}_{1-x}\text{Ti}_x\text{O}_3$  film under externally applied electric field. Thin germanium epitaxial films grown on  $\text{Si}(001)$  were investigated in our first SXRD experiment performed at 5ID-C using our psi-circle diffractometer and are described in section 2.5. In the first UHV X-ray scattering experiments the 1 ML and 0.5 ML coverage phases of tellurium adsorbed on  $\text{Ge}(001)$  were studied and described in Sec. 2.6.

## 2.0 Experimental results

### 2.1 XSW and XAFS analysis of $\text{InAsSb}/\text{InSb}(111)$ quantum-well

The XSW structural analysis of buried strained-layer III-V heterostructures grown by our collaborators (Wessel's group at NU) was performed as part of a commissioning stage to test all the components of the 5ID-C beam line including: X-ray optics, non-UHV diffractometer, diffraction and spectroscopic software. The scientific motivation of this work was a direct test on the microscopic level of the macroscopic elasticity theory applied to ultra-thin crystalline films down to the one monoatomic layer limit. The vapor phase epitaxy (VPE) samples (A and B) were grown as a single monolayer of  $\text{InAs}_x\text{Sb}_{1-x}$  buried in a  $\text{InSb}(111)$  matrix. The As concentration "x" in the buried monolayers was calibrated by x-ray fluorescence analysis to be 0.23 and 0.95 for A and B, respectively. For the XSW measurements, the APS undulator and  $\text{Si}(111)$  monochromator were tuned to an x-ray energy of 12.8 keV to excite As K fluorescence. The As fluorescence and the reflectivity R were recorded simultaneously during the angular scan through the  $\text{InSb}(111)$  rocking curve and then analyzed with dynamical diffraction theory derived reflectivity and yield curves (Fig. 3). The strong modulation amplitude (or high coherent fraction of  $f = 0.85$ ) of sample A indicates that a highly ordered  $\text{InAs}_x\text{Sb}_{1-x}$  monolayer was obtained at the lower As coverage of  $x = 0.23$ . An analysis of the modulation phase (or coherent position P) locates the As atomic layer with respect to the In layer of the  $\text{InSb}$  substrate (Fig. 4). Assuming

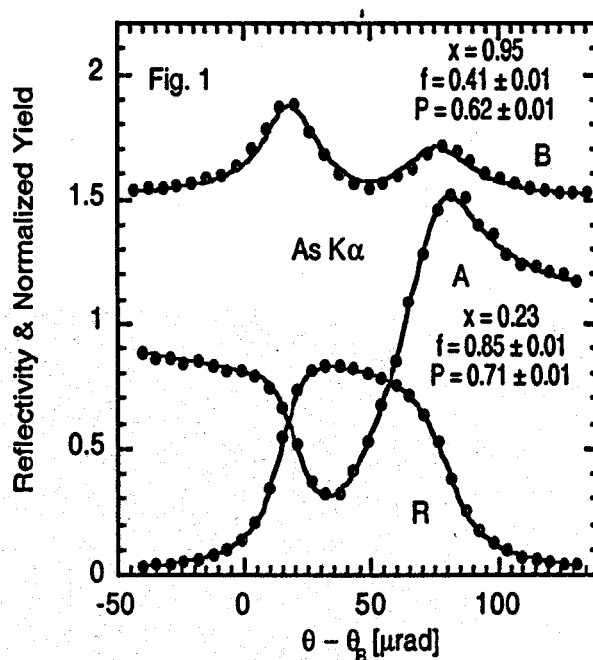


Fig. 3. The XSW As  $K_\alpha$  fluorescence yield curves for samples A and B and the reflectivity R (solid circles the experiment, solid lines - the fit using dynamical diffraction theory).

that the As occupy virtual crystal sites and that the strain effect is contained within one (111) d-spacing above and below the As layer, these XSW coherent positions correspond to vertical strains of  $-3.0\%$  and  $-6.9\%$  in the buried layers for sample A and B, respectively. Macroscopic elasticity theory, predicts that the vertical strains should be  $-1.0\%$  and  $-3.8\%$  for  $x = 0.23$  and  $0.95$ , respectively. This apparent disagreement may be explained by considering, on a more microscopic level, the bi-axial stress in the (111) plane and the anisotropic nature of the four bonds surrounding each As atom. For this, a random-cluster calculation utilizing a Keating force-field

approach was carried out for the  $\text{InSb}(111)/\text{InAs}_x\text{Sb}_{1-x}$  heterostructure. It predicts an In-As bond length split (or anisotropy), as large as  $0.05 \text{ \AA}$  at  $x = 1.0$ , between the bonds parallel to and nearly perpendicular to the  $[111]$  direction. This effect should cause a non-uniform deformation of the  $\text{InAs}_x\text{Sb}_{1-x}$  unit cell, where the average As position, as measured by XSW, should not be located at a virtual crystal site. The discrepancy between the data and the elasticity theory also suggests a larger strained region in the buried film. The off-normal (022) XSW data and analysis (not shown) for sample A agreed by the symmetry indicated in Fig. 4 with the (111) analysis and thereby confirmed that the As was confined to a pseudomorphic single layer. X-ray absorption fine structure (XAFS) measurements were also conducted at the DND CAT to independently confirm this bond-length anisotropy.

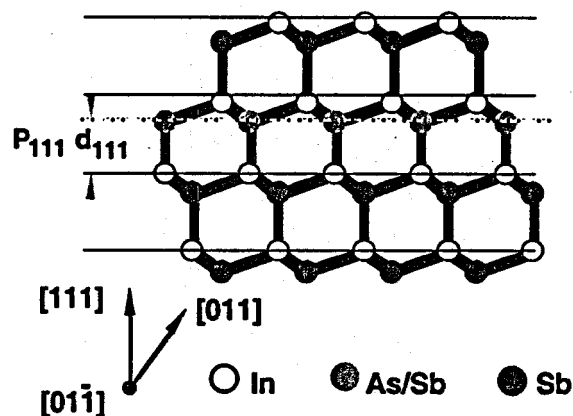


Fig. 4. Pseudomorphic As single layer buried in  $\text{InSb}(111)$  matrix. The coherent position  $P_{111} = 0.75$  for bulk Sb.

## 2.2 XSW studies of the passivated Ge surface prepared from aqueous solution

The chemical and electronic passivation of the surface of semiconductors is vital to their utility for device construction. Truncation of the surface of a crystal induces electronic defect states that allow carriers to recombine, and "fixed" charges residing at a semiconductor/insulator interface can hinder performance of a metal-insulator-semiconductor capacitor (and hence preclude construction of a working device). An effective passivation treatment should be chemically stable, protect the substrate from oxidation, remove surface-induced carrier recombination states, and exhibit a low density of fixed charges. For ease of processing, it is desirable that such a passivation reaction be carried out chemically at atmospheric pressure. In this work the structure of a passivating sulfide layer on  $\text{Ge}(001)$  was studied using X-ray standing waves and X-ray fluorescence (XRF) techniques [3].



The sulfide layer was formed by reacting clean Ge substrates in  $(\text{NH}_4)_2\text{S}$  solutions of various concentrations at  $80^\circ\text{C}$ . Our XRF results showed remarkably constant coverage of  $\Theta = 2$  to 3 ML under a wide range of concentration of  $(\text{NH}_4)_2\text{S}$  solution indicating that the sulfidation reaction is self-limiting. Perhaps the most striking result is the consistency of the coherent coverage, which is defined as the product of the coherent fraction and the total coverage,  $\Theta_c = F_{004} \cdot \Theta$ . This parameter is a measure of the total number of S atoms (in ML) that are located at a position in registry with the Ge substrate. We found that even though the concentration of the  $(\text{NH}_4)_2\text{S}$  solution was varied by a factor of 240, the resultant  $\Theta_c$  varied by only 10% from 0.35 to 0.39. Thus, the structure of the reacted layer that is correlated to the Ge substrate is highly regular and repeatable. Our XSW data supports bridge-site S atoms at an absorption height of  $1.27 \pm 0.02 \text{ \AA}$  above the last Ge layer. The remaining 1.5 to 2.5 ML of S occupy positions uncorrelated with the Ge lattice, consistent with bonding in a glassy network of  $\text{GeS}_x$ .

### 2.3 Probing the polarity of epitaxial ferroelectric thin films with X-ray standing waves:

#### **$\text{PbTiO}_3 / \text{SrTiO}_3(001)$**

Ferroelectricity in solids originates from the relative shifts of the anion and cation atomic sublattices along a certain crystallographic axis resulting in a net electronic dipole moment (spontaneous polarization). Under an externally applied electric field, the direction of the polarization vector can be switched. This bi-stable property provides the basis for nonvolatile random access memory (NVFRAM). In the ferroelectric  $\text{PbTiO}_3$  perovskite structure the dipole moment along the  $[001]$  polar axis is due to the displacements of the  $\text{Pb}^{2+}$  and  $\text{Ti}^{4+}$  sublattices with respect to the  $\text{O}^{2-}$  sublattice. In bulk ferroelectric crystals the dipole moments are perfectly aligned only within single domain volumes with typical sizes much less than the absorption length of x-rays in crystals. Therefore, it was widely accepted that the lattice polarization and its evolution under an external electric field could not be studied by x-ray diffraction techniques. Recently, single crystal ferroelectric thin films with thickness much less than the typical domain size in bulk crystals have been grown using various epitaxial methods. The ability to control the domain structure, together with a technique to precisely characterize the polarization state, would be a powerful combined approach for investigating such fundamental phenomena in ferroelectricity as switching and polarization degradation.

In this work [4] we applied a newly developed thin film XSW method to determine the polarity of thin  $\text{PbTiO}_3$  films grown by MOCVD on  $\text{SrTiO}_3(001)$ . This method is based on the excitation of an X-ray standing wave field inside the film as a result of the interference between the strong incident X-ray wave and the weak kinematically Bragg-diffracted X-ray wave from the film.

## 2.4 Thin Film XSW Investigation of Polarization Switching in PZT Thin Film Capacitors

The development of thin film x-ray standing waves and the successful application of this technique to probe the polarity of as-grown ferroelectric  $\text{PbTiO}_3$  films opened up new opportunities to study switching in thin ferroelectric films on an atomic scale. As a next step in this direction we studied polarity switching in ferroelectric  $\text{PbZr}_x\text{Ti}_{1-x}\text{O}_3$  (PZT) thin films, which are used in nonvolatile ferroelectric random access memory (NVFRAMs) [5]. Several PZT ferroelectric capacitor samples were prepared. The capacitors consisted of a 1300 Å thick bottom electrode composed of  $\text{SrRuO}_3$  (SRO), sputter deposited at Duke University on a  $\sim 2^\circ$  miscut  $\text{SrTiO}_3(001)$  substrate. The 200 Å thick PZT film with Zr composition  $x = 0.3$  was grown via MOCVD at MSD/ANL. Polycrystalline Ag pads (500 Å thick, 250  $\mu\text{m}$  diameter) were shadow mask deposited to complete the capacitors. Polarization I-V measurements were performed on the capacitors and they were left in either the "up" or "down" polarization state.

The switched samples were then examined at the Advanced Photon Source 5ID-C undulator station using the thin-film XSW method. The beam was

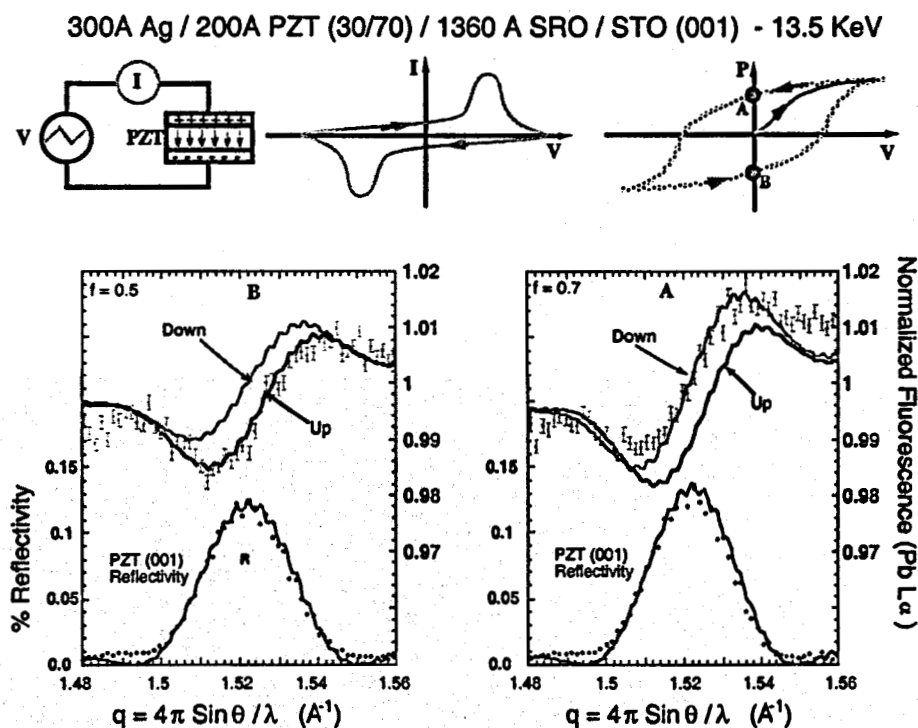


Fig. 5. XSW measurement of Pb-L fluorescence for the PZT(001) Bragg reflection measured from a PZT ferroelectric capacitor that was switched into the polarized up state (left) and down state (right). Best fit for the up and down polarization is shown as solid and dashed lines, respectively. This constitutes an atomic-scale quantitative measure of the displacement of the Pb ions.

monochromated to 13.55 keV in order to excite Pb-L fluorescence and was reduced in size by slits to select out individual 250 micron diameter pads. Each ferroelectric capacitor was scanned in angle through the PZT(001) reflection, while the Pb fluorescence was collected. The difference between the two polarization states of the PZT layer was determined via model-fitting of the calculated E-filed intensity to the modulation of the experimental fluorescence signal. The XSW method successfully determined the polarization of the PZT capacitors. The typical XSW response from the PZT capacitor switched into the polarized up state (left) and down state (right) are shown in Fig. 5. Future plans include studying the effect of hydrogen annealing on the structure of individual sublattices in PZT films and on the polarization of PZT-based capacitors.

## **2.5 X-ray scattering studies of Ge ultra-thin films on Si(001): the role of Bi as a surfactant**

The epitaxial growth of lattice mismatched heterostructures is a non-equilibrium process that is driven by surface kinetics and strain relief mechanisms. Surfactants have been shown to modify the morphology of lattice mismatched heterolayers. Under metastable growth conditions the surfactant causes a decrease in surface energy at the growth layer which aids the wetting process of the epilayer. While under equilibrium conditions the surfactant can promote wetting or islanding due to surface energy anisotropy.

The lattice mismatch between Si and Ge is 4% and the surface energy of Ge is lower than that of Si. Therefore, Ge growth on Si is in Stranski-Krastanov mode which is 2-dimensional layer-by-layer growth up to 3 monolayers (ML) and 3-dimensional or island-like growth thereafter. We studied the strain and morphology of pseudomorphic Si / Ge heterostructures grown on Si (001) with Bi as a surfactant, using a combination of grazing incidence x-ray diffraction (GIXD), x-ray reflectivity, and x-ray standing waves [6].

The samples were prepared by molecular beam epitaxy (MBE) in an ultra high vacuum (UHV) chamber with a base pressure of  $1 \times 10^{-10}$  Torr. The LEED and AES were used to monitor the structure and composition of the surface layer at different stages of the growth. The samples were prepared with and without Bi as a surfactant with Ge coverages ranging from 1 –10 ML. An epitaxial 10 nm thick Si layer was grown on top of the Ge film using an e-beam evaporator.

The x-ray measurements were performed at the 5ID-C beamline at the Advanced Photon Source on our psi-circle diffractometer. A platinum coated flat mirror was used to filter out the higher harmonic photons from the x-ray beam. X-ray reflectivity and radial H,K scans crossing the (22L) crystal truncation rod (CTR) at grazing incident angles were measured for a series of Si/Ge/Si(001) heterostructures. The set of samples had different Ge film thickness ranging from 1 to 10 ML. Samples were grown with and without Bi as a surfactant. (These measurements were also used as a stringent test of the psi-circle diffractometer.) In Fig. 6 the effect of the surfactant in growing planar heterolayer structures is clearly seen for the pair of 10 ML Ge samples. Both scans are

at  $L = 0.01$ , which is below the critical angle. The data for the sample with Bi (Fig. 6(a)) indicates a nearly perfect pseudomorphically grown strained layer heterostructure with only one peak appearing at a lattice constant corresponding to pure unstrained Si(220). Whereas, the same growth without Bi shows (Fig. 6(b)) two additional peaks: a broad weak peak at the Ge(220) bulk-like lattice position  $H=K=1.92$  and a peak at  $L=1.98$  which corresponds to the formation of a Ge-Si alloy. This formation of Ge islands and the Ge-Si intermixing results in a very rough and diffuse interface.

## 2.6 X-ray scattering studies of Ge(001):Te 1x1 surface structure

The search for effective ways to passivate the clean Si or Ge (001) surface, which is characterized by a pair of dangling bonds per surface atom on ideally truncated surface, revealed group VI Te as one of the best candidates. Te is known also as an effective surfactant that can be used for the growth of Ge/Si heteroepitaxial structures. We applied the XSW method to study the structure Te forms on the Ge(001) surface. Our (004) and (022) XSW data strongly support a two-fold bridge-site as the most favorable occupation site for the 1 ML Te/Ge(001) 1x1 phase. A random array of missing Te rows was proposed to explain the streaky (1x1) LEED pattern. The bridge-site Te occupation positions was later confirmed by total energy first-principle calculations. To further discriminate between two plausible modified bridge-

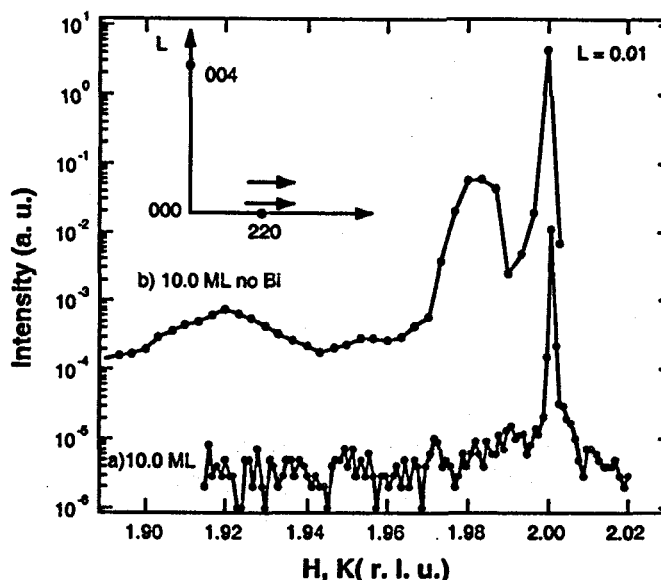


Fig. 6 Grazing incidence H,K radial scans through (22L) with  $L = 0.01$  for Si/ Ge/ Si(001) heterolayer structures with a) 10.0 ML of Ge grown with Bi and b) 10.0 ML of Ge grown without Bi. The scans are shifted vertically for clarity. The inset represents the path of the scan in reciprocal space.

site models: namely the 'zigzag' and missing row models supported by the first principles calculations, we performed X-ray scattering measurements at APS 5ID-C. This was the first UHV test of our psi-circle surface science diffractometer [7].

The sample was prepared in the main chamber of our UHV diffractometer by ion sputtering and annealing at 650°C to produce a clean dimerized Ge(001) surface with a sharp two-domain (2x1) LEED pattern. After Te deposition from an effusion cell the sample was heated for 20 min at 400°C to desorb loosely bound Te from the surface. The LEED showed a (1x1) streaky pattern indicating that the surface had 1x1 long-range order but was locally disordered. The incident 18.0 keV X-ray beam was monochromized by the Si(111) monochromator and focused on the sample by the horizontally focusing mirror. The 10L, 11L and 30L crystal truncation rods measured from the sample are shown in Fig. 7.

The CTR data was compared to the bridge-site, top-site, hollow-site, and anti-bridge-site models and the minimum  $\chi^2$  value was obtained for the bridge-site in agreement with our earlier XSW analysis. Furthermore, we found that within the bridge-site model, the missing row model gives a better fit than the 'zigzag' model. (See Fig. 7.) Combining the CTR and XSW analysis we were able to refine the atomic positions of the Te atoms, determine the relaxation of underlying Ge atomic layers, and determine the Te-Ge bond length. Finally, the local disorder of the Te atoms was taken into account.

## 2.7. High Energy X-ray crystallography of $\text{UPt}_3$

We have used high-energy x-ray diffraction to refine the crystal structure of  $\text{UPt}_3$  [8], a heavy Fermion material whose low-temperature properties (superconductivity and antiferromagnetism) have been widely studied but are incompletely understood. Nominally single crystal samples were grown by UHV float-zone refining, with subsequent annealing from 800° C to 1300° C, resulting in high crystal

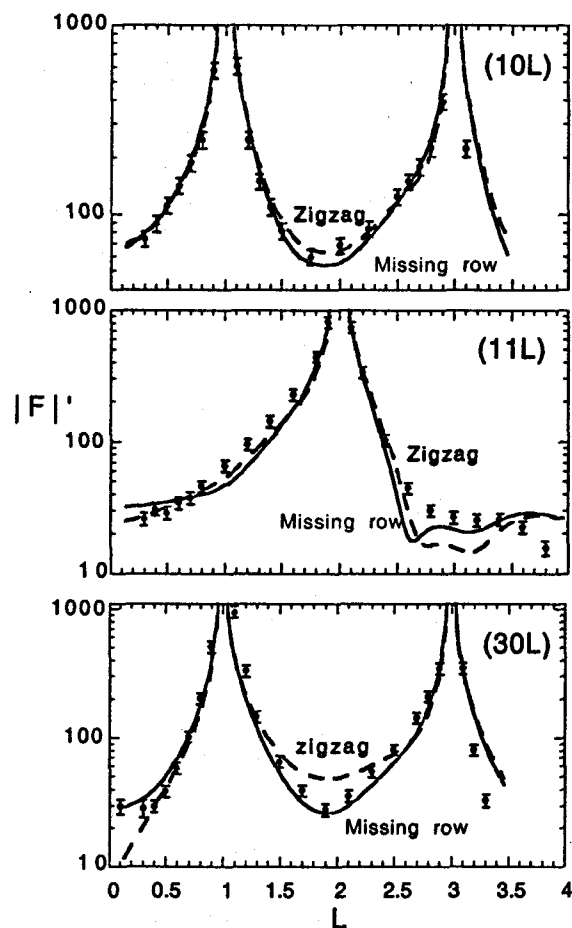


Fig. 7 The experimental 10L, 11L and 30L crystal truncation rods (circles) and calculation (line) for the Te missing-row model and calculation (dot) for the bridge-site model.

quality and very low impurity concentrations. X-ray diffraction measurements were performed at beamline 5BM-D of DND-CAT, Advanced Photon Source, Argonne National Laboratory. Data were collected using the oscillation method on a two-dimensional CCD camera with 75 keV x rays ( $\lambda=0.165\text{\AA}$ ). High-energy x-rays were needed to minimize absorption; given the large electron density of  $\text{UPt}_3$ , a high-resolution experiment was feasible only at a third-generation synchrotron source which could provide a sufficient intensity of high-energy x-rays. The experimental diffraction patterns (typically consisting of  $>1500$  independent reflections per sample) were of apparent Laue class  $6/mmm$ , which is inconsistent with the  $P6_3/mmc$  spacegroup traditionally assigned to  $\text{UPt}_3$ . That is, the systematically absent reflections required by  $P6_3/mmc$  symmetry were in fact present throughout the diffraction pattern. Furthermore, the data cannot be reconciled with *any* hexagonal space group. Instead, the atoms of the unit cell experience very slight vertical displacements, lowering the crystal's symmetry to the trigonal  $P\bar{3}m1$  spacegroup, as shown in Fig. 8. Careful data collection was required to accurately measure the displacements of the atoms from their "ideal" hexagonal positions (displacements were less than 1% of the unit cell length). The "single" crystal is, however, severely twinned by merohedry, with two evenly populated twins that give the appearance of hexagonal symmetry. With this lowering the symmetry of the space group, the symmetry of the U site (the source of the superconducting and magnetic properties) is substantially reduced from  $-6m2$  to  $3m$ . This result significantly restricts the possible symmetry of the superconducting order parameter (which in the past was often assumed to have six-fold symmetry) and will help complete the theoretical description of the low-temperature properties of this surprisingly complex material.

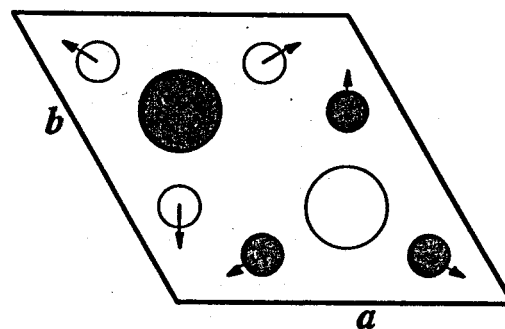


Fig. 8. Unit cell of  $\text{UPt}_3$  viewed down the  $c$  axis. Large circles are U, small circles are Pt. Dark and open circles are at heights  $+z$  and  $-z$ , respectively. For trigonal symmetry  $z_U \neq z_{Pt}$  in general, but for hexagonal spacegroups  $z_U = z_{Pt} = 1/4$ . Arrows indicate the possible in-plane displacement of Pt atoms, permitted in both hexagonal and trigonal systems.

**3.0 Publications which made use of instrumentation and/or personel supported by DoE Grant No. DE - FG02 - 96ER45588:**

- 1 "UHV Surface-Analysis Endstation with X-ray Scattering and Spectroscopic Capabilities". P.F. Lyman, D.T. Keane, and M.J. Bedzyk, *AIP Conference Proceedings* -- July 1, 1997 -- 417, 10-14 (1998).
- 2 "X-ray standing wave investigation of a single monolayer of  $\text{InAs}_x\text{Sb}_{1-x}$  buried in an  $\text{InSb}(111)$  matrix", T.-L. Lee, D.L. Marasco, P.F. Lyman, S. Therring, B. Wessels, D.T. Keane, and M.J. Bedzyk, in preparation
- 3 "Structure of passivated Ge surface prepared from Aqueous Solution", P.F. Lyman, O. Sakata, D.L. Marasco, T.-L. Lee, K.D. Breneman, D.T. Keane, M.J. Bedzyk, *Surf. Sci.* 462, L594-L598 (2000).
- 4 "Probing the polarity of ferroelectric thin films with x-ray standing waves", M.J. Bedzyk, A. Kazimirov, D.L. Marasco, T.-L. Lee, C.M. Foster, G.-R. Bai, P.F. Lyman, D.T. Keane, *Phys. Rev. B* 61, R7873-7876 (2000).
- 5 "X-ray standing wave observation of switching in ferroelectric thin films", D.L. Marasco, M.J. Bedzyk, A. Kazimirov, T.-L. Lee, O. Auciello, S. K. Streiffer, G. R. Bai, C.-B. Eom, in preparation.
- 6 "X-ray scattering studies of surfactant mediated epitaxial growth of  $\text{Si}/\text{Ge}/\text{Si}(001)$  heterostructures", W. Rodrigues, O. Sakata, T.-L. Lee, D.A. Walko, D.L. Marasco, M.J. Bedzyk, *J. Appl. Phys.* 88, 2391-2394 (2000).
- 7 "X-ray scattering study of the  $\text{Ge}(001)$ :  $\text{Te}(1\times 1)$  surface structure", O. Sakata, P.F. Lyman, B.P. Tinkham, D.A. Walko, D.L. Marasco, T.-L. Lee, and M.J. Bedzyk, *Phys. Rev. B* 61, 16692-16696 (2000).
- 8 "New crystal structure assignment for  $\text{UPt}_3$ ", D.A. Walko, J.-I. Hong, T.V. Chandrasekhar, Rao, Z. Wawrzak, D.N. Seidman, W.P. Haperin, and M.J. Bedzyk submitted to *Phys. Rev. Lett.*

**3.1 Northwestern University Thesis with partial support from this grant via instrumentation:**

- T. L. Lee, "High-Resolution Analysis of Adsorbate-Induced  $\text{GaAs}(001)$  Surface Structures and Strain in Buried III-V Semiconductor Heterolayers by X-Ray Standing Waves", Ph.D. Thesis, Materials Science and Engineering, December 1999.
- W. P. Rodrigues, "Growth and Characterization of  $\text{Si}/\text{Ge}$  Heterostructures on  $\text{Si}(001)$  Surface and Ge Nano-Dots on Patterned  $\text{Si}(001)$  Surface", Ph.D. Thesis, Physics and Astronomy, December 2000.