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Spontaneous Step Creation on (001) Silicon Surfaces Studied with Scanning Tunneling Microscopy and Low-Energy Electron Microscopy

DISSERTATION

Presented in Partial Fulfillment of the Requirements for the Degree Doctor of Philosophy in the Graduate School of The Ohio State University

By

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ABSTRACT

Ultra-high vacuum (UHV) scanning tunneling microscopy (STM) and low-energy electron microscopy (LEEM) were used to make the first conclusive and direct observation of a striped step phase on silicon (001) surfaces induced by a substrate imposed biaxial tensile strain and/or heavy boron doping.

For 5nm silicon (Si) films grown on relaxed silicon-germanium (SiGe) substrates, stable wavy-step structures in which large amplitude, periodic $S_B$-step undulations, alternate with straight $S_A$-steps are observed. These wavy-step structures evolve into elongated-step structures as the local terrace width increases, and for very wide terraces take on a striped-step surface structure. The observation of these wavy-steps and stripes, are explained in terms of strain-enhanced surface stress-relaxation effects stemming from the $\sim 1\%$ biaxial tensile strain at the surface.

Heavily B-doped Si(001) surfaces are also observed to form finely striped step structures ($\sim 10\text{nm}$ wide) on surfaces annealed below $800^\circ\text{C}$ in UHV, which grow in size with increasing annealing temperature and evolve into triangular-tiled structures above $900^\circ\text{C}$. In this case, the surface step structures are also explained in terms of stress-relaxation effects, with the temperature dependence arising from a temperature-dependent surface segregation of B. Surface segregation of B should produce a biaxial tensile strain, and uniaxial strain experiments suggest that B surface segregation also leads to $\sim 12\%$ increase in the surface stress anisotropy over that for
Si surfaces with low doping concentrations. Both the biaxial tensile strain and the increased stress anisotropy should enhance the stripe phase.

The shape of the structures observed for both Si/SiGe(001) and B-doped Si(001), are consistent with recent isotropic elastic calculations, which show the same progression of step structures from straight—triangular-tiled—striped, as the terrace width, surface stress anisotropy, or biaxial tensile strain is increased.

LEEM and STM have also been used to image residual uniaxial strain fields on SiGe(001) films, and a high degree of correlation was found between lateral strain variations and the surface cross-hatch morphology on strain-relaxed Si_{0.7}Ge_{0.3} substrates.
This work is dedicated to my family and friends,
and to the memory of Darrin Jones, Crystal Shrider, Tom Bucher,
Kendra Simmons, Monique Wireman, and Tim Gossard.
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The work presented in this document was not completed by one individual alone, but rather by the collective efforts of many. Therefore, I wish to acknowledge the contributions of those who helped to make this endeavor a success.

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Next I wish to acknowledge my collaborators, who have contributed so much to this work. Our early experiments would not have been possible without the Si/SiGe samples provide to us by Ya-Hong Xie and Paul Silverman of Lucent Technologies. Likewise I wish to thank Ying Hong and Dr. Ignatius Tsong for making my research visits at Arizona State University, to use the low-energy electron microscope during the summer of 1995, both enjoyable and productive. I would also like to thank Eugene Fitzgerald, Earnst Bauer, and George Gilmer for helpful discussions and insight. I sincerely thank Dr. Charles Ebner who, in addition to contributing greatly
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CHAPTER 1

OVERVIEW

In this work, ultra-high vacuum (UHV) scanning tunneling microscopy (STM) and low-energy electron microscopy (LEEM) were used to study the surfaces of Si grown under tensile strain on relaxed SiGe(001) substrates, relaxed Si$_{1-x}$Ge$_x$ layers ($x=0.25-0.8$), and heavily B-doped Si(001) surfaces.

The (001) surface of Si has been studied intensely, due in large part to its central role within the microelectronics industry. In chapter 2 of this document a review of the Si lattice and the structure of the Si(001) surface is given. The Si(001) surface forms a reconstruction to reduce the number of surface dangling bonds, resulting in (2x1) and (1x2) surface domain structures which possess anisotropic surface stress.

The theoretical work of Marchenko [1] in 1981 suggested that degenerate surface phases with differing stress tensors, could lower their surface energy by the formation of elastic stress domains. In 1988 Alerhand, Vanderbilt, Meade, and Joannopoulos (AVMJ) [2], (independently of Marchenko) proposed that stress relaxation effects at step edges, would make it energetically favorable for an initially flat Si(001)-(2x1) surface to spontaneously form extra up/down $S_A$ steps at the surface.

The AVMJ theory was quite successful in advancing the understanding of such observed phenomena as the Si(001) single—double height step transition and changes
in surface domain populations with an applied uniaxial strain. However, the central prediction of the AVMJ model, the spontaneous creation of extra up/down $S_A$ steps forming a striped surface phase, went without observation.

In this work, the first direct observation of striped step structures on Si(001) surfaces is reported, confirming the formation of elastic stress domains [1] by the AVMJ [2] mechanism.

These experiments were performed using a STM designed and constructed by this author, and described in detail in chapter 3. The system consists of three chambers connected in series, which may be isolated from one another. A sample introduction chamber permits samples and STM tips to be freely exchanged from atmosphere to the main chamber, where sample cleaning and tip preparation is carried out under UHV conditions. Prepared samples and tips can then be transferred to the STM, housed in a separate UHV chamber. The instrument is very flexible for a wide range of surface science experiments, and provides resolving power sufficient to image surface dimers (pairs of atoms) and fields of view $\geq 5\mu m$.

Presented in chapter 4, are the results of STM and LEEM experiments (done in collaboration with researchers at Lucent Technologies and Arizona State University respectively), where it is found that for Si grown under biaxial tensile strain on relaxed SiGe substrates, stable wavy-step structures are formed, which evolve into elongated step structures as the local terrace width increases [3]. These structures are postulated to be stabilized by strain-enhanced surface stress-relaxation effects.

Perhaps the most striking observations of this work, are of an equilibrium, quasi-periodic striped-step structure on heavily B-doped Si(001)-(2x1) surfaces annealed in UHV. This system shows a striking transitions in the size and shape of the step
structures as the sample temperature is changed [4]. It is proposed, that these structures result from Marchenko-Alerhand (M-A) stress relaxation effects [1, 2], enhanced by a temperature-dependent surface B segregation. The effect of B segregation is to produce a surface layer under a biaxial tensile strain (like that for Si/SiGe), and measurements reported here also indicate a ~ 12% increase in the surface stress anisotropy ($F_0$) over that for regular Si(001) surfaces. The combined effect of biaxial tensile strain and increased surface stress anisotropy, are argued here to cause the spontaneous formation of steps with very narrow periodicity, resulting in the observed striped surface phase.

The shape of the structures observed for both Si/SiGe(001) and B-doped Si(001), are consistent with recent isotropic elastic calculations of Ebner et al. [5], which show the same progression of step structures from straight—triangular-tiled—striped, as the terrace width, surface stress anisotropy, or biaxial tensile strain is increased.

In addition, LEEM (and STM) have been used to image residual uniaxial strain fields on SiGe(001) films, where a high degree of correlation is found between lateral strain variations (most likely due to sub-surface networks of misfit dislocations) and the surface cross-hatch morphology encountered on strain-relaxed Si$_{0.7}$Ge$_{0.3}$ substrates. This supports the proposal that crosshatch is produced by spatially varying strain fields.

A summary of current findings, as well as a discussion of future directions of experiments and research related to stress relaxation at surfaces, is presented in chapter 5.

The appendices contain detailed equipment drawings of individual STM components, circuit schematics, and computer listings. These sections are intended to provide a readily available source of information to those researchers that will use
the scanning tunneling microscope (and other related equipment) designed and constructed by this author for these experiments.
CHAPTER 2

INTRODUCTION

2.1 Motivation

The technological importance of silicon within the microelectronics industry for use in very large scale integration (VLSI), as well as a substrate for heteroepitaxial growth, has caused it to be one of the most intensely studied materials of all time. The (001) surface of Si is perhaps the most technologically important, being widely used for metal-oxide-semiconductor (MOS) structures. The continued demand for semiconductor devices with progressively smaller dimensions requires an increased understanding and control of surface structures. Much of the rich behavior observed on the Si(001) surface, is related to the anisotropic nature of the (2x1) surface reconstruction, leading to a variety of effects such as anisotropic surface diffusion and anisotropic surface stress. Although there are a wide array of factors which affect the physics at these surfaces, a number of important classes of surface structures which are produced and/or evolve on the Si(001) surface, may be directly influenced by the presence of bulk or surface stress.

In addition to the technical motivation for studying these systems, there is a basic interest in examining the interaction of stress and surface morphology. In essence, strained layer systems provide microscopic laboratories where the influence of surface...
stress/strain on the detailed step structures may be studied, providing an experimental test system to compare with theoretical predictions of stress relaxation at surfaces.

2.2 Si(001)

The surface stress relaxation effects to be discussed in this work, are intimately connected to the crystal structure and reconstruction of the silicon surface. A review of the silicon crystal lattice and the atomic structure of the low-index (001) surface will now be presented. Although both the (001) and (111) surfaces of Si have been heavily studied, this work deals exclusively with the (001) surface, which is used widely for MOS devices within the microelectronics industry.

The bulk structure of silicon is that of diamond, which is face centered cubic (fcc) with a basis of two atoms [6, 7, 8]. Figure 2.1 shows the bulk silicon unit cell, which has a lattice constant of 5.43Å, the length of the cube edge ($a_0$). The complete lattice structure is generated by one fcc lattice at the origin, along with another fcc lattice displaced by ($a_0/4, a_0/4, a_0/4$) (with respect to the coordinate system indicated) from the origin. Throughout this work the standard notation convention [6, 7] is employed, where $(xyz)$ is the plane normal to the $[xyz]$ direction, $\{xyz\}$ is the set of all planes equivalent to $(xyz)$, and $\langle xyz \rangle$ is the set of all directions equivalent to $[xyz]$. The equivalent directions and planes arise due to the cubic symmetry of the silicon lattice.

The {001} planes are the surfaces normal to the <001> directions, and are thus the faces of the unit cell. Examination of figure 2.1 reveals that equivalent (001) lattice planes are separated by $a_0/2 = 2.72\text{Å}$ in the $z$ direction. However the dual basis forms another (001) lattice plane (rotated by $90^\circ$) halfway between these, resulting
Figure 2.1: Crystal lattice structure of silicon. The bulk unit cell is shown, which consists of two interpenetrating fcc lattices. The coordinate system is shown on the right [from reference [8]].
in a monolayer (ML) spacing for the (001) surface of \( a_0/4 = 1.36\text{Å} \). In STM images then, step heights will be integral multiples of 1.36Å.

Referring once again to figure 2.1, we see that the nearest neighbor spacing on the (001) surface, is not the lattice parameter \( a_0 \), but rather the distance corresponding to 1/2 of the face diagonal. The surface lattice parameter \( a \) therefore is given by \( a = \frac{1}{2}(\sqrt{2}a_0) = \frac{3a_0}{\sqrt{2}} = 3.84\text{Å} \). The surface therefore has an atomic site density of 6.78x10^{14}\text{cm}^{-2}.

While in theory it is possible to produce a single perfect surface plane, in reality this situation is not realized. This is because samples are prepared by cutting a wafer from a large silicon ingot and then polishing the surface. This procedure always results in a surface with some misorientation with respect to the [001] direction. This misorientation is commonly referred to as “miscut”, the specification of which might be given as: \textit{Si(001) miscut 0.1° toward (110)}. The miscut necessarily results in the presence of surface steps as indicated in figure 2.2. If the surface normal is miscut by an angle \( \theta \) away from [001], a staircase of steps of height \( h \) results, with an average step separation \( l = h / \tan \theta \). If the miscut angle is in a \{110\} direction and greater than about 2° the surface forms double-height steps, for lower miscut angles single-height steps are observed, even though one double-height step has lower energy than two single-height steps \cite{9}. The reason for this double—single step transition, is itself related to stress relaxation effects and will be discussed in section 2.4.2.
Figure 2.2: Minor misorientation while cutting the wafer from the ingot and polishing results in the formation of surface steps of length $l = h / \tan \theta$. 
2.3 The Si(001)-(2x1) Surface

2.3.1 Reconstruction

The Si(001) surface described above in relation to figure 2.1, is what would result if the crystal was *bulk terminated*. For Si(001) this bulk termination is not observed, but rather a dimer *reconstructed* surface. Much of the rich behavior observed on the Si(001) surface is due to the anisotropic surface stress resulting from the (2x1) dimer reconstruction. In figure 2.3(a) an ideally bulk terminated Si(001) surface is shown. Due to the large number of dangling bonds, the surface undergoes a reconstruction, in which two surface atoms pair, forming “dimers”. These dimers line up in long rows parallel either to the [110] or the [110] crystal directions, as shown in figure 2.3(b). The dimerization of the surface results in an anisotropic surface stress, and causes the steps generated by the surface miscut to have distinct properties. The nature of these surface steps will be the subject of the next section.

2.3.2 Surface Steps on Si(001)

As mentioned earlier the effect of miscut is to introduce steps on the Si(001) surface. Four types of steps are found to occur on vicinal (001) surfaces miscut towards a (110) direction (examples of which may be found in figure 2.4): (1) $S_A$-step which is a single height step (height = 1.36Å for Si) in which the dimers on the upper terrace are parallel to the step edge, (2) $S_B$-step which is also single height, but with the upper terrace dimer row direction perpendicular to the step edge, (3) $D_A$-step which is a double dimer row direction perpendicular to the step edge, and (4) $D_B$-step which is also a double height, but with the dimer rows perpendicular to the step edge on the upper terrace side.
Figure 2.3: Illustration of the formation of the Si(001)-(2x1) surface reconstruction. The "ideal" bulk-terminated surface shown in (a), has a large number of dangling bonds, and may lower its surface energy by the formation of dimers which line up in long dimer rows shown in (b). Two single height surface steps may result, one of which is indicated in (b). The dimer reconstruction results in the dimers being under a larger tensile stress along the dimer bond than along the dimer rows. This \textit{surface stress anisotropy} is schematically indicated by the arrows at the bottom of (b), and it should be noted that the direction of the anisotropy rotates by 90° across a monolayer step.
Figure 2.4: Ball and stick models of the four types of steps studied by Chadi [9]. (a) and (c) show single height $S_A$ and $S_B$ steps respectively. The dimer rows at the upper terrace edge run parallel (perpendicular) to the step edge direction for $S_A$ ($S_B$) steps. Note that the dimer row direction rotates by 90° in crossing the single layer steps. (b) and (d) show double height $D_A$ and $D_B$ steps respectively. The dimer rows at the upper terrace edge run parallel (perpendicular) to the step edge direction for $D_A$ ($D_B$) steps. Note however that the dimer row direction remains unchanged in crossing the double layer steps [From reference [9]].
If the orientation of the miscut (azimuthal misorientation) is not directly towards a (110) direction, the resulting steps are no longer purely “A”-type or “B”-type, but are “mixed”. This means that along a given step edge there are segments of each type of step. For the special case of a single stepped surface with azimuthal miscut 45° away from the [110] direction (i.e., miscut toward [100]), each step has an equal amount of A and B step character.

Calculations by Chadi [9] in 1987 indicated that the step edge energies per unit length of the various configurations in figure 2.4 to be $E_{S_A} = 0.01, E_{D_B} = 0.05, E_{S_B} = 0.15$, and $E_{D_A} = 0.54$ eV/Å. On the basis of these calculations, one would expect to observe surfaces consisting entirely of double steps of type $D_B$, since the energy of a single $D_B$ step is much less than either a single $D_A$ step, or the combined energy of an $S_A$ and $S_B$ step. Single stepped surfaces require both the $S_A$ and $S_B$ steps, collectively having an energy higher than a single $D_B$ step, making the single stepped surface energetically unfavorable. We should point out that although experimentally determined values for the step energies [10, 11] differ from those calculated by Chadi, they follow the same ordering in energy.

While STM observations of the Si(001) surface for large miscuts [12], showed that double height $h = 0.272$ nm steps dominate, with only the low energy $D_B$-step observed. Something quite unexpected is observed for surfaces at lower miscuts. In contrast to the calculations of Chadi [9], as the miscut is lowered, first a mixed phase of $D_B$-steps and $S_A, S_B$-steps, followed by a single-height stepped surface with an alternating staircase of $S_A$ and $S_B$ single-layer (SL) steps were found [13]. Swartzenstruber et al. [13] found that even extended annealing of these lower miscut surfaces did not result in a surface of only $D_B$ steps. This suggested that the existence of the
single layer steps was not just due to kinetic limitations of moving surface adatoms across the now wider terraces. The single—double height step transition, as well as the very presence of $S_A$ and $S_B$ steps in the first place, presented a significant puzzle. We will see shortly that the resolution of this apparent problem comes, when the elastic relaxation of surface stress is taken into account [14, 15, 16].

For the low miscut surface (which will be the only one investigated here), the dimer-row direction rotates by 90° across SL steps [9], the result is that the surface consists of alternating “(2x1)” and “(1x2)” reconstructed terraces across vicinal, singly-stepped Si(001) surfaces [9, 17, 18]. This (2x1) dimer row structure also exists on Ge(001) [19] as well as on Si$_{1-x}$Ge$_x$ (001) alloy surfaces [20, 3].

An atomic resolution STM image of a “regular” Si(001) surface is shown in figure 2.5. The image is shown in “gray-scale”, where higher features appear brighter. The dimer row directions are indicated by the double arrows, and are observed to rotate by 90° across each monolayer step. The $S_A$ steps appear very smooth and relatively straight, while the $S_B$ steps are considerably rougher, and wander (for “regular” silicon) essentially randomly between adjacent $S_A$ steps. The difference in behavior of the two types of single layer steps, may be understood from the difference in relative step edge energies [10]. The $S_B$ step wanders by the formation of kinks, creating short segments of energetically inexpensive $S_A$ step along the side. However, wandering of the $S_A$ step, requires the creation of energetically expensive $S_B$ segments into the side, hence leading to straighter step edges.

At larger length scales, the $S_A$ and $S_B$ steps are observed to form a regular staircase of steps on the surface. Figure 2.6 show a large scale STM scan of a “regular” Si(001) surface. This image is displayed in derivative-mode (DM), where the difference in
Figure 2.5: ~1200 \times 1200 \text{Å}^2 atomic resolution STM scan of a "regular" Si(001)-(2x1) surface. The $S_A$ and $S_B$ steps are indicated, along with the direction of the dimer rows on the terraces. The $S_A$ step is smooth and has relatively few kinks, while the $S_B$ step is randomly rough and has many kinks. Note that the dimer row direction rotates by 90° across the single layer steps [uhv3d25/feb28s9].
height between two adjacent pixels is used. This technique serves to highlight the step edges, and one may view the image as if light were coming from the left at a low angle. The identification of $S_A$ and $S_B$ steps can be done by zooming in to atomic resolution, or we may use the observation that $S_A$ steps are smooth and $S_B$ steps are rough, to make the determination.

Figure 2.6: Large scale (approx. $1.8 \times 1.2 \mu m^2$) derivative-mode (DM) STM scan of the "regular" Si(001)-(2x1) surface. The $S_A$ and $S_B$ are indicated, and for well oriented samples, form a staircase of step across the surface [uhv3d25/feb23s7].
2.4 Stress Relaxation at Step Edges

2.4.1 Theory

Motivated by a desire to understand the experimental observations of Men et al. [21] for Si(001) surface under an applied uniaxial strain (these experiments will be discussed in section 2.4.3), stress relaxation effects at single layer (SL) steps were studied by Alerhand, Vanderbilt, Meade, and Joannopoulos (AVMJ) [2]. The results of the AVMJ calculations not only provided an explanation of Men et al.’s observations, but it also proposed a new class of ordered, self-organized equilibrium step structures on samples with sufficiently wide terraces.

In the early 1980’s Marchenko [1], presented very general theoretical arguments that surfaces for which energetically degenerate phases existed, but with different stress tensors, could lower their elastic energy by the formation of extra “domain boundaries”. Independently of Marchenko, Alerhand et al. [2] in 1988 made predictions for stress relaxation effects specifically for the Si(001)-(2x1) surface, that were quite successful in explaining the observation of Men et al. [21] reported earlier that year. In addition to explaining the observations of Men et al., Alerhand et al. made the further prediction that an initially flat Si(001)-(2x1) surface would undergo a transition to a novel “stripe”-like surface phase [2, 22], consisting of alternating up-and-down steps separating orthogonal “stress domains” [1].

To understand the origin of the stress relaxation effects postulated by AVMJ, it must be recognized that an important consequence of the dimer reconstruction at the Si(001) surface, is that the surface stress tensor is highly anisotropic, the dimers being under a larger tensile stress along the dimer bond direction $\sigma_\parallel$ than along the dimer row $\sigma_\perp$ [23, 24, 25, 26, 27]. This surface stress anisotropy $F_0 = (\sigma_\parallel - \sigma_\perp)$ is
schematically indicated in figure 2.3(b). Since the dimer row direction rotates by 90° across (SL) steps, the surface stress anisotropy rotates by 90° as well, and produces a force imbalance (often referred to as a “force monopole” (FM)) across a SL step. For an $S_A$ step the force monopole points toward the upper terrace edge as indicated in figure 2.7(a). The existence of FM’s at step edges can lead to the formation of elastic stress domains as discussed below.

The origin of the stress relaxation mechanism, is schematically illustrated in figure 2.7 (a), where a cross-section through an $S_A$ step is shown before relaxation occurs, essentially representing a perfectly rigid crystal. Due to the 90° rotation of the surface stress anisotropy in crossing the step edge, there is a larger stress (force) directed away from the step and toward the upper terrace side, and a smaller stress directed away from the step edge and toward the lower terrace side. The result is that there exists a net stress or force monopole (FM) on the step directed toward the upper terrace side.

If the step edge is now allowed to relax under this FM, the step edge may deform elastically, as illustrated in figure 2.7(b). As the step edge relaxes, the net stress across the step decreases, being balanced by a build-up of strain in the substrate, until the net stress across the step becomes zero. The result is a lowering of the stress energy. It is the stress relaxation at step edges, and the resulting interaction of strain fields that provide the explanation of the surface domain population changes observed by Men et al. for surfaces under an applied uniaxial strain (see section 2.4.3).

Alerhand went on to point out, that if the stress energy reduction due to step edge relaxation is more than the energy cost associated with the creation of the step, it would then be energetically favorable to create extra steps so that relaxation may
Figure 2.7: Cross-section of an $S_A$-step (a) before relaxation occurs, and (b) after relaxation. Because the dimer row direction rotates in crossing the step edge, there is a larger surface stress $\sigma_{||}$ at the upper terrace edge than at the lower terrace edge $\sigma_{\perp}$. If the energy associated with relaxation is more than that required to create the step edge, spontaneous step creation may take place.
occur. This process would then lead to \textit{spontaneous step generation}, as indicated in figure 2.8.

Alerhand \textit{et al.} [2], have considered the energetics for the transition from a perfectly flat surface to that of a striped surface shown in figure 2.8. Since for the striped surface one type of step is sufficient, only the lower energy $S_A$ step is considered here. The change in energy per unit length ($\Delta E_{\text{length}}$) from the AVMJ calculation is:

$$\Delta E_{\text{length}} = E_A - \alpha (F_0)^2 \ln \left( \frac{l}{\pi a} \right) \quad \text{(2.1)}$$

Where $E_A$ is the $S_A$ step creation energy per unit length, $\alpha = \frac{1 - \nu}{2\pi \mu}$ ($\nu$ and $\mu$ are the Poisson ratio and shear modulus respectively) is an elastic constant, $F_0 = (\sigma_\parallel - \sigma_\perp)$ is the surface stress anisotropy, $a$ is a microscopic cutoff length (on the order of the surface lattice constant), and $l$ is the stripe width. In equation 2.1 the first term represents the local step creation energy per unit length, while the second term represents a reduction in energy per unit length of a step of width $l$, due to elastic relaxation.

It is evident from equation 2.1 that $\Delta E_{\text{length}}$ decreases for increasing $l$ and becomes zero when $E_A = \alpha (F_0)^2 \ln \left( \frac{l}{\pi a} \right)$, or

$$l_* = \pi a \exp \left( \frac{E_A}{\alpha (F_0)^2} \right) \quad \text{(2.2)}$$

For $l < l_*$, there is not sufficient relaxation to justify the creation of a new step. On the other hand, for $l > l_*$, step creation follow by relaxation can take place. At first it would seem that the system will prefer large $l$, but as $l$ increases the number of stress relaxing step edges per unit area decreases (see figure 2.8). It is therefore most appropriate to look at the change in energy per unit area rather than the energy per unit length.
Figure 2.8: The work of Alerhand et al. [3], suggested that an initially flat surface of the Si(001) surface could lower its energy by the formation of extra up-down $S_A$-steps.
The change in energy per unit area \( \Delta E_{\text{area}} \) from the AVMJ calculation is:

\[
\Delta E = \frac{E_A}{l} - \frac{\alpha (F_0)^2}{l} \ln \left( \frac{l}{\pi a} \right) \tag{2.3}
\]

If \( l \) is varied, \( \Delta E_{\text{area}} \) is minimized for a particular \( l = L_0 \) given by:

\[
L_0 = \pi a \exp \left[ 1 + \frac{E_A}{\alpha (F_0)^2} \right] \tag{2.4}
\]

So for terraces wider than \( L_* \), there exists an optimal stripe width \( L_0 \) often referred to as the Alerhand critical terrace width, since these terraces are predicted to spontaneously form extra domain walls (steps).

### 2.4.2 The Double—Single Step Transition

We are now in a position to resolve the apparent problem of the double—single step transition occurring on the Si(001) surface. The difference in energy/length for the double and single stepped surface, where there is either one \( D_B \) step or a pair of \( S_A \) and \( S_B \) steps per length \( 2l \), is given by [14, 15, 16].

\[
\Delta E_{\text{length}}^{\text{double/single}} = [E_A + E_B - 2\alpha (F_0)^2 \ln \left( \frac{l}{\pi a} \right)] - [E_{D_B}] \tag{2.5}
\]

There is no stress relaxation term for the double step, since there is no rotation of the surface stress anisotropy in going across the \( D_B \) step, and therefore no force monopole [14, 15, 16].

Equation 2.5 may be re-written as

\[
\Delta E_{\text{length}}^{\text{double/single}} = [E_A + E_B - E_{D_B}] - 2\alpha (F_0)^2 \ln \left( \frac{l}{\pi a} \right) = E_A' - 2\alpha (F_0)^2 \ln \left( \frac{l}{\pi a} \right) \tag{2.6}
\]

and has the same form as equation 2.1. So even though the double steps are energetically less costly than the two single steps, the surface becomes unstable to the formation of single layer steps for sufficiently large \( l \). The stress relaxation term causes the single stepped surface to be the lower energy configuration for low miscut.
2.4.3 Effects of Applied Uniaxial Strain

As mentioned earlier, a very interesting strain related effect was demonstrated in some beautiful low-energy electron diffraction (LEED) experiments of Men et al. [21], Webb et al. [17], and subsequent STM experiments of Swartzentruber et al. [18]. These authors showed that annealing a Si(001) surface under an applied uniaxial strain, alters the relative widths (and hence areas) of the (2x1) and (1x2) terraces.

Men et al. [21] observed, that when a uniaxial strain was applied to a Si(001) sample, the surface domain areas changed such that terraces with the dimer row direction parallel (perpendicular) to the applied tensile (compressive) strain were favored. Men et al. surmised that this change in relative domain areas, was due to a lifting of the energy degeneracy between the (2x1) and (1x2) domains. However, they were unable to explain the observation that when the strain was removed, the surface returned to equal domain populations with the same relaxation time as when the strain was applied.

If the strain simply lifted the degeneracy of the two domain types, the only driving force for the domains to return to equal areas when the strain is removed, would be a purely entropic one [28]. Therefore one would expect different relaxation constants for the application and removal of strain. The fact that the relaxation constants were the same suggested that the same energetics were involved in both processes. It was the experimental observations of Men et al. [21], that led to Alerhand et al.'s [2] theoretical calculations, which provided an explanation of the observed experimental effects of uniaxial strain.
Alerhand [2] considered the case of an uniaxial strain ($\epsilon^{ext}$) applied along a symmetry direction (with respect to the surface stress anisotropy) which breaks the orientation degeneracy of the (2x1) and (1x2) domains, with the result that one domain population grows at the expense of the other. For consistency, we will refer to domains in which the dimer bond direction is parallel (perpendicular) to the applied uniaxial strain direction as 2x1 (1x2). Values of $\epsilon^{ext} > 0$ correspond to tensile strain causing the 2x1 regions to shrink and the 1x2 regions to grow, while values of $\epsilon^{ext} < 0$ corresponding to compressive strain cause the 1x2 regions to shrink and the 2x1 regions to grow. By generalizing the earlier presented stripe phase (which had equal domain populations) to a structure with alternating domains of unequal widths $(1 + p)l$ and $(1 - p)l$ (where the area fraction $p$ is defined by $\frac{(A_{2x1} - A_{1x2})}{(A_{2x1} + A_{1x2})}$, and $A_\xi$ are the terrace areas) allows equation 2.3 to be generalized to [2],

\[ \Delta E = \frac{1}{2} \epsilon^{ext} p F_0 + \frac{E_A}{l} - \frac{\alpha (F_0)^2}{l} \ln \left( \frac{l}{\pi a} \cos \left( \frac{\pi}{2} p \right) \right). \]  

Depending on the surface considered, $\Delta E$ may be minimized by varying $p$ while holding $l$ constant or by varying both $p$ and $l$, corresponding to local or global equilibrium respectively. The local equilibrium case should apply when there are steps imposed by the surface miscut or where surface kinetics permit equilibration only on the local scale, while the global equilibrium case should apply to low miscut surfaces where extra step generation takes place (see figure 2.9).

It is the combination of the external strain term coupled with stress relaxation effects at step edges, that determines the energetics for both the application and removal of uniaxial strain. Since the energetics are the same in each process, the relaxation mechanism and relaxation times, should be the same in each case as well.
Figure 2.9: Alerhand et al. [2] refer to "local" and "global" equilibrium at surfaces. Local equilibrium applies when the sample miscut results in surface steps which determine the adjacent step spacing (miscut fixes $l$). Global equilibrium applies when the sample miscut is zero (or sufficiently small) that spontaneous step generation takes place ($l$ is free to vary).
It should be noted that the elastic interaction between steps, leads to an effective "repulsive force" between the steps. This may be seen most easily by considering equation 2.7, with $\epsilon^{\text{eff}} = 0$. The energy is clearly minimized when $p = 0$, corresponding to equally spaced steps. Any variation in $p$ away from $p = 0$ leads to an increase in energy, and hence an effective repulsive force between steps. It is the elastic step repulsion which inhibits the surface from becoming uni-domain for arbitrarily small applied external strains.

2.4.4 Observations

The AVMJ theory advanced the understanding of several experimental observations for the Si(001) surface that were not well understood at the time, chiefly the double—single height step transition [29, 13], and the change in surface domain structure under an applied external uniaxial strain [28, 21, 17]. However, the predicted spontaneous generation of steps was not observed for the Si(001) surface.

There have been reports of highly elongated islands and step "fingers" on Si(001)-(2x1) following epitaxial growth [30], but these elongated structures have generally been attributed to non-equilibrium kinetic effects present during growth [31]. The observation of striped structures for O adsorbed on Cu(110) surfaces, while initially thought to result from stress-relaxation effects, were later proposed by Vanderbilt [32] to be spontaneous domain generation in this case not due to differences in surface stress, but rather due to work function variations.

In 1991 Tong and Bennett [29] carried out reflection high-energy electron diffraction (RHEED) on Si(001) samples with a range of miscuts. Their experiments indicated a large scale meander to the $S_B$ steps (larger than that expected from entropy
aione), although with a high degree of disorder. Tong and Bennett speculated that the meandering of the $S_B$ was mediated by strain effects. Although this work suggested the possibility of step meandering, no direct observations of steps were made.

In 1992 Tromp and Reuter [33] and Tersoff and Pehlke [34] published papers dealing with wavy steps on Si(001). Using LEEM, Tromp and Reuter observed a co-undulation of $S_A$ and $S_B$ steps on Si(001) surfaces miscut by $\sim 0.1^\circ$ (direction of miscut unspecified). Tersoff and Pehlke proposed that these observations were due to a "sinusoidal step instability", in which steps undulating together, effectively bring (2x1) and (1x2) domains closer (in a direction perpendicular to the global step edge direction), thus allowing for stress relaxation effects. This situation results in an Alerhand-type structure, but without the need for the creation of extra steps. The work of Tersoff and Pehlke, assumed that $S_A$ and $S_B$ steps undulated equally. This situation is rather artificial, because undulation of the $S_A$ step (requiring creation of high energy $S_B$ segments) is much less energetically favorable than undulation of the $S_B$ step (requiring creation of only low energy $S_A$ segments). Tersoff and Pehlke even point out that $S_B$ steps should have a larger undulation amplitude than $S_A$ steps. Although stress relaxation effects could be involved, there was still no evidence of the stripe-like surface phase postulated by AVMJ.

Zandvliet et al. [35, 36] provided some of the first evidence, at that time, that a periodic undulation of the $S_B$ step edge may exist on these surfaces. In their STM work on low miscut Si(001) surfaces, Zandvliet et al. studied the deviation-deviation correlation function $G(\tau)$ for $S_B$ steps, and found a decrease in $G(\tau)$ for step positions separated by 40-60 dimers (i.e., 150-230Å).
Although each of the preceding groups, reported evidence for stable “undulations” of step edges on slightly miscut Si(001) surfaces [29, 33, 34, 36] and pointed out that such undulations should be stabilized by surface stress effects similar to those considered by Alerhand et al. [2], the true stripe phase on Si(001) was not observed.

The first clear evidence of an equilibrium striped phase on the Si(001) surface, was provided by our earlier work [3], where we observed stripe-like structures and quasi-periodic “wavy” steps on Si(001)-(2x1) surfaces subjected to biaxial tensile stress from a relaxed Si$_{0.7}$Ge$_{0.3}$ substrate, and argued that the Marchenko-Alerhand (M-A) stress relaxation effects were enhanced by the applied tensile stress. More recently we [4] have made conclusive observations of an equilibrium M-A striped phase completely covering heavily B-doped Si(001)-(2x1) surfaces, with a characteristic stripe width $L_0$ as small as $\sim 10\text{nm}$. We have suggested [4] that the temperature dependent step shape transition observed in this system is due to biaxial tensile stress [37, 38, 39, 40] in the surface layer caused by temperature dependent B surface segregation [41, 42], an increase in the surface stress anisotropy (and possibly a decrease in the $S_A$ step-edge creation energy).

2.5 Stress Relaxation of 3-Dimensional Structures

It is clear from the preceding discussion, that stress relaxation effects can have significant effects on the properties of surface steps. In addition to influencing the evolution of the “2-D” surface, stress relaxation can also lead to some interesting “3-D” structural effects.
The importance of stress and strain during growth, has been appreciated for some time. There are three well known growth modes for epitaxial films: Frank-van der Merwe (FvdM), Volmer-Weber (VW), and Stranski-Krastinov (SK) [43, 44]. In the FvdM mode, the epitaxial film grows layer by layer to any thickness (generally observed for homoepitaxial growth). For VW growth the interface energy between the film and substrate is so high that the film does not "wet" the substrate and the film grows entirely by 3-D islanding (generally observed for heteroepitaxy with large lattice mismatch). The SK growth mode is essentially intermediate between FvdM and VW, in that the film initially forms a 2-D wetting layer (usually for 2-4 monolayers), but due to increasing strain energy in the surface layer, 3-D islanding begins. In general the islanding in SK growth begins as a result of strain relieving dislocations which form in the strained layer. The surface near the dislocations is (partially) relaxed, and therefore has a more favorable lattice constant for subsequent grown, and growth occurs preferentially there.

Recently however, Eaglesham and Cerullo [44] observed the coherent growth of 3-D islands of germanium on silicon. The term coherent, refers to the fact that transmission electron microscopy (TEM) performed during the initial stages of growth, showed no sign of dislocations, even though 3-D islands were observed. Eaglesham and Cerullo [44] proposed that this coherent growth occurs since a 3-D structure, even in the absence of dislocations, can lower its energy through strain relaxation at the island walls. Figure 2.10 schematically shows the 3-D relaxation process. For a film grown under compression, the three dimensional structure shown in (a) may relax elastically outward (b), lowering the strain energy. Since the island is at least
partially strain relieved by this deformation, the lattice constant on the island is better matched for subsequent growth, and it becomes a preferred site for future growth.

Snyder et al. [45, 46, 47] have observed evidence of this effect in InGaAs grown on GaAs. This system also forms strain relieved 3-D coherent islands. It should be noted that the strain is only partially relieved in these structures, so that extended growth results in increased strain energy, which ultimately leads to the introduction of strain relieving dislocations.

A further example of the importance of strain in thin film growth is provided by some recent work by Shiryaev et al. [48], where nucleation and growth of Ge clusters was found to take place preferentially along misfit dislocation lines on a strain relaxed SiGe substrate. This preferential nucleation in a regular arrangement, suggests a possible way of patterning an array of quantum dots on a surface.

Finally we note that at the μm length scale, stress induced changes in surface morphology continue to be of interest particularly for strain-relaxed SiGe films grown on Si(001) substrates, which have potential applications in high-speed electronic devices [49, 50, 51] and as lattice matched substrates for GaAs epitaxy. While great advances have been made in recent years in improving the bulk material quality of these relaxed SiGe(001) films [49, 50, 52, 53], these systems continue to exhibit a characteristic surface morphology known as “cross-hatch” [49, 50, 52, 53], consisting of μm-wide raised or depressed “bands” of material running along orthogonal ⟨110⟩ crystal directions. The resulting large-scale surface roughening may ultimately limit the utility of these materials in device structures. While it is known that there exists a close relationship between cross-hatch and the introduction of ⟨110⟩ oriented misfit
Figure 2.10: Formation of 3-dimensional islands during growth of lattice mismatched materials, allow for the reduction of strain even in the absence of strain-relieving dislocations. The side walls of a compressively strained island (such as Ge on Si) (a), are free to relax outward (b), reducing the strain. The surface of the island then has a larger effective lattice spacing, and acts as a preferred site for future growth.
dislocations in sub-surface layers [49, 50, 52, 53], the exact mechanisms connecting dislocations and the cross-hatch pattern are still not well established. Fitzgerald et al. [49] suggested that networks of misfit dislocations produce spatially-varying strain fields at the surface, which may locally modify growth rates during deposition, a position supported by our recent strain field imaging study [54].

2.6 Summary

Strain and stress on Si(001) surfaces, produce a wide range of interesting morphological effects, ranging from influencing step shapes to 3-D islanding. So much of the rich behavior at the Si(001) surface, arises because of the anisotropic surface stress from the dimer reconstruction. It is the stress relaxation at step edges (driven by the presence of a force monopole at single layer steps) [2], that provides an explanation of the single—double height step transition [14], the response of surface domains to an applied uniaxial strain [21], and recent observations of striped step structures on surfaces with a biaxial tensile strain [3, 4]. In addition to the “2-D” effects at step edges, coherent 3-D island occurring during growth [44, 46], may also be understood in terms of relaxation of island sidewalls to accommodate lattice mismatch.

2.7 Recent Results

Scanning tunneling microscopy (STM) and low-energy electron microscopy (LEEM) experiments of strained Si grown on relaxed SiGe(001) layers [3, 20, 54], as well as heavily B-doped Si(001) surfaces [4] are presented in this work. The step shapes observed on Si(001) surfaces observed by STM and LEEM, have been explained in
terms of a change in the relative terrace spacing with respect to fixed Alerhand critical terrace width $L_0$ for Si/SiGe, and as a change of $L_0$ with respect to fixed terrace width for B-doped Si. The evolution of steps on these surfaces is found to be from straight, to triangular tiled, to striped, and is consistent with recent isotropic elastic calculations by Ebner et al. [5] which include the effects of finite surface misorientation on the stress relaxation effects originally presented by Alerhand et al. [2]. New results of STM measurements of surface domain population changes in response to an applied uniaxial strain, indicate that the surface stress anisotropy ($F_q$) on heavily B-doped Si(001) surfaces is increased over that of "regular" Si(001) surfaces by about 12%. This increased $F_q$ and the approximate biaxial tensile strain ($\epsilon$), are used to make estimates based on the AVMJ theory of $L_0$, for comparison with experiment. In addition, large scale LEEM measurements at the surface of relaxed SiGe(001) structures show lateral variations in uniaxial surface strain over $\mu m$ sized areas, which are highly correlated with the "cross-hatch" morphology observed on graded layer systems.
CHAPTER 3

EXPERIMENTAL APPARATUS AND PROCEDURE

3.1 Overview

Since the invention of the Scanning Tunneling Microscope (STM) in 1982 by Binnig and Rohrer[55, 56], many useful references for STM have been published (STM)[57, 58, 59, 60], so only a brief overview will be presented here. STM is a technique for imaging conducting and semiconducting surfaces with atomic (or near atomic) resolution. To obtain these images, a sharp metallic “tip” is brought very close (but not touching) to the surface to be investigated. There is a (vacuum) barrier to electron flow from the tip to the sample, but for sufficiently narrow gaps between tip and sample there is an overlap of electronic wavefunctions. If a voltage is applied between the tip and sample, electrons may quantum mechanically tunnel through the barrier. Under “normal” operating conditions the tip is at ground potential and the sample bias $V_s = \pm$ a few volts. For $V_s > 0$ electrons tunnel from the tip to the sample (empty state imaging), while for $V_s < 0$ electrons tunnel from the sample to the tip (filled state imaging). The high spatial resolution of STM arises because the tunnel current is exponentially sensitive to the separation between the tip and the sample, and that the tunnel current density is highly localized to just a few atoms at the end of the tunneling tip (see figure 3.1).
3.2 STM

The STM used in this work was designed and constructed by this author, and will be described in some detail here. For drawings of individual STM and related components, the reader is referred to section A.

3.2.1 Scanner

The heart of the STM is the scanner assembly, which in this case consists of a piezoelectric tube as shown in figure 3.2. The scanner tube is extruded Lead Zirconia Titanate (usually referred to as PZT), having a solid inner electrode and an outer electrode sectioned into 4 quadrants. A positive potential applied from an outer quadrant to the inner electrode, causes the piezoelectric material to shrink in the direction perpendicular to the applied field and parallel to the axis of the tube. Applying equal and opposite voltages to the \(+x\) and \(-x\) (\(+y\) and \(-y\)) outer electrodes, result in
motion in the vertical (horizontal) plane (see figure 3.3). By adjusting the potential applied to the inner $z$ electrode, the overall length of the tube may be adjusted. The result is that an object (in this case the tunneling tip) located at the end of the tube may be accurately positioned in $x$, $y$, and $z$. The intrinsic piezo gains for the microscope are shown in table 3.1). For voltage sources with a range of 400 V and regulated to within 2 mV, the effective lateral ($x,y$) scan range is $\sim 6.7 \mu m$ with a resolution of $0.3 \AA$, and the effective vertical ($z$) scan range is $\sim 1.3 \mu m$ with a resolution of $0.06 \AA$.

<table>
<thead>
<tr>
<th>Piezo (Axis)</th>
<th>$x$</th>
<th>$y$</th>
<th>$z$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Intrinsic Gain $\AA/V$</td>
<td>168</td>
<td>168</td>
<td>31.7</td>
</tr>
</tbody>
</table>

Table 3.1: Intrinsic piezo gains for "UHV3".
Figure 3.3: Illustration of bending of piezo tube, when equal and opposite voltages are applied to opposite pairs of electrodes [from Ref. [60]].
The voltages on the outer electrodes are slowly changed to raster the tip over the sample surface, while the inner $z$ voltage is adjusted (with a feedback loop described in section 3.3.3 to keep the tunnel current constant. The feedback signal to the $z$ piezo is digitized and recorded by the computer and plotted as a function of lateral position of the tip. This is schematically indicated in figure 3.4.

Figure 3.4: Schematic representation of STM. The tunneling tip is positioned in $x$ and $y$ by voltages applied to piezo elements $P_x$ and $P_y$, while a feedback loop maintains a constant tunnel current by adjusting the tip’s height ($z$) above the surface. In this way contours of constant current density are produced. These contours may represent structural features (such as the surface step shown at $A$), and/or electronic features (such as the defect shown at $B$. [from Ref. [57]].
Capacitive Coupling to Tip

<table>
<thead>
<tr>
<th></th>
<th>+x</th>
<th>-x</th>
<th>+y</th>
<th>-y</th>
<th>z</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sapphire Disk Floating (\times 10^{-13} F)</td>
<td>3.04</td>
<td>3.54</td>
<td>4.02</td>
<td>2.71</td>
<td>0.157</td>
</tr>
<tr>
<td>Sapphire Disk Grounded (\times 10^{-15} F)</td>
<td>1.31</td>
<td>1.56</td>
<td>1.26</td>
<td>2.44</td>
<td>0.171</td>
</tr>
</tbody>
</table>

Table 3.2: Tip to piezo capacitive coupling. Grounding the sapphire disk results in two orders of magnitude decrease in capacitive coupling.

A possible problem exists, having the tip (measuring a small current) in close proximity to the changing potentials on the piezo electrodes. Capacitive coupling between the tip and the piezo can add an extraneous signal to the tunnel current. In order to reduce this problem, a ground plane has been placed between the scanner tube and the tip (see figure 3.5). The ground plane is a sapphire disk 3/8” in diameter on which is evaporated Cr followed by Au. This metal layer is then connected to the system ground. The measured capacitances for the disk grounded and floating are shown in table 3.2. Clearly the disk ground plane results in a dramatic lowering of the capacitive coupling of the tip and piezo. The piezo voltages are further shielded since the scanner tube is recessed into the invar scanner mounting block.

Connections to the scanner tube were made by soldering 3 mil Cu wires to each of the four outer electrodes and to the center electrode. The preferred way to make these solder connections is as follows. The area where the wire is to be attached is first tinned by dipping the end of the Sn/Ag solder wire in a standard paste solder flux. The solder/flux is applied to the tube with a soldering iron. The tube is then cleaned to remove all flux residue. Then a length of tinned/cleaned Cu wire is touched to the solder pad under the application of heat from the soldering iron. Care must be taken at each point in this process to avoid over heating the piezo material and causing it to “de-pole”.

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Figure 3.5: Top view of the custom designed and constructed STM used in these experiments [photo courtesy of Ben Kaczer].
It should be noted that "regular" 60/40—Sn/Pb eutectic solder is not used to make these connections, since Pb has a significant vapor pressure particularly at bake-out temperatures (see reference [61] for vapor pressure curves of the elements). The vapor pressure of Pb can therefore increase the system pressure, and also presents a possible source of contamination to our sample surfaces.

3.2.2 Tip Stage (z-axis)

The tip stage forms another critical part of the STM. Since the scanner has a limited range of at most several μm’s, a means of bringing the tip within several thousand angstroms of the surface (in a controlled manner) is required. To achieve this fine approach, the scanner assembly is mounted on an invar tip slider (see figure 3.5) which rests on 3 stainless steel ball bearings riding in “V”-groves on a cantilevered invar tip stage. The 3 ball bearings establish a stable plane for the slider and prevent rocking. The slider is held in firm contact with the tip stage, by 3 neodymium-iron-boron high energy density magnets (available from Edmund Scientific). These magnets act to essentially increase the effective spring constant of the mechanical loop, moving mechanical resonances to higher frequencies.

A Burleigh inchworm motor having a range of 5mm and a step size of the order of a nm, is fixed to the tip stage and coupled to the slider (see figure 3.5). The action of the inchworm motor will be described in section 3.4.1. The design is such that the tip slider can be pushed forward by the inchworm until it is in the proper position, then the inchworm may be withdrawn several steps without moving the slider, allowing the inchworm drive to be decoupled from the slider stage.
The tip stage itself rests along its front edge on two ball bearings riding in a V-groove on the STM stage base plate. The third point of the tip stage plane, is defined by a V-groove on the underside of the tip stage that rests on top of the vertical height adjust screw ball bearing.

### 3.2.3 Coarse Height Adjust (y-axis)

The coarse height adjust screw, permits the tip stage to perform another one of its key functions, providing coarse positioning of the tip in the vertical direction. This is accomplished by a fine thread screw which is used to raise and lower the back of the tip stage (farthest from the tip). The screw is turned by a rotational feed-through on the bottom of the 8" STM support flange, and is arranged so that the screw can be decoupled from the rotation mechanism.

### 3.2.4 Sample Stage (x-axis)

The sample stage serves three main purposes, (1) to provide a simple means of changing samples, (2) make electrical contact to the sample for sample bias and sample heating (hot STM), and (3) to allow the sample to be positioned in the horizontal direction (x) in front of the tip. The sample stage carries four electrical contacts, forming essentially a socket into which sample blocks may be “plugged”. The front two contacts are wired with two 5 mil Cu wires each and may carry approximately 0.25 Amps for sample heating. The back two contacts are each wired with a single 3 mil Cu wire, and are not currently used (these contacts were added to increase the versatility of the instrument in the future).

The sample stage, like the tip slider, is moved by an inchworm motor (see figure 3.5). The motor housing is clamped to the STM stage base plate, while the inner
motor push rod is attached at either end to a frame that surrounds the sample stage. An alumina ball at the center of the front edge of the frame, electrically isolates the frame from the STM stage base plate. This frame may be moved from side to side, pushing/pulling the stage. Some play is intentionally left between the frame and the sample stage, so that the motor may be reversed a few steps, thus decoupling the inchworm/frame from the sample stage. The resistance of an insulated wire attached to the frame, may be checked to determine if the stage is coupled or not.

The sample stage itself rests on three ball bearings, and the contact force with which it is held to the base plate is increased by three niodmium iron boron magnets recessed into the under side of the sample stage.

Sample Blocks

All samples are mounted on specially designed sample blocks, which can be transferred throughout the system. The sample blocks (see figures A.20—A.23) consist of a macor block, with a specially designed (and uniquely numbered) Invar pick-up bar across the top. The pick-up bar guides the STM and main grabber jaw-pieces into the proper location, providing a secure grip on the sample block during transfers. On either side of the back of the sample block, are Invar contacts which retain Mo contact clips and the W filament used for sample block outgassing and tip cleaning. On either side of the front of the sample block there are Ta (and in some cases Mo) bars retaining the Mo contact clips, and which act additionally as mounting bars for the sample. The sample block plugs directly in to the mating socket on the STM sample stage, where electrical contacts are automatically made to the sample upon insertion.
The sample rests across the ends of the front Ta bars, and (for all sample blocks other than those used for uniaxial strain experiments) is held in place by Ta sample clips spot welded to the Ta bars. A Mo heat shield covers the majority of the macor surface on the front side of the sample blocks. The heat shield reduces radiation heating of the macor surface, and protects the sample from outgassing products which might cause contamination of the sample surface.

The sample blocks used for uniaxial strain experiments use the same standard sample block design, but the sample mounting method is somewhat different. Along the right hand side a Ta clamping bar rigidly clamps the sample to the Ta sample bar (see figure A.33), while along the left hand side a Ta clip spotwelded to a flexible Cu braid, provides a (nearly) stress free contact to the left side of the sample. This arrangement allows the sample to be strained by loading the free end of the sample, creating a cantilevered beam arrangement.

3.2.5 STM Scan Stage Base Plate

The STM scan stage is a 0.5" thick x 4.6" diameter invar disk, to which is mounted the tip stage and the sample stage. Near the back of the base plate is a large macor block (see figure 3.5), which provides a surface for diffuse scattering of light onto the sample, improving the ability to view the tip — sample relative position during tip approach.

During sample/tip exchange, the STM stage base plate is clamped down to the outer support ring by a liner motion feed-through coming up from the 8" STM support flange. The system is arranged so that the clamp mechanism can be fully disengaged from the STM stage base plate during scanning.
3.2.6 Vibration Isolation

The exponential sensitivity of the tunnel current on tip — sample separation, makes the STM highly sensitive to external vibrations. Therefore, several levels of vibration isolation have been implemented. The first and most important vibration isolation mechanism, is to isolate the STM scan stage as much as possible from the outside world. This is achieved by (1) making all electrical connection to the STM stage with 3 or 5 mil wires, and (2) supporting the STM stage base plate by hanging it from three #027 viton O-rings. The second level of isolation consists of a flexible metal bellows connecting the STM and main chambers. Next the entire STM system is mounted on a TMC vibration isolation air table. Finally the entire experimental setup is located in the basement of the building to further isolate the system from machinery in other parts of the building.

The O-ring suspension used in this system, provides such effective vibration isolation, that in practice the air table was seldom raised. In fact atomic resolution images were obtained with this instrument even with the air table lowered and the external turbo pump connected and running. A question that one may ask, is "why not always raise the air table to provide some addition vibration isolation?". The answer is, that although the air table provides additional isolation, it also makes the system more susceptible to damage if the table is jarred while in the raised position. For this reason it was generally preferred to operate the system with the table lowered.

3.2.7 STM Wiring

In this section the physical layout of the wiring for the STM is described. A sketch of the 8" STM support flange as viewed from above is shown in figure 3.6.
Connectors J1 and J2 are each 10-pin connectors, while J3, J4, and J5 are shielded BNC type connectors. J1 carries all connections for both of the inchworm motors. J2 carries connections for the rear sample stage contacts, all voltages for the scanner, and the test connection for checking coupling of the sample stage to the inchworm frame. J3 and J4 are the front sample stage contacts, and J5 is the tip wire. Details of the connections are shown in table 3.3. All connections except for connections to the scanner tube (as discussed earlier) are spot welded connections.

![Top View of UHV3 STM 8" Base Flange](image)

Figure 3.6: Top view of the UHV3 STM 8" base flange. Connectors J1 thru J5 carry all signals to/from the STM. J1 and J2 are each 10-pin connectors, while J3, J4, and J5 are BNC-type shielded coaxial connectors.

The layout of the wiring within the STM was carefully planned to reduce undesirable cross talking of the various signal lines. The most important consideration was
Table 3.3: Detailed wiring layout for UHV3, the STM designed and constructed for these experiments.

<table>
<thead>
<tr>
<th>Wire Label</th>
<th>Connection</th>
</tr>
</thead>
<tbody>
<tr>
<td>J1 — A</td>
<td>z inchworm clamp 3</td>
</tr>
<tr>
<td>J1 — B</td>
<td>z inchworm center 2</td>
</tr>
<tr>
<td>J1 — C</td>
<td>z inchworm clamp 1</td>
</tr>
<tr>
<td>J1 — D</td>
<td>z inchworm clearance</td>
</tr>
<tr>
<td>J1 — E</td>
<td>y inchworm clamp 3</td>
</tr>
<tr>
<td>J1 — F</td>
<td>y inchworm center 2</td>
</tr>
<tr>
<td>J1 — G</td>
<td>y inchworm clamp 1</td>
</tr>
<tr>
<td>J1 — H</td>
<td>y inchworm clearance</td>
</tr>
<tr>
<td>J1 — I</td>
<td>No connection</td>
</tr>
<tr>
<td>J1 — J</td>
<td>No connection</td>
</tr>
<tr>
<td>J2 — A</td>
<td>Right hand rear sample stage</td>
</tr>
<tr>
<td>J2 — B</td>
<td>Left hand rear sample stage</td>
</tr>
<tr>
<td>J2 — C</td>
<td>+x Scanner</td>
</tr>
<tr>
<td>J2 — D</td>
<td>-x Scanner</td>
</tr>
<tr>
<td>J2 — E</td>
<td>+y Scanner</td>
</tr>
<tr>
<td>J2 — F</td>
<td>-y Scanner</td>
</tr>
<tr>
<td>J2 — G</td>
<td>z Scanner</td>
</tr>
<tr>
<td>J2 — H</td>
<td>Stage Frame Contact (couple test)</td>
</tr>
<tr>
<td>J2 — I</td>
<td>No Connection</td>
</tr>
<tr>
<td>J2 — J</td>
<td>No Connection</td>
</tr>
<tr>
<td>J3 — A</td>
<td>Right hand front sample stage</td>
</tr>
<tr>
<td>J4 — A</td>
<td>Left hand front sample stage</td>
</tr>
<tr>
<td>J5 — A</td>
<td>Tip wire</td>
</tr>
</tbody>
</table>

to locate the scanner lead wires (which carry time varying signals during scanning) as far away as possible from the tip wire. This reduces the amount of capacitive pickup in the tunnel current. In general all signal lines other than the tip are routed near the back side of the STM, while the tip wire is routed along the front side.
<table>
<thead>
<tr>
<th>Connection Pair</th>
<th>Coupling Capacitance</th>
</tr>
</thead>
<tbody>
<tr>
<td>J1A — J1D</td>
<td>4.10 nF</td>
</tr>
<tr>
<td>J1B — J1D</td>
<td>4.76 nF</td>
</tr>
<tr>
<td>J1C — J1D</td>
<td>4.21 nF</td>
</tr>
<tr>
<td>J1A — J1C</td>
<td>6.90 pF</td>
</tr>
<tr>
<td>J1B — J1C</td>
<td>29.3 pF</td>
</tr>
<tr>
<td>J1A — J1B</td>
<td>29.7 pF</td>
</tr>
<tr>
<td>J1E — J1H</td>
<td>4.79 nF</td>
</tr>
<tr>
<td>J1F — J1H</td>
<td>3.75 nF</td>
</tr>
<tr>
<td>J1G — J1H</td>
<td>4.66 nF</td>
</tr>
<tr>
<td>J1E — J1G</td>
<td>17.1 pF</td>
</tr>
<tr>
<td>J1F — J1G</td>
<td>92.3 pF</td>
</tr>
<tr>
<td>J1E — J1F</td>
<td>99.0 pF</td>
</tr>
<tr>
<td>J2C — J2G</td>
<td>1.77 nF</td>
</tr>
<tr>
<td>J2D — J2G</td>
<td>1.26 nF</td>
</tr>
<tr>
<td>J2E — J2G</td>
<td>1.92 nF</td>
</tr>
<tr>
<td>J2F — J2G</td>
<td>1.60 nF</td>
</tr>
</tbody>
</table>

Table 3.4: Connection capacitances for troubleshooting (all capacitances measured at 150 Hz).

All of the wiring to/from the STM stage base plate, is anchored on insulated stainless steel springs to the STM support ring before being spot welded to the electrical feed-throughs on the 8" STM base flange. This arrangement helps to damp any mechanical vibrations transmitted along the signal wiring.

### 3.2.8 Electrical Characteristics / Troubleshooting

Table 3.4 lists the measured capacitances between various connections, and may useful in troubleshooting the instrument. These capacitances provide rapid verification of connections to various elements of the microscope, as well as provide a check for cracked piezo elements in the inchworm motors.
It often proves useful to test these capacitances (1) before attaching the STM flange to the vacuum chamber, (2) after the STM is inserted but before tightening the flange, (3) after tightening and roughing, and finally (4) after baking is complete.

3.3 Scanning Electronics

There are a number of electronics modules that are required to operate the STM. For the most part all of these modules were designed and constructed in the Physics department’s Electronics Shop.

3.3.1 Tip Bias

Under normal tunneling conditions the sample is biased with respect to the tip (a virtual ground) by a precision voltage source which can be continuously varied from -10V to +10V. The bias circuitry is part of the feedback control module which will be discussed in section 3.3.3, and the schematic may be found in figure B.6.

3.3.2 Tip Pre-amplifier

The tunnel current used in these experiments was generally $\approx 1nA$. Because this very small current is used to adjust the feedback of the tip — sample distance, it is important that it be as noise-free as possible. To reduce the coupling of extraneous noise pick-up, it is desirable to amplify the tunnel current as close to the source (tip) as is possible, and then to use this amplified signal as the input to the feedback control unit. The ideal configuration would have a current amplifier inside the vacuum chamber, but placing such an amplifier in the UHV environment presents numerous problems. As a compromise, a current pre-amplifier with a gain of $10^6V/A$ is placed
just outside the vacuum wall. This amplifier produces a 100mV output when 1nA is applied to the input. A schematic of this low-noise pre-amplifier is shown in figure B.5.

3.3.3 Feedback Control Module

The feedback control module takes as its input, the output of the tip pre-amplifier (tunnel current), and produces an output signal which is proportional to the difference between the absolute value of the tunnel current and a user defined set-point current. The feedback system is a simple proportional feedback circuit with adjustable gain and time constant. It is desirable to operate the system with the gain set as high as possible, as this results in minimal error signal and thus reproduces surface features as accurately as possible. Too high of gain setting however, can result in self oscillation. Likewise it is desirable to keep the time constant of the feedback loop as small as possible, since this allows the system to slew at the highest possible rate, thus tracking sharp surface features accurately. But here again there is a trade-off since too short of a time constant again leads to self oscillation.

The output of the feedback control module is used as input for (1) the $z$ piezo driver, (2) the computer for digitization (after some signal conditioning), and (3) an oscilloscope to monitor the tunnel barrier and scanning process.

A separate unit in the feedback control module produces the sample bias voltage ranging from -10 to +10 volts.

3.3.4 Scanner HV Piezo Driver

The intrinsic piezo gains are relatively small (see table 3.1) so, in order to scan areas of several $\mu$m's, piezo voltages of the order of $10^2$V are required. Since the
general signal levels used within the control electronics are only of the order 10V a separate driver stage is required to provide the piezo voltages.

The Scanner Piezo Driver consists of 3 separate low-noise, high-voltage, dual-output, bi-polar, adjustable gain amplifier modules — one for each of the piezo degrees of freedom. The input for the z module is the feedback signal. The inputs for x and y each come from the computer’s digital-to-analog converter (ADC output is -10 to +10V).

An offset control on each module adds a user defined reference to each input, allowing the x and y center to be adjusted, and allowing the z piezo to be adjusted so that the error signal in the feedback loop is brought to zero.

Additionally each module also has a “MOD” (modulation) input that can be used to perturb the tip position for dithering or, more usually, to jog the tip to induce a tip change.

### 3.3.5 Feedback Signal Conditioner (SR560)

The output of the feedback control module (used to control the z height of the tip) is the signal that will be digitized and used to form the image of the surface. However, before digitizing the signal is pre-conditioned by the Stanford Research Model SR560. This unit provides a variety of filter setting to allow low, high, and bandpass filters of a wide range of cutoff frequencies, as well as variable gain. Since the DC component of the z signal as well as surface features up to the digitization rate are of interest, the SR560 is configured for low-pass filtering with a cutoff frequency of about 1kHz. The gain is adjusted to ensure that (nearly) the full dynamic range of the analog-to-digital converter is used. This provides the highest resolution in the recorded data.
The SR560 also serves to buffer the input to the analog-to-digital converter (the output of the SR560 saturates at ~ ±10V) protecting the ADC circuitry from possible overvoltage conditions.

3.3.6 Computer Controlled Analog-Digital Converter

During scanning all data to/from the computer is handled by a Quatech DAQ-16 16-bit data acquisition adapter. The conditioned z feedback signal is sent to one of the eight analog-to-digital converter (ADC) inputs (channel 1) which digitizes signals from -10 to +10V. The 16-bit resolution results in a measurement accuracy of 20V/2^{16}bit = 0.3mV/bit. For a z piezo gain of 31.7Å/V (see table 3.1), this is equivalent to ~ 0.01Å/bit.

3.3.7 Computer Controlled Digital-Analog Converter

In addition to the ADC, the DAQ-16 also provides two 12-bit digital-to-analog converters (DAC) (-5 to +5 volt output) which are programmed by the computer to control the x and y position of the tip. The resolution of the analog output, is therefore 10V/2^{12}bit = 2.4mV/bit. For x and y piezo gains of 168Å/V (see table 3.1), this produces a scan range of 1680Å with an uncertainty equivalent to ~ 0.41Å/bit, when the DAC output is passed through the scanner high-voltage piezo driver (see section 3.3.4), with unity gain (G = 1). The dynamic range may be extended by operating the piezo driver with larger gains, but with a corresponding increase in the uncertainty of the tip position due to the discrete nature of the DAC output.

The fact that the output of the DAC is discrete and not continuous, leads to some interesting (and undesirable) effects if the scan size and number of acquired pixels are not commensurate with the underlying resolution of the DAC. The result of this
incommensurate condition, is that some data points request an output from the DAC that is in the "gap" between allowed output values (fractional bit), and is therefore rounded to the nearest allowed value (nearest whole bit). This condition leads to the appearance of vertical bars in the acquired image. Proper selection of scan size and number of pixels can eliminate this problem. The "recipe" for "good" scan sizes and number of pixels is that

$$S_{\text{scan}} = \left[ \frac{N}{2^{12}} \right] \cdot g \cdot p \cdot (10V) = \left[ \frac{N}{4096} \right] \cdot g \cdot p \cdot (10V)$$

(3.1)

where $S_{\text{scan}}$ is the scan size (in Å), $g$ is the piezo gain (in Å/V), $p$ is the number of pixels, and $N$ is an even integer.

3.4 Approach Electronics

As mentioned earlier, the range of the tip scanner is limited, so a means of bringing the tip in close proximity to the sample is required. This task is accomplished by the approach electronics and the tip inchworm motor.

3.4.1 Inchworm Motor Electronics

The inchworm motor electronics are used to both coarsely move the tip (and sample) forward and backward to the position desired, as well as to advance the tip one step forward and then to probe forward with the scanner while checking for tunnel current. If no tunnel current is detected this process is repeated until tunneling is reached. The entire approach process is automated with the personal computer, by way of the General Purpose Interface Bus (GPIB). A listing of the approach program 'DJAPP5M.IBW' may be found in section C.1.
Inchworm GPIB Controller

The inchworm is controlled by sending simple GPIB commands to the inchworm controller. The controller has 6 transistor-transistor-logic (TTL) digital outputs, three of which are used (along with the inchworm HV driver) to control the clamping sequence of the inchworm motor to produce a step, and one which controls (by way of the feedback control module) retraction of the tip during a step. The remaining two TTL outputs are currently not used.

The size of an individual step is adjusted by a digital-to-analog converter.

Another section of this unit switches the output from the inchworm driver between either the tip or sample inchworm motors.

The sequence required to make one step of the inchworm motor is illustrated in figure 3.7 below. The inchworm has two piezoelectric clamps (clamp 1 and clamp 3, one at either end) which can contract circumferentially about the core, and one piezoelectric element (center 2) that can be contracted (and/or extended) in a direction along the axis of the core. The sequence may be repeated as necessary to move the core (relative to the exterior housing) over distances of several mm’s, or the direction of motion reversed simply by reversing the clamping order. The size of the step is controlled by adjusting the voltage on the center 2 piezo, and step sizes of the order 1nm may be obtained.

Inchworm HV Driver

The inchworm motor being a piezoelectric device much like the scanner, requires operating voltages higher than typical signal levels, therefore the inchworm also needs a separate high voltage driver module.
Figure 3.7: Schematic of the clamping sequence used to move the inchworm motor [from Ref. [60]].
The inchworm driver consists of four separate variable gain switchable (unipolar) amplifier channels. The channels for clamp 1 and clamp 3 are switched between either 0V or 600V by the digital output from the inchworm controller, while the channel for center 2 is switched between 0V and a level determined by the inchworm controller DAC output. The fourth channel is used to apply a voltage to the inchworm motor's clearance electrode, which essentially sets the ground level reference for the other piezo electrodes. This voltage is generally fixed at 200V, and aids the release of the end clamps.

**Feedback Control — Tip Retract**

In the approach process, the tip is retracted by applying a large negative voltage to the $z$ scanner piezo, then a forward step (of size smaller than the piezo range) is taken. The tip is then probed forward to test for tunneling. The tip retract/extend function is implemented by a TTL output from the inchworm controller which connects to a TTL input on the back of the feedback control module. When the input is pulled to ground the feedback control calls for negative absolute current, thus fully retracting the tip.

**Tunnel Current Detection (HP54601A)**

When the tip is probed forward to check for tunneling, the computer queries the oscilloscope via the GPIB bus and reads back the tunnel current, to see if it exceeds a user-defined threshold level.
3.5 Auxiliary Electronics

Aside from the electronic components listed above, which form the minimum necessary to approach and scan, there are several other units that are routinely used.

3.5.1 Tilt Compensator

Ideally the tip would be positioned with the $z$ axis normal to the sample surface, but in practice this is seldom the case. As a result, large excursions of the $z$ piezo are required as the tip is moved in $x$ and $y$, to track the surface plane. These $z$ excursions are undesirable for the following reasons: (1) the feedback voltage can easily exceed $\pm 10V$ for a $1\mu m$ scan (corresponding to a misalignment of $< 4$ deg), thus exceeding the maximum digitization voltage of the DAQ-16 ADC, causing an out-of-range condition, (2) the feedback controller is forced to continuously adjust the $z$ signal, and under some circumstances may exceed the slew rate of the circuit so that the tip no longer tracks the surface of the sample accurately, and (3) the feedback signal resulting from this misalignment contains no information of interest about the sample morphology.

To resolve this problem a tilt compensator is used to remove the the effects of the sample/tip misalignment. To do this, a fraction of each of the $x$ and $y$ piezo voltages ($V_x$, $V_y$) is added to the true $z$ signal ($V_z$), to form the new $z'$ signal ($V_{z'}$) which is sent to the $z$ scanner piezo. Mathematically, $V_{z'} = T_x V_x + T_y V_y + V_z$, where $-1 < T_z < +1$. A schematic of the tilt compensator may be found in figure B.7.
3.5.2 Sweep Rotator

It is often desirable to rotate the fast and slow scan directions to an arbitrary orientation with respect to the sample surface. To accomplish this operation, a sweep rotator was constructed which takes the original $x$ and $y$ signals from the computer, and applies a rotation matrix (through angle $\theta$) to generate a rotated signal set $x' = x \cos \theta - y \sin \theta$ and $y' = x \sin \theta + y \cos \theta$. A schematic of the sweep rotator is shown in figure B.8.

3.5.3 Tip Conditioning Electronics

The quality of STM images is highly sensitive to the atomic and electronic structure of the tip. In order to obtain the highest quality images, and to correct tip changes that occur spontaneously during scanning, deliberate alterations of the tip are frequently required. Two techniques for altering the tip are discussed below.

Tip Bias Ramp

Nearly all of the STM images in this work were taken with sample biases of $\approx -1.85V$. To improve the performance of the tip (i.e., to improve resolution) a technique commonly employed is to ramp the sample bias to $\approx -10V$ while scanning, hold it there briefly, and then lower the bias slowly to the original bias level. It is believed that the high field which exists during this process, causes some of the atoms from the shank of the tip to migrate to very end of the tip, causing it to elongate [60] as illustrated in figure 3.8.

The bias ramp may be performed manually, but since it is used so frequently, a separate tip bias ramp generator was designed. This unit outputs an up/down ramp
Figure 3.8: In order to obtain high resolution STM scans, it is often necessary to modify the tip slightly. One technique commonly used is to ramp the sample bias, perhaps causing atoms to migrate to the very end of the tip. [from Ref. [60]].

with variable amplitude, rate, and polarity. A schematic of the bias ramp generator may be found in figure B.1.

Tip $z$ Pulse

Another means of altering the tip properties, is to cause the tip to undergo a controlled collision with the sample [60]. To do this a square wave pulse of adjustable amplitude and duration is sent from the tip pulse generator (see figure B.3) to the modulation input of the $z$ piezo driver. This pulse causes the tip to be modified (perhaps by picking up a cluster of Si atoms) as suggested in figure 3.9.
Figure 3.9: In order to obtain high resolution STM scans, it is often necessary to modify the tip slightly. One technique commonly used is to pulse the $z$ position of the tip slightly, resulting in modification of the end of the tip. [from Ref. [60]].

3.6 Vacuum System

A photograph of the entire STM system, is shown in figure 3.10, and a schematic overview in figure 3.11. The system consists of a series of three connected chambers that can be isolated from one another by UHV gate valves.

3.6.1 Introduction Chamber

The introduction chamber allows samples and tips to be introduced and extracted from the main chamber by way of a magnetically coupled linear motion feed-through and sample introduction boat. This chamber is essentially a 4-1/2" 5-way cross. The top 4-1/2" port is removed to place samples/tips on the introduction boat, and is equipped with a viewport to allow easy inspection of boat contents. The right hand side 2-3/4" flange carries the magnetically coupled linear feed-through (introduction
Figure 3.10: Photograph of the vacuum system (UHV3) designed and constructed for these experiments.
Figure 3.11: Schematic overview of the vacuum system (UHV3) designed and constructed for these experiments.
transport arm) on the end of which is attached the introduction boat. The left hand side 2-3/4” flange attaches to the “air-lock gate valve”. The front and back 2-3/4” flanges are used for the roughing valve and convectron pressure gauge respectively.

**Turbomolecular Pump**

The introduction chamber is ‘roughed’ to a pressure of $\approx 10^{-8}$T by a 200l/s Balzers turbomolecular pump (TPU240), and is generally pre-baked (using heating tapes) without any samples/tips, to remove water vapor and other contaminants. The pre-baked and cooled introduction chamber and turbo, are then vented with dry nitrogen through the vent valve and roughing valve. Samples and tips are introduced/extracted with a continuous purge of dry nitrogen through the airlock. The introduction chamber is then re-roughed cold, to reduce the amount of residual hydrocarbon contamination [62] to samples inside.

**Sample/Tip Introduction Boat**

The sample/tip introduction boat has been designed to carry up to 2 sample blocks and up to 6 tips into or out of the system at a time. The sample blocks rest in recessed positions in an aluminum tray. The tips holders (with their integral magnets) are placed on short segments of aluminum-nickel-cobalt (AlNiCo) magnets held in Al recesses on the introduction boat. The existence of the AlNiCo magnets is ‘historical’, since the original tip holders had no magnets of their own (they were simply made of Invar). In any re-design of these items, one should consider replacing the magnetic holder on the boat, with Invar blocks that could carry even more tips.

It should also be noted that the current introduction boat has been milled to a beveled shape along the back side. This modification is required for the boat to be
able to clear the cylindrical mirror analyzer, which when installed extends well into the main chamber.

### 3.6.2 Main — Preparation Chamber

The main chamber consists of a Perkin-Elmer TNBX pump well with an upper bell jar originally designed for Auger electron and X-ray photoelectron spectroscopy. This chamber was modified to serve as a sample preparation chamber, where samples and tips could undergo outgassing and cleaning prior to being transferred to the STM for imaging. The chamber is equipped with several sample/tip manipulators (at present 3), as well as multiple viewports to aid sample/tip transfers, and to permit temperature measurements with an infrared pyrometer.

The main chamber is isolated from the introduction chamber by a 3-3/8" Huntington UHV gate valve, and from the STM chamber by an 4-1/2" VAT gate valve. This provides a means for maintaining the levels of cleanliness required for these surface studies from the relatively dirty environment of the introduction chamber, to the clean environment of the main chamber, and finally the ultra-clean environment of the STM chamber.

**Main Chamber Grabber**

Samples and tips are transferred between the introduction boat, main chamber sample/tip storage tray, and the STM transport boat, by a grabber assembly. The main chamber grabber consists of a claw mechanism with specially designed regions to engage the pick-up bar on top of the sample blocks, and a tweezer-like area for grabbing the tip mounts. The grabber is mounted on a precision \(x - y - z\) rotatable manipulator, with an extra linear degree of freedom used to open and close the grabber.
jaws. The grabber has been designed with the jaw pieces located off the axis of the main chamber, to allow access to the left and right sides of the sample storage tray, and to provide the correct standoff distance of a *grabbed* sample in front of the CMA for AES.

The grabber jaws collectively carry 4 high current contacts that engage the sample block contacts. This allows for sample/filament outgassing, as well as sample flashing to remove surface oxides and to clean the surface. The lead wires to the grabber consist of 4 pairs of solid Cu wires, each 25 mils in diameter, and are insulated from each other and the system ground by Nextel ceramic fiber sleeves (available from Ω-Engineering). The solid Cu wires are then connected to flexible Cu braids which are finally connected to the Cu-Be strips that make contact to the Mo sample contacts. The wiring system of the main grabber is capable of carrying sustained currents of 15 amperes to/from the sample.

**Sample/Tip Storage Tray**

A sample/tip storage tray mounted on a linear motion feed-through in the main chamber, allows 3 samples and 4 tips to be stored for eventual transfer to the STM or for extraction to the introduction chamber. The design of this *main tray* is much like that of the introduction boat, with recesses for the sample blocks (2 on the left hand side, 1 on the right hand side), and AlNiCo magnets to accept the tips.

The magnet position on the main tray furthest from the front, has been specially designed as a tip cleaning position. An extra long AlNiCo magnet mounted in the macor block attached to the main tray, is electrically connected to a separate feed-through to provide the bias needed during tip cleaning.
Additionally, this main tray has been modified to carry a Ta post that can be brought into contact with one side of a sample, providing a means of straining samples in situ. This will be discussed in more detail in section 4.2.1.

Auger Electron Spectroscopy (AES)

The main chamber has the capability to carry out Auger Electron Spectroscopy (AES) on samples used for STM. The only part of the AES system that must be installed in the vacuum system, is the cylindrical mirror analyzer (CMA). The CMA contains an integral electron gun to excite the Auger process, an electrostatic electron energy analyzer, and either a Faraday cup or electron multiplier for signal detection.

The remaining parts of the AES system are all electronics equipment which are outside of the vacuum wall. While the AES system does function (chemical peaks in the derivative spectrum appear in the correct positions), the output signal is noisy. Even using lock-in detection, the sensitivity of the current system is less than optimal. Much of the problem likely lies with the external electronics, which are over 20 years old, and contain several high-voltage batteries that introduce time-varying voltages on various control grids. While chemical identification can be done with this system, studies requiring quantitative analysis were done elsewhere. The infrequent use of the AES system prompted its substitution with a getter pump module which is described in section 3.6.2.

For completeness, a listing of the computer program written to acquire data from the system with the addition of a GPIB controlled lock-in amplifier, is included in section C.2.
Ion Pump

Under normal operating conditions, the main chamber is maintained at a pressure of \( \leq 2.5 \times 10^{-10} \) Torr, by a 200\( \text{m}^3 \text{s}^{-1} \) ion pump. For more information on ion pumps refer to references [61, 63]. The ion pump can be isolated from the upper section of the main chamber by a 12" poppet valve.

Within the ion pump well, there are 4 titanium sublimator filaments and a cryo-shield. The Ti sublimator is used periodically, and pumps by subliming a fresh reactive Ti film onto the surface of the cryo-shield, providing a gettering surface for reactive gases. Although the cryo-shield is designed to be operated at liquid nitrogen temperatures, in practice the sublimator is only run for several minutes at a time and cooling of the cryo-shield with a compressed air supply was found to be sufficient. Cooling to lower temperatures would likely improve the efficiency of this pumping system, but the added complexity of flowing liquid nitrogen through the cryo-shield seems to outweigh the potential benefits.

Getter Pump

The STM designed for this work was carefully planned to allow as much flexibility for future experiments as possible. Recently, three electron beam evaporation sources were added to the main chamber for deposition of metals for ballistic-electron emission microscopy (BEEM), and for co-deposition of materials such as Si and Ge. The evaporators add considerably to the total material enclosed in the vacuum vessel, and more importantly to the total internal surface area. The increased gas load, coupled with that fact that the evaporators placement restricts the pumping orifice of the ion pump, prompted the addition of another pump. To increase the pumping speed in
the main chamber, the Cylindrical Mirror Analyzer (CMA) used for Auger Electron Spectroscopy (AES) was removed (8" port), and a custom designed getter pumping system designed by this author was attached. The activated getter module, pumps hydrogen by volume desolution into the active material, and pumps reactive gases by adsorption at the surface. The active getter is #C-500-MK2/St707 from SAES Getters, consisting of a Zr-V-Fe powder-alloy on a constantan strip. The quoted pumping speed of this getter, is 1200l/s for H₂ (5400torr • l capacity) and 500l/s for CO (8torr • l capacity).

Regeneration of the pump is required whenever the volume of the getter is saturated with hydrogen, or the surface is fully reacted. The regeneration process consists of heating the getter under vacuum, which is achieved by passing a DC current through the heater cartridge (Ta filament) which runs along the axis of the getter cartridge. Heating the getter causes the dissolved hydrogen to be released, and the adsorbed reactive species to diffuse down into the interior of the active getter, thus exposing a fresh (reactive) surface layer.

To date the getter has only been generated one time (initial generation), but future regenerations should likely follow this procedure: (1) with system pre-baked and at (roughly) room temperature, begin (continue) turbo pumping system, (2) heat getter cartridge to ≈ 550°C for 2 hours, (3) cease heating and close getter gate valve when the pressure reaches ≈ 10⁻⁸T, (4) complete system bake-out, and (5) open getter gave valve. Data from the initial generation is shown in table 3.5. Detailed drawings of the getter pump module may be found in figures A.25—A.32.
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Table 3.5: Parameters from first generation of getter pump module on April 1, 1995.

Residual Gas Analyzer (RGA)

The main chamber is equipped with a quadrupole residual gas analyzer (RGA) (Ametek Dycor Model MA200M) with an analyzer range of 0 to 200 amu. The RGA is used during leak testing, sample outgassing, and to monitor general system cleanliness. The RGA is interfaced to the system personal computer via an RS-232 serial connection, and the "DyLink" executable program can be configured to automatically transfer the current mass spectra to an Excel worksheet. A representative background scan and a scan taken during sample block outgassing are shown in figures 3.12 and 3.13 respectively.
Figure 3.12: Residual gas analysis of system "background". The largest gas component is H\(_2\) at mass number 2.
Figure 3.13: Residual gas analysis during sample block outgas. The hydrogen peak is still dominant, but H$_2$O (at mass numbers 18, 17, 16, 15, etc.) is now a major partial pressure component.
3.6.3 STM Chamber

The STM chamber is basically an 8" six-way cross. The back port connects by way of an 8" VAT gate valve to the STM's own ion pump, which maintains the STM chamber at pressures $\leq 1 \times 10^{-10}$ Torr. On the left hand side is attached the rack-inpinion transporter, while on the right hand side a flexible bellows attaches the STM to the STM-main gate valve. The flange containing the actual STM bolts onto the STM chamber from below and is fully visible through the 8" viewport on the front. Sample/tip manipulations are carried out by the STM grabber attached to the top flange.

In addition four 2-3/4" ports also access the STM chamber. Two are used for viewports providing alternate viewing angles into the STM chamber, while the other (lower) two are used for a nude ionization gauge tube and pump-out port respectively.

Ion Pump

The STM chamber is pumped primarily by a 200L/s Perkin-Elmer ion pump (references [61, 63] provide useful background on UHV technology). Just as the main chamber ion pump, this pump is also equipped with a Ti sublimator and cryo-shield system. To protect the STM from Ti spray during sublimation, a baffle was added to the end of the cryo-shield to prevent direct line-of-sight from the Ti filaments to the STM. The baffle is positioned in such a way as to limit the restriction of the conductance into the pump.

STM Chamber Grabber

The STM grabber has much the same function as the main grabber described above, with the exception that the STM grabber does not carry any electrical contacts.
to the sample. The STM grabber is also positioned off the axis of the STM chamber, which allows the grabber to access both the sample stage for sample exchange, as well as to perform tip exchange from the end of the scanner.

Sample/Tip Boat

The rack-in-pinion transporter carries a sample boat with 2 recesses for sample blocks, and 4 AlNiCo magnets positions for holding tips. As mentioned earlier, the magnet positions are historical and could be replaced with Invar blocks to allow more flexibility in tip storage.

3.7 Tip Preparation

The tips used for this study were primarily custom fabricated from tungsten wire using an electrochemical etch procedure explained below.

3.7.1 Tip Etching

A length of tungsten (W) wire 10 mils in diameter is rinsed with ethanol followed by water, and then a long section (approx. 4cm) is pre-cleaned by electrochemically etching for about 30 seconds. The etching is carried out in a beaker lined with a stainless steel sleeve (see figure 3.14(a)) and filled with a 1 molar NaOH solution. A current is passed from the tungsten wire (+) to the sleeve (-), through the electrolyte solution (NaOH), by a constant voltage source which monitors the etch current. A schematic of the power supply used is shown in figure B.4.

The current flow produces W ions at the anode which results in erosion of the anode surface. The W ions combine with hydroxy ions and precipitate from the solution, while hydrogen is evolved at the cathode [64]. After pre-cleaning, the W
wire is removed from the etcher, rinsed with water, ethanol, and again with water. At this point the W wire is returned to the etcher where care is taken to suspend it perpendicularly to the liquid surface, with about 2mm of wire extending below the liquid surface. Due to the meniscus that forms where the wire penetrates the surface, there results a non-uniform current distribution at the wire surface causing the wire to be etched to a sharp point (see figure 3.14(b)). When the tip is fully etched the portion of the W wire below the meniscus drops off, and the tip-etcher halts current flow. The fully etched tip is finally rinsed with water followed by ethanol, and gently blown dry with nitrogen.

3.7.2 Tip Mounting

As etched tips are mounted in specially designed Invar tip holders (see figure A.24) fitted with set screws, and are transferred into the UHV chamber through the introduction chamber load-lock assembly.
3.7.3 Tip Cleaning

Prior to scanning, tips are cleaned by electron bombardment heating [65] in the main/preparation chamber. A schematic of the setup is shown in figure 3.15. The tip is placed on an insulated magnetic holder, positioned within a few mm's of a tungsten filament, and floated at +250V above the chamber/filament ground. The filament is slowly heated while monitoring the current flow through the tip, until electron emission is detected. The filament current is then adjusted for 2 mA of electron bombardment for a duration of 2 minutes. Tips prepared in this way, generally provide very hard tunnel barriers, although further conditioning is often required to obtain dimer resolution of the Si(001) surface.

It should be noted that if rather than floating the tip above ground, the filament is floated below ground, electron emission still takes place from the filament. However,
in this case the emitted electrons are accelerated to all chamber surfaces, so the tip bombardment current density is decreased, and the pressure increased due to electron stimulated desorption from the chamber walls. For these reasons, this configuration in not recommended for tip cleaning.

3.8 Sample Preparation

Preparation of samples is an important issue for surface studies, since improper techniques can result in surfaces with an excessive number of pinning sites or metallic contamination.

Samples are prepared by first cutting the appropriate sample wafer to size using a diamond tipped scribe. The optimal sample size is ≈5mm wide by 20mm long, although samples narrower than 5mm may be used. In addition, a specially designed sample block has been constructed that can accommodate samples with lengths as short as 8mm.

The as cut samples are then pre-cleaned by one of the following techniques, prior to being placed in the introduction chamber for transfer into the system.

3.8.1 Sample Cleaning (Conventional)

For conventional Si samples, a very simple ex situ clean is generally sufficient. The sample is rinsed with reagent grade methanol (Me-OH), and immediately blown dry with high-purity dry nitrogen (N₂).

3.8.2 Sample Cleaning (Si/Si₁₋₂Ge₂)

Although a considerable amount of work has gone into finding efficient means of cleaning Si[66, 13, 67] (and for that matter Ge[68, 69]), very little work has been done
to study cleaning of SiGe alloy films and strained Si layers grown on relaxed SiGe substrates. As will be discussed later, the techniques used for cleaning Si for STM studies, cannot be directly applied to the SiGe samples, so two alternative ex situ cleaning techniques were employed.

P-clean (Piranha-clean)

The first cleaning procedure evaluated for SiGe, used a standard, wet-chemical "p-clean" process to remove surface organics and grow a clean oxide film. The p-clean procedure was as follows:

- 2 minute immersion in a H$_2$SO$_4$:H$_2$O$_2$ (5:3) solution,
- de-ionized (DI) water rinse,
- 1 minute in an HF:H$_2$O (1:10) solution,
- DI water rinse,
- 2 minutes in a fresh H$_2$SO$_4$:H$_2$O$_2$ (5:3) solution,
- DI water rinse (3 times), and
- nitrogen gas blow-dry.

This wet chemical cleaning procedure is similar to that developed by Ishizaka and Shiraki [66] (often referred to as a Shiraki clean), which is commonly used to clean pure Si.

UVO-clean

In an alternate cleaning procedure for SiGe, samples were exposed for 90 minutes at room temperature to a Hg-UV lamp in a commercial (UVO) cleaner (Jelight Model
42) (located in Smith 3076 — Technical Operations Laboratory), under a continuous 1 SCFH flow of O₂ gas.

3.8.3 Sample Outgas and Oxide Removal

Samples blocks were passed into the UHV system through a pre-baked airlock [62] and outgassed, by heating the W filament at ~ 4.5A and resistively heating the sample to the desired outgas temperature (~ 350°C—~ 750°C), for 6–12 hours.

After outgassing and allowing the sample blocks to cool for several hours, the protective surface oxide layer is removed by rapidly heating the sample to temperatures in the range ~ 1025°C—~ 1250°C, depending on the sample.

Sample temperatures were measured by an infrared pyrometer whose measured value was adjusted for attenuation of the system viewports, by a calibration curve [70] derived from eutectic points of Au, Al, and Ag on Si(111), and external thermocouple calibrations performed by J. Seiple [70].
CHAPTER 4

RESULTS

4.1 Si/SiGe(001)

The microscope constructed was used first to study the surfaces of SiGe alloy films, and in particular Si grown under a biaxial tensile surface strain on relaxed SiGe layers. The interest in studying the surfaces of these systems, resulted from both a fundamental interest in studying the effects of strain on surface morphology [71, 50], as well as a technological interest stemming from the high channel mobility of strained channel Si-MOSFET [51, 49] devices.

4.1.1 SiGe Sample Geometry

All of the SiGe samples used for this work, were made at Lucent Technologies Bell Laboratories by molecular beam epitaxy (MBE). Three types of samples were studied (the basic sample geometry is shown in figure 4.1: (1) Si epi-layers (5 nm thick) grown under tensile strain on flattened, relaxed Si$_{1-x}$Ge$_x$ buffer layers [49] with $x = 0.25$ or 0.30, (2) fully relaxed ex-situ polished $[72]$ Si$_{0.7}$Ge$_{0.3}$ films, and (3) fully relaxed, ex-situ polished Si$_{0.3}$Ge$_{0.8}$ films. The relaxed Si$_{1-x}$Ge$_x$ films were deposited on compositionally-graded Si$_{1-y}$Ge$_y$ layers on Si(001) substrates, with Ge fraction $y$ graded from 0 to $x$ using procedures which relieve lattice strain via controlled
Figure 4.1: Schematic diagram of the structure of the strained Si samples used in these experiments. A graded layer starting at zero Ge content is grown with progressively increased Ge content, until the final composition is reached. This layer allows for the controlled introduction of dislocations. A thick uniform buffer layer with a lattice constant larger than Si is then grown, and the sample flattened by an *ex situ* chemimechanical polish. Then another SiGe buffer layer is grown on the flattened surface. Finally a thin Si cap layer is grown, which adopts the larger SiGe underlayer lattice constant, and is thus under a biaxial tensile strain.
introduction of misfit dislocations (figure 4.2 illustrates a dislocation in an ideal cubic lattice) during growth [49]. The strained Si cap layers were grown on relaxed Si\textsubscript{1-\textit{x}}Ge\textsubscript{\textit{x}} buffer layers, which in turn were grown on relaxed, flattened Si\textsubscript{1-\textit{y}}Ge\textsubscript{\textit{y}} graded layers.

4.1.2 Sample Cleaning

Cleaning procedures for Si [66, 13, 67] and Ge [68, 69] surfaces have been investigated for many years, due in large part to their importance for fundamental
surface science studies as well as technological applications. In contrast, little work has been done to study cleaning of $\text{Si}_{1-x}\text{Ge}_x$ alloy films and strained-layer films on SiGe substrates. With the rapid development of SiGe heterostructure materials in recent years, there is now an increasing need to develop and evaluate new cleaning procedures for these materials as well. From a fundamental viewpoint, we had considerable interest in studying the effects of strain on the structural properties of $\text{Si}_{1-x}\text{Ge}_x$ surfaces. From a technological viewpoint, we note that a procedure for producing flat, strain-relaxed SiGe substrates has recently been developed [72], which involves an ex-situ chemi-mechanical polish followed by a surface clean and deposition of a $\text{Si}_{1-x}\text{Ge}_x$ buffer layer. Since the quality of films grown on the chemi-mechanically polished substrates, depends critically on the cleanliness of the surface, it is essential to develop an effective method of cleaning the flattened substrates, which result in clean surfaces with low densities of residual "particulates".

Ultra-high vacuum scanning tunneling microscopy (UHV STM) was used to evaluate two cleaning procedures for strained Si(001) films grown on relaxed $\text{Si}_{1-x}\text{Ge}_x$ buffer layers, relaxed $\text{Si}_{1-x}\text{Ge}_x$ films grown on Si(001) substrates, and Si(001) substrates. We have found that ex-situ ultra-violet ozone (UVO) enhanced surface oxidation followed by in-situ desorption of the oxide film at moderate temperatures ($\sim 1025^\circ\text{C}$) is a simple, effective cleaning procedure for SiGe films with a wide range of Ge content, which consistently produces surfaces with a low residual density of surface particulates. An alternate method using standard wet-chemical treatments resulted in surfaces with a (generally) higher particulate density, and was sensitive to the purity of the chemicals and de-ionized (DI) water used in the cleaning process. The UVO procedure also results in few surface particulates on pure Si(001) substrates
(often less than 1 per 25\(\mu m^2\)), and hence should be considered as a lower temperature alternative to the standard 1200°C Si(001) clean [13, 67] commonly used to produce Si surfaces with low particulate density.

Two sample cleaning procedures were evaluated. The first used a standard, wet-chemical “p-clean” process to remove surface organics and grow a clean oxide film. The p-clean procedure used: (1) 2 minute immersion in a \(\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2\) (5:3) solution, (2) de-ionized (DI) water rinse, (3) 1 minute in an \(\text{HF}:\text{H}_2\text{O}\) (1:10) solution, (4) DI water rinse, (5) 2 minutes in a fresh \(\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2\) (5:3) solution, (6) DI water rinse (3 times), and (7) nitrogen gas blow-dry. In the second procedure, samples were exposed for 90 minutes at room temperature to a Hg-UV lamp in a commercial (UVO) cleaner (Jelight Model 42), under a continuous 1 SCFH flow of \(\text{O}_2\) gas. This resulted in an oxide layer of typical thickness of 1 - 2 nm, as determined by ellipsometry. Samples were passed into the UHV system through a pre-baked airlock [62] and outgassed at \(~350^\circ\text{C}\) for 6 - 12 hours prior to oxide removal by flashing at elevated temperatures.

We first consider cleaning of the Si-capped samples grown under tensile strain on relaxed \(\text{Si}_{1-x}\text{Ge}_x\) substrates. The conventional method for cleaning pure Si wafer surfaces for STM studies is to “flash” (i.e., quickly heat) the sample above 1200°C [13, 67], in order to produce flat, clean Si surfaces with few carbide particulates. These particulates can pin surface steps and hence severely affect surface morphology. However, we found that heating above 1100°C destroyed the heterostructure and severely roughened the surface, so instead we tried to grow a very clean room-temperature oxide film and then desorb it at lower temperatures, using a procedure very similar to the \textit{Shiraki} clean used for preparing Si wafers for MBE growth [66].
Figure 4.3 shows a 5x5μm² image of a strained-Si-cap sample cleaned using the "p-clean" procedure followed by a 2-minute flash at ~ 1025°C inside the UHV system. This image is shown in "derivative mode," [13] and should be viewed as if illuminated obliquely from the left. In addition to the striking step structures seen on the surface [3], we observe ~ 15 raised particulates with typical height of 5 nm. These are reminiscent of the particulates reported [13, 67] for pure Si surfaces flashed at temperatures below 1200°C. It should be mentioned that the effectiveness of the p-clean method is highly variable, likely due to variations in the quality of the DI water supply.

For comparison, figure 4.4 shows a 5x5μm² image of a strained-Si-cap sample which was cleaned using the UVO procedure described above, with the oxide removed by three successive 2-minute flashes at ~ 1025°C. We see that this procedure results in a surface with no raised particulates over the entire 5x5μm² scan. The atomic-resolution close-up image in figure 4.5 reveals a moderate point defect density of about 4% on the terraces. We do note that residual surface particulates can be found on these UVO cleaned samples (often in clusters of 2 - 5), but it is relatively easy to find extended areas larger than 25μm² with no particulates.

The UVO cleaning procedure was also evaluated for nominally relaxed Si₁₋ₓGeₓ(001) films directly following ex-situ polishing. Figure 4.6 shows a 1.8x1.8μm² image of a relaxed, polished Si₀.7Ge₀.3 sample following sample flashing at ~ 1025°C, and figure 4.7 shows the typical point defect density. While these samples clean relatively well, they do in general show a larger density of residual particulates (typically ~ 1/μm²) than found on the Si-capped samples. At this point, we do not know why.
Figure 4.3: 5x5μm² derivative mode image of a strained Si sample following p-clean procedure. There are ~ 15 raised particulates which may cause step pinning.
Figure 4.4: 5x5μm² derivative mode image of a strained Si sample following the UVO cleaning procedure. This cleaning procedure produces an extremely clean surface.
Figure 4.5: 64x64nm$^2$ gray-scale image of a strained Si sample following UVO cleaning.
Figure 4.6: 1.8x1.8μm² derivative mode image of a nominally relaxed Si₀.₇Ge₀.₃ sample following UVO cleaning. There are ~ 4 raised particulates.
Figure 4.7: 64x64nm² atomic resolution image taken near the center of figure 4.6.
Given our relative success in cleaning low Ge content films, we went on to study cleaning of higher Ge-content relaxed Si$_{1-x}$Ge$_x$ films. Figure 4.8 shows a 820x820nm$^2$ derivative-mode image of a relaxed, ex-situ polished Si$_{0.2}$Ge$_{0.8}$ film prepared with the UVO procedure followed by three successive 2-minute flashes at \( \sim 875^\circ C \). This large scale image shows a rough-looking surface with a number of raised “mounds”. However, the close-up scans shown in figures 4.9—4.11 reveal that the surface roughness between the mounds is due to irregular step edges and many ML-deep holes, but the point defect density on the terraces is actually quite low. Figure 4.11 in particular shows a surface dimer structure and defect density which is comparable to prior STM studies of pure Ge(O01) surfaces [73, 74]. We also note that the mounds are actually “volcano”-type structures, each with a central crater at least 4 nm deep. This indicates that they are not due to carbide particulates left by surface cleaning (which do not have central craters), but rather are due to bulk defects present within the relaxed Si$_{0.2}$Ge$_{0.8}$ films before cleaning. And in fact, Fitzgerald et al. [75] found that high Ge-content MBE-grown relaxed Si$_{1-x}$Ge$_x$ films sometimes contain “pit”-like growth defects with typical size and areal densities similar to the volcano-type structures noted here.

We also note that these cleaned Si$_{0.2}$Ge$_{0.8}$ films appear to exhibit a unique step roughness reversal [76] as compared with Si(O01) surfaces. To see this, note that almost all the ML-deep holes as well as the longest “fingers” extending from step edges are elongated perpendicular to the dimer-row direction on the upper surface. This indicates that the surface favors step edges with local “B-type” termination over “A-type” termination [9, 10], the opposite to what is observed on pure Si(O01) surfaces [10, 30, 77]. Step roughness reversal was previously reported when 1 - 2 ML
Figure 4.8: 820x820nm² DM image of a relaxed Si₀.₂Ge₀.₈ sample after UVO cleaning.
Figure 4.9: 95x95nm² image taken from the central part of figure 4.8, showing irregular step edges and elongated ML-deep holes. The dimer row direction is indicated by the white line.
Figure 4.10: 95x95nm² image taken from the central part of figure 4.8, showing irregular step edges and elongated ML-deep holes. The dimer row direction is indicated by the white line.
Figure 4.11: 30x30nm² close-up image showing low point defect density.
of pure Ge was grown on Si(001) surfaces [76], but in that case was attributed to the “2xn” reconstruction which formed on those highly-strained surfaces. However, figures 4.10 and 4.11 clearly show that for relaxed Si$_{0.2}$Ge$_{0.8}$ films, the step roughness reversal occurs even though there is no trace of a “2xn” structure. At present the origin of the step roughness reversal seen here is not known.

Finally we note that the UVO procedure works quite well for regular Si(001) surfaces. Figure 4.12 shows a 5x5$\mu$m$^2$ derivative mode image of a UVO-cleaned Si(001) surface following flashing at $\sim$ 1025°C, revealing a vicinal surface with a nearly uniform step density due to a $\sim$ 0.25° surface miscut. It is easy to find extended areas larger than 25$\mu$m$^2$ with no carbide particulates. The close-up inset shows the typical point defect density. We have also tested the p-clean process, and in general found a higher density of particulates with a large run-to-run variability. However, with very clean chemicals and DI water, we could at times obtain particulate densities as low as for the UVO process. These results show that (in contrast to earlier reports [13, 67]) it is possible to produce Si surfaces with few carbide particulates by flashing at temperatures well below 1200°C, provided that a sufficiently clean oxide film is first grown [66].

Overall, the UVO process combined with a moderate temperature ( $\sim$ 1025°C ) flash, is simpler, more consistent, and (in general) more effective than the comparable p-clean process for preparing clean Si$_{1-x}$Ge$_x$ surfaces with a wide range of Ge content. One drawback to the UVO process is that it does produce a more “robust” oxide film, which required a longer flash to completely desorb it, so wet chemical procedures [66] may still be preferable for applications which require lower integrated processing temperatures.
Figure 4.12: 5x5μm² DM image of UVO cleaned Si surface. Inset shows gray-scale atomic resolution scan of another UVO cleaned Si(001) sample.
An important consideration after heating these SiGe samples to remove the surface oxide layer, is whether the stoichiometry of the surface has been changed from that initially grown. To address this concern, a commercial depth-profiling XPS system was used to make ex-situ measurements to determine how the surface composition of the samples is affected by the cleaning process.

For the Si/Si$_{0.7}$Ge$_{0.3}$ samples, XPS measurements of the Si(2p) and Ge(3d) peaks indicated a surface Ge content of $\sim$12% averaged over the photoelectron escape depth ($\sim2$ - 4 nm), and rose to the expected value of $\sim$30% as the surface was sputter-etched into the relaxed Si$_{0.7}$ Ge$_{0.3}$ substrate. To improve the depth resolution at the top surface, we also used a procedure developed by Lu et al. [78], whereby the top $\sim$0.5 nm of the surface is oxidized by UVO. Oxidation of the surface layer causes a shift in energy of the Si(2p) and Ge(3d) spectral peaks to higher binding energy (oxygen essentially pulls electrons away from Si(Ge), making the remaining electrons more tightly bound). If now only the oxide-shifted Si and Ge XPS peaks analyzed, the chemical composition is determined only for the thin oxide layer at the surface. Using this procedure, the measured Ge content at the very top surface was $\sim$2%. From these measurements, we conclude that the Si/SiGe surface is under an approximate 1% surface tensile strain.

It should be pointed out that a shallow oxidation of the surface at room temperature is required for this process, since: (1) the depth resolution enhancement depends on the thickness of the oxide layer, (2) elevated temperatures may lead to diffusion of atoms at the surface resulting in an incorrect analysis of the surface composition. The UVO oxidation fulfills these requirements.
XPS measurements of the relaxed Si_{0.7}Ge_{0.3} samples (no Si cap layer) following cleaning, surprisingly look very similar to those of the Si-capped samples discussed above. The Ge content at the top surface was 12% (2% by Lu et al. method) as compared with ~30% deeper into the relaxed Si_{0.7}Ge_{0.3} film, indicating that the surface lost Ge during cleaning. Since Ge has a higher vapor pressure than Si [61], it is reasonable that significant Ge loss could occur during sample flashing. A residual gas analyzer was used to make thermal programmed desorption (TPD) measurements, and in fact observed Ge desorption at temperatures above 1000°C. We estimate that ~ 2 - 10 monolayers (ML) of Ge desorb during a 6 minute flash at ~ 1025°C, corresponding to a Ge depletion depth of 1 - 5 nm. Finally, we note that surface Ge loss during cleaning should result in tensile strain in the top layers of the sample. This would explain why these samples show very similar step structure to the strained-Si-capped samples discussed earlier, which are under tensile strain [3].

Based on the observations for the 30%Ge samples we expected to find a similar depletion of Ge at the surface of the 80% Ge samples. However XPS measurements indicate that the surface Ge composition profile of the Si_{0.2}Ge_{0.8} samples does not change significantly during cleaning, but rather remains within ~ 5% of the nominal 80% content in the relaxed layers. This smaller change in surface composition may be due to the lower flashing temperatures (~ 875°C) used, and/or to faster relative diffusion of Ge atoms towards the surface as compared with the Si_{0.7}Ge_{0.3} samples discussed above.
4.1.3 Wavy Steps — Strained Si on Relaxed SiGe

We now move on to the observations of the morphology of Si(001) surfaces under tensile strain. On a large scale, the Si/Si$_{0.7}$Ge$_{0.3}$ strained layer sample after surface cleaning (see figure 4.13) consists of "step bunched" regions generally running along [110] directions, which are probably related to the [110] - oriented "cross-hatching" present on relaxed SiGe surfaces [49], a topic which will be discussed later. Away from the step bunched regions are large (001) terraces, and extended areas with small local miscut (typically 0.05° - 0.4°) usually oriented towards a [110] direction. This unique morphology permits a direct investigation of the dependence of the step behavior on local misorientation.

Figure 4.14 is a 1.8 x 1.8 $\mu$m$^2$ scan, showing that the slightly miscut regions are covered with quasi-periodic "wavy" steps alternating with very straight steps. The local miscut in this scan ranges from 0.02° - 0.6°, corresponding to local step separations $L$ in the range 350 - 13 nm (here 2$L$ is the separation between adjacent straight steps). Figure 4.15 shows a 820x820nm$^2$ scan (displayed now as a gray-scale keyed to height) of the central part of figure 4.14, and figures 4.17—4.19 show atomic resolution close-up scans of the boxed areas in figure 4.15. On a large scale, note that: (1) both the amplitude $A$ and characteristic wavelength $\lambda$ of the wavy steps increase with $L$, (2) for $L > 20$ nm the wavy steps in general cover most ($> 80\%$) of the distance between adjacent straight steps, and (3) for $L < 20$ nm the periodicity becomes suppressed and/or masked by step meander. We also find that $\lambda$ increases more slowly with $L$ than does $A$, causing larger step undulations to assume elongated "finger"-like shapes. Analysis of many scans from several samples (see figure 4.16) suggests an approximate empirical relation $\lambda \approx (10\text{nm} \pm 3\text{nm}) \times (L/1\text{nm})^{0.5 \pm 0.1}$ for
Figure 4.13: 5x5μm² DM image of a strained Si surface after surface cleaning by UVO. There are step bunched regions separating regions of low local miscut.
Figure 4.14: 1.8x1.8μm² DM image of the central part of figure 4.13.
Figure 4.15: 820x820nm² gray-scale image of the central part of figure 4.14.
Figure 4.16: Wavy $S_B$ step wavelength $\lambda$ as function of terrace width $L$. 
$20\text{nm} < l < 120\text{nm}$, with about a $\pm 20\%$ scatter in the data. On much larger terraces ($l > 300\text{nm}$) highly-elongated "stripe"-like [2] step fingers are often observed. In this case the lateral separation of the fingers generally falls in the range of 100 - 200 nm with no systematic variation with terrace width. The exact same cleaning procedures to study unstrained vicinal Si(001) substrates with surface miscuts of $0.1^\circ$ - $0.3^\circ$ aligned towards a [110] direction and found essentially random $S_B$ step undulations, in agreement with previous studies [10, 79, 80].

The atomic structure of the steps is revealed by the close-up scans in figures 4.17—4.19. We see that the straight steps are nominally of the $S_A$ type, while the wavy steps are nominally of the $S_B$ type. From previous studies of unstrained, thermally cleaned Si(001) it is well known that $S_B$ steps are much rougher than $S_A$ steps [10] due to large step meander, with step kinks largely uncorrelated in direction, separation, and length [10, 79, 11]. In contrast, it is evident that the kinks on the larger wavy steps on the strained Si samples are highly correlated, particularly in direction.

Our atomic-resolution scans show that the vast majority of the fingers are not caused by defect-related step “pinning” [81, 70]. We also find that they are not in general “kissing sites” [33], i.e., step protrusions which extend completely to the next step edge and which are stabilized by anti-phase boundaries (APB’s) [82] in the (2x1) reconstruction on the lower terrace. Figures 4.17—4.19 directly show that there are no APB’s present under many of the fingers. This is also evident from figure 4.14, which shows that many fingers do not extend to the next step edge. Some APB’s can be found (see figure 4.19), but wavy $S_B$ steps many cycles long clearly do exist without them.
Figure 4.17: 100x100nm$^2$ close-up image of one of the boxed regions in figure 4.15.
Figure 4.18: 100x100nm$^2$ close-up image of one of the boxed regions in figure 4.15. This finger is highly elongated, incorporating long segments of A-type step along the sides.
Figure 4.19: 100x100nm² close-up image of one of the boxed regions in figure 4.15. The arrow indicates an anti-phase boundary (APB).
We should also consider whether the wavy $S_B$ steps are non-equilibrium structures formed by kinetically-limited processes during surface cleaning. This is important since various "kinetic instabilities" [31] could produce wavy step fronts during growth or etching, and since the longer step "fingers" do look qualitatively similar to growth structures observed during MBE [30] or CVD [83] homoepitaxy. To address this, we have performed experiments which show that the wavy step structures are quite robust, even after annealing for 2 hours at 720°C (as shown in figure 4.20), 10 min. at 875°C, or even after several 2 min. "reflashes" at 1025°C. For comparison, steps on unstrained Si(001) surfaces with miscuts in the range of 1° - 0.1° are commonly assumed to equilibrate during time scales of minutes at temperatures in the range 600° - 710°C [18, 29, 10, 11, 82]. This suggests that the steps on our samples are in local equilibrium, at least for local miscuts down to 0.1° [84]. On very large terraces (> 500nm) we do observe long fingers which do not completely cross the terrace, suggesting a kinetically-limited structural evolution. We have used LEEM to study the surface dynamics of these samples at elevated temperatures, and have observed step fingers grow across a terrace up to the point at which sublimation begins to result in monolayer deep holes and step edge retraction. We conclude from these observations that the step fingers are indeed equilibrium structures.

4.1.4 Alerhand Stress Relaxation Enhancement

We [3] have proposed that the wavy $S_B$ steps on these samples are stabilized by surface stress relaxation effects similar to those previously proposed for unstrained Si(001) [2, 29, 33, 34, 85], which in this case are enhanced by the applied tensile strain. Several aspects of these wavy $S_B$ steps are consistent with surface stress
Figure 4.20: 780x570nm² DM image of a strained Si sample after annealing for 2 hours at 720°C. Boxed area indicates two $S_B$ steps with peak-to-trough alignment.
effects. Firstly, the waviness is confined to $S_B$ steps. Since kinks in $S_B$ steps cost much less energy than kinks in $S_A$ steps [10, 11, 86], one would expect most strain-relieving undulations to be confined to $S_B$ steps [29, 34, 85]. Secondly, the strength of the waviness increases as the local step separation $L$ increases, consistent with the idea of a "critical" terrace width for the onset of step undulations [2, 34]. Thirdly, we often find that the "peaks" of a wavy step on one terrace are aligned with the "troughs" on an adjacent terrace, particularly when $L$ (and hence $\lambda$) is nearly the same on both terraces. The boxed area in figure 4.20 shows an example of this effect. This peak-to-trough alignment, results in a pattern of roughly triangular terraces which border on all three sides extended $SL$ steps, and turns out to be a particularly effective in relieving surface stress (this will be discussed in more detail in section 4.4). Finally, we find that on very large terraces (> 300nm) the wavy steps become elongated fingers with nearly parallel $S_A$-type edges, and their separation approaches a limiting value in the range of 100 - 200 nm. In this limit the local step structure appears to approach the "stripe" phase originally proposed by Alerhand et al. [2], i.e., an array of nearly parallel, periodic, alternating up and down $S_A$ steps. The lower-central part of figure 4.21 shows a particularly striking example of this stripe-like appearance. We will examine the change in step shape as a function of surface miscut in more detail in the section discussing recent elastic simulations by Ebner et al. [5].

To understand how the substrate induced biaxial tensile strain enhances the formation of the M-A surface phase, we note that on strained Si(001) surfaces the force monopole (FM) at a step actually has two components. Figure 4.22 shows how the addition of a biaxial tensile strain leads to an additional FM at a step edge. The first component, $F_0 = (\sigma_\parallel - \sigma_\perp)$, (shown in figure 4.22(a)) is due to the surface stress
Figure 4.21: 3.1x2.3μm² area of a strained Si sample, showing a striped step region. Note this sample was cleaned by the wet chemical (p-clean) process and has a number of large particulates at the surface.
Figure 4.22: Cross-section of an $S_A$-step before relaxation occurs (a) without and (b) with a substrate induced tensile strain. Because there is an extra half plane of material on the left, there is an added surface stress $\sigma_B$ which adds to the already large FM $F_0 = (\sigma_\parallel - \sigma_\perp)$. This results in a larger effective surface stress anisotropy $F_{Tot} = F_0 + \sigma_B$, and hence a larger driving force for the spontaneous creation of steps.
anisotropy arising from the 2x1 reconstruction [2, 23], which on Si(001) produces a FM directed towards the upper (lower) terrace at an \( S_A \) (\( S_B \)) step. The second component (see figure 4.22(b)), \( F_B = \sigma_B h \) is present at a surface step on any film under a net bulk applied strain [87], and is simply due to the fact that there is more material under stress on one side of the step than the other. This FM is directed towards the upper (lower) terrace for tensile (compressive) strain, and has approximate magnitude [87] \( F_B \approx \sigma_B h = B \varepsilon_B h \), where \( h = 0.138 \) nm is the SL step height, and the elastic modulus \( B \) [43] relates biaxial in-plane film stress \( \sigma_B \) to the applied bi-axial strain \( \varepsilon_B \). Hence, an applied tensile strain should increase the magnitude of the total FM at \( S_A \) step edges, resulting in a larger driving force for step undulations. We have suggested that these two contributions to the FM should add vectorially, so that the total effective FM is \( F_{T_{tot}} = F_0 + F_B \). With this total FM, the optimum terrace width for the Alerhand stripe phase [2] should become

\[
L_0 \approx \pi a \exp \left[ 1 + \frac{E_A}{\alpha (F_0 + B \varepsilon_B h)^2} \right]
\]  

(4.1)

where \( E_A \) is the \( S_A \) step energy, \( \alpha = \frac{1-\nu}{2\pi \mu} \) (\( \nu \) and \( \mu \) are the Poisson ratio and shear modulus respectively) is an elastic constant, and \( a = 0.384 \) nm is the surface lattice constant.

We assume here the following experimental values: \( E_A \approx 0.028 \) eV/a [10, 86], \( F_0 \approx 1.2 \) eV/a\(^2\) [21, 23], and \( B \approx 64 \) eV/a\(^3\) [43]. A complication arises in determining the value of \( \alpha \), which when calculated using isotropic approximations from the elastic stiffness constants \( C_{ij} \) (see [43]) of silicon, yields a value of \( 0.0042a^3/eV \leq \alpha \leq 0.0066a^3/eV \) [2, 43].

For this range of \( \alpha \)'s, Eq. (1) gives the order of magnitude estimates of \( L_0 \) with no strain, and for 1% tensile strain (\( B \varepsilon_B = 0.19F_0 \)) shown in table 4.1. These predicted
values compare with the typical observed values $\lambda \approx 50 - 200$ nm for wavy $S_B$ steps measured on our strained samples. It should be noted, that the estimates presented here are quite rough and sensitive the actual parameter values used. In particular we note that we have revised the value of $\alpha$ used here from that of a previous work [3], and that values used by other authors vary widely.

We comment here that although using $\alpha_{\text{avg}}$ from table 4.1 results in relatively good agreement with our experimental observations on the strained layer samples, it predicts a rather low value of $L_0 = 120$nm for unstrained Si. Since these wavy and striped-step structures have not previously been observed for low miscut unstrained Si, we actually expect $L_0$ to be significantly larger, on the order of $\sim 500$nm. However if we use $\alpha_{\text{min}}$ from table 4.1 to get a larger $L_0$ for unstrained Si, we get less good agreement with the observations on strained Si. The resolution of this inconsistency, may come from the fact that $L_0$ is exponentially sensitive to the parameters mentioned above. In particular the presence of Ge at/near the surface may result in small changes in these parameters, thus affecting $L_0$.

Overall, we find that even a small 1% biaxial tensile strain is sufficient to reduce $L_0$ by a factor of $\sim 2 - 4$, but we mention again that the estimates presented here are quite rough and sensitive to the actual parameter values used. Nevertheless the

<table>
<thead>
<tr>
<th>$\alpha$ (Å$^3$/eV)</th>
<th>$L_0^{\text{unstrained}}$ (nm)</th>
<th>$L_0^{1% \text{strain}}$ (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\alpha_{\text{min}} = 0.0042$</td>
<td>336</td>
<td>85</td>
</tr>
<tr>
<td>$\alpha_{\text{avg}} = 0.0054$</td>
<td>120</td>
<td>42</td>
</tr>
<tr>
<td>$\alpha_{\text{max}} = 0.0066$</td>
<td>62</td>
<td>26</td>
</tr>
</tbody>
</table>

Table 4.1: Calculated $L_0$ for Si/SiGe with and without 1% biaxial tensile strain.
results strongly suggest that moderate bi-axial tensile strain should enhance step waviness on Si(001) surfaces, and the results are within an order of magnitude of the observed values.

4.1.5 Kink Counting

In a beautiful set of experiments, Xie et al. [71] found the surface roughness of strained layer growth of Si$_{0.5}$Ge$_{0.5}$ on relaxed Si$_{1-\xi}$Ge$_{\xi}$, to depend on the sign of the strain. Layers grown under tensile strain were found to be smooth, while those grown under compressive strain were found to be rough. Xie et al. [71] argued that tensile strain acts to increase the energy of $S_B$ steps, thus inhibiting step generation and limiting roughening, while compressive strain lowered the $S_B$ step energy and lead to increased step generation and rough surfaces.

In this section, we make note of an interesting preliminary observation that is not yet fully understood. Motivated in part by the work of Xie et al. [71] and by our own observations that the $S_A$ steps on Si/SiGe surfaces seemed unusually straight, we analysed several images to try to determine if the relative energy per unit length of the $S_B$ steps was increased. The method used was that developed by Swartzentruber et al. [10] and later extended by Zandvliet et al., to experimentally determine $E_A$ and $E_B$ for unstrained Si.

A kink in an $S_A$ step consists on a corner and a segment of length $n$ of $S_B$ step. Swartzentruber et al. [10] argued that the energy of this kink is just $E(n) = nE_B + C$, where $n$ is the length of the kink, and $C$ is the effective “corner” energy. Applying a Boltzman distribution to the kinks such that the probability of a kink of length $n$ is given by $P(n) \propto \exp^{nE_B+C}$, permits a calculation of the probability distribution of
kinks along the step edge. There is a complication on these surfaces however, there is (nearly) always an azimuthal miscut that forces a certain number of kinks per unit length to exist. These "forced" kinks are not thermally generated, but rather are necessary in order to accommodate the azimuthal misorientation. Swartzentruber et al. attempted to circumvent this problem by assuming that the number of thermally excited "un-forced" kinks were independent of the miscut. Using this assumption

$$\frac{E_B}{k_B T} = \ln[\exp^{-k_B T(1/P_{un} - 2)} + 1],$$

where $P_{un}$ is the probability of an un-forced kink of any length.

Zandvliet et al. later pointed out that forced kinks act to suppress the number of un-forced kinks, which would cause the Swartzentruber method to over estimate $E_B$. Zandvliet et al. derived an expression (based upon the Burton, Cabrera, and Frank (BCF) theory [88]) for

$$\frac{E_Y}{k_B T} = -\ln \frac{n+1}{n_0},$$

where $E_Y = 4E_B - 2C$, $n_0$ is the number of sites without any kinks, $n_{+1}$ is the number of un-forced kinks of length one, and $n_{-1}$ is the number of forced kinks of length one.

Table 4.2 contains results of kink counting for strained and unstrained silicon, as well as the values from Zandvliet et al. [11] and Swartzentruber et al. [10].

The value of $E_B$ for Si/SiGe calculated by the Zandvliet method, is higher than that found by Zandvliet et al., in agreement with the Xie et al. prediction that surface tensile strain should increase $E_B$ due to increased bond stress at the step edge. However our control experiments on regular unstrained Si also indicate a higher $E_B$ than that previously found. Our results calculated by the Swartzentruber method, show little difference between $E_B$ for for strained and regular Si, but both are consistently higher than that reported by Swartzentruber et al. On the basis of these preliminary results, definitive statements about changes in $E_B$ for surfaces under tensile strain
### Table 4.2: Results of kink counting analysis of strained Si and regular Si. Values of $E_b$ resulting from Zandvliet ("Z") and Swartzentruber ("S") type analysis are shown, for a freeze-in temperature of 775K († values shown have been adjusted from Swartzentruber's assumed freeze-in temperature of 873K to Zandvliet's freeze-in temperature of 775K, to allow direct comparison).

<table>
<thead>
<tr>
<th>Data</th>
<th>“Z” — Analysis</th>
<th>“S” — Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si (Swartzentruber [10])</td>
<td>—</td>
<td>0.08 ± 0.01</td>
</tr>
<tr>
<td>Si (Zandvliet [11])</td>
<td>0.06 ± 0.01</td>
<td>—</td>
</tr>
<tr>
<td>Strained Si — Si/Si$<em>{0.7}$Ge$</em>{0.3}$ (This work)</td>
<td>0.10 ± 0.01</td>
<td>0.11 ± 0.02</td>
</tr>
<tr>
<td>Si$_{\text{LowT}}$ (This work)</td>
<td>0.08 ± 0.01</td>
<td>0.12 ± 0.01</td>
</tr>
<tr>
<td>Si$_{\text{HighT}}$ (This work)</td>
<td>0.07 ± 0.01</td>
<td>0.12 ± 0.01</td>
</tr>
</tbody>
</table>

cannot be made. The discrepancy in the values for regular Si, also cannot currently be explained. The results presented here while not conclusive, are highly suggestive of a change in $E_b$ for the strained Si samples. Additional experiments should be done to allow more definite conclusions to be drawn regarding this issue.

#### 4.2 B-doped Si(001)

Given the results observed for Si/SiGe, it seems likely that striped and wavy step structures should exist on other Si(001) surfaces with biaxial tensile stress. One such system is heavily boron doped Si(001), because it is well known that high levels of boron at the Si surface induces significant tensile strain [37, 38, 39, 40], due to the smaller B-Si bond length as compared to the Si-Si bond length. This particular system turns out to manifest the M-A striped phase in a truly spectacular way, with a temperature dependent step shape transition from straight to triangular-tiled to striped steps as the sample annealing temperature is reduced.
The low-energy electron microscope (LEEM) images shown in figure 4.23, illustrate the essential features of the M-A phase and the stripe-to-triangular-tiled transition. Figures 4.23(a)-(e) show LEEM images of a particular 7μm-diameter surface area as the temperature is lowered from ~ 970°C to room temperature. The images were taken with the (1/2,0) diffraction beam such that terraces on which the dimer rows run approximately left-right ["(2x1)" domains] or up-down ["(1x2)" domains] appear bright or dark, respectively [89]. From the direction of step migration during sublimation [89], we determined that the "step-down" direction is from top to bottom. At 967°C, the smooth $S_A$ and rough $S_B$ steps appear qualitatively similar in shape to that previously observed for low miscut Si(001) surfaces [90, 91]. However, figure 4.23(b) shows that dramatically different behavior is seen as the sample is cooled to ~ 930°C. The $S_A$ steps remain nominally straight, but the $S_B$ steps assume a quasi-periodic "sawtooth" shape, with a clear terrace-to-terrace phase alignment, forming a triangular-tiled arrangement of (2x1) and (1x2) domains [3, 92]. As the sample is cooled further (figure 4.23(c)) the typical width of the triangular domains decreases, resulting in more "acute" triangular shapes, which then (figure 4.23(d)) evolve into narrower rectangular or "stripe"-like domains at lower temperatures. Real-time LEEM images reveal that this narrowing is accomplished either by growth of a new finger between two existing fingers or by a finger splitting in two. On other sample areas with different miscut and/or cooling conditions, we have also observed domain splitting via nucleation and growth of isolated islands or depressions. For $T < 800°C$ (figure 4.23(e)), the stripe spacing becomes smaller than the ~ 15nm lateral resolution of the LEEM, causing the finely-striped terrace regions to appear gray.
Figure 4.23: Sequence of LEEM images from a single 7μm diameter area of a heavily B-doped Si(001) sample as the temperature is varied. (a) At 967°C, where step flow occurs due to Si sublimation from the surface. By viewing the time sequence of a series of images at this temperature, the global step down direction was determined to be from the top to bottom of the figures. (b) — (e) Show a cooling sequence where the terrace structure becomes progressively more finely striped, until all domain contrast is lost in (e). (f) — (h) The features coarsen upon re-heating.
Remarkably, this structural narrowing can be largely reversed simply by re-heating the sample. Figures 4.23(f)-(g) show the same sample area after heating up to 888°C and 913°C, and a clear coarsening of striped structure has occurred. This continues for \( T = 940°C \) (figure 4.23(h)), where triangular tiling is starting to re-establish itself. Upon further heating, the step structure returns closely to that seen in figure 4.23(a).

Figures 4.24—4.32 show room-temperature STM images of similarly-prepared samples. Figure 4.24 shows a \( \sim 1.8\mu m \) area of a well-oriented sample region which was flashed at 1200°C, annealed for 2 hrs. at \( \sim 720°C \), and then quenched to room temperature. The finely-striped terrace structure of long narrow step fingers is evident. Note that this scan is displayed in “derivative mode”, and should be viewed as if illuminated obliquely from the left. A close-up scan of a finger “tip” (from another sample) shown in figure 4.25 confirms that the dimer rows run parallel to the long axis of the fingers, indicating that the stripe edges are formed almost entirely of \( S_A \) step edges. In figure 4.24, the lateral periodicity is \( \lambda \approx 21nm \) and the typical stripe width is \( 11 - 15nm \), which means that the stripes are only \( \sim 14 - 20 \) dimer rows wide (see figure 4.26). Figures 4.27-4.28 show \( \sim 820nm \) images of different samples after flashing and a 10 - 15 min. anneal at \( \sim 720°C \), and exhibit a similar lateral periodicity. Here, elongated islands and/or depressions can be seen, as well as substantial “cross-fingering” on sample areas on which the surface miscut is not aligned along a \((110)\) direction.

Other LEEM and STM images also indicate that if the terrace width \( l \) (imposed by the local miscut) varies across the surface, then the domains in general become more triangular in shape, and their lateral spacing decreases on regions with smaller \( l \). An STM image illustrating this behavior is shown in figure 4.29. The misorientation
Figure 4.24: 1.8x1.8μm² DM room temperature STM image of a highly B-doped Si(001) sample annealed at 720°C for 2 hours and quenched rapidly to room temperature.
Figure 4.25: ~ 55x55nm² atomic resolution STM image of the tip of a finger on a heavily B-doped Si(001)-(2x1) surface.
Figure 4.26: ~ 120x120nm$^2$ image of step stripes on a heavily B-doped Si(001) surface. The stripes are extremely narrow, and take the form of alternating up and down $S_A$ steps in agreement with the structure proposed by Alerhand et al. [2].
Figure 4.27: 820x820nm² DM image of another B-doped Si surface after annealing at 720°C for 15 minutes.
Figure 4.28: 820x820nm\textsuperscript{2} DM image of a B-doped Si surface after annealing at 720°C for 10 minutes. A small local misorientation produces cross-fingering (more clearly indicated in figure 4.29.)
angle becomes steeper in going from the bottom of the image to the top, and there is a continuous evolution of the surface step shapes from a striped structure to roughly triangular (or wavy) step shapes at higher miscuts. This dependence of step shape and size on L is in qualitative agreement with behavior reported by Jones et al. [3] for Si(001) surfaces under tensile strain on relaxed SiGe substrates.

These novel surface structures are almost certainly due to the spontaneous step formation predicted by Marchenko [1] and Alerhand et al. [2]. The striped phase seen at lower temperatures has the exact form predicted by Alerhand et al., i.e., an array of nearly parallel, periodic, alternating up-and-down steps (of the lower energy $S_A$ - type) with a characteristic “Alerhand” width $L_0$ on sample areas where $l >> L_0$. No specific predictions were made for situations where $l \approx L_0$, but it is physically reasonable that the stripes should evolve into some other form of stress domain tiling when the maximum length of a stripe is constrained by the surface miscut to be comparable with or less than its width. The surprising result that triangular stress domains appear on these (001) surfaces with biaxial symmetry, will be discussed in more detail in section 4.4. We note that roughly triangular step structures have also been reported for Si(001) films under tensile strain [3] and for Ge films under compressive strain by Chen et al. [92].

It is now well known that boron exhibits a temperature-dependent segregation at Si surfaces [41, 42]. For the Si(111) surface, the equilibrium surface B concentration has been reported to increase by a factor of $\sim 2$ as the temperature is lowered from 900°C to 750°C [41], and similar changes have been reported for Si(001) [42]. So if the M-A phase is induced by a high concentration of B at the Si(001) surface, then it follows logically that it should be enhanced at lower sample annealing temperatures.
Figure 4.29: ~ 1.8x1.8μm² image of a misoriented region of a heavily B-doped Si(001) wafer. Cross fingering occurs when the azimuthal miscut angle changes.
Using secondary-ion mass spectrometry (SIMS), we have found a significantly larger B concentration at the surface \( n_B, \approx 5 \times 10^{20} \text{cm}^{-3} \) than in the bulk \( n_B, \approx 8 \times 10^{19} \text{cm}^{-3} \) for samples which have been flashed and annealed in UHV. However, because the SIMS analysis was conducted ex situ with a native surface oxide present, it was not possible to clearly distinguish whether the surface B accumulation was larger for the samples annealed at lower temperatures.

To look for in situ evidence of top-layer B surface segregation, we have also made atomic resolution STM measurements. Figures 4.30—4.32 show 1.8 µm STM images and corresponding atomic-resolution insets measured on samples with different flashing/annealing histories (see the figure caption for details). We find that (a) the concentration of "dark" point defects on these heavily B-doped samples is in general larger on samples which have developed more finely-structured stripes, and (b) the defect density is always larger than that found on lightly-doped Si(001) surfaces (see for example figure 2.5) processed the same way. This is consistent with the idea that B atoms at the surface are responsible for both the dark defects and the enhanced M-A surface phase. In this regard, we note that Wang et al. [39, 40] have recently reported that CVD deposition of B onto Si(001) does produce B-related dark point defects. In our case, however, we have not observed the B-induced (4x4) structures reported by Wang et al. at high B coverage, suggesting that in our samples the B has not segregated laterally into dense patches. We have also found that the large-scale step structure and the point defect density both depend on the sample annealing history. In particular, when samples are cycled through several flash/annealing treatments, they more readily develop fine step stripes and high defect density when annealed at 700—750°C. This may reflect the fact that B builds up at the surface via out-diffusion.
Figure 4.30: 1.8x1.8\(\mu\text{m}^2\) STM image with atomic resolution inset, of B-doped Si(001) surface. This sample was flashed at \(\sim 1250^\circ\text{C}\) and quenched to room temperature.
Figure 4.31: 1.8x1.8μm² STM image with atomic resolution inset, of B-doped Si(001) surface. This sample was flashed, then cooled slowly from 950°C to room temperature in 45 seconds.
Figure 4.32: 1.8x1.8 $\mu m^2$ STM image with atomic resolution inset, of B-doped Si(001) surface. This sample was flashed, annealed at 700°C for 10 minutes, and then quenched to room temperature.
from the bulk during each annealing cycle, and that the high-temperature flashes are of too short duration to allow re-equilibration of the surface B content to near bulk values.

As discussed section 4.1.4 dealing with Si/SiGe, the presence of biaxial strain was suggested [3, 4] to increase the driving force for step relaxation. There is already strong evidence [37, 38] that high B-doping at Si surfaces does produce substantial bulk tensile strain. Based on our SIMS measurements and references [37] and [38], we estimate that B segregation produces ~ 0.25% bulk biaxial tensile strain near the surface. Using equation 4.1, and the same parameter estimates as in section 4.1.4, we estimate $L_0$ as shown in table 4.3. While it is possible that SIMS under estimates the

<table>
<thead>
<tr>
<th>$\alpha$ (a$^3$/eV)</th>
<th>$L_0$ unstrained (nm)</th>
<th>$L_0$ 0.25%strain (nm)</th>
</tr>
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<tbody>
<tr>
<td>$\alpha_{min} = 0.0042$</td>
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<td>$\alpha_{avg} = 0.0054$</td>
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</tr>
<tr>
<td>$\alpha_{max} = 0.0066$</td>
<td>62</td>
<td>48</td>
</tr>
</tbody>
</table>

Table 4.3: Calculated $L_0$ for B-doped Si(001) with and without 0.25% biaxial tensile strain.

surface B concentration, it seems that this level of bulk strain by itself is probably not sufficient to produce the extremely narrow stripes observed on our samples. We now examine other effects that could lead to smaller $L_0$. Referring once again to equation 4.1, we see that in addition to bulk tensile strain $\epsilon$, a B-induced decrease in $E_A$ as well as a B-induced increase in $F_0$, would both produce a strong decrease in $L_0$, and hence strongly enhance the M-A phase.
In figure 4.33 we present a modified surface structural model of the B-doped Si(001) surface, based in part on the work of Wang et al. [39, 40]. We consider the possibility that surface boron lowers the \( S_A \) step energy \( E_A \). The left part of figure 4.33 shows one possible configuration of B atoms at an \( S_A \) step edge. It is not immediately obvious whether or not such a structure should lower \( E_A \), we only point out that the possibility exists and warrants investigation.

Figure 4.33: Ball and stick model of B-reconstructed Si(001)-(2x1) surface, showing B incorporation in the “A” subunit ([39, 40]) in the dotted rectangle, and at an \( S_A \) step edge.
4.2.1 Determining $F_0$ on B-doped Si(001)

Structural Model

We turn now to the question of how the surface stress anisotropy is affected by high B doping. In an earlier work, we [4] argued that B may increase the surface stress anisotropy $F_0$. This seemed intuitively likely, based on specific structural models proposed by Wang et al. [39, 40], Zhang et al. [93], and Chang et al. [94] for B incorporation into the Si(001)-(2x1) surface. For the purpose of illustration, we here consider only the model proposed by Wang et al. The basic building block of their model (which they refer to as an "A" sub-unit) is shown in the dotted rectangle in figure 4.33. It consists of a missing surface Si dimer, with B atoms replacing the four adjacent Si atoms in the next lower atomic layer. We see from Fig. 4.33 that 2/3 of the highly strained B-Si bonds (drawn as heavy lines) are oriented in a plane parallel to the direction of the surface dimer bonds, and hence should contribute more tensile stress parallel to the dimer bond direction than perpendicular to it. Since the Si(001) surface stress is already more tensile parallel to the dimer bond direction than perpendicular to it (even on un-doped Si) [2, 9, 23], it appears likely that a high B-surface density would increase the surface stress anisotropy $F_0$.

It should be noted that while the structural model of Zhang et al. differs from that of Wang et al., it also would suggest an increase in the surface stress anisotropy. Additionally, recent work by Chang and Stott [94], suggests that the Wang et al. model may not be the lowest energy configuration for the Si(001)/B surface, nevertheless our experimental measurements indicate that there is an increase in the surface stress anisotropy $F_0$, as will be discussed next.
Uniaxial Strain Experiments to Determine $F_0$ on B-doped Si(001)

We have performed uniaxial strain experiments to measure the surface stress anisotropy. The technique used is based on some beautiful LEED experiments of Men et al. [17, 21], Webb et al. [17, 21], and subsequent STM experiments of Swartzentruber et al. [17, 18]. It was shown that annealing a Si(001) surface under an applied uniaxial strain alters the relative widths (and hence areas) of the (2×1) and (1×2) terraces, a finding that was not understood at the time, but the solution to which was also provided in the work by Alerhand et al. [2].

Alerhand [2] considered the case of an uniaxial strain ($\varepsilon^{\text{ext}}$) applied along a symmetry direction (with respect to the surface stress anisotropy) which breaks the orientation degeneracy of the (2×1) and (1×2) domains, with the result that one domain population grows at the expense of the other. For consistency, we will refer to domains in which the dimer bond direction is parallel (perpendicular) to the applied uniaxial strain direction as 2×1 (1×2). Values of $\varepsilon^{\text{ext}} > 0$ correspond to tensile strain causing the 2×1 regions to shrink and the 1×2 regions to grow, while values of $\varepsilon^{\text{ext}} < 0$ corresponding to compressive strain cause the 1×2 regions to shrink and the 2×1 regions to grow. By generalizing the earlier presented stripe phase (which had equal domain populations) to a structure with alternating domains of unequal widths $(1 + p)l$ and $(1 - p)l$ (where the area fraction $p$ is defined by $(A_{2\times1} - A_{1\times2})/(A_{2\times1} + A_{1\times2})$) allows equation 2.3 to be generalized to [2],

$$\Delta E = \frac{1}{2} \varepsilon^{\text{ext}} p F_0 + \frac{E_A}{l} - \frac{\alpha (F_0)^2}{l} \ln \left( \frac{l}{\pi a} \cos \left( \frac{\pi p}{2} \right) \right).$$  (4.2)

Depending on the surface considered, $\Delta E$ may be minimized by varying $p$ while holding $l$ constant or by varying both $p$ and $l$, corresponding to local or global equilibrium.
respectively (see section 2.4.3). The local equilibrium case should apply when there 
are steps imposed by the surface miscut or where surface kinetics permit equilibration 
only on the local scale, while the global equilibrium case should apply to low miscut 
surfaces where extra step generation takes place. For low strain values, the functional 
form of $p(e^{ext})$ is identical for both local and global equilibrium and is given by

$$p = \frac{-2l e^{ext}}{\pi^2 \alpha F_0^2}$$  \hspace{1cm} (4.3)$$

with $l$ being the step spacing due to miscut or $L_0$ for local or global equilibrium re-
spectively. Then by varying the applied strain and measuring the domain population 
 asymmetry, one may extract the surface stress anisotropy $F_0$.

We have adopted an approach similar to that used by Men et al., [21]. A silicon 
 wafer 4mm wide and 20mm long is rigidly clamped at one end and has a flexible 
 contact for sample heating at the other end. The sample is first cleaned by flashing 
 to 1200°C, and quenched to room temperature. Then a precision machined tantalum 
 post (see figure A.34) is used to deflect the free end of the silicon sample. In the 
 experiments by Men et al. the strain was determined by measuring the deflection of 
 the free end of the sample, and using the formula for a loaded beam from elasticity 
 theory. In our system, we could not directly generate a known deflection using the 
 linear feed-through, so we developed a new means of determining the strain applied. 
 A laser beam is directed into the main chamber through the right side viewport, and 
 is traversed across the sample surface. The reflected laser beam is directed through 
 the left side viewport onto a split photo-diode detector. The split photo-diode forms a 
 position sensitive detector (PSD) that is used to measure the local surface curvature 
 as indicated in figure 4.34, allowing calculation of the strain applied. The sample 
 is annealed at 700°C for 20 minutes under the applied strain, quenched to room
temperature, and the strain relieved. The surface curvature is then re-profiled to be certain that no plastic deformation has occurred. Large scale STM images are taken, and digitized to allow calculation of the areas of 2x1 and 1x2 domains, and step spacings $l$.

Figure 4.34: Arrangement for measuring the uniaxial strain of the cantilevered samples. A laser is traversed across the surface ($\Delta L_{\text{laser}}$) through a viewport, and the reflected beam position ($\Delta L_{\text{detector}}$) measured with a split photo-diode (position sensitive detector).

We have studied both heavily B-doped Si(001) and “regular” (i.e., low doping level) Si(001) surfaces. Figure 4.35 shows a regular Si(001) surface with no strain and an applied uniaxial strain. The insets are false color images in which (2x1) domains have been set to black and (1x2) domain to white. The applied strain causes the surface domain structure to become asymmetric. By counting the number of black and white pixels, the area of each domain is determined and a population asymmetry $p$
calculated. The same experiments for heavily B-doped Si are also shown in figure 4.35.

From data like that shown in figure 4.35 at various tensile and compressive strains, $p(e^\text{ext})$ is determined, and the results plotted in figure 4.36. The fitted slopes together with $l_{\text{regular}} \approx 30\text{nm}$ and $l_{\text{boron}} \approx 22\text{nm}$, result in: $F_0^{\text{regular}} = 1.09 \pm 0.12 \frac{eV}{\AA}$ (consistent a previously reported value [17, 21] of $F_0^{\text{Weib}} = 1.03 \pm 0.15 \frac{eV}{\AA}$) and $F_0^{\text{boron}} = 1.22 \pm 0.09 \frac{eV}{\AA}$, or $\frac{F_0^{\text{boron}}}{F_0^{\text{regular}}} = 1.12 \pm 0.14$. The surface stress anisotropy is then approximately 12% higher on the B-doped Si(001) surface than on regular Si(001), in agreement with the qualitative argument presented in section 4.2.1. We point out that the surface stress anisotropy, just as the biaxial tensile stress, should be a function of the annealing temperature if it is due to B accumulation. Although we have not measured the temperature dependence of the surface stress anisotropy, we expect $F_0$ to be larger for lower temperature anneals, and smaller for higher temperature anneals.

This increased surface stress anisotropy acts then as an extra driving force, in addition to the biaxial tensile strain, for the AVMJ step creation/relaxation mechanism. Using the parameter values given earlier, we estimate the values of $L_0$ shown in table 4.4, for a biaxial strain of 0.25% and a surface stress anisotropy equal to $1.12F_0^{\text{regular}}$. These predicted values should be compared with the observed values

<table>
<thead>
<tr>
<th>$\alpha$ ($a^2$/eV)</th>
<th>$L_0^{\text{unstrained}}$ (nm)</th>
<th>$L_0^{0.25%\text{strain}}$ (nm)</th>
<th>$L_0^{0.25%\text{strain}+1.12F_0}$ (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\alpha_{\text{min}}$ = 0.0042</td>
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<td>222</td>
<td>98</td>
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<tr>
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<td>46</td>
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<tr>
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<td>62</td>
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<td>28</td>
</tr>
</tbody>
</table>

Table 4.4: Calculated $L_0$ for B-doped Si(001) including increased stress anisotropy.
Figure 4.35: STM scans of regular and B-doped Si(001) surfaces for no strain and applied strain (tensile strain direction indicated by the arrows). Insets have been processed to show the (2x1) and (1x2) terraces as black and white respectively.
Figure 4.36: Data images like those in figure 4.35 are analysed to find the area fraction as a function of applied strain. The results are plotted here for both regular and heavily B-doped Si(001) surfaces annealed at 700°C.
\( l_{\text{boron}} \approx 22\text{nm} \) on these surfaces.

Clearly the increased surface stress anisotropy leads to a significant reduction in the value of \( L_0 \). We comment again that although using \( \alpha_{\text{max}} \) from table 4.4 results in relatively good agreement with our experimental observations on the B-doped samples, it predicts an \( L_0 \) for regular Si which seems too small. On the other hand using \( \alpha_{\text{min}} \) predicts a larger \( L_0^{\text{regular}} \) for unstrained Si (explaining the lack of previous observation of the striped phase), but it leads to a value of \( L_0^{\text{boron}} \) larger than what we observe. The exponential sensitivity of \( L_0 \) to the parameters in equation 4.1, may account for this problem. B-induced changes to \( E_A \) or even to \( \alpha \) may be enough to bring the calculations into better agreement with the experimentally observed behavior. Nevertheless these calculations show that the combination of biaxial tensile strain and increased surface stress anisotropy can result in a dramatic lowering of the value of \( L_0 \), and are within an order of magnitude of the observed values.

4.3 Strain Field Imaging on Si(001) Surfaces

We now turn to a different topic area that grew out of some interesting observations we noted while studying the stability of \( S_B \)-step fingers on the Si/SiGe samples discussed in section 4.1.3.

While in the last section the effects of an applied uniaxial strain were used to affect the relative domain populations at the surface (to determine the surface stress anisotropy), here we use observed domain population asymmetry to infer the presence of an intrinsic uniaxial strain on Si(001) surfaces. In particular, the surfaces of Si/SiGe samples provide evidence for a spatially varying uniaxial strain component at the surface that is highly correlated to the underlying cross-hatch pattern.
Since a uniaxial strain as small as 0.1% directed along a (110) direction (on samples with low-miscut) produces a surface with > 90% of the area consisting of terraces with the dimer row direction parallel (perpendicular) to the applied tensile (compressive) strain [17, 18, 21], the degree of uniaxial strain present near a surface may be monitored simply by observing variations in the local (2x1) and (1x2) domain populations. The original experiments by Men et al., used LEED to provide an average relative domain population over large areas, but it could not resolve microscopic lateral variations. Low-energy electron microscopy (LEEM) on the other hand, can form diffraction contrast images of the surface, allowing spatially resolved imaging of (2x1) and (1x2) domain populations with a routine spatial resolution of ~15nm [95]. In this way, LEEM may be used to image small lateral variations in the strain, essentially creating a two-dimensional (2D) map of local uniaxial strain fields at the surface.

Figure 4.37(a) shows a 7 μm field-of-view LEEM image of such a Si/Si0.7Ge0.3 sample after flashing to ~ 1025°C [20], in which (2x1) and (1x2) reconstructed regions appear dark and bright respectively [89]. At this length scale, we observe wavy step “fingers” and stripe-like surface structures [20, 3], which were discussed in section 4.1.3. STM experiments [20, 3, 4] indicate that the long axis of these step fingers and stripes are parallel to (110) directions, thus allowing determination of the surface crystal directions as shown.

Visible along the right hand side and bottom of figure 4.37(a), are two dark “bands” ~ 1μm wide running in the [110] and [110] directions, respectively. These bands are composed of extended areas in which nearly all adjacent (001) terraces exhibit the same dimer orientation, indicating that they are primarily separated by even-multiple-height steps (i.e., double-height, quadruple-height, etc.). As discussed
Figure 4.37: LEEM images of Si/Si$_{0.7}$Ge$_{0.3}$ cleaned by heating to 1025°C. (a) 7μm field of view showing fingered and striped step structures, and spatially separated uniaxial strain domains (USD's) which appear as dark or bright bands. (b) 20μm, and (c)&(d) 30μm field of view images. The USD's form ordered intersecting bands running parallel to the (110) directions.
above, this asymmetry away from equal areal densities of (2x1) and (1x2) domains indicates that a uniaxial strain is present at the surface [17, 18, 21]. Within a band, typically > 90% of the domains are of one type, suggesting a local uniaxial strain ≥ 0.1% [17]. Along the left hand side and near the center of figure 4.37(a), there are other areas with the opposite type of population asymmetry, which appear nearly all bright. This variation in the population asymmetry directly indicates that the uniaxial strain varies across the surface. We will refer to these dark/bright bands as “uniaxial strain domains” (USD’s). We note that residual stress due to sample mounting is not sufficient to explain these observations, since it would in general produce regions of uniaxial strain much larger in size than the μm-scale USD’s seen here.

The next question is whether these USD’s are connected with cross-hatch. Figure 4.37(b) shows a ~20 μm and figures 4.37(c) and 4.37(d) show larger (30 μm) LEEM images of different sample areas, and illustrate more clearly the dark/bright banding of the surface into different USD’s. A striking feature at these larger length scales is that the USD’s are arranged into a quasi-regular, rectangular pattern of intersecting bands aligned along ⟨110⟩ directions, with spacing and orientation very similar to the cross-hatch morphology typically observed on relaxed SiGe substrates [49, 50, 52, 53]. Figures 4.38(a) and (b) show 5 x 5 μm² STM and 10 x 10 μm² AFM images, respectively, of Si/Si₀.₇Ge₀.₃ samples prepared in an identical way to that shown in figure 4.37. These topographic measurements show a clear cross-hatch surface morphology, composed of locally elevated or depressed bands of material ~ 1 μm to 6 μm wide and up to 5-10 nm high, oriented parallel to ⟨110⟩ directions in a regular rectangular network. It is clear that the USD bands seen in figure 4.37 have essentially
the same lateral size, spacing, and orientation as the cross-hatch structures seen in figure 4.38. In passing, we note that it is possible to produce 2D USD maps of the surface directly from scanned probe images. In atomic resolution STM images, the domain asymmetry can be determined directly from the local dimer-row direction. In larger scale STM and AFM images (in which dimer rows are not directly resolved) USD’s may be monitored provided that well-resolved ML steps are visible, since the shape of a step edge indicates the dimer orientation on the upper terrace (see figure 4.39). Rough/wavy (smooth) step edges indicate the upper terrace dimers are oriented perpendicular (parallel) to the step edge direction.

The spatially-varying strain fields observed here, are almost certainly due to networks of sub-surface misfit dislocations, as was first suggested by Fitzgerald et al. [49, 50]. More recently, Shiryaev and co-workers [52] observed (particularly for lower-temperature growth conditions) that cross-hatch was associated with “self-organized” networks of misfit dislocations which often had the same Burger’s vector. Such organized dislocation networks are expected to produce long-range strain fields, which should extend to the surface [49]. The measurements presented here confirm that these strain fields do exist, and give rough estimates of their magnitudes and lateral spatial variations. They also suggest that real-time LEEM measurements could in the future prove to be a powerful method for studying large-scale strain relaxation phenomena during growth and annealing in the SiGe(001) system. We also note that recent studies of self-organized Ge clusters on Si(001) substrates indicate that lateral strain variations are critical to the self-ordering process [96]. The real-time evolution
Figure 4.38: (a) 5x5µm² STM image, and (b) 10x10µm² AFM image of another flashed Si/Si$_{0.7}$Ge$_{0.3}$ sample. Elevated (depressed) formed at the surface, form an intersecting network with long range order. It is this type of structure which leads to the crosshatch pattern observed on relaxed SiGe layers.
Figure 4.39: (a) the 5x5μm² STM image from figure 4.38 showing surface topography, and (b) the same image in which regions identified as (2x1) have been colored black, and regions identified as (1x2) have been colored white.
of such strain variations as well as their effect on cluster nucleation should be observable by the methods described here, provided that these strain variations have a uniaxial component.

4.4 Elastic Calculations for Miscut Surfaces

We now return to the interesting observation of the evolution of step shapes from straight, to triangular-tiled, to striped, for decreasing surface miscut on Si/SiGe, and for decreasing annealing temperature on B-doped Si(001) surfaces. All of the theoretical calculations presented previously, were based on the original Alerhand model geometry, which assumed a perfectly flat initial surface on which extra up and down steps are generated, resulting in a striped step surface. While Marchenko specifically mentions “parquet” structures [1], and Alerhand et al. present other specific tiling arrangements [14], these tiling patterns are still considered in the context of a perfectly flat or zero miscut surface. Although well suited for computation, surfaces with zero miscut are virtually impossible to access experimentally. Indeed all of the images presented so far have some finite surface misorientation which imposes a fixed number of surface steps per unit length. It therefore becomes important to consider surface misorientation within the elastic calculations. Recently, Ebner et al. [5] have performed isotropic elastic calculations for Si(001) surfaces with finite miscut and applied biaxial strain. We now review these results and compare them to the experimental observations.

Figure 4.40 shows the step configurations considered for a (001) surface misoriented towards a [110] direction. In the ideal (“regular”) case, the result is a vicinal surface with evenly spaced, parallel $S_A$ and $S_B$ steps alternating down the surface, as
indicated in figure 4.40(a). As mentioned earlier, the effect of strain relaxation at the step edges, may cause the initially straight step edges to wander (wavy steps). Tersoff and Pehlke [34] have previously shown that a surface on which $S_A$ and $S_B$ steps undulate together sinusoidally, should be lower in energy than straight steps within the framework of classical elasticity theory. However, since the energy required to create a segment of $S_B$ step is so much higher than that to create a segment of $S_A$ step, we expect the straight $S_A$ steps to remain essentially straight, while the rough $S_B$ steps are much more free to wander by the incorporation of $S_A$ segments into the $S_B$ step edge. The step configurations shown in figure 4.40 are a representative sample of the kinds of low energy excitations of the $S_B$ steps consistent with the imposed surface miscut. We should point out that Chen et al. [92, 97], have performed elastic calculations for the figure in 4.40(e) and found it to be lower in energy than the straight steps shown in figure 4.40(a), but the other step configurations shown in figure 4.40 were not considered.

In order to calculate the reduction in elastic energy due to step relaxation, specific step geometries must be assumed. Ebner et al. [5], has considered the step configurations shown in figure 4.40. The vicinal straight step case is shown in Fig. 4.40(a), a parallel striped structure consisting of rectangular steps arranged in-phase as shown in figure 4.40(b) was found to always be higher in energy than the out-of-phase structure shown in figure 4.40(c). Since the experimental observations show a high degree of stability of triangular step shapes, these geometries have also been investigated. Figure 4.40(d) is an approximation to a triangular step in which a finite number ($N$) of $S_A$ segments are inserted into an $S_B$ step. Figures 4.40(e) and (f) are effectively large $N$ in-phase and out-of-phase versions of figure 4.40(d) respectively. As in the
Figure 4.40: Step configurations for which Ebner et al. [5] have calculated the energy. (a) is the initial miscut surface, (b) is the striped phase with adjacent stripes in phase, (c) is the same as in (b) but with adjacent stripes out of phase, (d) is a triangular phase including kinks (small $N$ version of triangles) in an out of phase alignment, (e) is a triangular arrangement in phase, and (f) is a triangular arrangement out of phase.
case of the rectangular steps the in-phase structure was found to be higher in energy than the out-of-phase arrangement. We therefore may limit our discussion to the configurations shown in figures 4.40(a), (c), and (d).

The results of the elastic calculations for the case of biaxial tensile strain of 1% (and parameter values comparable to those presented earlier) are shown in figure 4.41. The upper solid, dashed, and dotted lines correspond to the surface energies as a function of $S_A$ step separation ($L$) for the straight, triangular-tiled, and striped surface phases respectively. For small $L$ (high miscut) straight steps are preferred, but with increasing terrace widths the triangular-tiled phase, in which both $\lambda$ and $N$ have been varied to minimize the surface energy, becomes energetically favored. Finally for large $L$ (low miscut), the triangular-tiled structure (with $N = 2$) merges with the "Alerhand" striped phase to be the most stable surface configuration. The lower two curves of figure 4.41 indicate how $\lambda$ varies as $L$ is changed. The predicted behavior of $\lambda$-vs-$L$ does not agree well with our experimental observations. In particular, we note that while the observe period of the triangular (wavy step) structure is an increasing function of $L$, it is not observed to have a larger period than the striped-step structure (see figure 4.16). Ebner et al. point out that the energy minimum with respect to $\lambda$ for the triangular structure is weak and very shallow, so that even small corrections (such as those coming from including the full crystal symmetry) may be enough to correct for this discrepancy.

If finite temperature effects are included in the calculations, the curves shown in figure 4.42 result. The finite temperature effects come from allowing configurational changes to the step structures, increasing the entropy, and hence lowering the overall
Figure 4.41: The surface energies of the original configuration [figure 4.40(a)] (solid line), the triangular configuration [figure 4.40(d)] including kinks and corners (upper dashed line), and the striped configuration [figure 4.40(c)] (upper dotted line) are given as functions of $L$ for $F_0 = 1.2\, \text{eV}/a^2$, $\sigma = 0.28$, $\mu = 23\, \text{eV}/a^3$, and $\Delta = 0.20$. Also shown are the equilibrium values of $\lambda$ for configurations [figure 4.40(d)] (lower dashed line) and [figure 4.40(c)] (lower dotted line). The latter two curves are referred to the right-hand vertical axis [from Ebner et al. [5]].
Figure 4.42: The surface energies of configuration [figure 4.40(d)] (dashed line) and [figure 4.40(c)] (dotted line) are shown as functions of $\lambda$ for $L = 200 \text{ nm}$. The various parameters are as in figure 4.41 [from Ebner et al. [5]].
free energy. From these calculations, Ebner et al. find that the triangular-tiled phase is stabilized (due to configurational entropy) over a wider range of terrace widths.

In addition to studying the energies as a function of terrace width, Ebner et al. have also investigated the dependence on changes in the surface stress anisotropy. Figure 4.43 shows this dependence for $L = 100\text{nm}$, indicating that there is a progression from straight steps to triangular-tiled to striped, as the surface stress anisotropy is increased. A similar calculation for $L = 300\text{nm}$ is shown in figure 4.44, and shows the same progression of straight—triangular-tiled—striped behavior.

We are now in a position to understand the observed step shape transition on both the Si/SiGe surface, as well as the heavily B-doped Si(001) surface. Referring once again to figure 4.14 we find the essential features of straight steps at small terrace widths which take on a triangular shape and finally a very elongated (stripe-like) nature as the terrace width increases. The out-of-phase triangular tiling arrangement in more apparent from figure 4.20 where there are several terraces of equal intermediate width. For the very wide ($<1\mu\text{m}$) terraces of figure 4.21 the striped step structure is observed. For the case of B-doped Si the step shape as a function of temperature shown in figure 4.23, again shows the progression from straight to triangular-tiled to striped as the annealing temperature is lowered [see in particular figure 4.23(a), (b), and (d)]. In this case the lower annealing temperatures result in higher surface B accumulation and hence larger biaxial tensile strain and larger surface stress anisotropy.

We may effectively restate the essential physics of the step shapes as follows: for $L << L_0$ the surface has already achieved full stress relaxation in the direction normal to the global step direction, for $L >> L_0$ the surface creates Alerhand stripes which
Figure 4.43: The surface energies of configuration [figure 4.40(a)] (solid lines), [figure 4.40(d)] (upper dashed lines), and [figure 4.40(c)] (upper dotted lines) are shown as functions of $F_0$ for $\sigma = 0.28$, $\mu = 23\,\text{eV/Å}^3$, and $\Delta = 0.0$ at $L = 100\,\text{nm}$ Also shown, and referred to the right-hand vertical axes, are the equilibrium values of $\lambda$ for configuration [figure 4.40(d)] (lower dashed lines) and [figure 4.40(c)] (lower dotted lines) [from Ebner et al. [5]].
Figure 4.44: Same as figure 4.43, but with $L = 300 \text{ nm}$ [from Ebner et al. [5]].
allow for full stress relaxation now in a direction parallel to the global step direction, and for the intermediate range of $L \approx L_0$ the triangular-tiled structure acts as a bridge between the other extrema. We may cross from one regime to the next either by (1) as in the case of Si/SiGe varying $L$ (local miscut) while $L_0$ remains fixed, or (2) at fixed $L$ (constant miscut) varying a surface parameter (such as surface B accumulation) which results in a change in $L_0$. 
CHAPTER 5

CONCLUSIONS

The theoretical work of Marchenko [1] along with Alerhand, Vanderbilt, Meade, and Joannopoulos (AVMJ) [2], has provided much insight into explaining various phenomena at Si(001)-(2x1) surfaces. We have seen that the single—double height step transition, as well as the observed changes in domain populations under the application of a uniaxial strain, may be understood in terms of stress relaxation effects at step edges. However, the central prediction of the AVMJ model, the spontaneous creation of extra up/down $S_A$ steps forming a striped surface phase, went without observation for many years. This work has provided the first direct observation of striped step structures on Si(001) surfaces, confirming the formation of stress domains [1] by the AVMJ [2] mechanism.

5.1 Major Findings

For the case of Si grown under biaxial tensile strain on relaxed SiGe substrates, we have found stable wavy-step structures which evolve into elongated step structures as the local terrace width increases [3], and have suggested that these structures are stabilized by strain-enhanced surface stress-relaxation effects. We have proposed a
physical mechanism to explain why biaxial tensile strain should enhance spontaneous step creation on these surfaces.

Perhaps the most striking observations of this work, are of an equilibrium, quasi-periodic striped-step structure on heavily B-doped Si(001)-(2×1) surfaces annealed in UHV. This system shows a striking transitions in the size and shape of the step structures as the sample temperature is changed [4]. We have proposed here, that these structures result from Marchenko-Alerhand (M-A) stress relaxation effects [1, 2], enhanced by a temperature-dependent surface B segregation. The effect of B segregation is to produce a surface layer under a biaxial tensile strain (like that for Si/SiGe), and measurements reported here indicate a ~ 12% increase in the surface stress anisotropy ($F_q$) over that for regular Si(001) surfaces. The combined effect of biaxial tensile strain and increased surface stress anisotropy, have been argued here to cause the spontaneous formation of steps resulting in the observed striped surface phase.

The shape of the structures observed for both Si/SiGe(001) and B-doped Si(001) are consistent with recent isotropic elastic calculations of Ebner et al. [5], which show the same progression of step structures from straight—triangular-tiled—striped, as the terrace width, surface stress anisotropy, or biaxial tensile strain is increased. This transition may be understood in terms of a relative change in magnitude of the miscut imposed terrace width $L = d_A/2$, and the Alerhand critical terrace width $L_0$. Figure 5.1 shows the correspondence between the structures observed, and the most stable structures considered by Ebner et al. [5]. For $L_0 >> d_A$ stress relaxation is already taken care of by the steps imposed by the surface miscut and the surface has the regular straight $S_A$-step alternating with a randomly rough $S_B$-step configuration.

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Figure 5.1: Comparison of observed and modeled step shapes as a function of the relative magnitudes of $L$ and $L_0$. 

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For $L_0 \approx d_A$ triangular steps result, now with a straight $S_A$-step alternating with a triangular (wavy) $S_B$-step. Finally for $L_0 << d_A$, the surface takes on the true Alerhand stripe phase appearance, with periodic up/down $S_A$ steps in a direction perpendicular to the miscut direction.

In addition, we have shown that LEEM (and with considerably more difficulty STM/AFM) can be used to image residual uniaxial strain fields on SiGe(001) films, and find a high degree of correlation between lateral strain variations (most likely due to sub-surface networks of misfit dislocations) and the surface cross-hatch morphology on strain-relaxed Si$_{0.7}$Ge$_{0.3}$ substrates.

5.2 Future Directions

The value of $F_0$ for the striped surface phase calculated in this work, assumes that the original theory developed by Alerhand et al. is valid for surfaces with an imposed surface miscut. This is certainly an area in which further study may lead to a better understanding of these effects. At present Dr. Charles A. Ebner and co-workers are investigating the interaction of applied uniaxial strain and stress relaxation for miscut surfaces. The results of their work may provide helpful insight for interpreting this and future data.

The question raised in section 4.2.1 as to whether high B doping may change the $S_A$ step edge energy, remains an open question. Studying the detailed dynamics of B-doped Si(001) step edges, using real time STM at elevated temperatures, may provide valuable information about the $S_A$ step edge energy, as well as detailed insight into the initial stages of spontaneous step creation.
It is also of interest to conduct experiments aimed at correlating the point defect density, finger spacing, annealing temperatures, and annealing times for heavily B-doped Si(001) surfaces. These experiments should provide additional information regarding the mechanism of B-induced changes in surface tensile strain and stress anisotropy.

There are several interesting directions for future research in this area. One very elegant experiment that would seem to settle once and for all, the question of why the AVMJ striped phase has not previously been observed on regular Si(001) surfaces, is to externally biaxially strain a thin Si(001) wafer and look for the presence of step stripes. A similar experiment has already been done for Si(111) by Wei, Li, and Tsong [98], looking for a surface reconstruction phase transition from (7x7) to c(2x8). The proposed experiment would involve mounting a thin Si wafer membrane in front of an UHV-STM and using a piston from behind to biaxially strain the sample. One would ideally like to flash a Si(001) sample (without strain) and image the surface to verify that the observed step structure is that of regular Si. One would then anneal the same sample, now with biaxial strain, and image the surface. If stripes were observed, then it seems clear that the biaxial strain serves to "tip the balance" in favor of spontaneous step creation.

Another experiment that our group has considered is to deposit a sub-monolayer coverage of Ge on one of the B-doped Si(001) samples with triangular steps. The tip of the step triangles should be maximally strain relieved, so they would likely be preferential sites for Ge nucleation [96, 92]. There is currently much interest in creating self-assembled nanostructures, and this system would seem to hold considerable promise for generating a regular array of "quantum-dots", with spacing that is easily
selected. This work has already shown that the finger spacing on the B-doped Si(001) surface may be readily changed simply by varying the annealing conditions.

Clearly the effects of surface stress and strain upon the surface morphology of 2-D and 3-D structures is a rich and diverse area, and one which will likely provide many more exciting possibilities for future research.
APPENDIX A

UHV3 STM COMPONENT DRAWINGS

The following figures provide detailed drawings of individual STM and related components, designed by this author for the work described in this document. Every attempt has been made to provide as complete drawing set as possible, however there are several components for which detail drawings do not exist at this time.
**Figure A.1: STM stage.**

*All holes are 3/32 tapped unless otherwise specified.

*Center hole (marked by ○) is 6-32 tapped.
Figure A.2: STM clamp down ring.
Figure A.3: STM vertical height screw.
Figure A.4: STM vertical height screw plug.
Figure A.5: STM clamp down ring spacers.
Figure A.6: STM rotation adaptor of vertical height adjust screw.
Figure A.7: STM retainer ring.

Type 304 Stainless
Make 1
Scale 1:1
Figure A.8: Tip stage scanner base block.

Scale 2:1
Material = "Super Invar"
Figure A.9: Tip stage scanner inchworm motor shaft mounts.
Figure A.10: Tip stage scanner slider stage.
Figure A.11: Tip stage scanner inchworm motor housing mount.
Figure A.12: Sample stage contacts.
Figure A.13: Sample stage contact insulators.
Figure A.14: Sample stage contact supports.
Figure A.15: Sample stage retainers.

Holes are #2-56
Clear, Counter bored
for socket cap screws
as shown.

Type 304 Stainless

1. Make one part with $A = 0.350$ $B = 0.100$
2. Make one part with $A = 0.450$ $B = 0.200$

Scale 4:1 [Not all features are to scale]
Figure A.16: Free floating sample stage.
D. Jones 7/30/96 Free Float Sample Stage

<table>
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<th>Datum A</th>
<th>Datum B</th>
<th>Datum C</th>
<th>Datum A</th>
<th>Datum B</th>
<th>Datum C</th>
<th>Datum A</th>
<th>Datum B</th>
<th>Datum C</th>
<th>Datum A</th>
<th>Datum B</th>
<th>Datum C</th>
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<td>0.125</td>
<td>approx. 0.300</td>
<td>-0.050 Clearance Vent Hole</td>
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<td>0.125</td>
<td>approx. 0.300</td>
<td>-0.050 Clearance Vent Hole</td>
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<td>0.900</td>
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<td>3/16&quot; Magnet Recess</td>
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<td>3/16&quot; Magnet Recess</td>
<td>0.700</td>
<td>0.812</td>
<td>0.031</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3/16&quot; Magnet Recess</td>
<td>0.900</td>
<td>0.833</td>
<td>0.031</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3/16&quot; Magnet Recess</td>
<td>1.150</td>
<td>0.187</td>
<td>0.031</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3/16&quot; Magnet Recess</td>
<td>1.150</td>
<td>0.500</td>
<td>0.031</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>3/16&quot; Magnet Recess</td>
<td>1.150</td>
<td>0.812</td>
<td>0.031</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Note: The Left and Right Sides each have features as shown, formed with a 60 degree included angle countersink tool to a depth of 0.094.
Figure A.18: Sample stage slider frame.
Figure A.19: Sample stage inchworm motor mount clamp.
Figure A.20: Sample block.
Figure A.21: Sample block pick-up bar.
Figure A.22: Sample block contact clips.
Figure A.23: Sample block heat shield.
Figure A.24: Tip holder.
Figure A.25: Getter pump heat shield # 2.
Figure A.26: Getter pump heat shield # 1.
Figure A.27: Getter pump support brackets.
Figure A.28: Getter pump nipple.
Figure A.29: Getter pump base support flange.
Type 304 Stainless

Figure A.30: Getter pump heat shield # 3.
Figure A.31: Getter pump heating flange.
Figure A.32: Getter pump heater coil.
Figure A.33: Uniaxial strain clamp.

Material is 2mm Thick Tantalum Stock

* Make 2 as follows:
  1 - with S = 0.750, L = 0.906 (Shown)
  1 - with S = 0.781, L = 0.937 (Not shown)
Figure A.34: Uniaxial strain post.
# Packing Slip and Certificate of Test

## Customer Information
- **Customer:** Ohio State University
- **Contact:** Darrell Jones
- **Address:** 174 W 18th Ave, Columbus, Ohio 43210

## Packaging and Certificate of Test
- **Date Shipped:** 3-28-93
- **Order No.:** 10202

## Alloy
- **Alloy:** Super Invar
- **Condition:** Hot Rolled Annealed & Descaled

## Item Specifications
<table>
<thead>
<tr>
<th>Item</th>
<th>Material</th>
<th>Pieces</th>
<th>Weight (lbs)</th>
<th>Size</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>31928</td>
<td>1</td>
<td></td>
<td>4.60 x 1/2&quot; TH</td>
</tr>
<tr>
<td>1</td>
<td>1</td>
<td>1</td>
<td></td>
<td>1/2 x 6 x 6&quot;</td>
</tr>
</tbody>
</table>

## Chemical Analysis
<table>
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<tr>
<th>Item</th>
<th>C</th>
<th>Fe</th>
<th>Mn</th>
<th>Ni</th>
<th>Cu</th>
<th>Cr</th>
<th>Fe</th>
<th>*/o</th>
<th>BAL</th>
<th>Al</th>
<th>Ti</th>
<th>B</th>
<th>Zr</th>
<th>Mg</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.42</td>
<td>.020</td>
<td>.013</td>
<td>.002</td>
<td>.14</td>
<td>.25</td>
<td>31.70</td>
<td>.11</td>
<td></td>
<td></td>
<td>.02</td>
<td>.04</td>
<td>.01</td>
<td>.01</td>
<td></td>
</tr>
</tbody>
</table>

## Physical Properties
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>1.42</td>
<td>0-50°F</td>
<td>.050</td>
<td>.122</td>
<td>.00</td>
<td>.153</td>
<td>.150</td>
<td>.263</td>
</tr>
</tbody>
</table>

[Figure A.35: Invar specification sheet]
**PROCEDURE, DATA, USV INCHWORMS**

**Model #:** UHVM 050  **Serial #:** M59P115  **PCT Tube Vendor:** Staglum  **PCT Tube PO #:** 743-1  **Date: 16-JUN-1993 ECN #:** 2191

<table>
<thead>
<tr>
<th>Parameter Tested</th>
<th>Description of Test</th>
<th>Measured Result</th>
<th>Required Result</th>
<th>Tested By</th>
<th>Date Tested</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surface Defect</td>
<td>Visual inspection</td>
<td>✓, Minor visible</td>
<td>Run the computer as noted on the PCT tube procurement drawings.</td>
<td></td>
<td>7/493</td>
</tr>
<tr>
<td>Tube Movement At 1000 Volts</td>
<td>Use interferometer to measure actual (ALT) movement, measure 4 sections 25% sample from each batch of PCT tube assembly.</td>
<td>7.9 mm, acceptable</td>
<td>Use the requirements as noted on the PCT tube procurement drawings.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Contact Area of Clamps</td>
<td>Measure shall also meet and maintain the percent of contact area for each clamp</td>
<td>90% x Clamp 1</td>
<td>For information only.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Capacitance Clamp 1: Center: Clamp 2:</td>
<td>Measure with capacitance meter</td>
<td>4.70 mF, mF, mF</td>
<td>For information only.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Maximum Forward Speed (1)</td>
<td>Use a stop watch to record the time required to travel a small distance.</td>
<td>10.5 sec.</td>
<td>&gt;1.0 m/sec.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Maximum Backward Speed(1)</td>
<td>Use a stop watch to record the time required to travel a small distance.</td>
<td>12.0 sec.</td>
<td>&gt;1.0 m/sec.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Maximum Push Force (Hold Line)(1)</td>
<td>Push the motor at maximum speed and measure the maximum push force at still with the spring inside.</td>
<td>43.0 kgf</td>
<td>&gt;5.0 kgf</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Rest Force(1)</td>
<td>Measure force required to slides the motor with clamp at 0 Volts and all springs compressed at 12VDC.</td>
<td>5.0 kgf</td>
<td>For information Only.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Power Off Force</td>
<td>Measure force required to slides the motor with clamp at 0 Volts and all springs compressed at 12VDC.</td>
<td>12.0 kgf</td>
<td>For information Only.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Maximum Forward (Hold)(1)</td>
<td>Measure motion Profile and confirm forward throttle is used (blue line).</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Motion Profile**

---

**Figure A.36:** Inchworm motor specification sheet.
Figure A.37: STM transfer boat.
Figure A.38: STM grabber body.
Figure A.39: STM grabber rod.
Figure A.40: Main grabber coupler/clamp.
Figure A.41: Main grabber feed-through collar.
Figure A.42: Turbo pump port blank.
Figure A.43: Main grabber slider rod and pin.
Figure A.44: Main chamber sample tray.
Figure A.45: Sample tray mount.

Clearance Slots, Counter-Sunk, to accept #10-32 (25° Slot) located on 0.500 radius from datum -A-

Drill and Tap for #4-40 thread depth of 0.500

Outer Holes should be drilled Clear thru Center Holes should be drilled Clear until penetration of the middle slot.

Scale 2:1

Aluminum
Figure A.46: Tip bomber insulator block.
Figure A.47: Tip bomber contact clamp.
APPENDIX B

STM ELECTRONICS SCHEMATICS

The following figures provide circuit schematics of some of the STM electronics. This is not a comprehensive compilation of circuit schematics, additional drawings should be available from the Physics Department Electronics Shop.
Figure B.1: Tip bias ramp generator # 1.
Figure B.2: Tip bias ramp generator # 2.
Figure B.3: Tip pulser.
Figure B.5: Tip pre-amplifier.
Figure B.7: Tilt compensator.
Figure B.8: Sweep rotator.
APPENDIX C

COMPUTER PROGRAM LISTINGS

C.1 Inchworm Approach Program

The following program (djapp5m.ibw) was written to control the clamping sequence of the inchworm motors used to position the sample, and to approach the tip to the sample for tunneling. The program runs under Hewlett-Packard instrument basic, which is a Windows based GPIB interface control package.

```
10 REM  This program written 03/15/93 by Darrell Jones
20 REM  Modified 04/21/93 by P. Grillot
30 REM  Modified 05/26/93 by D. Jones for tip retract
40 REM  and IH end commands appended to dig stmts
50 REM  Modified 07/08/93 by D. Jones to move all
60 REM  DAC parameter variables to header
70 REM  Requires HP-54601A Scope with GPIBaddr=3
80 REM  and OSU-M588K GPIB Inch-Worm Motor controller
90 REM  with GPIBaddr=4
100 REM  *******************************
110 OPTION BASE 1
120 DIM Buf$(100]
130 DIM Butn$(1)[30]
140 DIM Strup$(80]
150 DIM Step_no$(30]
160 REM  INITIALIZE INCH WORM CONTROLLER
170 ASSIGN @Scope TO 1403
180 ASSIGN @Iworm TO 1404
190 REM  Set-up inchworm motor with one end clamped
200 REM  and the center electrode voltage set to zero
210 OUTPUT @Iworm:" JOG1IH "
220 OUTPUT @Iworm:" IHTL000 
230 Tip_sam_flag$="Tip" ! Initialize default setting
240 Dac_current$=" L000 " ! Initialize the DAC current setting
250 Dac_set$=" L000 " ! Initialize the DAC set-point value
260 Step_direction$="Neither" ! Initialize step direction
```
REM  ******** Set-up DAC stepsizes for all inchworm steps
*
REM
320 Tip_auto_fwd$= "LDAO" !was 600 WAS 240
330 Sam_auto_fwd$= "I400" !WAS 800
340 Tip_auto_rev$ = "L078" !WAS 800
350 Sam_auto_rev$ = "L078" !WAS 800
360 Tip_multi_fwd$= "LDD9" !was 400
370 Sam_multi_fwd$= "LC19" !was 400
380 Tip_multi_rev$= "L999" !was 8DA
390 Sam_multi_rev$= "L8DA"
400 Tip_small_fwd$= "L400"
410 Sam_small_fwd$= "L800"
420 Tip_small_rev$= "L078"
430 Sam_small_rev$= "L426"
440 Tip_single_fwd$= "I400"
450 Sam_single_fwd$= "I400"
460 Tip_single_rev$= "L078"
470 Sam_single_rev$= "L078"
480 REM
490 Dac_settle_time=.1
500 Tip_retract_time=.5 !WAS .5
510 Waste_factor=2000 !was 2000
520 REM
530 Threshold_volt=.02 !was 0.005
540 REM
550 REM
560 REM  ******** -- ---------------------------------------
570 REM
580 REM
590 REM SET UP CONTROL PANEL
600 ASSIGN @Panel TO WIDGET "PANEL";SET("TITLE":"Micro InchWorm Controller and Auto-Approach")
610 CONTROL @Panel;SET("BACKGROUND":15,"X":25,"Y":20,"WIDTH":600,"HEIGHT":350,"RESIZABLE":0,"VISIBLE":0)
620 REM
630 REM
640 REM WRITE LABELS TO PANEL
650 Assign @L1 TO WIDGET "LABEL";PARENT @Panel,SET("X":0,"Y":60,"BORDER":0,"WIDTH":600,"BACKGROUND":15,"VALUE":"Fastest")
660 Assign @L2 TO WIDGET "LABEL";PARENT @Panel,SET("X":0,"Y":260,"BORDER":0,"WIDTH":600,"BACKGROUND":15,"VALUE":"Slowest")
670 Assign @L3 TO WIDGET "LABEL";PARENT @Panel,SET("X":105,"Y":35,"BORDER":0,"BACKGROUND":15,"WIDTH":70,"HEIGHT":16,"VALUE":"REVERSE")
680 Assign @L4 TO WIDGET "LABEL";PARENT
@Panel, SET("X":220,"Y":35,"BORDER":0,"BACKGROUND":15,"WIDTH":70,
"HEIGHT":16,"VALUE":"FORWARD")
690 ASSIGN @L5 TO WIDGET "LABEL"; PARENT @Panel,
SET("X":130,"Y":65,"BORDER":0,"BACKGROUND":15,"WIDTH":100,
"HEIGHT":16,"VALUE":"SINGLE")
700 ASSIGN @L6 TO WIDGET "LABEL"; PARENT @Panel,
SET("X":127,"Y":105,"BORDER":0,"BACKGROUND":15,"WIDTH":100,
"HEIGHT":16,"VALUE":"MULTIPLE")
710 ASSIGN @L7 TO WIDGET "LABEL"; PARENT @Panel,
SET("X":127,"Y":145,"BORDER":0,"BACKGROUND":15,"WIDTH":100,
"HEIGHT":16,"VALUE":"SMALL")
720 ASSIGN @L8 TO WIDGET "LABEL"; PARENT @Panel,
SET("X":127,"Y":185,"BORDER":0,"BACKGROUND":15,"WIDTH":100,
"HEIGHT":16,"VALUE":"AutoApp")
730 ! REM ASSIGN @L9 TO WIDGET "LABEL"; PARENT @Panel,
SET("X":127,"Y":225,"BORDER":0,"BACKGROUND":15,"WIDTH":100,
"HEIGHT":16,"VALUE":"1 nm")
740 ! REM ASSIGN @L10 TO WIDGET "LABEL"; PARENT @Panel,
SET("X":127,"Y":265,"BORDER":0,"BACKGROUND":15,"WIDTH":100,
"HEIGHT":16,"VALUE":"1 A")
750 ASSIGN @L11 TO WIDGET "LABEL"; PARENT @Panel,
SET("X":245,"Y":65,"BORDER":0,"BACKGROUND":15,"WIDTH":100,
"HEIGHT":16,"VALUE":"SINGLE")
760 ASSIGN @L12 TO WIDGET "LABEL"; PARENT @Panel,
SET("X":237,"Y":105,"BORDER":0,"BACKGROUND":15,"WIDTH":100,
"HEIGHT":16,"VALUE":"MULTIPLE")
770 ASSIGN @L13 TO WIDGET "LABEL"; PARENT @Panel,
SET("X":242,"Y":145,"BORDER":0,"BACKGROUND":15,"WIDTH":100,
"HEIGHT":16,"VALUE":"SMALL")
780 ASSIGN @L14 TO WIDGET "LABEL"; PARENT @Panel,
SET("X":245,"Y":185,"BORDER":0,"BACKGROUND":15,"WIDTH":100,
"HEIGHT":16,"VALUE":"AutoApp")
790 ASSIGN @L15 TO WIDGET "LABEL"; PARENT @Panel,
SET("X":0,"Y":105,"BORDER":0,"BACKGROUND":15,"WIDTH":100,
"HEIGHT":16,"VALUE":"TOGGLE")
800 ASSIGN @L16 TO WIDGET "LABEL"; PARENT @Panel,
SET("X":0,"Y":155,"BORDER":0,"BACKGROUND":3,"WIDTH":100,
"HEIGHT":16,"VALUE":"TIP")
810 REM
**********************************************************************************
820 REM
**********************************************************************************
830 REM SET UP BUTTON SHAPE, ETC.
840 ASSIGN @Button1 TO WIDGET "PUSHBUTTON"; PARENT @Panel
850 CONTROL @Button1; SET("BACKGROUND":2,"LABEL":"","HEIGHT":30,
"WIDTH":30,"X":110,"Y":60)
860 ASSIGN @Button2 TO WIDGET "PUSHBUTTON"; PARENT @Panel
870 CONTROL @Button2; SET("BACKGROUND":2,"LABEL":"","HEIGHT":30,
"WIDTH":30,"X":110,"Y":100)
880 ASSIGN @Button3 TO WIDGET "PUSHBUTTON"; PARENT @Panel
890 CONTROL @Button3; SET("BACKGROUND":2,"LABEL":"","HEIGHT":30,
"WIDTH":30,"X":110,"Y":140)
900 ASSIGN @Button4 TO WIDGET "PUSHBUTTON"; PARENT @Panel
910 CONTROL @Button4; SET("BACKGROUND":2,"LABEL":"","HEIGHT":30,
"WIDTH":30,"X":110,"Y":180)
920 ASSIGN @Button5 TO WIDGET "PUSHBUTTON"; PARENT @Panel
930 CONTROL @Button5; SET("BACKGROUND":2,"LABEL":"","HEIGHT":30,
"WIDTH":30,"X":220,"Y":60)
940 ASSIGN @Button6 TO WIDGET "PUSHBUTTON"; PARENT @Panel
950 CONTROL
@Button6;SET("BACKGROUND" :2 , "LABEL" :"", "HEIGHT" :30,
"WIDTH" :30 , "X" :220 , "Y" :100)
960 ASSIGN @Button7 TO WIDGET "PUSHBUTTON";PARENT @Panel
970 CONTROL
@Button7;SET("BACKGROUND" :2 , "LABEL" :"", "HEIGHT" :30,
"WIDTH" :30 , "X" :220 , "Y" :140)
980 ASSIGN @Button8 TO WIDGET "PUSHBUTTON";PARENT @Panel
990 CONTROL
@Button8;SET("BACKGROUND" :2 , "LABEL" :"", "HEIGHT" :30,
"WIDTH" :30 , "X" :220 , "Y" :180)
1000 ASSIGN @Button9 TO WIDGET "PUSHBUTTON"; PARENT @Panel
1010 CONTROL
@Button9;SET("BACKGROUND" :2 , "LABEL" :"", "HEIGHT" :30,
"WIDTH" :30 , "X" :220 , "Y" :120)
1020 ASSIGN @Button10 TO WIDGET "PUSHBUTTON";PARENT @Panel
1030 CONTROL
@Button10;SET("BACKGROUND" :3,"LABEL" :"QUIT", "X" :350 , "Y" :260)
1040 REM
1050 ASSIGN @List TO WIDGET "LABEL";SET("VALUE" :"Status Log"),PARENT @Panel
1060 CONTROL
@List;SET("X" :350 , "Y" :20 , "WIDTH" :230 , "HEIGHT" :20 , "BORDER" :0)
1080 REM *****************************************************
1090 REM ASSIGN BUTTON ROUTINES
1100 CONTROL @Panel;SET("VISIBLE" :1)
1110 ON EVENT @Button1."ACTIVATED" GOSUB Singlerev
1120 ON EVENT @Button2,"ACTIVATED" GOSUB Multirev
1130 ON EVENT @Button3,"ACTIVATED" GOSUB Smallrev
1140 ON EVENT @Button4,"ACTIVATED" GOSUB Auto_rev
1150 ON EVENT @Button5,"ACTIVATED" GOSUB Singlefwd
1160 ON EVENT @Button6,"ACTIVATED" GOSUB Multifwd
1170 ON EVENT @Button7,"ACTIVATED" GOSUB Smallfwd
1180 ON EVENT @Button8,"ACTIVATED" GOSUB Auto_fwd
1190 ON EVENT @Button9,"ACTIVATED" GOSUB Toggle
1200 ON EVENT @Button10,"ACTIVATED",VAL(SYSTEM$("SYSTEM PRIORITY")) +1 GOSUB End_program
1210 REM
1220 REM ************************************************************************
1230 LOOP  ! Wait for the user to
1240 END LOOP  ! Click on an application button
1250 REM
1260 REM
1270 Auto_fwd:
!  
1280 Step_direction$="forward"
1290 IF TipSam_flag $="Tip" THEN
1300 Dac_set$=Tip_auto_fwd$
1310 ELSE
1320 Dac_set$=Sam_auto_fwd$
1330 END IF
1340 GOSUB Dac_setup
1350 GOSUB Auto_step
1360 RETURN
1370 REM
1380 REM ************************************************************************
1390 Auto_rev:
1400 Step_direction$="reverse"
1410 IF Tip_sam_flag$="Tip" THEN
1420 Dac_set$=Tip_auto_rev$
1430 ELSE
1440 Dac_set$=Sam_auto_rev$
1450 END IF
1460 GOSUB Dac_setup
1470 GOSUB Auto_step
1480 RETURN
1490 REM
1500 REM
1510 Multifwd:
1520 Step_direction$="forward"
1530 IF Tip_sam_flag$="Tip" THEN
1540 Dac_set$=Tip_multi_fwd$
1550 ELSE
1560 Dac_set$=Sam_multi_fwd$
1570 END IF
1580 GOSUB Dac_setup
1590 GOSUB Mult_step
1600 RETURN
1610 REM
1620 REM
1630 Multirev:
1640 Step_direction$="reverse"
1650 IF Tip_sam_flag$="Tip" THEN
1660 Dac_set$=Tip_multi_rev$
1670 ELSE
1680 Dac_set$=Sam_multi_rev$
1690 END IF
1700 GOSUB Dac_setup
1710 GOSUB Mult_step
1720 RETURN
1730 REM
1740 REM
1750 Smallfwd:
1760 Step_direction$="forward"
1770 IF Tip_sam_flag$="Tip" THEN
1780 Dac_set$=Tip_small_fwd$
1790 ELSE
1800 Dac_set$=Sam_small_fwd$
1810 END IF
1820 GOSUB Dac_setup
1830 GOSUB Mult_step
1840 RETURN
1850 REM
1860 REM
1870 Smallrev:
1880 Step_direction$="reverse"
1890 IF Tip_sam_flag$="Tip" THEN
1900 Dac_set$=Tip_small_rev$
1910 ELSE
1920 Dac_set$=Sam_small_rev$
1930 END IF
1940 GOSUB Dac_setup
1950 GOSUB Mult_step

225
1960  RETURN
1970  REM
1980  REM
1990  REM
2000  Step_direction$="forward"
2010  IF  Tip_sam_flag$="Tip" THEN
2020    Dac_set$=Tip_single_fwd$
2030  ELSE
2040    Dac_set$=Sam_single_fwd$
2050  END IF
2060  GOSUB Dac_setup
2070  GOSUB Sing_step
2080  RETURN
2090  REM
2100  REM
2110  REM
2120  Step_direction$="reverse"
2130  IF  Tip_sam_flag$="Tip" THEN
2140    Dac_set$=Tip_single_rev$
2150  ELSE
2160    Dac_set$=Sam_single_rev$
2170  END IF
2180  GOSUB Dac_setup
2190  GOSUB Sing_step
2200  RETURN
2210  REM
2220  REM
2230  Forwardstep:  !  Was Forwardstep
2240  OUTPUT @Iworm;" G3 "
2250  GOSUB Delay
2260  REM actuate clamp3, (clamp1 on, center contracted)
2270  OUTPUT @Iworm;" G7 "
2280  GOSUB Delay
2290  REM release clamp1, (clamp3 on, center contracted)
2300  OUTPUT @Iworm;" G6 "
2310  GOSUB Delay
2320  REM extend center2, (clamp3 on, clamp1 off)
2330  OUTPUT @Iworm;" G4 "
2340  GOSUB Delay
2350  REM actuate clamp1, (clamp3 on, center extended)
2360  OUTPUT @Iworm;" G5 "
2370  GOSUB Delay
2380  REM release clamp3, (clamp1 on, center extended)
2390  OUTPUT @Iworm;" G1IH "
2400  RETURN
2410  REM
2420  REM
2430  Reversestep:  !  Was Reversestep
2440  REM clamp3 on, center extended
2450  OUTPUT @Iworm;" G5 "
2460  GOSUB Delay
2470  REM  (clamp1 off, center extended)
2480  OUTPUT @Iworm;" G4 "
2490  GOSUB Delay
(clamp3 on, contract center)
2510  OUTPUT @Iworm;" G6 "
2520  GOSUB Delay
2530  REM  (clamp1 on)
2540  OUTPUT @Iworm;" G7 "
2550  GOSUB Delay
2560  REM  (clamp3 off)
2570  OUTPUT @Iworm;" G3 "
2580  GOSUB Delay
2590  REM  (center extended)
2600  OUTPUT @Iworm;" GlIH "
2610  RETURN
2620
2630  REM  **************************************************************
2640  Delay:  !
2650  FOR Waste=1 TO Waste_factor
2660   NEXT Waste
2670  RETURN
2680  **************************************************************
2690  REM  **************************************************************
2700  Toggle:  !
2710  IF Tip sam_flag$="Tip" THEN
2720   OUTPUT @Iworm;" IHS "
2730  Tip sam_flag$="Sample"
2740  ASSING @L16 TO WIDGET "LABEL";PARENT @Panel_set("X":0,"Y":155,"BORDER":0,"BACKGROUND":2,"WIDTH":100,
2750  "HEIGHT":16,"VALUE":"SAMPLE")
2760  ELSE
2770   OUTPUT @Iworm;" IHT "
2780  Tip sam_flag$="Tip"
2790  ASSIGN @L16 TO WIDGET "LABEL";PARENT @Panel_set("X":0,"Y":155,"BORDER":0,"BACKGROUND":3,"WIDTH":100,
2800  "HEIGHT":16,"VALUE":"TIP")
2810  END IF
2820  RETURN
2830  REM  **************************************************************
2840  Dac_setup:  !
2850  IF Dac current$<>Dac_set$ THEN
2860   OUTPUT @Iworm;Dac_set$
2870   WAIT Dac_settle_time
2880   Dac current$=Dac_set$
2890  END IF
2900  RETURN
2910  REM  **************************************************************
2920  Auto_step:  !
2930  Halt$="false"
2940  ASSIGN @Button20 TO WIDGET "PUSHBUTTON";PARENT @Panel
2950  CONTROL @Button20;SET("BACKGROUND":3,"LABEL":"HALT","X":137,"Y":260)
2960  ON EVENT @Button20,"ACTIVATED",VAL(SYSTEM$("SYSTEM
2970  PRIORITY""))+# GOSUB Halt
2980  CLEAR @Scope
2990  I=0
3000  Volts=0

227
3000 OUTPUT @Scope: " :MEAS:SOUR CHAN1"
3010 OUTPUT @Scope: " :MEAS:VAVERAGE?"
3020 ENTER @Scope;Volts
3030 Volts=ABS(Volts)
3040 IF Volts>9.9E+30 THEN Volts=1
3050 WHILE I<2000 AND Volts<Threshold_volt AND Halt$="false"
3060 OUTPUT @Iworm; " JOIH " ! Retract the tip
3070 WAIT Tip_retrct_time
3080 IF Step_direction$="forward" THEN
3090 GOSUB Forwardstep
3100 !
3110 !
3120 !
3130 !
3140 Str_fwd_rev$="F# meas #"
3150 ELSE
3160 GOSUB Reversestep
3170 Str_fwd_rev$="R# meas #"
3180 END IF
3190 I=I+1
3200 OUTPUT @Iworm; " JOIH " ! Extend the tip
3210 WAIT Tip_retrct_time
3220 OUTPUT @Scope: " :MEAS:SOUR CHAN1"
3230 OUTPUT @Scope: " :MEAS:VAVERAGE?"
3240 ENTER @Scope;Volts
3250 Volts=ABS(Volts)
3260 IF Volts>9.9E+30 THEN Volts=1
3270 OUTPUT Buf$ USING " #,K,DDDD,3X,Z.6D";Str_fwd_rev$;I,Volts
3280 CONTROL @Text;SET("APPEND TEXT":Buf$)
3290 END WHILE
3300 BEEP
3310 BEEP
3320 BEEP
3330 OUTPUT @Iworm; "JOIH" ! Retract the tip
3340 Butn$(I)="Continue"
3350 Stru$p$="Auto Approach Completed - Please pull back tip"
3360 DIALOG "INFORMATION",Stru$p$;SET("X":100,"Y":275,"DIALOG BUTTONS":Butn$(*)
3370 GOSUB Halt
3380 RETURN
3390 REM
3400 REM
3410 Multi_step: !
3420 Halt$="false"
3430 ASSIGN @Button20 TO WIDGET "PUSHBUTTON";PARENT @Panel
3440 CONTROL @Button20;SET("BACKGROUND":3,"LABEL":"HALT","X":137,"Y":260)
3450 ON EVENT @Button20,"ACTIVATED",VAL(SYSTEM$("SYSTEM PRIORITY"))+1 GOSUB Halt
3460 OUTPUT @Iworm; "JOIH"
3470 DIALOG "STRING","Number of Steps",Button;RETURN("VALUE":Steps$),SET("TITLE":","RESIZABLE":0,
3480 "MOVABLE":0,"BACKGROUND":15)
3490 SELECT Button
3500 CASE =0
3510 IF Steps$="" THEN
3520 I=1
3530 Steps=VAL(Steps$)
3540 WHILE I<(Steps+1) AND Halt$="false"
C.2 Auger Data Collection Program

The following program (auger07.ibw) was written to allow automated collection of Auger electron spectroscopy (AES) data.

229
10 OPTION BASE 1
20 DIM Str2$(100)
30 DIM Evolts(4096)
40 REAL Ampx(4096)
50 REAL Ampy(4096)
60!
70 DIM File$(12]
80 DIM Span$(12)
90 Str1$=" File Name "
100 Str2$=" Number of data points (4096 max) "
110 ASSIGN @Input TO WIDGET "STRING"
120 CONTROL @Input;SET("TITLE":Str1$)
130 ON EVENT @Input,"RETURN" GOTO Endfilename
140 LOOP
150 END LOOP
160 Endfilename: !
170 STATUS @Input;RETURN("VALUE":File$)
180 File$=File$&".dat"
190 GRAPHICS OFF
200!
210 ASSIGN @Input TO WIDGET "STRING"
220 ! CONTROL @Input;SET("TITLE":Str2$&" for ",File$)
230 ! ON EVENT @Input,"RETURN" GOTO Endnoofo points
240 LOOP
250 ! END LOOP
260 Endnoofo points: !
270 ! STATUS @Input;RETURN("VALUE":Points$)
280 ! GRAPHICS OFF
290 ! Points=VAL(Points$)
300!
310 ASSIGN QLia TO _1423
320 OUTPUT QLia,"x5",";1 ! set mod freq to 10.0kHz
330 WAIT 1
340 OUTPUT QLia,"x6",";0
350 WAIT 1
360 OUTPUT QLia,"X1" ! read in lower eV limit
370 ENTER QLia;Evmin
380 OUTPUT QLia,"x6",";10
390 WAIT 1
400 OUTPUT QLia,"X1" ! read in upper eV limit
410 ENTER QLia;Evmax
420 OUTPUT QLia,"x6",";0 ! reset to lower eV limit
430 WAIT 1
440 Ev_range=Evmax-Evmin
450 OUTPUT QLia,"F"
460 ENTER QLia;Freq
470 !OUTPUT QLia;"S2"
480 No_points=5
490 Scale=10.24/No_points
500 ASSIGN QPlot TO WIDGET "STRIPCHART";SET("VISIBLE":0)
510 CONTROL QPlot;SET("TITLE":File$)
520 CONTROL QPlot;SET("POINT CAPACITY":No_points)
530 DIM Ydata(2)
540 CONTROL QPlot;SET("CURRENT AXIS":"X")
550 CONTROL QPlot;SET("ORIGIN":Evmin*200)
560 CONTROL QPlot;SET("RANGE":Ev_range*220)
570 CONTROL QPlot;SET("CURRENT AXIS":"Y")
580 CONTROL QPlot;SET("ORIGIN":-0)
590 CONTROL QPlot;SET("RANGE":.2)
CONTROL @Plot;SET("AUTOSCALE":1)
CONTROL @Plot;SET("VISIBLE":1)
FOR Z=1 TO No_points
  Ramp=Z*Scale
  OUTPUT @Lia USING "K,DD.DDDD";"X6,";Ramp
  OUTPUT @Lia;"X1","QX","QY"
  ENTER @Lia;Volts,Amplt(Z),Ampy(Z)
  Evolts(Z)=Volts*200
  CONTROL @Plot;SET("POINT LOCATION":Evolts(Z))
  Ydata(1)=Amplt(Z)
  Ydata(2)=Ampy(Z)
  CONTROL @Plot;SET("VALUES":Ydata(*))
NEXT Z
OUTPUT @Lia;"X6","0
CREATE "c:\auger\"&File$,.1
ASSIGN @File TO "c:\auger\"&File$;FORMAT ON
FOR I=1 TO No_points
  OUTPUT @File USING "DD.DDD,DD,XXX,SD.DDDE,XXX,SD.DDDE";Evolts(I),Amplt(I),Ampy(I)
NEXT I
ASSIGN @File TO *
END LOOP
END LOOP
END
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