High Resolution X-Ray Diffraction Applied to Strain Relaxation of Lattice Mismatched Semiconductor Films

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Abstract

Epitaxially grown lattice mismatched semiconductor structures are increasingly important for microelectronic and optoelectronic applications. Recently, a great deal of research has been done on strain relaxation mechanisms in lattice mismatched epitaxial films. Here we describe two high resolution x-ray diffraction experiments performed to study strain relaxation mechanisms and dislocation formation in Si$_{1-x}$Ge$_x$ alloys grown on (001) Si substrates. At small lattice mismatch (<2%), two different mechanisms of strain relaxation are observed, depending on the growth temperature and the magnitude of the strain. At higher growth temperatures or larger lattice mismatch, strain relaxation occurs initially by surface roughening. Subsequently, 60° misfit dislocations nucleate in regions of high strain. At smaller lattice mismatch or lower growth temperature, the surface of the film doesn’t roughen and the 60° misfit dislocations are formed primarily by Frank-Read multiplication. Triple-axis x-ray diffraction reciprocal space maps taken at grazing incidence on very thin epitaxial films can easily distinguish between these two mechanisms. In this geometry, thickness broadening of the x-ray peak is eliminated since the film is essentially infinitely thick parallel to the Si surface. It was also found that films having higher Ge content, which would normally relax by roughening, relax by the multiplication mechanism when the alloy composition of the Si$_{1-x}$Ge$_x$ layer is graded continuously or in steps. Triple-axis reciprocal space maps of a series of step-graded structures, each having an additional Si$_{1-x}$Ge$_x$ step, show that strain relaxes continuously during the growth of the step-graded structures. In such structures, therefore, dislocation nucleation occurs at low mismatch strain, even in layers of high Ge content.

Introduction

Electronic circuits utilizing heterojunction bipolar transistors having a strained Si$_{1-x}$Ge$_x$ layer as the base region and switching speeds in excess of 100 GHz are now being fabricated for applications in wireless telecommunications [1]. These devices are relatively complex from a device processing perspective but relatively simple from the perspective of lattice mismatched heteroepitaxial growth. In contrast, field effect transistors (FETs) are relatively simple devices and
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are, therefore, less expensive to fabricate. However, to include a strained Si or Si$_{1-x}$Ge$_x$ layer as the hole or electron channel of an FET, which increases the carrier mobility by a factor of five to ten, requires a substrate having the lattice parameter of bulk Si$_{0.7}$Ge$_{0.3}$ [2,3]. Since bulk Si$_{0.7}$Ge$_{0.3}$ wafers are not commercially available in sizes suitable for circuit fabrication in a standard silicon manufacturing line, a great deal of work has been done to achieve strain-relaxed Si$_{1-x}$Ge$_x$ buffer layers on (001) Si substrates which have characteristics suitable for application to high-speed FETs [4]. The growth conditions and the design of the buffer layer structure must be carefully controlled in order to achieve the necessary degree of strain relaxation while minimizing the density of dislocations threading up through the active device layers which are grown on top of the relaxed buffer layer.

A great deal of work has been done as well to understand strain relaxation mechanisms and dislocation nucleation in the Si$_{1-x}$Ge$_x$/Si(001) system [4]. The lattice mismatch strain in a coherent epitaxial film is defined as the fractional difference between the in-plane lattice parameter of the substrate crystal and that of the deposited material. In the case of Si and Si$_{1-x}$Ge$_x$, which are cubic materials, the mismatch strain in a pseudomorphic film is \((a-a_s)/a_s\), where \(a\) is the lattice parameter of bulk Si$_{1-x}$Ge$_x$ and \(a_s\) is the lattice parameter of bulk Si. As indicated in Fig. 1, two different strain relaxation mechanisms have been observed in films having <2% mismatch strain, depending on the growth temperature and the mismatch strain, \(\varepsilon\). At higher mismatch strain and growth temperature, the film initially relaxes by surface roughening [5-7], as shown schematically in Fig. 2. The lattice planes bend in such a way that the thicker regions have an in-plane lattice parameter greater than that of the Si substrate and the strain is therefore partially relaxed. However, the thinner regions of the film have an in-plane lattice parameter smaller than that of the Si substrate and thus are very highly strained. Additional strain relaxation occurs by the nucleation of 60° misfit dislocations in these highly strained regions [5]. At lower mismatch strain and/or lower growth temperature, the surface of the epitaxial layer remains smooth, and strain relaxation occurs by the introduction of 60° misfit dislocations, primarily by a Frank-Read mechanism [5,8,9]. This multiplication mechanism results in a lower density of threading dislocations at the surface of the film and is therefore preferable for device applications. In this paper we review two recent experiments which demonstrate the usefulness of high-resolution x-ray diffraction (XRD) measurements for the study of strain relaxation mechanisms and dislocation nucleation in Si$_{1-x}$Ge$_x$/Si(001).

Fig. 1 Phase diagram showing the growth conditions for two different strain relaxation mechanisms observed in Si$_{1-x}$Ge$_x$/Si(001). Symbols indicate experimental data. The dashed and dotted lines are guides for the eye.
Fig. 2. Schematic diagram showing strain relaxation by surface roughening. Note the larger in-plane lattice spacing in the thicker regions of the layer and the smaller lattice spacing in the thicker regions.

Fig. 3. Schematic diagram of the grazing-incidence geometry.

**Grazing Incidence X-Ray Diffraction Measurements (GIXD)**

Conventional XRD has limitations for the measurement of lattice parameters in thin films because of both the broadening of the diffraction peak of a very thin epitaxial layer and the presence of the much more intense diffraction peak from the substrate, which has almost identical lattice parameters. Grazing incidence geometry [10], shown schematically in Fig. 3, can be used to increase the path of the x-rays in a thin film and decrease the contribution from the substrate. The penetration depth normal to the surface is proportional to the sine of the angle of incidence, as long as the angle of incidence is larger than the critical angle, the angle of total external reflection. The problem of thickness broadening, which is clearly demonstrated by the rocking curve simulations in Fig. 4, is also eliminated in this geometry, since the film “thickness” parallel to the wafer surface is essentially infinite. The in-plane lattice parameters are measured directly, thus the relieved strain is easily determined. The amount of strain remaining in the film, or the fractional relaxation, can also be determined if the composition is known or if the out-of-plane lattice parameter is also measured.
GIXD measurements were made at Beamline X-20A at the National Synchrotron Light Source. A triple-axis configuration was used to eliminate mosaic broadening of the layer peak, which is significant in strain-relaxed films. A double-crystal Ge(111) monochromator set to ~8 kV, the sample, and a Si(111) analyzer were positioned in an approximately non-dispersive geometry. The symmetric (004) and low-exit angle (044) reflections of the Si substrate were used to determine the orientation matrix for the samples for alignment at grazing incidence. When the substrate was miscut, one of the four equivalent {004} reflections was chosen so that the sample face was open to the x-ray beam in the grazing incidence position. Samples for GIXD measurements were uniform composition SiGe layers grown on Si(001) wafers by ultra-high vacuum chemical vapor deposition (UHV/CVD).

Triple-axis radial scans for two samples which relaxed by roughening are shown in Fig. 5. The left-hand plot is for a rough sample which had no observable dislocations by cross-sectional transmission electron microscopy (TEM). The broad layer peak indicates that this film has a wide range of lattice parameters. Note that the maximum of the layer peak is at a lower absolute value of H than the narrow Si substrate peak, indicating that on average this film has a larger in-plane lattice parameter than the substrate. Note also that the distribution of lattice parameters includes values at higher |H| than the Si peak, indicating that regions of the layer are very highly strained. Fig. 5(b) is for a somewhat thicker sample grown under the same conditions. Again we see the very broad layer peak and narrower substrate peak. In this sample however, the maximum of the layer peak has shifted farther away from the substrate peak than for the thinner film, indicating that more strain relaxation has occurred in this thicker film than for the thinner one. Cross-sectional TEM images of this thicker film show the presence of dislocations. By varying the angle of incidence of the x-ray beam (varying L), the penetration depth of the x-rays can be varied, as is shown clearly in the right-hand plot. In this case, the layer was thick enough that the Si substrate peak could be entirely eliminated at very small values of L.
Figure 6 shows reciprocal space maps for the same samples. The broadening of the contours in H, just as in the single scans of Fig. 5, indicates the large variation in the in-plane lattice parameter of the layer. Broadening of the contours in K is also observed and is due to the bending of the lattice planes as indicated schematically in Fig. 2. The broadening in K is larger for the thicker film because of additional mosaic broadening caused by the strain fields of the dislocations.

Fig. 5. H scans at different L values for two Si_{0.7}Ge_{0.3} layers. The 200 Å-thick layer in (a) relaxed by roughening and the 500 Å-thick layer in (b) relaxed further by the introduction of misfit dislocations.

Fig. 6. H-K reciprocal space maps of the same two samples as in Fig. 5. The contours indicate the diffracted intensity on a logarithmic scale. Note the additional broadening in K due to the presence of misfit dislocations in (b).
Fig. 7. H-K reciprocal space maps of two sample which relaxed by Frank-Read multiplication. The sample in (a) is a 700 Å-thick Si$_{0.76}$Ge$_{0.24}$ layer and that in (b) is a 2500 Å-thick Si$_{0.85}$Ge$_{0.15}$ layer. The relieved strain is 0.0003 and 0.0004 in (a) and (b) respectively, due to the different film thickness, indicating that the misfit dislocation densities are comparable. The diagonal feature indicated by the arrows is an instrumental artifact (analyzer streak).

Fig. 8. H-K reciprocal space maps of a 1475 Å-thick Si$_{0.73}$Ge$_{0.27}$ layer which relaxed by Frank-Read multiplication. The data were taken at two different grazing incidence angles (α in Fig. 3). In plot (a), taken at lower incidence angle (lower L), the layer peak dominates the spectrum, whereas in plot (b), taken at higher L, the substrate peak is dominant. The relieved strain in this sample is 0.002; therefore, the dislocation density in this sample is about five times greater than in the samples of Fig. 7.
Reciprocal space maps of two smooth films which relaxed by the introduction of misfit dislocations by Frank-Read multiplication are shown in Fig. 7. In contrast to the rough samples, the contours in these maps have narrow extensions along the four \(<110>\) directions forming an “X” pattern. Although the alloy composition of these two layers is different, the relieved strain and therefore the misfit dislocation densities are comparable. Reciprocal space maps of a film similar in composition to that in Fig. 7(a) but thicker, and therefore much more relaxed, are shown in Fig. 8. The two maps were taken at different incident angles (L values). Fig. 8(a), taken at lower angle of incidence, shows the position of the layer peak more clearly, whereas Fig. 8(b) emphasizes the position of the substrate peak. Note that although the contours have broadened significantly, the “X” pattern is still observable. The “X” pattern arises from the long misfit dislocation segments running in the \(<110>\) directions, as discussed in detail in Ref. 10. Broadening of the “X” pattern in samples having higher misfit dislocation densities occurs when there are many intersecting misfit dislocations.

GIXD reciprocal space maps very clearly distinguish between the two different strain relaxation mechanisms observed in these Si$_{1-x}$Ge$_x$/Si(001) layers. Unlike cross-sectional TEM, GIXD is non-destructive and yields a quantitative measurement of the strain relaxation, and thus the dislocation density, in these epitaxial films. When either the alloy composition or the out-of-plane lattice parameter is also measured, the residual strain remaining in the films can be determined.

Step-Graded SiGe Buffer Layers

A strain-relaxed Si$_{0.7}$Ge$_{0.3}$ buffer layer is needed as a “substrate” for FETs having strained Si or Si$_{0.3}$Ge$_{0.7}$ quantum wells as active carrier channels [2,3]. The Frank-Read multiplication mechanism for strain relaxation is preferred for buffer layers, since this mechanism results in orders of magnitude fewer threading dislocations in the active device layers and smoother surfaces. From Fig. 1 we see that a uniform composition Si$_{0.7}$Ge$_{0.3}$ layer relaxes by this mechanism, only when grown at very low temperature. However, it has been found that when the alloy composition is graded, continuously or step-wise, from pure Si up to the desired Si$_{1-x}$Ge$_x$ composition, the buffer layer relaxes by dislocation multiplication, even at higher growth temperatures [8]. Since relaxation presumably occurs continuously during the growth of the graded region, it would therefore follow that dislocation nucleation always occurs at low mismatch strain in such structures, consistent with the data in Fig. 1. This was verified by the following XRD experiment.

A series of step-graded buffer layer structures was grown at 560°C, each sample having an additional step as shown in Fig. 9 [4,11]. Cross-sectional TEM images showed that the sample with three layers has no dislocations, that with four layers has only a few dislocations, but those having five or more layers have high densities of misfit dislocations [4,11]. All of these samples relaxed by dislocation multiplication. Triple-axis (004) radial scans for several samples are shown in Fig. 10. In addition to the Si substrate peak, only a single narrow peak was seen for sample 3, which has three steps. The first two steps are too thin to give visible peaks. The spectrum for sample 4, which has
four steps, shows two narrow x-ray peaks from the $\text{Si}_{1-x}\text{Ge}_x$ layers. The peak for layer 3 is at the same position as the single layer peak in sample 3. The data for sample 5 shows x-ray peaks from three $\text{Si}_{1-x}\text{Ge}_x$ layers; however, the two corresponding to layers 3 and 4 are now shifted closer to the Si substrate peak indicating that significant strain relaxation of those layers has occurred. As additional layers are added to the structure, further relaxation occurs as is seen for samples 6 and 7. In all the samples, the uppermost layer appears to remain somewhat strained, whereas the lower layers appear to have substantially relaxed.

![Graph of Ge profile for step-graded structures](image)

**Fig. 9.** Ge profile for a series of step-graded structures, each having an additional layer.

![Graph of XRD peaks for Si$_x$Ge$_y$/Si samples](image)

**Fig. 10.** (004) radial scans of a series of step-graded $\text{Si}_{1-x}\text{Ge}_x$/Si samples. The number of the XRD peak corresponds to the layer number in Fig. 9.
This trend is also clearly seen in the reciprocal space maps of the 044 reflection shown in Fig. 11. The map of sample 3 shows two narrow peaks for the Si substrate and layer 3, indicating that both have the same in-plane lattice parameter and thus no strain relaxation occurred. Three narrow peaks are seen for sample 4, and the data show that both SiGe layers have the same in-plane lattice parameter as the Si substrate, i.e. essentially no strain relaxation has occurred in this sample as well. However, the broadening of the low intensity contours perpendicular to the $q_x=q_y$ line indicates the presence of some dislocations in this sample, consistent with the TEM images. In contrast, the very broad contours of the map of sample 4 show that there is a high density of dislocations present in this sample. The large shift of the layer peaks towards the $q_x=q_y$ line shows that a significant change in the in-plane lattice parameter has occurred during the growth of layer 5. Maps of samples 6 and 7 look similar to that of sample 5 and in all the samples the uppermost layer is less relaxed than the underlying ones. These data clearly show that after the onset of dislocation formation, the strain relaxes continuously as additional layers are added to the structure.

![Fig. 11. Reciprocal space maps of step-graded samples 3, 4 and 5. $q_x$ is inversely proportional to the out-of-plane lattice parameter and $q_y$ is inversely proportional to the in-plane lattice parameter. The peak numbers correspond to the layer numbers in Fig. 9.](image-url)
The XRD data was used to determine the composition and strain in each \( \text{Si}_{1-x}\text{Ge}_x \) layer. From these data, the strain in the top layer at the onset of growth was determined. Since sample 3 did not relax at all, the mismatch strain at the start of growth of layer 3 is the mismatch strain between \( \text{Si}_{0.87}\text{Ge}_{0.13} \) and Si. The same is true for layer 4, i.e the mismatch is that between \( \text{Si}_{0.85}\text{Ge}_{0.17} \) and Si. Since negligible relaxation occurred in sample 4, the initial mismatch for layer 5 is that between \( \text{Si}_{0.79}\text{Ge}_{0.21} \) and Si. Since significant relaxation occurred during the growth of layer 5, however, the mismatch strain at the start of growth of layer 6 is reduced. Results for the whole series are given in Table I and Fig. 12. Initially the strain builds up, but once relaxation begins, the strain at the start of growth of each new layer is essentially constant, and the value is well within the range for the dislocation multiplication mechanism at this growth temperature (see Fig. 1). This experiment shows that dislocation nucleation occurs continuously during the growth of step-graded buffer layer structures and, therefore, the mismatch strain always remains relatively low [4,11]. Since mismatch strain is the driving force for surface roughening, it does not occur in these structures, even though a uniform composition layer of this alloy composition would roughen at this grow temperature. Because surface roughening is suppressed, dislocation nucleation occurs primarily by the Frank-Read multiplication mechanism in graded buffer layers grown by UHV/CVD. Thus step-grading is the method of choice for relaxed SiGe buffer layers for quantum well FETs.

Table I. Data for step-graded samples having the Ge profiles shown in Fig. 9.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Composition of top layer, x</th>
<th>Mismatch strain at start of growth of top layer</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>0.13</td>
<td>0.0052</td>
</tr>
<tr>
<td>4</td>
<td>0.17</td>
<td>0.0069</td>
</tr>
<tr>
<td>5</td>
<td>0.21</td>
<td>0.0083</td>
</tr>
<tr>
<td>6</td>
<td>0.26</td>
<td>0.0060</td>
</tr>
<tr>
<td>7 (solid line)</td>
<td>0.35</td>
<td>0.0067</td>
</tr>
<tr>
<td>7 (dashed line)</td>
<td>0.31</td>
<td>0.005</td>
</tr>
<tr>
<td>10</td>
<td>0.50</td>
<td>0.005</td>
</tr>
</tbody>
</table>
Conclusions

High resolution x-ray diffraction is a useful technique for the study of strain relaxation and dislocation nucleation in lattice mismatched semiconductors. Unlike cross-sectional TEM, high-resolution x-ray diffraction is non-destructive and also gives a quantitative measure of the strain relaxation. The volume of the sample probed by the x-ray beam is usually larger than that in a TEM image, so that the strain measured is the average over a substantial region of the sample. Here we have described two experiments, performed on $\text{Si}_{1-x}\text{Ge}_x/\text{Si}(001)$ structures, as examples of the power of triple-axis x-ray diffraction measurements. Grazing incidence reciprocal space maps of uniform composition $\text{Si}_{1-x}\text{Ge}_x$ layers give a visual "finger-print" indicating which of two different relaxation mechanisms have occurred. The use of grazing incidence geometry eliminates thickness broadening of the x-ray peaks. (044) reciprocal space maps of a series of step-graded layers show that these structures relax continuously during growth. The x-ray measurements verified that dislocation nucleation occurs at low mismatch strain, even when these structures include $\text{Si}_{1-x}\text{Ge}_x$ layers having large values of $x$. 

Fig. 12. Plot of the strain at the start of growth of the top layer of several samples as determined from the x-ray diffraction data. The solid circles are for the layers shown with a solid line in Fig. 9 and the open circles are for those shown by the broken line.
References


