Characterization of SiGe/Ge Heterostructures and Graded Layers Using Variable Angle Spectroscopic Ellipsometry

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Variable angle spectroscopic ellipsometry (VASE) has been used to characterize $Si_xGe_{1-x}/Ge$ superlattices (SLs) grown on Ge substrates and thick $Si_xGe_{1-x}/Ge$ heterostructures grown on Si substrates. Our VASE analysis yielded the thicknesses and alloy compositions of all layers within the optical penetration depth of the surface. In addition, strain effects were observed in the VASE results for layers under both compressive and tensile strain. Results for the SL structures were found to be in close agreement with high resolution x-ray diffraction measurements made on the same samples.

The VASE analysis has been upgraded to characterize linearly graded $Si_xGe_{1-x}$ buffer layers. The algorithm has been used to determine the total thickness of the buffer layer along with the start and end alloy composition by breaking the total thickness into many (typically > 20) equal layers. Our ellipsometric results for 1 $\mu$m buffer layers graded in the ranges $0.7 \leq x \leq 1.0$ and $0.5 \leq x \leq 1.0$ are presented, and compare favorably with the nominal values.

INTRODUCTION

Characterization of semiconductor superlattice (SL) structures is typically done by x-ray diffraction (XRD) and transmission electron microscopy (TEM). Both XRD and TEM can be used to quantify SL ordering and periodicity. In addition, XRD can give the average value of the composition. Both techniques have limitations; TEM is destructive, XRD requires relatively thick samples (~1000 Å), and neither technique can be used to quantify individual interfaces or quantum wells. Recently, variable angle spectroscopic ellipsometry (VASE) has been shown to be a powerful, non-destructive technique for the post-deposition characterization of $Si_xGe_{1-x}/Si$ SLs and other multilayer heterostructures [1,2]. In these studies VASE has been used to determine layer thicknesses, alloy composition, oxide thickness, number of superlattice periods, and sample homogeneity. In this current work we will concentrate on Ge rich $Si_xGe_{1-x}/Ge$ SLs and heterostructures.

Graded composition $Si_xGe_{1-x}$ layers are used in the base of $Si_xGe_{1-x}/Si$ heterojunction bipolar transistors to increase device speed. Graded composition $Si_xGe_{1-x}$ layers (graded to achieve $x \approx 0.3$) are also used as buffers to relieve strain in the growth of n-type $Si_xGe_{1-x}/Si$ modulation doped field effect transistors (MODFET) structures. In previous VASE analysis of MODFETs, only the high energy portion of the ellipsometric spectra was used. Thus, a graded composition layer analysis was avoided, since the graded layer was buried below the optical penetration depth of the probing light at these energies. In this work a simple algorithm for the calculation of the Fresnel reflection coefficients of a linearly graded $Si_xGe_{1-x}$ layer has been developed. This allowed a graded $Si_xGe_{1-x}$ layer to be characterized in terms of its thickness, and Si content at the substrate and ambient surfaces.

BACKGROUND

To determine the properties of multilayer structures the measured ellipsometric angles, $\{\tan \Psi(\lambda), \cos \Delta(\lambda)\}$, must be compared to results calculated from well defined models. Linear regression analysis (LRA) is used to minimize the unbiased estimator, $\sigma$:

$$
\sigma^2 = \frac{1}{n - m - 1} \sum_{i=1}^{n} \{ (\tan \Psi_i - \tan \Psi_i^*)^2 + (\cos \Delta_i - \cos \Delta_i^*)^2 \}
$$

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where $n$ is the number of observed data points and $m$ is the number of free parameters used in the model. The superscripts $e$ and $c$ refer to the experimentally observed data and the corresponding results calculated by the model, respectively. In order to construct a model, the dielectric function of all the constituent materials must be known. The dielectric function of composite layers can be determined using effective medium approximations (EMAs) [3]. The energy shift algorithm [4] is used to interpolate between Si$_x$Ge$_{1-x}$ dielectric functions published at discrete alloy compositions [5]. The algorithm requires a knowledge of the functional dependence of the critical point (CP) energies with the alloy composition, usually the $E_c(x)$, $E_l(x)$, and $E_T(x)$ CP energies. To improve the analysis of Ge rich Si$_x$Ge$_{1-x}$ materials the energy shift algorithm has been modified to include the effects of the $E_1 + \Delta_1$ CP energy [6].

Fig. 1 shows the $E_1$ and $E_1 + \Delta_1$ CP energies for strained and relaxed Si$_x$Ge$_{1-x}$ layers. The in-plane strain, $\varepsilon$, in the strained Si$_x$Ge$_{1-x}$ layer results from the lattice mismatch between the layer and the substrate. The shifts in the $E_1$ and $E_1 + \Delta_1$ CP energies for a biaxial (001) strain are given by [7]:

$$\Delta E_1 = \frac{\Delta_1}{2} + E_H - \frac{1}{2} \left( \Delta_1^2 + 4E_3^2 \right)^{1/2}$$  \hspace{1cm} (2)

$$\Delta (E_1 + \Delta_1) = \frac{-\Delta_1}{2} + E_H + \frac{1}{2} \left( \Delta_1^2 + 4E_3^2 \right)^{1/2}$$  \hspace{1cm} (3)

where the hydrostatic shift, $E_H$, and uniaxial shear, $E_S$, are given by:

$$E_H = 2\varepsilon (1 - C_{12}/C_{11})$$  \hspace{1cm} (4)

$$E_S = \frac{2}{3}\left(2/3\right)^{1/2} D_3^2 (1 + 2C_{12}/C_{11}) \varepsilon$$  \hspace{1cm} (5)

Table I: Comparison of target sample structure with that determined by HRXRD and VASE. The period is the sum of the Ge and SiGe layer thicknesses, and $x($avg$)$ is the average silicon content in one period. The parameters of the VASE analysis are the oxide, Ge and SiGe layer thicknesses, and the silicon content, $x$, of the SiGe layer.

<table>
<thead>
<tr>
<th>Sample Source</th>
<th>Period</th>
<th>$x($avg$)$</th>
<th>$d$(Ge)</th>
<th>$d$(SiGe)</th>
<th>$x$</th>
</tr>
</thead>
<tbody>
<tr>
<td>HA57 target</td>
<td>178</td>
<td>6.2$^a$</td>
<td>128</td>
<td>50</td>
<td>22.0</td>
</tr>
<tr>
<td>HA57 HRXRD</td>
<td>201.1</td>
<td>8.0</td>
<td>141.8$^b$</td>
<td>59.3$^b$</td>
<td>26.6$^b$</td>
</tr>
<tr>
<td>HA57 VASE$^c$</td>
<td>202.8</td>
<td>8.4$^a$</td>
<td>127.5</td>
<td>75.3</td>
<td>22.7</td>
</tr>
<tr>
<td>HA58 target</td>
<td>201</td>
<td>7.8$^a$</td>
<td>142</td>
<td>59</td>
<td>26.6</td>
</tr>
<tr>
<td>HA58 HRXRD</td>
<td>202.1</td>
<td>8.3</td>
<td>141.8$^b$</td>
<td>60.3$^b$</td>
<td>27.5$^b$</td>
</tr>
<tr>
<td>HA58 VASE$^d$</td>
<td>204.5</td>
<td>8.4$^a$</td>
<td>126.9</td>
<td>77.6</td>
<td>22.1</td>
</tr>
</tbody>
</table>

$^a$Calculated from $d$(Ge), $d$(SiGe), and $x$.
$^b$Estimated from the period, $x($avg$)$, and shutter opening/closing times.
$^c$Calculated from $d$(Ge), $d$(SiGe), and $x$.
$^d$Estimated from the period, $x($avg$)$, and shutter opening/closing times.

$C_{ij}$ are the elastic stiffness constants, $E_1$ is the hydrostatic deformation potential, and $D_3^2$ is an intraband deformation potential for the $\Delta_3$ valence band for a [001] uniaxial strain. The energy shift algorithm assumes the functional dependence of the relaxed $E_1$ and $E_1 + \Delta_1$ CP energies shown in Fig. 1. Therefore, when modeling strained Si$_x$Ge$_{1-x}$ layers the strain induced shifts in the $E_1$ and $E_1 + \Delta_1$ will cause the value of the Si content, $x$, to be over- or under-estimated for a compressive or tensile strain, respectively.

SiGe/Ge SUPERLATTICE STRUCTURES ON Ge SUBSTRATES

Two fifty period Si$_x$Ge$_{1-x}$/Ge SL structures, HA57 and HA58, were grown by Si molecular beam epitaxy (MBE) on Ge (100) substrates at Hughes Research Laboratories. The samples were first characterized by high resolution x-ray diffraction (HRXRD) [8], the results are shown in Table I along with the target structures. Each sample was then measured by VASE at several angles of incidence (69°, 73°, and 77°) which were chosen to increase the sensitivity of the ellipsometric angles to the sample structure parameters [9]. The measured ($\tan \Psi$, $\cos \Delta$) spectra for sample HA57 are shown in Fig. 2 along with the spectra generated from the best fit model. Due to the strong absorption in the low wavelength region, discrepancies between the measured and calculated spectra in this region are a result of devia-
Figure 2: Comparison of measured VASE data (symbols) with that determined from the best fit model (lines) for the Si$_x$Ge$_{1-x}$/Ge SL sample HA57. Sample HA58 shows similar fitting. The structure parameters found from the best fit model are shown in Table I.

Figure 3: Structure of sample CL141 showing the target structure parameters and those determined from the VASE analysis.

SiGe/Ge HETEROSTRUCTURES ON SI SUBSTRATES

Two Si$_x$Ge$_{1-x}$/Ge heterostructures, CL141 and CL171, were grown on Si substrates at the University of California at Los Angeles. The nominal structures for sample CL141 and CL171 are shown in Fig. 3 and Fig. 4, respectively. The Si content of the stepped buffer was changed from 100% to 20% or 30% in approximate steps of 25%. Therefore, it would require six parameters to completely characterize the buffer layer. Characterization of the buffer layer is further complicated by the fact that it is buried deep in the structure. As a result sample CL141 was modeled over the range 300 nm to 550 nm to ensure that the top step of the buffer would act as the substrate. Over this range the maximum penetration depth is ~1000 Å in Si$_0.3$Ge$_{0.7}$ and Ge, respectively. The oxide has been modeled as a mixture of Si$_x$Ge$_{1-x}$ (x same as underlying layer) and GeO$_2$ using the Bruggeman EMA; this mixture simulates a surface roughness. The surface of these samples is expected to be rough due to the large lattice mismatch between the Ge layer and the underlying Si substrate (~4%). The VASE results, shown in Fig. 3, agree with in the 90% confidence limits with the nominal structure.

The second sample, CL171, was modeled over the spectral range 300 nm to 760 nm. Over this range the Si$_x$Ge$_{1-x}$ layer will act as the substrate due to the increased thicknesses of both the Ge and SiGe layers. The measured VASE data is shown in Fig. 5 along with spectra generated from the best fit model; the resulting structure parameters are shown in Fig. 4. Modeling the
Table II: Results of VASE analysis of graded Si_{x}Ge_{1-x} layers on Si substrates. The nominal thickness of all samples is 1 μm. The samples were linearly graded from 100% Si at the substrate to x_{n}(nom) at the surface. The parameters of the VASE analysis are the oxide thickness, buffer layer thickness (D), and the Si content at the surface (x_{n}). The Si content at the substrate was held constant at 100%. A value of n = 30 was used to model all three samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>D(oxide) (μm)</th>
<th>D (μm)</th>
<th>x_{n} (%)</th>
<th>x_{n}(nom) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>HA82</td>
<td>0.0522 ± 0.04</td>
<td>1.04 ± 0.04</td>
<td>76.7 ± 0.6</td>
<td>70</td>
</tr>
<tr>
<td>HA83</td>
<td>0.0524 ± 0.03</td>
<td>1.02 ± 0.03</td>
<td>66.7 ± 0.6</td>
<td>50</td>
</tr>
<tr>
<td>HA17</td>
<td>0.0202 ± 0.01</td>
<td>0.97 ± 0.01</td>
<td>58.7 ± 0.3</td>
<td>50</td>
</tr>
</tbody>
</table>

Figure 4: Structure of sample CL171 showing the target structure parameters and those determined from the VASE analysis.

Figure 5: Comparison of measured VASE data (symbok) with that generated from the best fit model (lines) for the thick Si_{x}Ge_{1-x}/Ge heterostructure sample CL171. The best fit model is shown in Fig. 4 along with the resulting values of the structure parameters.

SiGe GRADED LAYERS

Three continuously, linearly graded Si_{x}Ge_{1-x} layers were grown by Si MBE on Si (100) substrates at Hughes Research Laboratories; the nominal structures are described in Table II. Graded layers are simulated in the VASE model by breaking the layer into n + 1 sub layers. The thickness of the i-th sub layer is:

\[ d_{i} = \begin{cases} 
\frac{D}{2n} & i = 0, n \\
\frac{D}{n} & 1 \leq i < n 
\end{cases} \]  

(6)

where D is the total layer thickness. For a linearly graded layer the Si content of the i-th layer is given by:

\[ x_{i} = \frac{(n-i)x_{0} + ix_{n}}{n} \]  

(7)

where x_{0} and x_{n} are the Si content at the substrate and surface, respectively. As n → ∞ this model will more closely approximate a continuously graded layer. However, values of n > 20 were found to yield nearly identical results in the VASE analysis for the graded samples used in this study.

The graded samples were measured by VASE at three angles of incidence: 70°, 75°, and 77°. Fig. 6 shows the experimental data for sample HA17 along with the ellipsometric angles generated from the best fit model. The fitting is nearly perfect in the cos Δ spectrum and in the long wavelength region of the tan Ψ spectrum. The poor fitting in the low wavelength region of the tan Ψ spectrum is most likely due to poor surface
quality. Because the graded layers are uncapped, the surface region of the graded layers are expected to be strained which can cause the surface to become rough. Table II summarizes the results of the VASE analysis. It can be seen from this table that the thickness values are very close to the nominal values. The values for the Si content at the surface ($x_n$), however, are consistently much higher than the nominal values. These discrepancies can be caused by the compressive strain in the surface region of the layer which would result in an overestimate of $x$. The growth log notes that the surface morphology of sample HA83 is poor when compared with sample HA82. This may be the cause of the larger discrepancy in $x_n$ for sample HA83.

CONCLUSIONS

VASE has been used to characterize Si$_x$Ge$_{1-x}$/Ge SL structures grown on Ge substrates. The values of the period and $x$(avg) determined by VASE show excellent agreement with those same values determined by HRXRD. Due to strain effects or interfacial layers not accounted for in the VASE models, the individual layer thicknesses and $x$ determined by the two methods do not agree as closely. Thick Si$_x$Ge$_{1-x}$/Ge heterostructures grown on Si substrates have also been characterized by VASE. Results show close agreement with the nominal structure. In addition VASE analysis provides a qualitative assessment of the surface roughness and strain in the various layers of the structures. Finally, VASE has been used to characterize continuously, linearly graded Si$_x$Ge$_{1-x}$ layers in terms of the layer thickness and Si content at the surfaces. Results agree extremely well with the target structure, especially when strain in the surface region of these uncaped graded layers is taken into account.

REFERENCES
