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Ellipsometric study of $Si_{0.5}Ge_{0.5}$/Si strained-layer superlattices

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We present an ellipsometric study of two $Si_{0.5}Ge_{0.5}$/Si strained-layer superlattices grown by MBE at low temperature (500 °C), and compare our results with x-ray diffraction (XRD) estimates. Excellent agreement is obtained between target values, XRD, and ellipsometry when one of two available Si_xGe_{1-x} databases is used. We show that ellipsometry can be used to nondestructively determine the number of superlattice periods, layer thicknesses, Si_xGe_{1-x} composition, and oxide thickness without resorting to additional sources of information. We also note that we do not observe any strain effect on the E_1 critical point.

Semiconductor superlattices (SL) and strained-layer superlattices (SLS) are most commonly characterized by x-ray diffraction (XRD) and transmission electron microscopy (TEM). Neither of these methods are ideal. The first technique is very accurate, but only directly gives the SL period and an average value of composition,[1,2] while the second technique requires very tedious sample preparation and is destructive. In this letter, we will show that variable angle-of-incidence spectroscopic ellipsometry (VASE) as applied to $Si_{0.5}Ge_{0.5}$/Si SLS yields simultaneously the SL period, the layer thicknesses, the Si_xGe_{1-x} layer composition, the number of periods, and the overlayer oxide thickness. In addition, strain effects on the dielectric function of the Si_xGe_{1-x} layer will be estimated by VASE. The effect of strain on the dielectric function of Si_xGe_{1-x} grown on silicon is presently an area of active research. Both single wavelength[3,4] and spectroscopic[5–7] ellipsometry, as well as photoreflectance[8] have been used on high Si concentration ($x>0.75$) Si_xGe_{1-x} thin films, showing conflicting results. Most spectroscopic measurements,[6–8] which give an overall picture of the strain on the dielectric function, have estimated the effect of strain on the critical points, particularly E_1 and E_0'. The E_1 critical point is a crucial parameter in the energy shift algorithm[9] and a change in its position will be reflected in a change of the estimated value of x.[10] Published results claim either no change in E_1,[6,8] decrease in E_1 with increasing strain,[7] or an effective increase.[5] In this work, the low Si content ($x \approx 0.5$) and small Si_xGe_{1-x} layer thickness (~ 5 nm) of our SLS should make any lattice mismatch effect on E_1 readily detectable.

Two nominally identical 15 period $Si_{0.5}Ge_{0.5}$/Si SLS samples, identified here as samples A and B, were grown by molecular beam epitaxy[2] (MBE) using a Perkin–Elmer (model 430S) Si MBE system. The growth temperature was 500 °C. The upper layer of each SLS was silicon. Target parameter values are given in Table I. Both samples were measured by XRD which, together with the knowledge of the shutter opening times, gives the SLS period, the average Si concentration throughout the SLS, and the Si_xGe_{1-x} layer thickness. The XRD pattern for sample B was of excellent quality, while that of sample A was somewhat degraded. However, an accurate estimate of the SLS period and average Si concentration in the SLS was obtained for both samples. All results are included in Table I. The average Si concentration is given as x(avg); values determined using shutter timing information are labelled with asterisks. The XRD results show excellent agreement with the target values for both samples.

The VASE measurements were taken with a rotating analyzer ellipsometer described elsewhere.[11] This instrument measures the complex reflection ratio $\rho=\tan(\Psi)e^{i\Delta}$ where Ψ and Δ are the conventional ellipsometric parameters used to represent the amplitude and phase of ρ. Measurements of the SLS samples were taken over the spectral range 300–760 nm for sample A and 300–780 nm for sample B, both in 5 nm increments. Each sample was measured at three angles-of-incidence: 75°, 76°, and 77°. These incident angles were selected to be near to the principal angle for most of the spectral range, providing maximum sensitivity.[12] Experimental results of $\tan(\Psi)$ and $\cos(\Delta)$ for sample A are shown in Fig. 1; the results for sample B are very similar.

Analysis of VASE data involves a least squares fit of the data to an appropriate model, with the quality of the fit defined by a mean-square-error σ.[10] A four parameter model was used to fit the SLS samples. The parameters were: Si and Si_xGe_{1-x} layer thicknesses, silicon concentration x in the Si_xGe_{1-x} layer, and the native oxide thickness. All periods of the superlattice were assumed to be exactly identical in terms of thicknesses and optical properties. The optical constants of silicon were taken from Ref. 13. There are currently two available databases for Si_xGe_{1-x} in the literature.[14,15] J. Humlíček *et al.*[14] have published the results of measurements of bulk Si_xGe_{1-x} grown by the Czochralski method and also of thick films grown by liquid-phase epitaxy. These measurements cover the entire compositional range. Optical measurements were taken[14]

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TABLE I. VASE and XRD analyses of the SLS samples. The ellipsometric fitted parameters are the oxide, Si and SiGe layer thicknesses (d), and the silicon concentration x of the SiGe layer. The period is the sum of the Si and the SiGe layer thicknesses. x(avg) is the average silicon concentration in one period.

Sample	Source	$d(SiO_2)$ (nm)	Period (nm)	d(Si) (nm)	d(SiGe) (nm)	x(avg)	x	σ
···	target	···	35.0	30.0	5.0	0.929	0.5	···
A	XRD	···	35.3	30.0[a]	5.3[a]	0.922	0.47[a]	···
A	VASE	1.89	34.8	30.11	4.67	0.942	0.5690	0.0021
	Ref. 14[b]	±0.08		±0.08	±0.05		±0.0035	
A	VASE	1.83	34.8	29.88	4.96	0.926	0.4709	0.0013
	Ref. 15[c]	±0.04		±0.06	±0.04		±0.0037	
B	XRD	···	38.3	32.8[a]	5.5[a]	0.931	0.50[a]	···
B	VASE	3.08	37.6	32.42	5.19	0.945	0.6031	0.0025
	Ref. 14[b]	±0.08		±1.00	±0.06		±0.0022	
B	VASE	2.96	37.7	32.36	5.33	0.931	0.5145	0.0016
	Ref. 15[c]	±0.06		±0.07	±0.05		±0.0031	

[a] Estimated from growth times.
[b] Fitted $\lambda < 650$ nm.
[c] All data fitted.

using a rotating analyzer ellipsometer (RAE). There are several difficulties with this database: first, because RAE does not measure low absorption substrates accurately, data at high wavelengths are unreliable.[14,15] Probably for this reason, data are available in the infrared only down to 1.7 eV (729 nm). This is a problem for our analyses, as the silicon layer of the first period is only penetrated for $\lambda > 370$ nm, so that the information on the superlattice is contained mostly in the high wavelength measurements. Second, the compositions were determined only by ellipsometry,[14] using the measurement at the He-Ne line (632.8 nm), where the RAE is inaccurate. Recently, Jellison, Haynes, and Burke[15] have published an independent database using the results of measurements of thick (7–8 μm on Si) relaxed Si_xGe_{1-x} films grown by conventional high-temperature chemical vapor deposition. Optical measurements were taken using a two-channel spectroscopic polarization modulation ellipsometer (2-C SPME) which measures low absorption substrates accurately;[15] data are available to 840 nm. Compositions were determined by electron microprobe and Rutherford backscattering measurements. This database also covers the entire compositional range. However, there is a variation between a sample grown on a silicon substrate and another sample of similar composition grown on a germanium substrate. Therefore, in our analyses, only Ref. 15 spectra taken from samples grown on silicon will be used. Because the databases of Refs. 14 and 15 show significant differences, each SLS was analyzed using each database and the results will be compared. In order to use the databases, an algorithm for interpolating between the compositions is needed. The energy shift algorithm[9] which has been applied previously to Si_xGe_{1-x} using the Ref. 14 database[5,6] was used. This algorithm requires the location of the main critical points to be expressed as a function of alloy composition. Because of the significant differences between the databases of Refs. 14 and 15, different critical point functions must be used. For both databases, the indirect band gap function $E_0(x) = 0.68 + 0.44x$ was used.[16] This function, which is a simple linear interpolation between the fundamental indirect band gaps of silicon and germanium, was found to give identical results in our analyses, as compared with more complex $E_0(x)$ functions, because the range of measurement is far away from $E_0(x)$. $E_1(x)$ for the database of Ref. 14 was

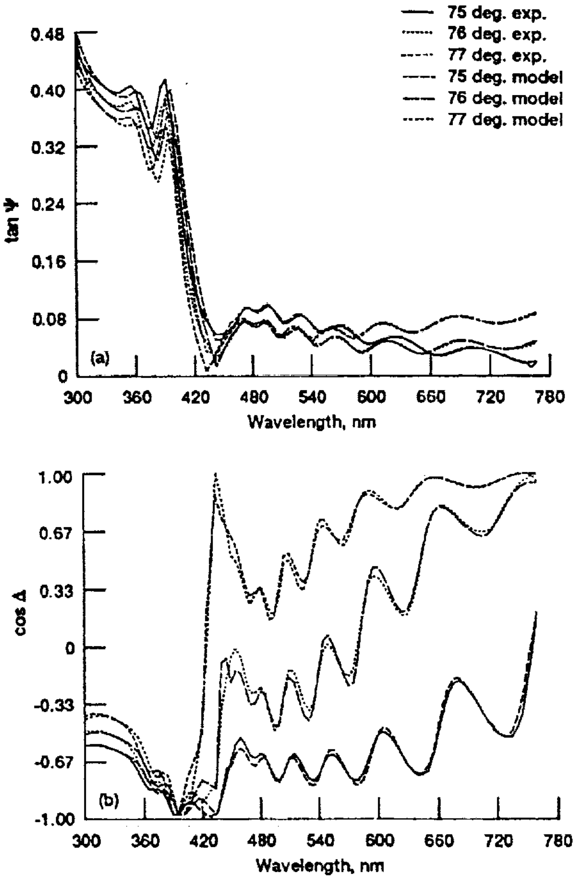

FIG. 1. Sample A experimental data vs model for the four parameter fit (using Ref. 15 database). Graphs show (a) tan(Ψ) and (b) cos(Δ).

given in that reference, while $E_2(x) = 4.39 + 0.03x$ was taken from Ref. 6. For the database of Ref. 15, critical points $E_1(x)$ and $E_2(x)$ were obtained by fitting the spectra to critical point line shapes.[17] $E_2(x)$ was found to be approximately constant at 4.30 eV, while the linear fit $E_1(x) = 2.357 + 0.9393x$ was found to describe $E_1(x)$ well, especially in the range $0.47 < x < 0.85$.

Comparison of experimental VASE and best fit model results using the Ref. 15 database for sample A are shown in Fig. 1. Full numerical VASE results for both samples are given in Table I along with the associated 90% confidence limits. Note that analyses using Ref. 14 are limited to $\lambda < 650$ nm, due to the range of reference data and the nature of the energy shift algorithm. Analyses using Ref. 15 include all measured data. In all cases, the values determined by XRD were used as initial conditions. Statistical analyses of the fits indicated no significant parameter correlations. The results using both Si_xGe_{1-x} databases are very similar, except for the x value of the Si_xGe_{1-x} layers. The composition obtained using Ref. 15 data agrees very well with the XRD results, while the x value obtained using Ref. 14 is about 0.1 higher for both superlattices. This, along with the higher σ values obtained using the Ref. 14 database, supports our belief in the greater accuracy of the Ref. 15 database. The XRD data and the VASE results all show a 0.03 difference in the x values between the two samples, strongly indicating this difference is real. This indicates that the precision of both the XRD and VASE techniques for determining the value of Si_xGe_{1-x} composition x is better than 0.03.

XRD can verify the number of periods provided the sample is of extremely good quality. To examine the ability of VASE to verify this parameter, we refitted sample B using the Ref. 15 database assuming 14, 16, and 17 periods. For the 14 period model, σ is a factor of 3 higher than the 15 period model and x increases to 0.607. For 16 periods and 17 periods, the x value decreased below 0.47 (the lowest available reference spectrum) and had to be held constant there: the resultant σ were 1.2 and 4.2 times higher, respectively. Adding or subtracting a period clearly degrades the fit, either by increasing σ or by unreasonably decreasing the composition; we thus conclude VASE can be used to verify the number of periods.

To examine potential parameter correlations, the measurement of sample B was refitted using an initial Si_xGe_{1-x} layer thickness of 6.6 nm and concentration $x = 0.60$, maintaining the XRD value $x(\text{avg}) = 0.93$. The fit converged to the same values as given in Table I to four significant digits. This means VASE can separate the Si_xGe_{1-x} layer composition and thickness, i.e., they are not significantly correlated. VASE thus provides an independent verification of shutter opening times, which are used in the XRD analysis.

The strain effect, if any, on the dielectric function will be observed as a shift in the x value. As seen from Table I, using the Ref. 15 database which is deemed more reliable in our wavelength range, we do not observe any strain effect. The good agreement between XRD and the fit using Ref. 15 indicates that the E_1 critical point is not strongly affected by the strain, since the energy shift algorithm used was based upon this critical point.[9] The $E_1 + \Delta_1$ and E_0' critical points are not checked with this method, as they are weaker and influence the spectrum only above the E_1 energy region, where the top period silicon layer largely shields the superlattice.

In conclusion, we have obtained excellent agreement between XRD and VASE results for two $Si_{0.50}Ge_{0.50}/Si$ SLS samples using Ref. 15 data for unstrained Si_xGe_{1-x}. We have shown that VASE can be used to determine the number of periods, layer thicknesses, Si_xGe_{1-x} layer composition, and native oxide.

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