Effect of hydrostatic pressure on the Raman spectrum of Ge_nSi_m multiple quantum wells with $n \leqslant 4$ and $m \leqslant 7$

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We report a Raman study of the effects pressure has on the vibrational structure of Ge_nSi_m multiple quantum wells (MQW's) with $n \leqslant 4$ and $m \leqslant 7$. Three primary phonon bands are studied: Ge-Ge within the germanium layers, Si-Si within the silicon layers, and the Ge-Si interface mode. Pressure shifts each of these bands consistent with a mode-Grüneisen constant of unity for all samples and laser excitations used. We observe resonance effects with the confined Ge-like E_1 transition for the Ge_4Si_5 sample. The transition is near 2.4 eV at ambient pressure and blueshifts at $\approx 4 \pm 1$ meV/kbar. This pressure coefficient is smaller than the corresponding quantity in bulk germanium. This is attributed to the fact that silicon dictates the in-plane contraction of the Ge layer that is at a smaller rate than the corresponding quantity in bulk germanium. We see no evidence of resonance enhancement in samples with thinner Ge layers in each MQW period. This implies that at least four Ge atoms are necessary to form the states producing the E_1 transition, consistent with previous studies. An additional feature seen in the spectra near 310 cm^{-1} is identified by the pressure study to be 2TA Raman scattering from silicon. [S0163-1829(98)04232-5]

I. INTRODUCTION

A great deal of effort has been invested in producing high-quality heterostructures of silicon and germanium.[1] Raman scattering has been exploited as a microscopic probe of the integrity of superlattices[2–10] (SL's) and, to a much lesser extent, multiple quantum wells[11–14] (MQW's) composed of silicon and germanium. This interest is rooted in the capability of Raman studies for probing the layers of silicon and germanium (or, to some extent, Si_xGe_{1-x} alloy), and the *interfaces* binding them. Careful studies have led to the conclusion that optic phonons for layers of each type of atom are confined.[11] While this is not surprising for the vibrations of silicon, which lie beyond the phonon spectrum of germanium, it is unexpected for the germanium bands that overlap with (non-zone-center) modes in the silicon spectrum. Raman studies are also interesting because electronic transitions can lead to resonances in the spectra. This provides information about the electronic structure and electron-phonon interactions. However, whether the resonance enhancement observed in Raman spectra of these and similar samples between 2.2 and 2.5 eV stems from the Ge-like E_0 or E_1 parent transitions is still controversial.[15]

When taken in the backscattering configuration from (001)-grown MQW's or SL's, Raman spectra exhibit three bands (excluding the folded acoustic phonons[4]), as seen in the $P=0$ spectrum in Fig. 1. Longitudinal optic (LO) modes, i.e., with vibrational amplitude along the (001) direction, from the Ge layers are seen near 304 cm^{-1} and from the silicon layers near 510 cm^{-1}. These are respectively denoted Ge-Ge and Si-Si. Raman measurements of both these bands show confinement effects.[5] Near 417 cm^{-1} we observe the interface mode, also longitudinal in this scattering configuration, designated Si-Ge.

Application of hydrostatic pressure will have several interesting consequences for Ge_nSi_m MQW's. First, pressure diminishes the interatomic spacing. Second, the differences between the bulk moduli of silicon and germanium gradually diminishes the strain on the Ge layers. When grown pseudomorphically on silicon substrates, the germanium layers are under biaxial-compressive strain of $\approx 3.8\%$.[16] This reduces to $\approx 2.8\%$ at a pressure of 120 kbar. Both of these effects will alter the vibrational and electronic band structures. While there have been a few studies of the pressure effects on the optical phonon spectrum,[17,18] we find no reports concerning resonance-Raman processes in Ge_nSi_m MQW's under pressure.

In this paper we report the results of a Raman investigation of thin ($n \leqslant 4$, $m \leqslant 7$) Ge_nSi_m MQW's under hydrostatic pressure. The measurements were done with several visible excitation sources, so that resonance enhancement is observed with Ge-like transitions. We find that the observation of a resonance depends on the number of germanium monolayers in each MQW period. Following a brief statement of the experimental methods used, we discuss the effect

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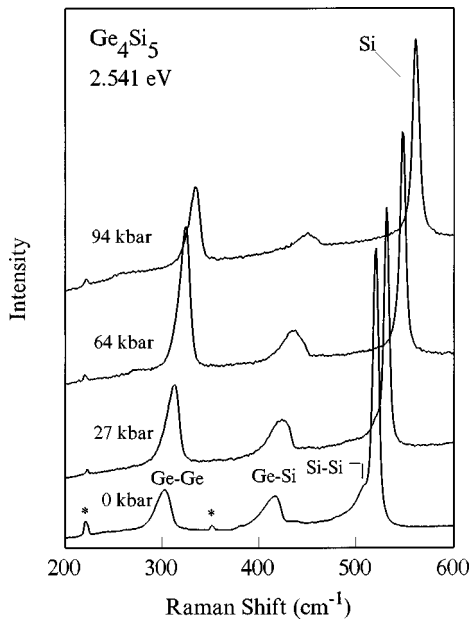

FIG. 1. Room-temperature Raman spectra at various pressures for the Ge_4Si_5 MQW. The principal vibrational modes discussed in the text are Ge-Ge near 300 cm^{-1} and Ge-Si near 420 cm^{-1}. These bands show a clear enhancement in the 50 – 70 kbar range, relative to the silicon substrate/spacer phonon near 520 cm^{-1} at ambient pressure. The low-wave-number shoulder to the latter (near 510 cm^{-1}) is identified as the Si-Si band from the thin MQW silicon layers. Asterisks denote laser plasma lines.

pressure has on the vibrational structure of our samples. We then turn our attention to the observed resonance effects. Results are then summarized.

II. EXPERIMENTAL DETAILS

The Ge_nSi_m MQW's were grown on (001)-oriented silicon substrates at low temperature using molecular-beam epitaxy.[19] The structural building blocks of the MQW's are composed of n and m monolayers of Ge and Si, respectively, repeated five times. These are in turn repeated ten times with spacers of silicon of 300 or 500-Å thick. Values for n, m, and the spacer thickness are given in Table I, with n and m taken from our Raman measurements using the prior work correlating layer thicknesses and phonon confinements.[5] Previous Raman studies indicate the high interface quality achieved in these samples.[14]

TABLE I. Sample designations according to number of monolayers in each MQW period, spacer thickness between repeated units. Raman peak positions (to ± 1 cm^{-1}) for the three main features (see Fig. 1) studied in each sample at ambient presure. Numbers in parentheses are the confinement-induced shifts for the Ge-Ge and Si-Si bands.

Sample	Spacer thickness (Å)	$\nu_{\text{Ge-Ge}}$ (cm^{-1})	$\nu_{\text{Ge-Si}}$ (cm^{-1})	$\nu_{\text{Si-Si}}$ (cm^{-1})
Ge_4Si_5	300	303 (−12)	417	509 (−11)
Ge_3Si_5	300	299 (−16)	418	510 (−10)
Ge_3Si_7	500	299 (−16)	419	512 (−8)

Hydrostatic pressure was applied using standard diamond-anvil cell techniques with a 4:1 mixture of methanol and ethanol as the pressure transmitting medium. Since the Raman band of the silicon substrate was present in all spectra (520 cm^{-1} in the ambient-pressure spectrum of Fig. 1), we use it as an internal measure of the pressure with the established pressure coefficient of 0.51 cm^{-1}/kbar.[20] Ruby dust was used to verify the pressure.[21] Raman spectra were taken at room temperature using the 514.5-nm (2.410 eV), 488.0-nm (2.541 eV), and 457.9-nm (2.708 eV) lines from an argon-ion laser and the 647.1-nm (1.916 eV) krypton line. A micro-Raman instrument was used to focus the excitation onto the sample and gather scattered light in a direct backscattering configuration. Collected light was passed through a holographic notch filter, then analyzed using a 0.5-m spectrometer and detected by a cooled charge-coupled-device (CCD) detector.[34] Signal collection times ranged from 1 to 30 min.

III. EFFECT OF PRESSURE ON THE VIBRATIONAL SPECTRUM

For each sample studied we observed the Si-Si band to shift with the silicon substrate/spacer phonon as pressure varied. We were not able to satisfactorily resolve these bands, so may only state that the pressure shifts are approximately equal. The Si-Si modes are down-shifted from that of the bulk silicon phonon by confinement. The ambient-pressure Raman spectrum (such as in Fig. 1) thus permits us to check the silicon layer thicknesses. The results are listed in Table I, where the values for m and n in Ge_nSi_m for each sample are from the Raman analysis.

The peak position of the Ge-Ge band likewise permits estimation of the thicknesses of the germanium layers of our MQW's. Results (n in Ge_nSi_m) are listed in Table I. Most relevant to us is the observation that two of the samples have slightly thinner germanium layers in each MQW period. This turns out to be important in the resonance-Raman measurements, to be discussed in the next section. The Ge-Ge band blueshifts under pressure for each sample studied, as is seen in Fig. 1 for the Ge_4Si_5 sample. Figure 2 shows the pressure-induced vibrational energy shifts. We graph $\Delta\nu(P)=\nu(P)-\nu(0)$ versus the silicon substrate/spacer phonon energy shift $\Delta\nu_{\text{Si}}(P)=\nu_{\text{Si}}(P)-\nu_{\text{Si}}(0)$ for the Ge-Ge and Ge-Si bands. Table II summarizes the shift rates for each sample obtained from least-squares linear fits to the data, as in Fig. 2. Also listed in Table II are the implied pressure coefficients $\partial\nu/\partial$P and the shift rates scaled to the $P=0$ phonon energy $(\nu_{\text{Si}}/\nu)(\partial\nu/\partial\nu_{\text{Si}})$. The Ge-Ge band shifts at a consistent rate for all three samples. The scaled coefficient is ≈ 1.3 in all cases. This is due to the smaller elastic moduli of germanium compared to those of silicon.[16] Taking this into account, we conclude that the germanium layers in the MQW samples have Grüneisen parameters within a few percent of that of bulk silicon.

Figure 2 also includes the pressure-induced shift of the Ge-Si interface vibrational band, with fit results in Table II. The shift rate is between that of the silicon band and the Ge-Ge mode. We also notice that $\partial\nu_{\text{Ge-Si}}/\partial\nu_{\text{Si}}$ varies slightly from sample to sample. The scaled pressure coefficient implies deformation constants of the Ge-Si interface bonds as

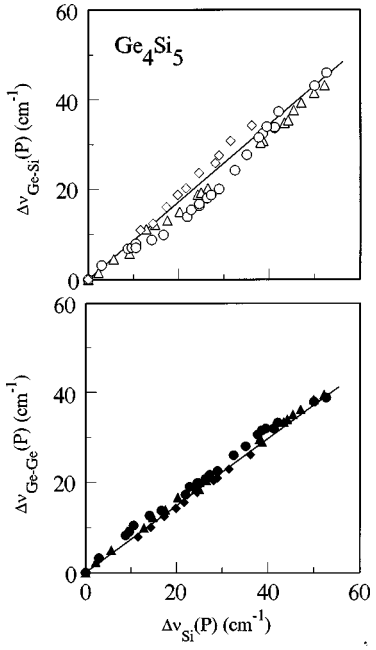

FIG. 2. Pressure-induced shifts in the Raman energies $\nu(P) - \nu(0)$ vs the shift in the silicon substrate/spacer $\nu_{Si}(P) - \nu_{Si}(0)$ phonon. The upper panel is for the Ge-Si interface vibrational band and the lower panel is for the Ge-Ge line. Excitation sources are 2.541 eV (circles), 2.410 eV (diamonds), and 2.708 eV (triangles). Results of least-squares linear fits to the data are in Table II.

being intermediate to those of pure germanium and silicon. This is as anticipated, and is consistent with what can be concluded from hydrostatic pressure studies of Si_xGe_{1-x} alloys.[22]

IV. PRESSURE-TUNED RESONANCE EFFECT

Evident in Fig. 1 is an increase in intensity of the Ge-Ge band and, to a lesser extent, the Ge-Si band relative to the silicon substrate/spacer line. Although the Raman intensity of silicon will vary slightly over the large pressure range studied, we use it as an internal standard. Previous, ambient-pressure work identified resonance enhancement near 2.4 eV as originating from the E_1 transition in the germanium MQW layers.[14,12,3] This value is shifted from the E_1 gap in bulk germanium by strain and quantum confinement. Based on this, we interpret the enhancement seen in Fig. 1 for

TABLE II. Pressure-induced shifts for the Ge-Ge and Ge-Si modes relative to the silicon substrate/spacer phonon band, implied pressure shift rates, and the reduced pressure shift for comparison to silicon.

Sample	Mode	$\partial\nu/\partial\nu_{Si}$	$\partial\nu/\partial$P (cm^{-1}/kbar)	$(\nu_{Si}/\nu)(\partial\nu/\partial\nu_{Si})$
Ge_4Si_5	Ge-Ge	0.74±0.01	0.39±0.01	1.32±0.02
	Ge-Si	0.86±0.03	0.44±0.02	1.08±0.04
Ge_3Si_5	Ge-Ge	0.72±0.04	0.37±0.02	1.27±0.05
	Ge-Si	0.90±0.03	0.46±0.02	1.13±0.04
Ge_3Si_7	Ge-Ge	0.77±0.03	0.39±0.02	1.32±0.04
	Ge-Si	0.94±0.02	0.48±0.01	1.18±0.03

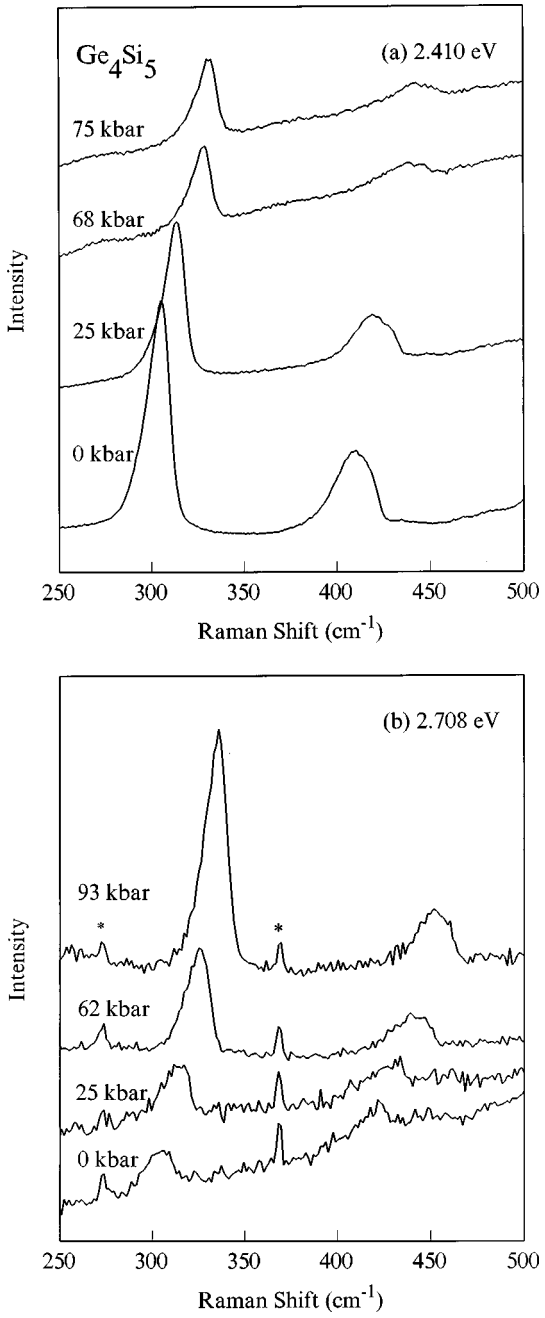

FIG. 3. Raman spectra of the Ge_4Si_5 MQW sample at various pressures comparable to those in Fig. 1 and for two different laser excitations: (a) $\hbar\omega_L = 2.410$ eV and (b) $\hbar\omega_L = 2.708$ eV. The intensities are normalized relative to the silicon substrate/spacer band.

Ge-Ge and Ge-Si to be resonance scattering as *pressure* tunes the electronic transition within the Ge layers through the laser photon energy ($\hbar\omega_L = 2.541$ eV).[23]

Figure 3 shows Raman spectra for the other laser photon energies used here. Spectra are normalized to silicon substrate/spacer phonon intensity (not shown). For the 2.410-eV excitation we observe the intensities of both the Ge-Ge and Ge-Si bands to diminish as pressure shifts the transition out of resonance with $\hbar\omega_L$. When using the 2.708-eV laser source, enhancement of the two bands is seen in Fig. 3 at high pressures. Thus, pressure shifts the resonating energy gap of the germanium layers into resonance with this larger $\hbar\omega_L$. Unfortunately, the silicon and germanium

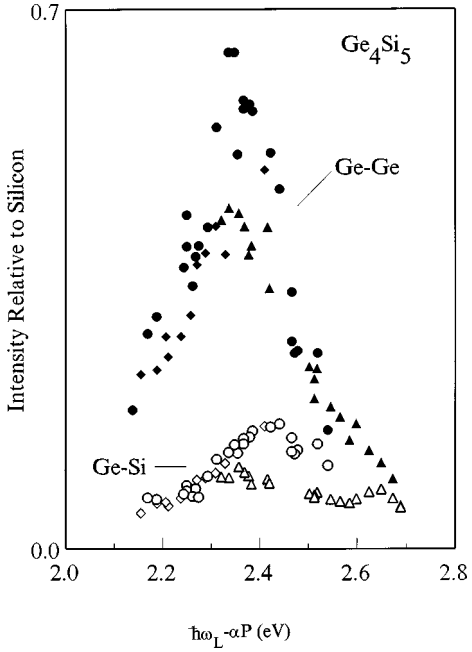

FIG. 4. Relative intensities of the Ge-Ge band (filled symbols) and Ge-Si band (open symbols) for the Ge_4Si_5 MQW. These are graphed vs energy according to Eq. (1) so that results from three laser excitations may be compared. Same symbols as in Fig. 2.

structural phase transitions[24,25] occur at pressures that prohibit us from observing the expected drop in intensity if the resonance were to shift well beyond 2.708 eV.

For the Ge_4Si_5 MQW we graph in Fig. 4 the intensities of the Ge-Ge and Ge-Si bands, relative to that of silicon, versus the quantity

$$E=\hbar\omega_L-\alpha P. \tag{1}$$

Here, $\hbar\omega_L$ corresponds to the excitation photon energy and α is the pressure coefficient of the resonating electronic transition. When treated this way, data from the three laser photon energies may be compared. With $\alpha\approx 4\pm 1$ meV/kbar we obtain the best agreement for the intensity profiles of all three excitation lines. The maximum enhancement resulting from this analysis is near 2.4 eV, as in the ambient-pressure investigation.[14] This pressure coefficient α is lower than the accepted value of 8.5 ± 0.2 meV/kbar for the E_1 transition in bulk germanium.[26] The difference can be partially accounted for according to the following analysis. Since the in-plane lattice constant of the Ge layers is constrained to match that of the surrounding silicon, the change in each due to pressure will be the same as, and determined solely by, the properties of silicon. Since silicon has a larger bulk modulus than germanium (984 versus 750 kbar),[24] the former will show a smaller change in the lattice constant, Δa, than the latter for a given applied pressure. In other words, the Ge layers in our MQW's will exhibit a smaller $\Delta a_{\parallel}^{\mathrm{Ge}} = \Delta a^{\mathrm{Si}}$ in-plane deformation than bulk germanium when subjected to the same pressure. This corresponds to a reduced effect of pressure on the Ge layers imposed by the stiffer silicon. Using

$$\epsilon_{xx}(P)=\frac{a^{\mathrm{Si}}(P)-a^{\mathrm{Ge}}(P)}{a^{\mathrm{Ge}}(P)} \tag{2}$$

for the in-plane strain and the above bulk moduli, we estimate that the E_1 transition in our MQW will exhibit $\approx 74\%$ of the pressure shift seen in bulk Ge. Based on this we arrive at an expected 6.3 meV/kbar pressure coefficient. This partially explains our reduced measured value compared to bulk Ge, substantiating the identification of the resonating electronic transition as the E_1 band of germanium. The remaining departure between the MQW and bulk pressure shifts may be due to differences between elastic moduli and deformation potentials of thin Ge layers compared to the bulk values.

We note that the pressure-tuned resonance enhancement seen in Fig. 4 has the effect of increasing the intensity of the Ge-Ge band by a factor of ≈ 5 versus the $P=0$ intensity. This modest enhancement factor is in approximate agreement with the results of Schorer *et al.* for Ge_5Si_5 strain-symmetrized superlattices.[10] Furthermore, we see that the Ge-Si mode enhancement is weaker than that observed for the Ge-Ge band, differing by a factor of ≈ 5. This is because the Ge-Si mode is localized to the interface regions, while the Ge-like electronic transition is localized to the Ge layers. Thus, the overlap between the electronic and vibrational wave functions is smaller for the interface modes than for the Ge-Ge mode. This accounts for the relative strengths of the resonances of the Ge-Ge and Ge-Si with the E_1-like transition.

Significant differences were observed between the Raman spectra of the Ge_4Si_5 and both the Ge_3Si_5 and Ge_3Si_7 samples. Most notably, we did not observe the pressure-induced resonance enhancement in either of the latter samples. The fact that the germanium layers are slightly thinner (three versus four monolayers) suggests that a certain mimimum thickness is necessary in order to establish energy bands. This agrees with both theory[27] and previous ambient-pressure experiments.[10,14] Our measurements support a minimum thickness of ≈ 4 monolayers of germanium are needed for the resonance-Raman process with the E_1 transition. That the electronic states are borderline in their formation of energy bands between $n=3$ and $n=4$ supports the notion that the elastic constants and deformation potentials may vary from those of bulk germanium, as suggested by the E_1 pressure shift interpretation.

Several interesting observations can be made when comparing the Raman spectra of our Ge_4Si_5 and those of the Ge_3Si_5 or Ge_3Si_7 MQW's. Since the Raman spectra from the latter two were nearly identical, we focus on the Ge_3Si_5 sample. Figure 5 shows spectra for this sample at various pressures comparable to the range examined in Figs. 1 and 3. Two weak features are present, one above the Ge-Ge band and one above the Ge-Si band at ambient pressure. The latter is believed to be due to a slight amount of mixing of silicon and germanium at the interfaces.[28] Under pressure, it shifts with the Ge-Si band, which is what we would expect if the above interpretation is correct. The most striking difference between the spectra in Fig. 5 and in Figs. 1 and 3 is the additional feature near the Ge-Ge band. At ambient pressure this feature is just above the Ge-Ge phonon. Pressure causes this band to redshift at a rate of -0.63 ± 0.07 cm^{-1}/kbar. Both the energy and pressure shift identify this line as second-order, zone-edge scattering by $2\mathrm{TA}(X)$ and, possibly, $2\mathrm{TA}(\Sigma)$ phonons in *silicon*.[20] We are unable to say whether

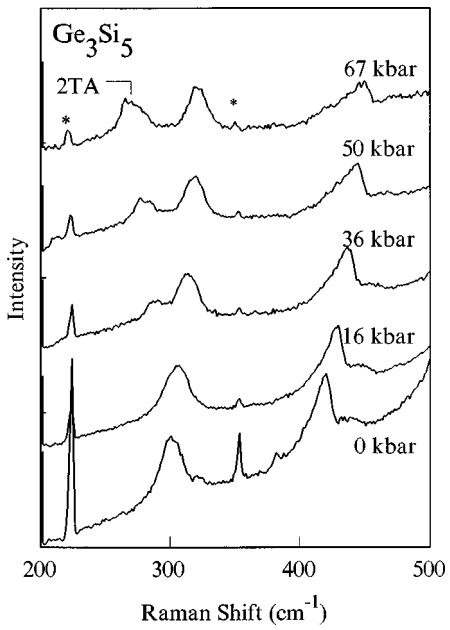

FIG. 5. Effect of pressure on the Raman spectrum of our Ge_3Si_5 MQW. The band just above 300 cm^{-1} in the $P=0$ spectrum is seen to red shift, identifying it as $2TA(X,\Sigma)$ scattering in silicon.

the 2TA comes from the silicon in the MQW or from the spacer/substrate material. We also observe the 2TA feature in the Ge_4Si_5 MQW spectra, but the Ge-Ge and Ge-Si lines are significantly stronger than the 2TA band. This is because in all cases shown (Figs. 1 and 3) the Ge-Ge and Ge-Si bands are enhanced by the resonance with E_1. We verified this by examining the Ge_4Si_5 sample using the off-resonance 1.916-eV (647.1-nm) excitation. Under these conditions, relative intensities for the 2TA and Ge-Ge (and Ge-Si) bands of the Ge_4Si_5 MQW were in close agreement with all spectra measured from the Ge_3Si_5 and Ge_3Si_7 samples. Furthermore, the absolute intensities of the 2TA bands were consistent in spectra from each sample for a given laser excitation and, to a slightly lesser extent, across excitation sources.

The last differences between the Ge_4Si_5 and the two samples with thinner germanium layers (Ge_3Si_5 and Ge_3Si_7) concern the line shapes. We observe that under all conditions for which we studied the Ge-Ge band from the Ge_4Si_5 sample (i.e., three laser lines and pressures up to the structural phase transitions), it remained asymmetric. This was the case even when passing through the resonance in Fig. 4. In contrast, the Ge-Ge Raman band was symmetric under all conditions for the other two samples. Symmetric Ge-Ge bands have been interpreted as stemming from a narrow distribution of Ge layer widths with smooth interfaces. Asymmetric peaks signify ''rougher'' interfaces and a distribution of layer widths. Evidently, the shape of these bands is determined by these factors, plus strain and confinement effects, and is unperturbed by pressure. This makes sense, since we do not expect pressure to alter the basic MQW structure below the phase transition region (≈ 120 kbar) and in the absence of line dislocations caused by pressure.[29]

The Ge-Si interface band exhibits a different behavior. For the Ge_3Si_5 and Ge_3Si_7 MQW's, this band is asymmetric under all conditions studied (Fig. 5). The linewidth is consistently ≈ 17 cm^{-1}. These factors have been associated with smooth interfaces.[7,14] The tale for the Ge_4Si_5 sample is different. At ambient pressure the Ge-Si band is asymmetric and broader (≈ 21 cm^{-1}) in this sample, indicating a higher degree of interface roughness. Most interesting is the effect of pressure, which, in all cases, causes the Ge-Si band in Ge_4Si_5 to become more triangular (Figs. 1 and 3). The linewidth remains approximately constant. This trend prevails regardless of the relationship between the excitation photon energy and the pressure-tuned E_1 transition. A factor that plays a role in optical phonon linewidths is two-phonon resonance.[20,30–32] Since two-phonon decay processes limit the phonon lifetime, resonance with lower-lying combinations that have high densities of states will broaden the optic phonons. For the Ge-Si interface vibration, the closest match with bulk germanium and silicon vibrations is the $TO^{Si}(X)+TA^{Ge}(X)$ combination at frequency 422 cm^{-1} in the bulk matarials.[33] Pressure will shift the interface band out of near resonance with the combination. However, this is most relevant when dealing with Raman modes for which strain, locally inhomogeneous conditions, alloying, isotopic substitution, and other line-shape broadening effects are small. Since this is not the case in our experiments, the line-shape variation seen due to pressure in this sample set is not understood.

V. SUMMARY

The pressure dependences of three primary Raman bands are examined, corresponding to longitudinal vibrations within the germanium layers, within the silicon layers, and at the interface. The pressure shifts agree with a previous study,[17] and are consistent with mode-Grüneisen constants for both the silicon and germanium layers close to that of pure silicon. The Ge-Si interface mode likewise has a similar mode-Grüneisen parameter, provided the elastic constants are an average of those for bulk Ge and Si.

We find that resonance effects dominate the Raman spectrum of the Ge_4Si_5 MQW when measured with any of the argon-ion laser lines as excitation. This can be seen in Figs. 1–3. The resonance involves the germaniumlike, confined E_1 transition near 2.4 eV. The observation that the resonance effect dominates the Raman spectrum for any blue/green excitation is important, since these sources are typically used in Raman studies. Relative intensities of the Ge-Ge and Ge-Si bands may be strongly enhanced and their line shapes altered by the resonance over the more straightforward, volume-related intensities measured far from resonance. The other two MQW's examined consisted of thinner germanium layers and showed no resonance under any conditions examined. The differences in the germanium-layer thickness are small, nominally just 1 ML. Our examination implies that terracing or short-range interface roughness is not solely responsible for the absence of resonance effects within the thinner Ge layers. Rather, it is the absence of an E_1-related energy band that prohibits the resonance. Under all conditions studied, Raman spectra for these samples were like those of the Ge_4Si_5 MQW when it was excited far from resonance with E_1. The resonance-Raman study estimates the E_1 gap to pressure shift at 4 ± 1 meV/kbar. This value is small compared to the pressure shift of the E_1 transition in bulk Ge. We attribute the difference to the larger bulk modulus of silicon, which mediates the pressure-induced lattice

contraction in the germanium layers. Variations in other relevant quantities, such as the elastic constants and deformation potentials, which may occur in extremely thin layers, could also contribute to this reduced pressure shift.

We observe an additional feature near the Ge-Ge vibrational band ($\approx 310\ \mathrm{cm}^{-1}$). These are seen in Figs. 4 and 5 and, to a much lesser extent, in Figs. 1 and 3. This band is strong relative to the Ge-Ge and Ge-Si lines for nonresonant excitation. The systematic redshift allows us to identify the band as 2TA scattering from silicon.

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