Polarized Raman spectroscopy of multilayer Ge/Si(001) quantum dot heterostructures

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Polarized Raman spectroscopy in backscattering geometry has been applied here for the investigation of Ge/Si(001) quantum dot multilayer structures (with the number of layers ranging from 1 to 21) grown by the Stranski-Krastanov technique. The characteristic Raman spectra of the dots have been obtained by taking the difference between the Raman spectra of the dot sample and the reference Si substrate, taken under the same excitation/scattering conditions. We found that the Raman spectra of Ge/Si dots obtained in such a manner are strongly polarized, in particular, for the Ge-Ge (at ~295 cm⁻¹) and Si-Ge (at ~413 cm⁻¹) vibrational modes. The dependence of peak intensity and peak position of the Ge-Ge and Ge Raman bands versus the number of dot layers has been analyzed. It was found that studied quantum dot (QD) systems possess prominent anisotropic intermixing. This results in the Si content in the dots being high and this increases with the number of QD layers. At the same time, the increase of the number of layers was followed by a reduction in the compressive stress within the dots. © 2004 The American Physical Society.

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I. INTRODUCTION

In recent years, a considerable amount of work has been devoted to the study of semiconductor self-assembled quantum dots (QDs). Self-assembled QDs can be grown with III-V, II-VI, and Group IV lattice-mismatched semiconductors using the Stranski-Krastanov growth technique (see review 1 and references therein). Ge/Si self-assembled QDs are attracting specific interest because of their compatibility with Si-based electronics. Growth of high-quality Ge/Si self-assembled QDs can be achieved either by molecular beam epitaxy or chemical vapor deposition. 3 Naturally, the electronic properties of the nanostructures depend on many parameters, including the size, shape, strain profile, and composition of the QDs. A knowledge of these parameters is of crucial importance for future optoelectronic applications of QD nanostructures.

The structure of Ge quantum dots and other nanostructures formed on silicon has been studied using atomic force microscopy, transmission electron microscopy, x-ray photoemission spectroscopy, and other techniques. Raman spectroscopy was also found to be a powerful tool for the characterization of the nanostructure formations. The position, intensity, and width of Raman lines allow one to obtain information on the composition, strain, and quantum confinement in the nanostructures. 2–4 However, there is controversy regarding the analysis of Raman spectra of Ge/Si QDs due to overlapping of the LO-like phonon mode of the Ge dots with the two-phonon spectrum (transverse acoustical, TA modes) of the Si substrate (or Si spacers) at ~435 and 302 cm⁻¹ (see discussion in Refs. 5 and 6) which often dominate the Raman spectra of Ge/Si nanostructures in this region. This fact has been largely ignored in many studies. Because the two-phonon spectrum of Si is strongly polarized, 7 several authors recently used polarized Raman measurements of Ge/Si nanostructures in a configuration which allows the gradual suppression of the contribution of the silicon acoustic phonons to the Raman spectra in the region of ~200–460 cm⁻¹. 6–8 Some aspects of this polarization technique were analyzed in our recent investigations of Si/Ge QD nanostructures. 9 It should be noted that during the preparation of this paper, results on polarized Raman investigations of small, self-assembled Si/Ge dots were published. 10 The dots investigated have a height less than 3 nm and a high areal density when interaction between the dots may be essential. These dots were grown at a relatively low substrate temperature, explaining why the Ge composition obtained was so high. However, to the best of our knowledge, very little work to date has been performed in

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order to determine the composition profile or the strain state inside large size isolated Si/Ge dots grown in multilayer structures.

In this work we use comparative polarized Raman measurements of samples of dots and a Si reference substrate to extract characteristic Raman spectra from QDs for the study of dot composition and strain in Ge/Si quantum dot multilayer structures. The paper also reports on the intermixing effect and stress in QD multilayers (with the number of layers ranging from 1 to 21) grown by the Stranski-Krastanov method.

II. EXPERIMENT

Ge dots were grown in an ultrahigh-vacuum chemical vapor deposition system using SiH$_4$ and GeH$_4$ as gas sources. The growth system has a base pressure of less than $1 \times 10^{-10}$ Torr, and the pressure during growth was about $5 \times 10^{-4}$ Torr. Details of the experimental setup and the growth conditions have been reported elsewhere.\textsuperscript{11}

All samples were grown under approximately identical conditions, the main difference between the samples investigated is the number of the Ge/Si periods. To obtain Ge islands of practically identical size in each period, the Ge amount deposited in each period was adjusted to the effective reflection high-energy electron diffraction critical thickness, according to a method recently proposed.\textsuperscript{12} The Ge islands thus formed have a pyramidal shape and are highly oriented along the [100] directions of the Si substrate.\textsuperscript{13}

The growth conditions and properties of these samples are summarized in Table I. AFM image and a schematic shape of Ge dots are shown in Figs. 1(a) and 1(b), respectively. Finally the Raman spectra of a number of thin (10 nm) SiGe alloys grown by molecular beam epitaxy on Si substrates were also investigated for comparison.

Raman spectra were registered in backscattering geometry using a RENISHAW 1000 micro-Raman system equipped with a Leica microscope. To prevent sample heating the power density was kept below $10^5$ W/cm$^2$. The measurements were performed at room temperature with an Ar$^+$ laser, with a wavelength of 514.5 nm. An 1800 lines/mm grating was used for all measurements, giving a spectral resolution of $\sim 1$ cm$^{-1}$.

During the Raman measurements we used different polarization configurations according to selection rules to distinguish the signals from the Ge dot layers and the Si substrate. The position of the sample, the directions of the Si wafer crystallographic axes and the polarizations of the incident and scattered light are shown in Fig. 2. Therefore, Raman spectra were obtained in two configurations, VV, or [001](110,110)00-1 (Porto’s notation) where scattering by the 2TA mode (peak at $\sim 302$ cm$^{-1}$) is allowed, and in VH, or [001](100,010)00-1 where scattering by the 2TA mode is

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### TABLE I. Parameters of the samples with Ge QDs multilayers.

<table>
<thead>
<tr>
<th>Sample name</th>
<th>Number of layers</th>
<th>Si, barrier Dots (height/base)</th>
<th>Si cap</th>
<th>Areal density (cm$^2$)</th>
<th>Ge content x (Raman)</th>
<th>Strain $\varepsilon$</th>
</tr>
</thead>
<tbody>
<tr>
<td>S21</td>
<td>21</td>
<td>30</td>
<td>5/100</td>
<td>$\ldots$</td>
<td>$6 \times 10^8$</td>
<td>$0.30 \pm 0.04$</td>
</tr>
<tr>
<td>S10</td>
<td>10</td>
<td>22</td>
<td>5/100</td>
<td>$\ldots$</td>
<td>$2.5 \times 10^9$</td>
<td>$0.31 \pm 0.03$</td>
</tr>
<tr>
<td>S10a</td>
<td>10</td>
<td>30</td>
<td>5/100</td>
<td>30</td>
<td>$2.5 \times 10^9$</td>
<td>$0.36 \pm 0.04$</td>
</tr>
<tr>
<td>S5</td>
<td>5</td>
<td>22</td>
<td>5/100</td>
<td>22</td>
<td>$2.5 \times 10^9$</td>
<td>$0.38 \pm 0.04$</td>
</tr>
<tr>
<td>S3</td>
<td>3</td>
<td>160</td>
<td>5/100</td>
<td>$\ldots$</td>
<td>$1.4 \times 10^9$</td>
<td>$0.48 \pm 0.05$</td>
</tr>
<tr>
<td>S2</td>
<td>2</td>
<td>22</td>
<td>5/100</td>
<td>22</td>
<td>$1.4 \times 10^9$</td>
<td>$0.44 \pm 0.04$</td>
</tr>
<tr>
<td>S1</td>
<td>1</td>
<td>22</td>
<td>3/50</td>
<td>$&lt; 20$</td>
<td>$1.5 \times 10^9$</td>
<td>$0.50 \pm 0.04$</td>
</tr>
<tr>
<td>S1a</td>
<td>1</td>
<td>22</td>
<td>14/95</td>
<td>22</td>
<td>$3 \times 10^9$</td>
<td>$0.52 \pm 0.04$</td>
</tr>
</tbody>
</table>

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FIG. 1. AFM image (a), schematic shape of Ge dot (b), and TEM image (c) of Ge/Si multilayer heterostructures with ten layers of quantum dots (sample S10a, see Table I).
dark contrast correspond to the thin Ge wetting layers.

Regions of at 520 cm$^{-1}$, become noticeable. Features at weak features in the Raman spectrum of the Si substrate, single layer of Ge dots. Under such experimental conditions, to noise ratio and to obtain reliable spectra even from a number of accumulations allowing us to increase the signal region 250–700 nm. As mentioned earlier in this paper, the geometries is presented in Fig. 3 for the sample S10a in the Si substrate and spacer layers.

and Ge islands, while light contrast regions correspond to the thick black line which coincides for S10a and Si

III. RESULTS AND DISCUSSIONS

Figure 1(c) shows a representative cross-sectional TEM image of a sample with 10 Ge/Si bilayers. Regions of dark contrast correspond to the thin Ge wetting layers (WLs) and Ge islands, while light contrast regions correspond to the Si substrate and spacer layers.

A set of the Raman spectra measured in the VV and VH geometries is presented in Fig. 3 for the sample S10a in the region 250–700 nm. As mentioned earlier in this paper, the Raman spectra of all the samples were collected with a high number of accumulations allowing us to increase the signal to noise ratio and to obtain reliable spectra even from a single layer of Ge dots. Under such experimental conditions, weak features in the Raman spectrum of the Si substrate, which is always dominated by a strong LO-like phonon band at 520 cm$^{-1}$, become noticeable. Features at $\sim$300, 435, 620 cm$^{-1}$ and at higher frequencies were revealed and described in detail in Ref. 7. Those bands assigned to the two-phonon (or density of states) Raman spectra of Si were found to be strongly polarized.$^7$ In order to avoid any possible confusion of the Raman spectra of Ge/Si quantum dots at the frequencies of interest ($\sim$430 and $\sim$300 cm$^{-1}$) with the two-phonon spectra of Si, spectra of the samples and a Si substrate were measured in VV and VH configuration under the same experimental conditions. The Raman spectra of the substrate are also presented in Fig. 3. A prominent difference is clearly discernible between the sample and reference (Si) spectra in the region 250–500 cm$^{-1}$. Although the 2TA band is observed at $\sim$300 cm$^{-1}$ in both spectra at the VH geometry, the Ge-Ge peak with an intensity sufficient for analysis appears as a low-energy shoulder of the more intense Si 2TA peak in the sample spectrum. Note that the high orientation of the Ge islands on the Si substrate in the samples studied results in the highest intensity of the LO-phonon-like Ge-Ge peak. For random orientation of the islands (for example, dome-shaped islands) the Raman signal will be at least half this intensity and may be too weak for analysis.

Therefore, the characteristic Raman spectra of Ge dots $I_{\text{dots}}$ can be obtained by taking the difference between Raman spectra of the dot sample $I_{\text{sample}}$ and the reference Si substrate $I_{\text{Si}}$ registered in VH geometry,

$$I_{\text{dots}} = I_{\text{sample}} - f I_{\text{Si}},$$

where $f$ is the subtraction factor which can be obtained by taking the ratio of the LO-like bands of the Si substrate at 520 cm$^{-1}$ in the Raman spectra of the dot sample and the reference one $f = (I_{\text{sample}}/I_{\text{Si}})_{520}$. $I_{\text{dots}}$ will hereafter referred to as the “difference spectrum.” We believe that by using this approach the contribution of both the Si substrate and Si spacers to the Raman spectrum of sample with dots will be taken into account. It should be noted that great care must be taken when choosing the reference for subtraction. We observed during this work that if the Si substrate or Si buffer layer are heavily doped, their TA phonon Raman spectrum will be slightly different from that for the normally doped Si substrate. In such a case the reference sample must be specially grown with the same structure of Si layers. Only then can the subtraction be done correctly.

The difference Raman spectrum of a sample with ten layers of Ge dots (sample S10), together with the original spectrum of the sample and that from a Si substrate in VH polarization are shown in Fig. 4. Three features at frequencies of $\sim$413, $\sim$435, and $\sim$298 cm$^{-1}$ are seen in the difference spectrum [Fig. 4(b)]. Assignment of the bands is well established: 2,14 the two high frequency peaks belong to Si-Si local and Si-Ge modes, respectively, while the low frequency peak belongs to the Ge-Ge mode. The difference spectra for a number of samples in the region 250–470 cm$^{-1}$ are shown in Fig. 5. In order to compare the intensities of the Raman signals from different samples, the spectra were normalized to the intensity of the Si LO-phonon peak of the substrate. As can be seen, the Raman spectra differ from each other depending on number of layers deposited. The dependence of the Si-Ge and Ge-Ge peak integrated intensities and the peak positions for the samples studied are summarized in Fig. 6.

FIG. 2. Schematic of electric vector orientation in the incident and scattered light channels during polarized Raman measurements.

FIG. 3. Raman spectra of sample S10a and the Si substrate collected at VV (thick black line which coincides for S10a and Si) and VH (thick and dashed gray lines, respectively) polarization.
Surprisingly, the intensities of both peaks increase with the number of layers, demonstrating a weak saturation only for the sample with 21 layers of the Ge dots (Fig. 6). A simple estimation (see Fig. 7 and Table II) of the depth of light penetration into Ge is given by

\[ d_p = 2.3/\alpha, \]

where \( \alpha_{\text{Ge}} = 600,000 \text{ cm}^{-1} \) for the wavelength of \( \lambda = 514 \text{ nm} \) used in our experiment gives us a value of \( d_p(\text{Ge}) = 19.2 \text{ nm} \) [note, \( d_p(\text{Si}) = 763 \text{ nm} \) for Si where \( \alpha_{\text{Si}} = 15,080 \text{ cm}^{-1} \)]. It is obvious that the intensities of the dot related Raman bands vs number of dot layers must undergo saturation in the region of three to four layers. However, as can be seen from Figs. 6(a) and 6(b) this is not the case. There are two possible explanations for this effect: (i) the Raman signals come mainly from the surface or local vibrational modes of the Ge WLs (Fig. 7), linearly increasing in intensity with number of WLs, or (ii) the dots contain a large amount of Si due to its diffusion into Ge islands, the so-called intermixing effect, resulting in an increase of the penetration depth for the multilayer heterostructures studied.

As far as the Raman signal from the wetting layer is concerned, we can exclude the surface modes, which can propagate in such layers, since they are forbidden in the backscattering geometry and their peak positions are shifted to lower frequencies \((390 \text{ cm}^{-1} \text{ for Si-Ge and } \sim 288 \text{ cm}^{-1} \text{ for Ge-Ge peaks})\) as compared with those observed by us. As for the local modes, we can refer readers to the papers, where the results on Raman line-mapping experiments on Ge islands with small areal density \( \sim 9 \times 10^8 \text{ cm}^{-2} = 9/\mu\text{m}^2 \) were published. In both reports no Ge-related signal was detected between these islands, i.e., from the WL region, despite the sensitivity of the Raman probe being comparable to that used in this work where the signals of the Ge-Ge and Si-Ge modes have been reliably registered from the single dot layer. Therefore, we can conclude that the major contribution to the Raman signal from the one layer sample studied originates from the dots. To estimate a possible contribution of the WL related modes to the Raman signals from samples with 10–21 dot layers, we measured the Raman spectra from a sample containing one 10 nm thick Si\(_{0.64}\)Ge\(_{0.36}\) layer. The thickness of this layer corresponds to that of 30–60 WLs. We found that the intensities of Raman signals from the layer are comparable with those from a sample with one dot layer. Therefore, we conclude that the Raman signals in question arise mainly from the dot layers.

On the other hand, the Raman intensity dependence on the number of QD layers can be explained by the intermixing effect that causes a reduction of the light absorption by the Ge-Si dots and hence an increase of the depth of penetration of the laser light. Indeed, the depth of penetration of 514 nm radiation into Si\(_{1-x}\)Ge\(_x\) layers can be easily estimated by the use of the well-known expression

\[ d_p(\text{Si}_{1-x}\text{Ge}_x) = (1-x)d_p(\text{Si}_{1-x}) + xd_p(\text{Ge}_x), \]

which shows that at \( x = 0.5 \), \( d_p(\text{Si}_{0.5}\text{Ge}_{0.5}) = 391.1 \text{ nm} \). This value is large enough to allow the light to pass through all 21 layers of Ge dots and Si spacers.

Therefore Ge-Si intermixing is expected to occur in the Ge/Si quantum dot structures studied and will modify the Raman spectra of the Ge dots. As shown in a number of papers, this effect depends on the Ge and Si layer deposition conditions and for Ge dots grown at high temperature we can expect that more than 50% Si will diffuse into the dot interface. For one layer of dots, Ge/Si intermixing may occur at both interfaces: at the dot/substrate interface, intermixing is mainly thermally activated while at the interface between the dots and cap layers, it may be enhanced by the strain field distributed over the dot surface.

Based on Raman measurements the intermixing effect can be found from the ratio of the integrated intensities of the Ge-Ge and Si-Ge peaks,

\[ I_{\text{Ge-Ge}}/I_{\text{Si-Ge}} = Bx/(1-x), \]

where \( x \) is the Ge content and \( B \) is a constant which depends on the experimental conditions. We must stress that Eq. (4)
was obtained and checked for a number of SiGe alloys using nonpolarized Raman spectra. Since we used polarized Raman measurements in our study, we initially checked the validity of Eq. (4) for a number of thin SiGe layers (embedded between Si epitaxial layers) with a known Ge content. In this way, we determined a coefficient $B$ of 1.5 for our experimental conditions. Calculations performed for the set of samples with multilayers of Ge dots show that the Ge content reduces as the number of layers increases (see Table I) from ~50\% for one layer down to 30\% for 21 layers, i.e., the dots are in reality the Ge/Si ones. The high content of Si in the dot is in agreement with data obtained for similar samples in Ref. 18 by the selected area transmission electron diffraction of a single quantum dot and with results obtained by various analytical methods for Ge dots grown by other techniques.\textsuperscript{19,20} Because of the increasing number of QDs in the laser beam waist, both the Ge-Ge and Ge-Si band intensities simultaneously increase as the number of QD layers increases from 1 to 21 layers [Figs. 6(a) and 6(b)]. However, comparing the data shown in these figures it is seen that the intensity of the Ge-Ge band increases by approximately three times whereas that of Ge-Si band increases by approximately eight times, even though the ratio of these intensities changes by only three times. This fact qualitatively supports the assumption that the Raman band intensities are proportional to the number of corresponding bonds, on which relation (4) is based.

As can be seen from Figs. 3 and 4, the band at ~413 cm\(^{-1}\), corresponding to the local Si-Ge modes is strongly polarized in the Raman spectra of Ge/Si dots while this band was found to be mainly depolarized in the Raman spectra of isotropic bulk Si\(_{1-x}\)Ge\(_x\) alloys and demonstrate a partial polarization in the Raman spectra of thin (10 nm) alloy layers embedded between the Si layers (Fig. 1). We believe that the strong polarization of the band could indicate the ordering of the Si-Ge bonds in the dots due to an anisotropic intermixing effect when the Si atoms penetrate the dots from the buffer and Si cap layers, mainly along the sample growth direction. This is not unexpected because the height of the Ge-Si islands (6 nm) is much smaller than their lateral size (50–100 nm). The same effect leads to a partial polarization of the Si-Ge band in Raman spectra of the thin alloy layer where the distribution of Si atoms also becomes slightly anisotropic.

Other features indicating Si/Ge intermixing in the QD systems studied is that the energy of the Ge-Ge peak is lower than that of the Ge bulk LO phonon (300 cm\(^{-1}\)). In pure Ge systems it indicates that the Ge dot layer is under tensile stress. However, this conclusion contradicts the results obtained for analogous systems by other methods, and in particular by TEM and by photoluminescence measurements, which show that the stress is in fact compressive.
for Si 1–
and Si-Ge Raman bands, we utilize an approach developed dot from the Raman data, namely, from the shift of Ge-Ge

fore, in order to extract information about stress in the Ge

s

dots from the bulk value is mainly due to the three reasons:

(i) a confinement effect, (ii) an intermixing effect, and (iii)
stress within the layer. The confinement effect shifts the po-
tion of the Ge-Ge phonon peak downwards.24,25 However,
the size of Ge dots appears too large to expect a substantial
confinement effect especially in the plane of growth. As was
shown in Refs. 24 and 25, this effect can play a crucial role
for Ge nanostructures with sizes of less than 2–3 nm. There-

fore, in order to extract information about stress in the Ge
dot from the Raman data, namely, from the shift of Ge-Ge
and Si-Ge Raman bands, we utilize an approach developed
for the Si1–xGex alloys.23

The value of stress can be estimated using the expres-
sions for Si-Ge ($\omega_{Si-Ge}$) and Ge-Ge ($\omega_{Ge-Ge}$) peak positions
for Si1–xGex alloys23 if the intermixing effect (or Ge content, x)
is known,

$$\omega_{Si-Ge} = 400.5 + 14.2x - 575\epsilon,$$

(5)

$$\omega_{Ge-Ge} = 282.5 + 16x - 384\epsilon,$$

(6)

where $\epsilon$ is strain, which is directly related to the biaxial
stress $\sigma$. Since we obtained the $x$ values for our samples by
taking the ratio of the integrated intensities of Si-Ge and
Ge-Ge peaks, we can easily estimate the corresponding values
of $\epsilon$ based on expressions (5) and (6). The arithmetic
average values of $\epsilon$ calculated from the $\omega_{Si-Ge}$ and $\omega_{Ge-Ge}$
data are listed in Table I and shown in Fig. 8 as a function of
the number of layers. From these values we can conclude
that the stress is compressive and that with the increase in
the number of the layers, the structure becomes more relaxed.
This is supported by the evolution of the $\omega_{Si-Ge}$ and $\omega_{Ge-Ge}$
peak frequencies which approach the values corresponding
to the fully relaxed SiGe alloy. This is seen from Figs. 6(c)
and 6(d) where dashed lines show these frequencies calcu-
lated using Eqs. (5) and (6) for the fully relaxed ($\epsilon = 0)$ Si0.5Ge0.5 alloy.

The Raman data indicate that even in 21 layer Si-Ge
heterostructures the dots are still not fully relaxed. At the
same time, we would like to note that in our case the Raman
data give us the stress value and intermixing averaged over
all QD layers because of the large light penetration depth
into the samples. It is possible that in the multilayer systems
studied, the stress value for the upper QD layers is smaller
than those for lower ones and the top layer is fully relaxed.
However, it is not possible to check using our Raman analy-
sis. Nevertheless, in Ge dot systems without Si-Ge intermix-
ing, where high absorption of light by the dots limits the
penetration depth, such analysis is possible with at least three
to four layer resolution.

IV. CONCLUSION

We have shown that polarized Raman spectroscopy can be
used for the study of evolution of composition and stress of
Ge QDs in multilayer Si-Ge structures with the number of
layers varying between 1 and 21. Analysis of polarization,
integrated intensities, and peak positions of the Ge-Ge and
Si-Ge bands in the Raman spectra of the dots has been car-
ried out. We have found a pronounced anisotropic Si-Ge in-
termixing effect which results in an increase of the Si content
in the dots from 0.5 to 0.7 with an increasing number of QD
layers. At the same time a reduction of the compressive
stress in the dots with an increasing number of layers has
been revealed. These results are in agreement with data ob-
tained by other methods.

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