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Elastic relaxation of dry-etched Si/SiGe quantum dots

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Elastic relaxation of the compressive strain due to the lattice mismatch between SiGe and Si has been studied with both x-ray diffraction and Raman scattering in small (30–100 nm) dry-etched Si/SiGe quantum dots fabricated from high-quality multilayers grown on (001)-oriented Si. The Raman spectroscopic investigations showed that the dot alloy layers have relaxed by approximately 65% from their fully strained value and that a compensating tensile strain has been induced in the Si layers. The relaxation is essentially independent of the dot size and the values derived experimentally compare well with analytical and numerical model calculations. [S0163-1829(98)07731-5]

I. INTRODUCTION

The SiGe material system has been widely investigated because of its high degree of compatibility with Si technology. So far, the band offsets of SiGe with respect to Si have been utilized to enhance the performance of electrical devices such as heterobipolar transistors, but there is also a lot of interest in fabricating Si-based optical devices. Optical absorption devices such as SiGe infrared detectors have been realized.¹ Optical emission from SiGe is hampered by the indirect band gaps of both constituents and hence has a low efficiency. However, the electroluminescence emission efficiency of SiGe superlattices can be increased by orders of magnitude if they are structured into arrays of quantum dots with dot diameters below 100–200 nm.^{2,3}

In the growth of SiGe on Si, the offsets in the valence and conduction bands as well as the band gap itself strongly depend on the strain in the layer.^{4,5} Therefore, for a proper interpretation of optical and electrical experiments, a knowledge of the strain status of the layers is required. The latter can be evaluated by x-ray diffraction and vibrational Raman spectroscopy. X-ray diffraction is sensitive to lattice constants and does not allow a direct determination of the strain status of the Si and SiGe layers separately. Raman scattering, however, does offer such a material resolution.

Raman spectroscopy has been applied previously to analyzing the strain in a variety of SiGe mesa and wire heterostructures^{6–8} with modeling of the results based on the finite element method.^{9–11} Comparatively little Raman work has been performed on SiGe dot heterostructures,^{12–14} where dots with a small (50–60 nm) or large (300–1000 nm) diameter were investigated. In the large dots, only small shifts of the Raman vibrational frequencies from the pseudomorphic values were obtained, due to the edge induced relaxation.¹⁴ In the small dots, a 50% relaxation of the alloy layer was observed.¹³ No information was available in these studies from simultaneous x-ray-diffraction analyses.

High-resolution x-ray diffraction can be used for the assessment both of the coherent strain due to the elastic relaxation of dry-etched semiconductor nanostructures and of random strain fields due to defects induced by the fabrication process.^{15–18} In the case of laterally periodic structures such as the studied arrays of quantum dots, the superlattice peaks occurring in x-ray diffraction split up into a series of lateral dot satellites, which necessitates the two-dimensional mapping of the intensity distribution in reciprocal space.

In this article we report on reciprocal space mapping and Raman investigations of the elastic relaxation of Si/SiGe quantum dots with diameters ranging from 30 to 100 nm and its simulation using both an analytical isotropic model and finite element calculations. The large alloy layer relaxation is found to be essentially independent of the dot diameter and an appreciable compensating tensile strain is evident in the Si layers. Both are in agreement with the continuum-elastic model predictions.

II. EXPERIMENT

The sample was grown by solid-source molecular beam epitaxy in a VG Semicon V80 system at a growth temperature of 600 °C.¹⁹ It consists of ten periods of nominally 23 nm of Si and 4 nm of SiGe with a Ge concentration of 35% on a (001)-oriented Si substrate. The excellent quality of the sample is illustrated in Fig. 1, where an x-ray rocking curve of an as-grown reference sample is shown along with its simulation using dynamical x-ray-diffraction theory. Since no broadening of the superlattice satellites occurs up to the eighth and ninth order, the composition and layer thicknesses are homogeneous throughout the multiple quantum well stack. The parameters derived from the dynamical fit are 22.4 nm for the Si barrier thickness, 3.0 nm for the thickness of the SiGe wells, and 25% for their Ge concentration.

Four quantum dot arrays with a size of 1 mm² were defined by electron beam lithography and subsequent reactive

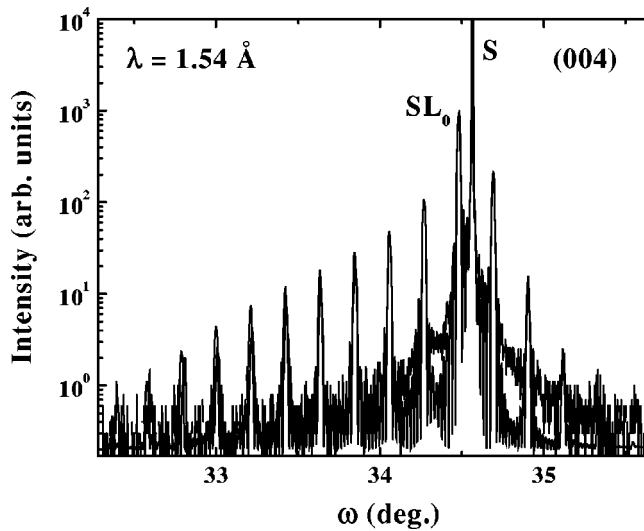


FIG. 1. (004) rocking curve of the unetched reference sample (crosses) along with its dynamical simulation (full line).

ion etching using SiCl_4 .⁷ The diameters of the quantum dots in the square arrays were nominally 30, 40, 60, and 100 nm, respectively. The spacing of the dots, i.e., the lateral period, was five times the respective dot diameter. Scanning electron microscope investigations of the 100 nm dot sample showed that the dots were etched uniformly across the array, were 120 nm across the top, and had a slight (7°) outward taper with depth from the surface. The etching depth was approximately 225 nm and nominally one period of the multiple quantum well structure remained unetched. Figure 2 shows a sketch of the dot geometry.

The x-ray experiments were performed at the bending magnet beam line ROEMO I of HASYLAB, Hamburg. The beam line was equipped with an asymmetrically cut double crystal monochromator and a wavelength of 1 \AA was used. A sufficient angular resolution was achieved by using a narrow slit in front of the NaI scintillation detector. The measurements of the unstructured reference sample were done with a Philips MRD diffractometer with a four-crystal Ge(220) monochromator.

The angle between the incident x rays and the sample surface is denoted as ω and the angle between the primary

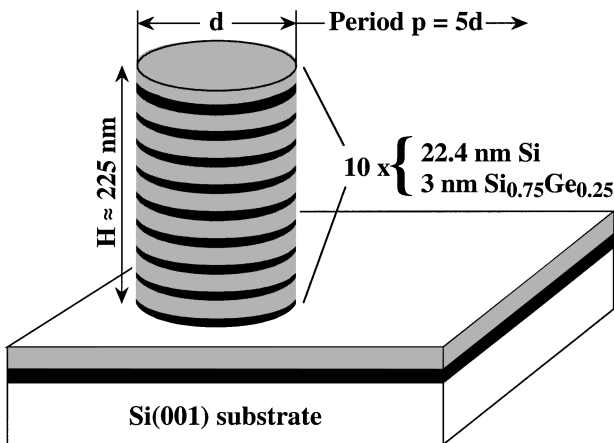


FIG. 2. Sketch of the dot geometry. About one period of the superlattice remained unetched.

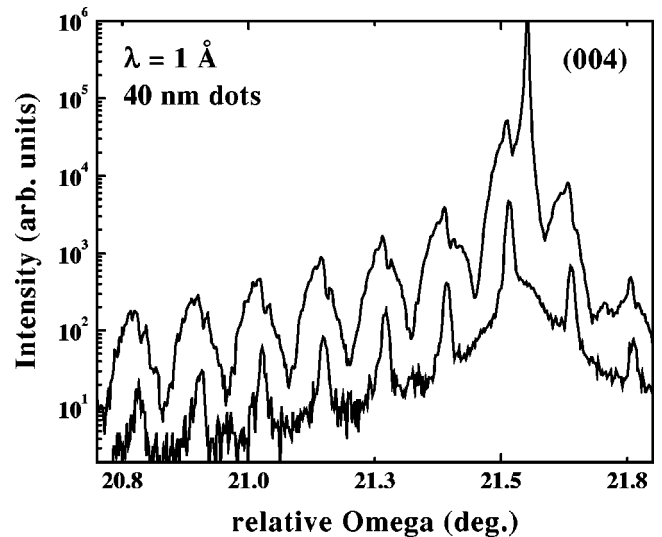


FIG. 3. $\omega - 2\theta$ scans through the zeroth-order (upper curve) and first-order (lower curve) dot satellites.

and the diffracted x-ray wave vectors as 2θ . Reciprocal space mapping is achieved via independent variation of ω and 2θ .

The Raman experiments were carried out at room temperature in a quasibackscattering geometry²⁰ using an angle of incidence of 77.7° for the incoming laser light on the (001) surface. The Raman spectrum was excited with 30 mW of 457.9 nm argon laser light, analyzed with a Spex 14018 double monochromator, and detected with a cooled RCA 31034A photomultiplier. The incident light was polarized in the scattering plane, while the scattered light was collected without polarization analysis.

III. RESULTS

A. X-ray diffraction

An $\omega - 2\theta$ scan of the 40 nm dot array analogous to the rocking curve in Fig. 1 is depicted in Fig. 3 (upper curve). The lower curve is an $\omega - 2\theta$ scan through the first-order dot satellites. As can be seen, the width of the dot satellites is significantly smaller than the width of the zeroth-order satellites. The latter apparently consist of two peaks: a narrow peak (with a full width at half maximum identical to the width of the first-order satellites) superimposed on a broader one. We attribute this feature to the incomplete etching of the superlattice. The remaining two-dimensional part of approximately two unetched periods gives rise to broader multiple quantum well peaks at positions nearly identical to the dot satellites.

In Fig. 4 reciprocal space maps (RSM's) around (004) of the 30 nm and the 100 nm dot array are shown. The different lateral periods of 5 times the dot diameter give rise to lateral satellites with a spacing inversely proportional to the real space periods of 150 nm and 500 nm, respectively. The larger width of the satellites in the RSM of the 30 nm array is due to a larger detector-slit width, which was chosen to increase the detected intensity.

The streak denoted by E traces the Ewald sphere in the reciprocal plane and is due to a limited angular resolution of

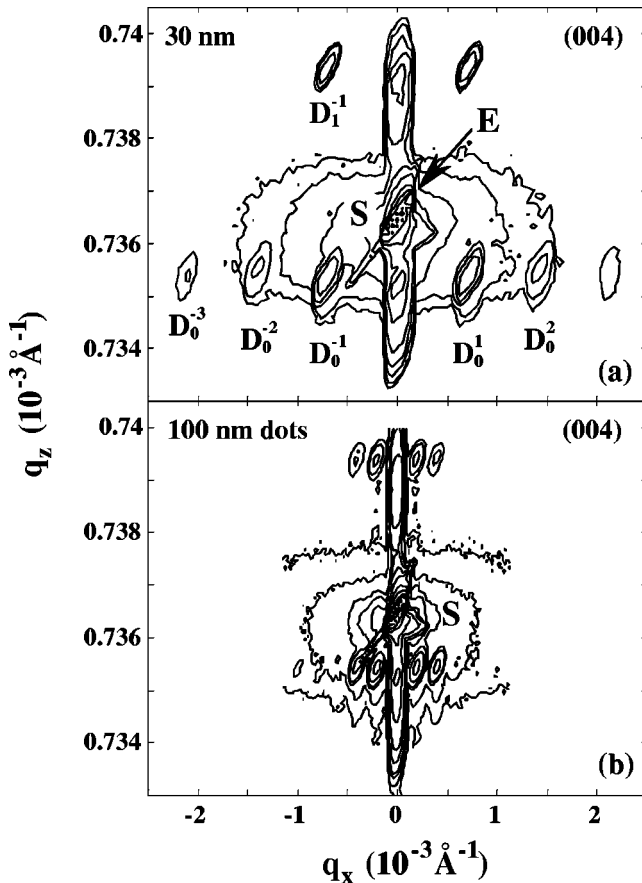


FIG. 4. Reciprocal space maps around (004) of the (a) 30-nm and (b) 100-nm dot arrays.

the detector setup. S denotes the Si substrate peak and D_i^j are the dot satellites of respective order. The first index i marks the order of the corresponding superlattice satellite and the second index j labels the lateral order of the dot satellite.

Diffuse scattering centered around the substrate peak is observable, which is very likely the result of lattice defects in the Czochralski-grown Si substrate. It obscures potential diffuse scattering from the quantum dots and thus prevents the detection and the structural analysis of a possibly present damaged layer at the sidewalls of the dots. Such a damaged sidewall layer would manifest itself in diffuse scattering around and between the coherent dot satellites.¹⁶

In contrast to symmetrical reciprocal lattice points (RLP's) such as (004), the diffraction around asymmetrical RLP's is also influenced by the in-plane lattice constant in the layers and hence the lateral strain. A lateral strain relaxation increases the in-plane lattice constant compared to its value in an unetched, fully strained, and pseudomorphic layer. This increase of the in-plane lattice constant in the quantum dots induces a shift of the envelope of the dot satellites in the direction of smaller reciprocal in-plane coordinates $|q_x|$. The shift is proportional to the increase of the average in-plane lattice constant above the value of the unstrained Si substrate.

In Fig. 5, RSM's around the asymmetric (115) RLP are shown for the 60 nm and the 100 nm dot array. For geometrical reasons concerning the experimental setup, the investigation of RLPs with a more distinct asymmetry than (115) was not possible. The streak through the substrate peak towards

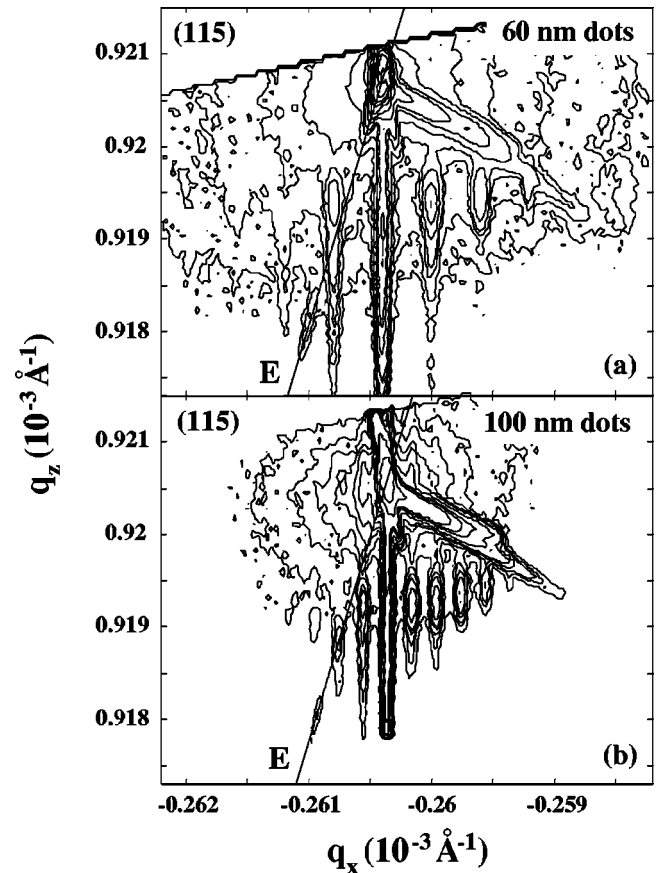


FIG. 5. Reciprocal space maps around (115) of the (a) 60-nm and (b) 100-nm dot arrays.

the lower left corner of the map again follows the Ewald sphere. Another streak that is nearly symmetric to the first one with respect to the substrate truncation rod is attributed to a monochromator streak.²¹ The more prominent one, which runs only on the right-hand side of the substrate peak, is approximately parallel to the [111] direction and may be due to stacking faults.

In the (115) RSM of the 100 nm diameter dots (Fig. 5), satellites on the left and right hand sides of the substrate peak are also visible. We attribute their existence to a periodic strain field induced by the relaxing dots in the unetched part of the multilayer and the Si substrate.

The shift of the envelope of the dot satellites towards $q_x = 0$ is clearly visible, as more satellites are present on the right hand side than on the left hand side of the substrate q_x position. The peak shift has been determined for all four dot arrays and the corresponding values for the average strain relaxation expressed as in-plane lattice constants are depicted in Fig. 6 (lower curve). As the number of observable satellites decreases with decreasing dot period, the error bars for smaller dots are larger.

Due to the relaxation, a tensile strain is induced in the Si layers and the compressive strain in the etched SiGe layers is reduced. However, the relaxing dots also cause a tensile strain in the unetched superlattice periods and the Si substrate directly underneath the dots. Moreover, the unetched regions between the dots are compressively strained. The range of these strain fields in the unetched material scales with the dot diameter and is larger for the 100 nm dots than

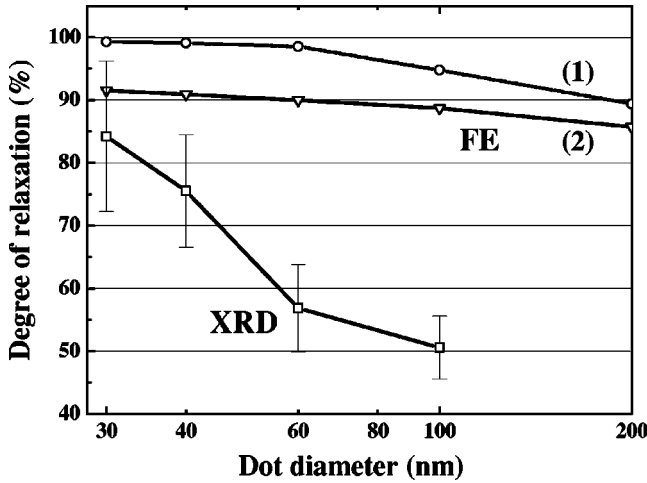


FIG. 6. Average elastic relaxation of the quantum dots as obtained from the reciprocal space maps (lower curve, labeled XRD) and the finite element calculations (upper curves, labeled FE).

for the 30 nm dots. This is reflected in the RSM's, where the aforementioned lateral satellites of the substrate peak are visible for the 60 nm and the 100 nm dot arrays, but not for the 30 and 40 nm dots. For the latter, the satellite peaks are hidden in the diffuse scattering.

As a consequence of the tensile and compressive strain fields induced in the unetched material, the average in-plane lattice constants as detected by x-ray diffraction and the corresponding elastic relaxation are smaller than expected for the etched part alone and the discrepancy increases with increasing dot diameter. The data points in Fig. 6 therefore represent lower limits for the dot relaxation.

B. Raman spectroscopy

Raman spectra obtained from the reference sample and the dot arrays are shown in Fig. 7. The spectrum of the reference sample exhibits a series of eight sharp peaks at frequencies up to 100 cm^{-1} that are due to superlattice folded longitudinal acoustic (FLA) modes. The appearance of such a large number of FLA peaks together with their sharpness indicates a high-quality superlattice with sharp

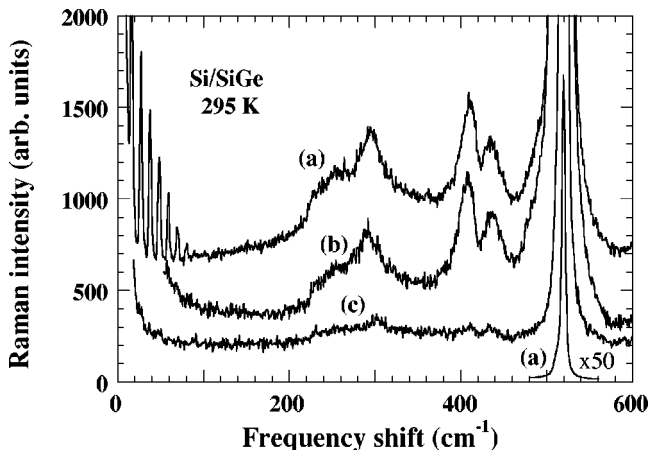


FIG. 7. Room temperature Raman spectra from (a) the as-grown superlattice, (b) the 60 nm dot array, and (c) the etched surface between the dot arrays. The spectral resolution was 3 cm^{-1} .

interfaces.²⁰ The frequencies of the FLA modes are very well reproduced in the Rytov theory^{20,25} taking the x-ray values for the layer parameters and using known or interpolated values of $v_{\text{Si}}=8433\text{ m/s}$ and $v_{\text{SiGe}}=7553\text{ m/s}$ for the layer sound velocities, $\rho_{\text{Si}}=2330\text{ kg/m}^3$ and $\rho_{\text{SiGe}}=3088\text{ kg/m}^3$ for the mass densities, and $n_{\text{Si}}=4.66$ and $n_{\text{SiGe}}=4.70$ for the refractive indices at 457.9 nm . A perfect fit is obtained with the Rytov model by increasing the superlattice period (22.4 nm) by 0.2 nm. Such a slight increase in the period predicted by Raman results over the value determined by x-ray diffraction has been observed before for $\text{InSb}/\text{In}_{1-x}\text{Al}_x\text{Sb}$ superlattices.²⁶ It was attributed to the literature sound velocities used in that study being slightly too high. The present result shows that the small difference between x-ray and Raman results for the period is more systematic and warrants further investigation.

Also shown in Fig. 7 is a series of longitudinal optic (LO) phonon peaks due to lattice vibrations within the Si and SiGe layers. For the as-grown superlattice [Fig. 7(a)], three peaks occur at 295.3 , 410.3 , and 514.2 cm^{-1} (shoulder feature) associated with the so-called Ge-Ge, Ge-Si, and Si-Si modes,²⁷ respectively, of the strained alloy layers and at 520.2 cm^{-1} in the unstrained Si layers and Si substrate. In the 60 nm dots, the respective peaks occur at 291.9 , 408.2 , 514.3 , and 520.1 cm^{-1} [see Fig. 7(b)]. However, analysis of the dot results requires some care because of two factors: First, the samples are incompletely etched through leaving at least one two-dimensional superlattice alloy layer and second, segregated Ge during dry etching recombines with the surface materials forming a diluted SiGe layer coating the entire structure.^{7,12,28} Evidence for this comes from the Raman spectrum shown in Fig. 7(c), which is taken from the unmasked and etched surface area in between the four arrays of dots on the sample. Weak peaks at 302 , 409 , and 515.5 cm^{-1} (shoulder) due to SiGe alloy formation are evident along with the intense 520.2 cm^{-1} line and some second-order scattering from the Si substrate. Thus, in the $500\text{--}550\text{ cm}^{-1}$ region of the dot Raman spectrum there are actually four peaks: two Si-Si alloy peaks coming from the dots and SiGe coating plus unetched alloy layers, respectively, one Si substrate peak, and one dot Si-layer peak. Curve resolving this frequency region lead to a ready identification of the four bands. In the least squares fits, the frequency, width, and line shapes of the Si substrate and background Si-Si alloy peaks were fixed using parameter values obtained from fits to the reference spectra. The frequencies of the other two modes, as obtained from the curve fitting, are plotted versus dot diameter in Fig. 8. The alloy layer Si-Si frequencies are considerably lower in the dots than in the fully strained superlattice case, while the Si layer frequencies are reduced from that of bulk Si. These results indicate considerable strain relaxation effects in the dots. Similar strain relaxation induced shifts in the Si-Si line frequency have been observed previously for $50\text{--}60\text{ nm}$ dots,^{12,13} but the frequency shifts in the Si layers of the Si/SiGe superlattice samples were not resolved in that case.

IV. MODEL CALCULATIONS

A. Finite element calculations

Finite element calculations of the elastic relaxation of the quantum dots have been performed with the commercial pro-

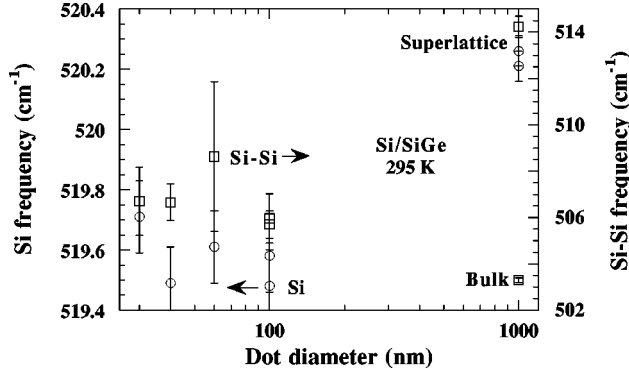


FIG. 8. Raman line frequencies of the Si (○) and Si-Si (□) modes in the Si and SiGe layers, respectively, of the dots. The equivalent superlattice and bulk frequencies are plotted at the right.

gram NASTRAN. The following values for the lattice constants and the elastic constants have been assumed:^{22,23} $a_{\text{Si}}=5.43105 \text{ \AA}$, $a_{\text{Ge}}=5.6579 \text{ \AA}$, $C_{11,\text{Si}}=165.8 \text{ GPa}$, $C_{12,\text{Si}}=63.9 \text{ GPa}$, $C_{44,\text{Si}}=79.6 \text{ GPa}$, $C_{11,\text{Ge}}=128.5 \text{ GPa}$, $C_{12,\text{Ge}}=48.3 \text{ GPa}$, and $C_{44,\text{Ge}}=68.3 \text{ GPa}$. The values for $\text{Si}_{0.75}\text{Ge}_{0.25}$ were obtained by linear interpolation.

The calculations were done with two different geometrical models. In the first model, the vertical superlattice structure was replaced by a homogeneous layer of the same thickness and with the average composition of the superlattice. This was done to reduce the number of node points necessary for the discretization of the structure.

In the second model, we took the compositional modulation of the multiple quantum well into account and obtained results close to those of the less accurate first model. In both models, the stress in the unetched material induced by the relaxing quantum dot was accounted for by modeling a large part of the substrate between the dots.

The numerical values of the average elastic relaxation of the quantum dots following from the two models are plotted in the upper curves of Fig. 6 [labeled (1) and (2)] for comparison with the experimental ones. A cylindrical shape of the dots was assumed in both cases after we had verified that the slight taper of the dot shape has only a very small influence on the relaxation (<4%).

B. Analytical isotropic model

For a second analysis, we have adapted an analytical model developed for shrink fit problems of long solid circular cylinders by Barton.²⁴ This model assumes elastic isotropy and the small difference in the elastic constants of Si and $\text{Si}_{0.75}\text{Ge}_{0.25}$ is not accounted for. The boundary conditions are that the shear stress σ_{rz} vanishes at the sidewall and the normal stress σ_{rr} is equal to the misfit stress, which varies according to the chemical modulation along the growth direction.

The main advantages of this analytical model is that the stresses and strains can be determined quickly at any point and with any desired numerical accuracy without the necessity of solving large linear matrix equations. The main disadvantage is that the influence of the unetched material, which slightly hinders the elastic relaxation at the bottom of the dots, cannot be taken into account.

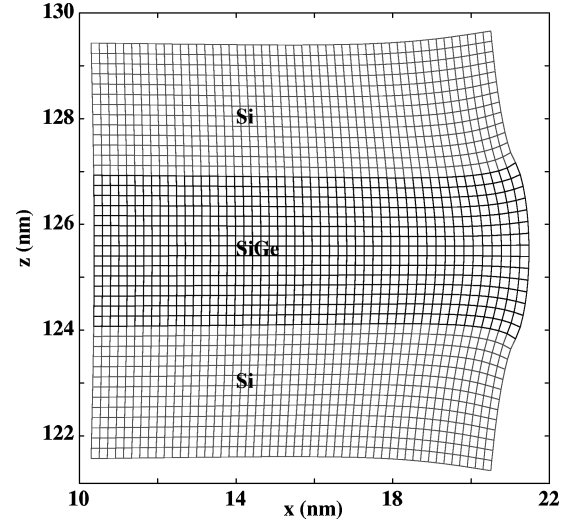


FIG. 9. Calculated displacement field (scale 20:1) of a SiGe layer in the region close to the sidewall of a relaxed 40 nm dot. The abscissa is the radial direction and the z axis corresponds to the superlattice growth direction.

We have used the values of Si for Young's modulus and the Poisson ratio: $E=130.25 \text{ GPa}$ and $\nu=0.279$. The average relaxation of the dot resulting from the analytical calculation is practically identical to the corresponding finite element curve [labeled (2) in Fig. 6]. The displacement field close to the sidewall of an elastically relaxed 40 nm dot is shown in Fig. 9. Only the displacements of one SiGe layer in the center of the dot and about 3 nm of the surrounding Si barriers are plotted. The ratio between position and plotted displacement is 1:20.

V. DISCUSSION

The compressive parallel strain $\epsilon_{\parallel}^{\text{SiGe}}$ in the as-grown alloy epilayer of the superlattice, as calculated from the lattice mismatch between the epilayer and the substrate, is

$$\epsilon_{\parallel}^{\text{SiGe}} = (a_{\text{Si}} - a_{\text{SiGe}}) / a_{\text{SiGe}}, \quad (1)$$

where a_{Si} is the Si substrate lattice constant and a_{SiGe} is the alloy bulk lattice constant. The bulk lattice constant as a function of alloy composition is²⁷

$$a_{\text{SiGe}}(x) = 0.5431 + 0.02x + 0.0026x^2 \quad (\text{nm}), \quad (2)$$

which for $x=0.25$ yields $a_{\text{SiGe}}=0.5483 \text{ nm}$. Hence the expected average parallel strain is $\epsilon_{\parallel}^{\text{SiGe}} = -0.009484$ in our as-grown superlattice. The Raman LO-phonon frequencies can also be used to estimate $\epsilon_{\parallel}^{\text{Si}}$ in the case of the Si layers from the relationship²⁷

$$\epsilon_{\parallel}^{\text{Si}} = -\delta\omega_{\text{Si}} / 715, \quad (3)$$

where $\delta\omega_{\text{Si}}$ is the frequency shift in wave numbers from the bulk Si frequency of 520.2 cm^{-1} . For the Si-Si line in the alloy layers $\epsilon_{\parallel}^{\text{SiGe}} = \delta\omega_{\text{Si-Si}} / b$,²⁷ where

$$b = -715 - 825x \quad (x < 0.35) \quad (4)$$

and hence

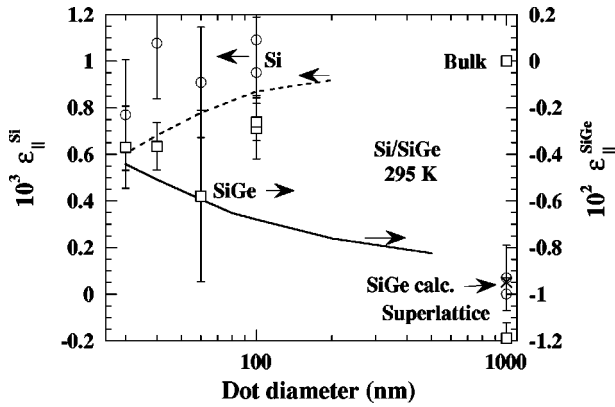


FIG. 10. In-plane strain $\varepsilon_{||}$ in the Si (\circ) and the SiGe (\square) alloy layers of the dots, as determined by Raman scattering. The equivalent superlattice and bulk frequencies are plotted at the right. The calculated $\varepsilon_{||}^{\text{SiGe}}$ for the superlattice is denoted by \times . The full (broken) line represents the parallel strain in the relaxed (strained) SiGe (Si) layers calculated with the analytical model.

$$\varepsilon_{||}^{\text{SiGe}} = -\delta\omega_{\text{Si-Si}}/921 \quad (5)$$

for $x=0.25$. The required bulk frequency value $\omega_{\text{Si-Si}}^0$ of the Si-Si line in a $\text{Si}_{0.75}\text{Ge}_{0.25}$ alloy was estimated from the data of Lockwood and Baribeau²⁷ to be 503.3 cm^{-1} . This agrees well with predictions based on more recent results for the concentration dependence obtained by Tsang *et al.*²⁹ and also those obtained earlier by Byra.³⁰

The experimentally determined average $\varepsilon_{||}$ in the Si and SiGe dot layers, based on Eqs. (3) and (5) and the frequency data of Fig. 8, are given in Fig. 10. For the SiGe layers the Raman value for $\varepsilon_{||}$ in the fully strained as-grown superlattice is a little higher than that predicted from Eq. (1), but the difference lies within the errors associated with determining b and $\delta\omega_{\text{Si-Si}}^0$ for $x=0.25$. The parallel strain in the dots is considerably reduced compared to the as-grown superlattice and, within errors, is independent of the dot size (up to 100 nm). On average, the strain in the alloy layer has relaxed by approximately 65% due to the fabrication of dots from the superlattice. In comparison, the Si layers in the dots have

become tensilely strained relative to the Si substrate, with $\varepsilon_{||}^{\text{Si}} \approx 0.001$ as compared with $\varepsilon_{||}^{\text{SiGe}} \approx -0.0035$. Thus an appreciable compensating strain is set up in the Si layers when alloy layer relaxation occurs during dot formation. Such an effect has been theoretically predicted,¹¹ but until now has been difficult to observe directly in such small dots.

The full line in Fig. 10 represents the strain in the SiGe layers calculated with the analytical model. It agrees well with the results obtained for 30, 40, and 60 nm dots within experimental error, but less so for the 100 nm dots. The calculated parallel strain in the Si layers is approximately 25% smaller than the Raman results.

The Raman results indicate that the dots are essentially fully relaxed (averaged over the whole dot) independently of dot diameters in the investigated range. This is in agreement with the continuum elastic model calculations for dot diameters up to about 200 nm (see Fig. 6). The elastic relaxation derived from the x-ray reciprocal space maps is systematically smaller, which is attributed to the averaging over the strain fields both in the dots and in the unetched material below.

VI. CONCLUSIONS

The elastic relaxation of dry etched Si/SiGe quantum dots has been investigated by means of both x-ray reciprocal space mapping and inelastic light scattering spectroscopy. Both techniques show a similar large and essentially size-independent relaxation for dot diameters up to 100 nm. The Raman results provide confirmation of earlier theoretical predictions of a compensating tensile strain induced in the Si layers by SiGe layer relaxation. The observed strain ratio of 1:−3 in the Si/SiGe layers is well reproduced by continuum-elastic model calculations.

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