Depth profile of strain and composition in Si/Ge dot multilayers by microscopic phonon Raman spectroscopy

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(Received 7 June 2005; accepted 1 November 2005; published online 9 December 2005)

We characterized strain and Ge content depending on depth in a self-assembled Si/Ge dot multilayer by scanning a microscopic Raman probe at a (110) cleavage plane. The multilayer structure was deposited by molecular-beam epitaxy on a (001) Si substrate and consisted of 80 periods, each of them composed by 25 nm Si spacers and 8 monolayer Ge forming laterally and vertically uncorrelated islands with a height of 2 nm and a lateral diameter of about 20 nm. An average biaxial strain of -3.5% within the core regions of islands is determined from the splitting of longitudinal and transversal optical Ge-Ge phonon modes observed in polarized Raman measurements. The absolute mode frequencies further enable analysis of a Ge content of 0.82. The analyzed strain and composition of islands are nearly independent from depths below the sample surface. This indicates well-controlled deposition parameters and negligible intermixing during deposition of subsequent layers. These Raman results are in agreement with x-ray diffraction data. Small, local Raman frequency shifts were observed and discussed with respect to partial elastic strain relaxation of the multilayer stack after cleavage, undefined Raman-scattering geometries at the sample edge, and local heating by the laser probe. © 2005 American Institute of Physics. [DOI: 10.1063/1.2140078]

I. INTRODUCTION

Because of the large lattice mismatch between Ge and Si, self-organized Ge dots can be fabricated using the Stranski-Krastanov growth mode.^{1,2} The growth conditions of Si/Ge dot layers severely affect the island formation process and the intermixing of Si and Ge. Thus size, shape, composition, and strain of Si/Ge islands are influenced by important parameters such as substrate temperature, Ge coverage, and the thickness of Si spacer layers. These structural characteristics determine the optical and electronic properties of Si/Ge dots.² Self-assembled Si/Ge nanostructures have been intensively studied for potential applications in Sibased integrated optoelectronics for many years. Many Ge dot layers separated by Si spacer layers are often deposited in multilayer structures for the purpose of increasing the optoelectronic response of such structures.³⁻⁶ Depth profiles of the structural island properties are very desirable in order to characterize the homogeneity of multilayers which may be severely disturbed by spatially correlated formation of islands caused by local strain fields penetrating the Si spacer layers up to the epitaxial growth front.^{2,7} High-resolution phonon Raman scattering is a nondestructive and fast tool to characterize the average structural properties of Si/Ge dot structures in backscattering geometry from the sample surface.^{3,4,8-13} In this paper, microscopic polarized Raman measurements were performed on a polished (110) cleavage plane of Si/Ge dot multilayers. Depth profiles of strain and

^{a)}Present address: State Key Laboratory for Superlattices and Microstructures, Institute of Semiconductors, Chinese Academy of Sciences, Beijing, 100083, P. R. China; electronic mail: pinghengtan@hotmail.com composition in Si/Ge dot multilayers are extracted from LO-TO phonon mode splittings while scanning the probe along the growth direction. The influence of extrinsic sample parameters on Raman-backscattering spectroscopy of dot multilayers on cleavage planes is also discussed here in detail.

II. EXPERIMENT

The sample was grown in a solid source molecular-beam epitaxy (MBE) system on (001) Si substrate at a substrate temperature of 520 °C. A Si buffer layer with a thickness of 150 nm was deposited, followed by 80 periods of 8 monolayer (ML) Ge dot layers separated by 25 nm Si layers and a 100-nm Si cap layer. The sample parameters were optimized with respect to a high density of homogeneous Ge dots and avoiding correlated island growth or defect formation. Atomic force microscopy of an uncapped reference structure reveals a lateral size of about 20 nm, a height of about 2 nm, and a sheet density of Si/Ge islands of about 1.5 $\times 10^{11}$ cm⁻². The average Ge content in the core of Si/Ge dots is as high as 0.82, corresponding to our previous Raman studies by backscattering from the (001) surface.¹⁰

Raman measurements were carried out using a triple Raman spectrometer equipped with a microscope scan setup and a liquid-nitrogen-cooled Si charge-coupled device camera (CCD). The 514.5-nm line of an argon-ion laser with about 1.2 mW power focused to a spot size of about 1 μ m was used for excitation. The labels *x*, *y*, *z*, *x'*, and *y'* used in the polarized Raman measurements refer to directions parallel to [1 0 0], [0 1 0], [0 0 1], [1 –1 0], and [1 1 0], respectively. The polarization of exciting and detected light could be changed independently. The *z*- or *x'*-polarized phonon modes are selectively probed by backscattering in $y'(x',x')\overline{y'}$ and $y'(x',z)\overline{y'}$ geometries, respectively.¹⁴ These

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FIG. 1. (a) Measured (004) Θ -2 Θ XRD scan of the Si/Ge dot multilayer sample along with multilayer simulation results using an average Ge content *x* and thickness *d* of Ge dot layers; (b) *x*=1.0, *d*=1.1 nm, and (c) *x*=0.54, *d*=2.0 nm.

two modes are assigned to LO and TO phonon modes, respectively, in this paper in order to be consistent to conventional Raman backscattering from the (001) surface. The sample was mounted on a piezodriven xyz scanner with 0.1 μ m step resolution. The Raman probe was scanned on a polished (110) plane along the [001] direction with a step size of 0.3 μ m.

III. RESULTS AND DISCUSSIONS

The Si/Ge dot multilayer structure was first characterized by high-resolution x-ray diffraction (XRD) measurements as shown in Fig. 1. Well-defined periodic multilayer satellite peaks up to the ninth order are identified, which indicate a very good periodicity and homogeneity of the Si/Ge multilayer. A slight broadening of observed XRD peaks may be due to incoherent scattering by local inhomogeneities in strain and composition that are caused by island formation and local elastic relaxation, but a significant correlation of dots in subsequent layers of the 80-period structure and a resulting drastic reduction of homogeneity can be excluded. From the angular spacing between neighboring multilayer reflection peaks and the one between the zerothorder layer and the Si substrate peaks we deduce a structural periodicity of 26.3 nm and an average vertical strain of about 0.26% relative to the bulk Si lattice parameter. These values correspond to a total thickness of the 80-period multilayer of 2.1 μ m and an average vertical strain of about 3.5% in a 2–nm-thick dot alloy layer. The lower two curves (b) and (c) in Fig. 1 depict the dynamical XRD simulations of a perfect pseudomorphic Si/SiGe layer structure using two sets of parameters: the nominal thickness of a pure 8-ML Ge layer (1.1 nm) and a SiGe alloy layer with a Ge content of 0.54 and a thickness of 2 nm given by the height of free-standing Si/Ge



FIG. 2. Raman spectra from (a) Ge-Ge and Ge-Si and (b) Si-Si LO phonons of the Si/Ge dot multilayer as a function of probe position z at the cleavage plane [depth underneath (001) sample surface]. $z=0.0 \ \mu m$ roughly corresponds to the sample surface. The two dashed gray curves in (a) and (b) are Raman spectra of TO phonons illustrating the LO-TO splitting at $z=0.9 \ \mu m$. The inset shows the linewidth of Si-Si modes vs probe position z. The vertical dashed lines indicate the bulk phonon frequencies of Ge and Si.

dots. Both simulation results are in excellent agreement with the experimental results. XRD thus shows a perfectly periodic 2.1- μ m-thick multilayer structure. The well-defined satellite peaks indicate high homogeneity in periodic layer compositions and strain throughout the 80 periods. However, considering small, flat Si/Ge dots assembled in a high areal density, simulations show that XRD is rather insensitive to the composition and effective thickness of a Si/Ge dot layer within one period of the multilayer structure. Structural information on several dot layers can be deduced from the following Raman phonon scattering study obtained by scanning along the growth direction on a (110) cleavage plane.

Figure 2 shows the Raman spectra of LO Ge-Ge, Ge-Si, and Si-Si modes obtained by scanning the microscopic probe to different depths below the surface. $z=0.0 \ \mu m$ roughly corresponds to the sample surface. In order to get the intrinsic Raman spectra of Ge dots, the spectrum of pure bulk Si has been subtracted from the measured spectra in Fig. 2(a). All the remnant Raman signals can then be assigned to the core regions within Si/Ge dots, as Ge wetting layers and Si spacer layers do not significantly contribute to the Ge-related modes of this multilayer structure characterized by a very high areal density of well-defined islands in each layer.¹⁰ The spectra showing the Si-Si mode are depicted in Fig. 2(b) as measured. They are mainly due to the Si spacer layers, the cap layer, or the substrate.¹⁰ Even allowing for some intermixing, the Si/Ge dots contribute negligibly to the Si-Si mode Raman signal, as the Ge content is still high¹⁰ and the integrated volume of dots is very small. The presence of a sharp Si-Ge mode originating from the core of Si/Ge dots¹⁰ indicates high Ge content in the dots. The Ge-Ge modes show a quite constant shift with respect to the measured Ge bulk value. Si-Si modes also are slightly shifted compared to bulk Si between 0.0 and 2.1 μ m, but in the opposite direction of the Ge-Ge shift. At each probe position, the TO pho-



FIG. 3. The frequency and intensity of LO and TO (a) Si-Si and (b) Ge-Ge modes in Si/Ge dot multilayers as a function of displacement position at the cleavage plane.

non spectrum was also measured. As an example, the TO phonon spectra at a depth of $z=0.9 \ \mu\text{m}$ are shown in dashed curves in Figs. 2(a) and 2(b).

The peak intensities of the Ge-Ge and Si-Si LO and TO modes shown in Fig. 2 are plotted versus probe position in Fig. 3. The Ge-Ge mode shows a nearly constant intensity between about 0.0 and 2.0 μ m. It decreases to about half the intensity at $z=2.1 \ \mu m$, which thus corresponds to the lower edge of the multilayer structure. The observed intensity behavior nicely fits the intensity profile calculated for a Raman probe of about 1 μ m diam. Surprisingly the Ge-Ge mode intensity stays rather high at $z \le 0 \mu m$. In fact, one can observe a plateau of about 2.5 μ m width (full width at half maximum), which is larger than the thickness of the multilayer determined by XRD. Also, the intensity of the Si-Si mode decreases with a weak oscillatory behavior at the sample surface edge. Both results suggest complex paths for exciting and scattered light close to the sample edge. Light might be reflected at or transmitted through the (001) sample surface, leading to complex Raman-scattering geometries or interference effects. This is more remarkable for the dot multilayer system than the usual Si/Ge superlattices, because of the lesser absorption of Raman signal in a dot layer than a pure Ge layer.

Figure 3 also shows the phonon frequencies of the Ge-Ge and Si-Si LO and TO modes from Fig. 2 versus probe position. A splitting of several cm⁻¹ is observed between LO and TO Ge-Ge phonon modes whereas it is quite small for Si-Si modes. Furthermore, the absolute frequencies of both LO and TO Si-Si phonons decrease significantly by about $0.5-1.0 \text{ cm}^{-1}$ within the Si/Ge dot multilayer region between z=0.0 and 2.1 μ m compared to surface and substrate regions. The LO-TO frequency splitting of Si-Si and Ge-Ge modes from Fig. 3 is summarized in Fig. 4(a). It shows a quite constant, large splitting of about 4.2 cm⁻¹ between LO and TO Ge-Ge phonon modes. This splitting is significantly reduced by up to 0.5 cm^{-1} close to the sample surface for $z \leq 0.3 \mu$ m. The splitting between the LO and TO Si-Si phonon modes, in contrast, is small with about -0.25 cm^{-1} .



FIG. 4. (a) The LO-TO splitting of Si-Si and Ge-Ge modes and (b) the lattice strain and Ge concentration within the core regions of Si/Ge dots dependent on the displacement position at the cleavage plane.

The observed splittings and shifts compared to the bulk values of Ge-Ge and Si-Si modes may originate in three intrinsic physical properties of the Si/Ge dot multilayer: the lattice strain, the composition, and the size of Si/Ge dots. Strain lifts the degeneracy of the optical phonons at momentum $q_z=0$. Thus the measurement of the LO-TO splitting of Ge-Ge and Si-Si modes through a (110) facet under different polarizations represents an attractive tool for probing a strain depth profile of the Si/Ge dot and Si spacer layers in a multilayer structure. For our structure we consider biaxial strain within the dot layers, which is caused by the difference in lattice parameter during coherent growth of Ge dots on a (001) Si substrate. The LO-TO splitting of the Si-Si mode is constant, small, and of opposite sign compared to Ge-Ge mode splitting. We consider extrinsic sample properties, which will be discussed later, to be responsible for this splitting. As a consequence, the Si spacer layers are unstrained. The biaxial strain has been confirmed to be valid in the spatially uncorrelated Si/Ge dot multilayers.¹⁰ In fact, the Raman analysis of Ge content is only little falsified by strain relaxation. For example, for about 8% lateral and 20% vertical relaxation, representing typical local values within small Si/Ge dots, our calculation shows that the Ge content will be only 5% higher than the original value under an assumption of the biaxial case, which is within the present experimental errors. Based on the above assumption, the frequency shift of the LO and TO Ge-Ge modes induced by biaxial strain within the (001) plane is then given by¹⁵

$$\delta\omega_{\rm LO} = \frac{1}{2\omega_0 C_{11}} (2qC_{11} - 2pC_{12})\varepsilon_{xx},\tag{1}$$

$$\delta\omega_{\rm TO} = \frac{1}{2\omega_0 C_{11}} [q(C_{11} - 2C_{12}) + pC_{11}]\varepsilon_{xx},\tag{2}$$

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$$\omega_{\rm LO} - \omega_{\rm TO} = \frac{1}{2\omega_0 C_{11}} (2C_{12} + C_{11})(q - p)\varepsilon_{xx},\tag{3}$$

where C_{11} and C_{12} are the stiffness coefficients, *p* and *q* are deformation potentials of the optical phonons in Ge, ω_0 is the optical-phonon frequency in bulk SiGe alloy, and ε_{xx} is the biaxial strain given by the lattice mismatch $(a_{Si}-a)/a$ with a_{Si} and *a* being the bulk Si and the (alloyed) Ge island lattice parameter, respectively. Using the parameters listed in Table I of Ref. 15, we get the following equation:

$$\omega_{\rm LO} - \omega_{\rm TO} = -122\varepsilon_{\rm xx} \,\,(\rm cm^{-1}). \tag{4}$$

The deduced biaxial strain within Si/Ge dot layers is shown in Fig. 4(b) versus probe position. The error bars correspond to an experimental uncertainty of ± 0.1 (± 0.2) and ± 0.2 (± 0.3) cm⁻¹ for determining the LO and TO mode frequencies in the center (edge) region of the multilayer structure, respectively. The analyzed biaxial compressive strain is constant at about ε_{xx} =-3.5% within the dot multilayer structure, but it appears to decrease to about -3.2% towards the sample surface $z \approx 0.0 \ \mu$ m. This variation close to the sample surface is small and within the error bars, but the trend is clearly visible.

Having determined the strain depth profile in Si/Ge dot layers through the LO-TO splitting one can now consider the shift of the Ge-Ge mode with respect to bulk material. Apart from the shift caused by biaxial compressive strain it reflects the influence of composition and dot size. In a Si/Ge dot structure with an effective dot height of about 2 nm the Ge-Ge LO and TO phonons are localized within the Ge-rich core regions of the dots. The resulting finite momentum q_z and the negative parabolic dispersion of optical phonons cause a reduction of both confined modes by about -1.0 cm^{-1} (Ref. 8). This constant frequency shift is taken into account for all Ge-Ge modes and does not affect the mode splitting. The residual shift with respect to the observed bulk Ge-Ge mode frequency should only be related to the composition of Si/Ge dots. The Ge content x_{Ge} within the core regions of Si/Ge dots is then given by the relation $\Delta \omega = -37(1-x_{\text{Ge}}) \text{ cm}^{-1}$ (Ref. 10). Figure 4(b) shows x_{Ge} versus probe position. The uncertainty is induced by the strain determination. In analogy to the strain depth profile, the concentration profile is very homogeneous around a mean value $x_{\text{Ge}} = 0.82$ apart from a slight increase near the sample surface.

The values of strain and Ge content observed within the multilayer region confirm a detailed Raman study in backscattering from the (001) surface, which only probed the approximate 15 topmost layers.¹⁰ By now scanning along a [001] axis, we observe intermixing in Si/Ge dot layers during growth, leading to a quite homogeneous Ge content x_{Ge} =0.82 in the core region of dots throughout the whole multilayer of 80 periods, corresponding to a total thickness of 2.1 μ m. Furthermore, the Ge content within Si/Ge dots of 0.82 corresponds to a biaxial compressive strain -3.5% within a simple model for biaxial strain that takes into account the lattice mismatch and neglects island formation and strain relaxation. This model applies to the studied islands, as they are small, rather flat, of very high areal density, and are coherent to the substrate without plastic relaxation. Thus (elastic) strain relaxation of the Ge dots is weak and nearly all the strain expected from the nominal thickness and the lattice mismatch of Ge layers contributes to the Raman Ge-Ge mode shifts, as well as to the XRD peak shifts discussed before. Within this model of pseudomorphic strained Si/Ge dot layers on a Si substrate, a fixed Si-Si mode frequency corresponding to bulk Si is expected for the unstrained Si spacer layers of the multilayer. But mode frequencies reduced by 0.5-1.0 cm⁻¹ and a LO-TO splitting of about -0.25 cm⁻¹ are observed. Probing the LO-TO splitting of the Ge-Ge mode during a scan along the growth axis thus proves to be a reliable tool to obtain strain depth profiles for periodic Si/Ge nanostructures. Information about nanostructure sizes or composition can then be deduced. However, this study also shows that one has to care about possible deviations due to extrinsic sample characteristics while applying this method on a cleaved plane of Si/Ge dot structures.

In the following we will address the unexpected behavior of the Si-Si mode and the reduction of LO-TO splitting of the Ge-Ge mode near the sample surface. Elastic strain relaxation of the Ge dots in the bulk of the sample should result in (average) compressive strain within the neighboring Si. This would cause an increase in Si-Si phonon frequencies and a positive LO-TO mode splitting that is limited to a small region of Si surrounding the dots, which is in contrast to the observed mode shift, splitting, and line broadening. Furthermore, the results described above and in an earlier Raman study with backscattering from the sample surface¹⁰ consistently indicate that strain relaxation of the Si/Ge dots is negligible. Thus, we attribute the unexpected behavior of both Si-Si and Ge-Ge modes to extrinsic sample properties. A shift of the observed phonon Raman frequency may have several origins beyond the main multilayer parameters, as there may be (1) unintended Ge alloying of Si layers in the multilayer, (2) uniaxial strain relaxation at the (110) cleavage plane, (3) local heating of the sample by the focused laser spot, and (4) instrumental errors in spectral accuracy caused by drift or inhomogeneous illumination of the spectrometer entrance slit. The latest was carefully avoided and checked by reference measurements, but small spectral shifts of typically $\pm 0.1 \text{ cm}^{-1}$, especially at the sample edge (z=0 μ m), cannot be fully excluded.

The reduced Si-Si LO and TO mode frequencies could in principle result from Ge alloying of the Si spacer layers by segregation or interdiffusion during MBE deposition. A frequency shift of about -0.6 cm^{-1} , however, would require a rather high Ge concentration within the spacer layers of about x=0.015. This corresponds to a large fraction of the deposited Ge material to be distributed homogeneously within the Si spacer layers. Furthermore, the tensile strain indicated by the observed LO-TO mode splitting is contradictory to alloyed pseudomorphic SiGe spacer layers. Thus, we do not expect significant contributions of Ge alloying to the observed Si-Si mode frequency reduction.

Another mechanism could be strain relaxation due to cleaving the sample in a (110) plane at which the Raman probe is scanned in depth. In general, the cleaved surface causes modified boundary conditions of a (110) plane free of

stress and results in local strain relaxation. Simple numerical finite element simulations of elastic relaxation at the sample edge show that a single strained Ge (dot) layer embedded in Si relaxes strain nearly completely at the cleavage plane in the [110] direction (along y'). The relaxed region extends into the neighboring Si, but its dimensions scale with the layer thickness and are in the nm range. This very local strain relaxation of each Ge layer after cleavage is not resolved by the Raman probe averaging within the volume given by the micron-sized probe diameter and the optical absorption length of about 0.7 μ m. Regarding the whole 2.1 μ m multilayer as a homogeneous SiGe layer with an average biaxial strain of about -0.17%, however, shows a similar uniaxial relaxation of strain in the y' direction that is around 60% within the multilayer and reaches degrees beyond 80% at the sample edge. The partial relaxation takes place at the cleaved surface of the whole multilayer and extends to depths and lateral regions in the Si substrate up to a few μ m apart. Relaxation along the x' direction is negligible. This relaxation results in tensile strain within the Si spacer layers that is roughly uniaxial in the y' direction $\varepsilon_{y'y'}=0.1\%$ and causes a Si-Si LO-TO splitting of about -0.3 cm⁻¹ (Ref. 16). This value agrees well with the observed Si-Si mode splitting, but the observed reduction of both LO and TO frequencies within the multilayer region is larger than expected for this strain.

Thus, additional origins for a frequency reduction of about -0.5 cm^{-1} for both modes in Si have to be discussed. A reasonable candidate is local heating of the probed sample region by the focused laser spot. Model calculations for a laser spot with 1 μ m diam, 1.2 mW power, and a penetration length of $\alpha^{-1}=0.7 \ \mu m$ reveal a Gaussian-like temperature profile in bulk Si with a maximum rise of about 4 K at the spot center.¹⁷ Recent studies of the anisotropic thermal conductivity of Si/Ge layer and dot multilayer structures show that the in-plane thermal conductance of a similar dot multilayer is more than five times lower than the bulk Si value of 156 W/mK and the vertical conductivity across the interfaces is even lower.¹⁸ Thus, a local temperature rise up to typically 20 K may be expected for scanning the Raman probe from the Si substrate into the multilayer region. This enhanced temperature is calculated to cause a frequency reduction of all Si Raman modes of about -0.35 cm⁻¹ and an increase of Raman linewidth of typically 0.3 cm⁻¹ by homogeneous and inhomogeneous broadening.¹⁹ Both values are in reasonable agreement with the residual frequency shift and the line broadening observed for the Si modes in the multilayer region (see inset of Fig. 2). Laser heating appears to affect the Raman results even at such low laser power if thermal conductivity is low and position dependent, as in the studied Si/Ge multilayer stack. A laser power of about 1 mW, however, is required in order to ensure an accurate determination of Raman mode frequencies without severe noise contributions.

We already discussed the relaxation of strain at the cleavage plane and its effect on Si spacer layers. This relaxation will also reduce compressive strain within the Ge islands at the same extent due to the coherence of the epitaxial structure. The expected change in Ge-Ge mode splitting of about 0.15 cm^{-1} , however, is much smaller than the shifts caused by the predominant biaxial compression and by alloying. The resulting small corrections in analyzed strain and Ge content are about 0.2% and 0.01, respectively. Thus strain relaxation at the cleavage plane has only little influence on the properties deduced from Raman results of our multilayer, but it may considerably affect the Raman analysis in thick multilayer structures with a smaller ratio of Ge and Si layer widths.

Finally, we discuss the significant reduction of LO-TO splitting of Ge-Ge modes up to -0.5 cm^{-1} that was observed close to the sample surface around $z=0.0 \ \mu\text{m}$. This reduction is larger than calculated even for full relaxation of the Si/Ge multilayer to the average lattice constant in the y' direction ($\varepsilon_{y'y'}=0.17\%$). As laser heating should not affect the LO-TO splitting, it is tentatively explained by complex geometries and buckled Raman-scattering paths possible at the sample edge. Besides the plateau broadening and the oscillatory behavior in the depth profile of Raman intensity observed in Fig. 3, they may also result in relaxed Raman polarization selection rules and much higher Ge-Ge mode intensity at $z < 0 \ \mu\text{m}$, and may reduce the observed mode splitting by inhomogeneous mixing of LO and TO modes.

IV. CONCLUSIONS

In summary, we studied a $2.1-\mu$ m-thick 80-period multilayer structure of self-assembled Si/Ge dots by phonon Raman backscattering at a cleaved (110) plane. An average biaxial strain ε_{xx} =-3.5% and a mean Ge content x=0.82 within the core regions of Si/Ge dots are determined from the splitting and the frequency of the longitudinal and transversal optical Ge-Ge phonons. Scans of the Raman probe as well as x-ray diffraction results confirm constant biaxial strain and composition throughout the structure and exclude structural correlation between dot layers. Our results show that polarized Raman spectroscopy on a cleavage plane can be effectively used to characterize the spatial distribution of strain and composition of semiconductor dot multilayers along the growth direction. We also point out that one has to be aware of small deviations induced by unavoidable extrinsic sample properties, such as strain relaxation after cleavage, relaxed Raman polarization selection rules at the sample edge, and position-dependent local heating by the laser probe due to very inhomogeneous thermal conductances in Si/Ge nanostructures.

ACKNOWLEDGMENT

This work was financially supported by the DFG under Grant No. BR1960/1-4.

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