Optical-geometrical effects on the photoluminescence spectra of Si nanocrystals embedded in SiO₂

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We demonstrate that thickness, optical constants, and details of the multilayer stack, together with the detection setting, strongly influence the photoluminescence spectra of Si nanocrystals embedded in SiO₂. Due to multiple reflections of the visible light against the opaque silicon substrate, an interference pattern is built inside the oxide layer, which is responsible for the modifications in the measured spectra. This interference effect is complicated by the depth dependence of (i) the intensity of the excitation laser and (ii) the concentration of the emitting nanocrystals. These variations can give rise to apparent features in the recorded spectra, such as peak shifts, satellite shoulders, and even splittings, which can be mistaken as intrinsic material features. Thus, they can give rise to an erroneous attribution of optical bands or estimate of the average particle size, while they are only optical-geometrical artifacts. We have analyzed these effects as a function of material composition (Si excess fraction) and thickness, and also evaluated how the geometry of the detection setup affects the measurements. To correct the experimental photoluminescence spectra and extract the true spectral shape of the emission from Si nanocrystals, we have developed an algorithm based on a modulation function, which depends on both the multilayer sequence and the experimental configuration. This procedure can be easily extended to other heterogeneous systems. © 2005 American Institute of Physics. [DOI: 10.1063/1.2115100]

I. INTRODUCTION

Si nanocrystals (Si-ncs) in a silicon oxide matrix are attractive for optoelectronics and photonics as a consequence of their visible and tunable photoluminescence (PL) and electroluminescence (EL), optical gain, and nonlinear optical properties. The emission bands of these Si–SiO₂ composites have been studied as a function of silicon excess for materials obtained from different fabrication methods. The emission band coming from Si nanocrystals embedded in SiO₂ appears after Si excess precipitation into nanocrystals during a high-temperature annealing and it peaks between 1.4 and 1.7 eV. During nucleation and growth of the Si-ncs, their emission intensity increases with annealing time, until a saturation regime is reached when most of the Si excess has precipitated and interfacial defects have been annealed out.

Several authors have reported shifts of the emission peaks, appearance of satellite bands, and even the splitting of the main emission band as a consequence of material variations such as nanocrystal size and density. Shifting shoulders around the main band have been attributed to intrinsic properties such as the existence of optically active defects. Together with specific structural properties, these PL spectra modifications have been used to tentatively validate the mechanisms of emission based on quantum confinement, interfacial radiative defects, or others. The correlation of the PL spectra with material structure and emission mechanism which has been provided by many authors is usually self-consistent, especially when they compare samples with similar thickness and fabricated in the same experimental conditions. However, comparisons between the results obtained by different groups are always difficult because experimental values of peak energies, widths, and intensities of spectral bands are scattered over a wide range. Moreover, several research groups have tentatively tried to reproduce the observed position and shape of the PL band from the measured spread in size distribution. For this purpose they made use of theoretical calculations of the variation of the electronic band gap of the nanocrystalline silicon with the particle size, based on the quantum confinement model.

On the other hand, only scarce information is available in the literature about how the PL spectra can be dramatically influenced by optical-geometrical features such as layer thickness, refractive index, multilayer sequence and geometry of the excitation, and collection setups. These effects arise as a consequence of the optical response of the layered system, considering for the interference patterns both the profile of the excitation radiation and the depth distribution of the emitting Si-ncs. Oscillation patterns in the emission spectra of Si–SiO₂ samples grown on a Si substrate and their absence in identical samples formed on a silica substrate have been mentioned by Iwayama et al. However, they do not provide with a dedicated study of this phenomenon. Nesheva et al. have pointed out that oxides thinner than 0.2 μm must be chosen to avoid interference effects. Orbons et al. have reported the distortion of the PL spectra as a function of sample thickness only for a single Si ion implantation in...
II. EXPERIMENT

A 300-nm-thick buried layer with uniform Si excess in SiO$_2$ has been obtained by performing Si$^+$/multi-implantation into two different substrates: 2-mm-thick fused silica (FS) glass or 2-$\mu$m-thick SiO$_2$ thermally grown on Si thermal oxide (TO) wafers. TRIM simulations have provided the correct doses and energies of implantation. Three samples for each type of substrate have been synthesized with different Si excesses (from 7% up to 13%). The phase separation and precipitation of Si-ncs have been attained by annealing the samples at 1100$^\circ$C for 4 h. As we have previously demonstrated, this treatment ensures the precipitation of most of the Si excesses.$^7$ X-ray photoelectron spectroscopy (XPS) measurements have been carried out to estimate the actual Si excess and its in-depth profile. Transmission electron microscopy (TEM) has been used to image the Si-ncs and check the uniformity of the size distribution.

Photoluminescence measurements have been performed by using for excitation a 30 mW He–Cd laser emitting at 325 nm. In order to vary the incident and collection angles, the sample has been positioned onto a goniometer placed in the focus of a spherical mirror, which collects the light emitted from the sample and focuses it at the entrance of a 0.5 m monochromator. A GaAs photomultiplier tube has been used as a detector and standard chopper/lock-in synchronous detection has been applied to minimize the noise. Great care has been taken in measuring and correcting the spectral response of the whole detection system. Figure 1 shows the schematics of the experimental setup. One of the critical parameters to control is the detection angle $\theta$ (see Fig. 1 for details). Since the spherical mirror used for collecting the emitted light has an aperture of about 16°, which sets the angle resolution of the system, the value of $\theta$ has to be taken as an averaged value. The influence of this aperture on the experimental spectra has been checked out and consists of a small increase in the width of the emission band, while the position of the maximum does not show any variation.

III. PHYSICAL AND OPTICAL CHARACTERIZATIONS

Figure 2 shows a typical XPS profile which can be briefly described as a central flat region with thickness $d_x$ = 300 nm of Si excess starting at a depth $d_0$ of about 50 nm. For each implanted dose the actual Si excesses measured are 7%, 9.5%, and 13%, respectively. A dark-field TEM picture of the sample with 13% of Si excess is shown in Fig. 3. Si nanocrystal size varies between 3.6 nm for the 7% Si excess and 4.6 nm for the 13% Si excess, as indicated in Table I. Within the 300 nm region of the flat profile, size distribution is independent of depth. By assuming a perfect phase separation and by combining Si excess and size measurements, we have estimated a density of about 4 $\times$ 10$^{18}$ Si-nc/cm$^3$, which appears roughly independent of the Si excess. All these data will be used in the following to perform the simulations. Exhaustive details of these measurements and additional secondary-ion-mass spectroscopy (SIMS) and Rutherford backscattering spectroscopy (RBS) experiments on the same samples are provided elsewhere.$^{19}$

Figure 4 shows the modification of the PL spectrum when varying the Si excess in thermal oxide and fused silica samples, respectively, for a quasinormal incidence/detection setting. The intensities have been normalized to magnify the shift as a function of Si excess. Even if the following discussion will be focused mainly just on the spectral features, in general a stronger emission has been observed from the...
samples grown on a silicon substrate. The Si substrate reflects backwards the weakly absorbed excitation radiation, enhancing the power density available for the excitation of the nanocrystal ensemble and resulting in a PL emission efficiency up to the double of the corresponding layer buried in fused silica. In Fig. 4 it is apparent that for increasing Si excess, i.e., Si grain size, the peak energy position of the FS spectra redshifts, in agreement with the quantum confinement theory. It is worth to stress that these spectra show no distortion coming from interference effects in the layers. The redshifts are much more reduced in the PL spectra of the corresponding TO samples, which show distortions and a pronounced shoulder in the high-energy side of the spectra, for this particular chosen experimental configuration. Moreover, contrary to the TO spectra, emission bands from FS samples can almost perfectly be fitted by a Gaussian shape for this particular chosen experimental configuration. More- over, for the corresponding TO sample. The shape spectacularly changes with the detection angle: the peak maximum shifts, for the corresponding TO sample. The shape spectacularly changes with the detection angle: the peak maximum shifts, for the corresponding TO sample. The shape spectacularly changes with the detection angle: the peak maximum shifts, for the corresponding TO sample. The shape spectacularly changes with the detection angle: the peak maximum shifts, for the corresponding TO sample. The shape spectacularly changes with the detection angle: the peak maximum shifts, for the corresponding TO sample. The shape spectacularly changes with the detection angle: the peak maximum shifts, for the corresponding TO sample. 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can conclude that the origin of this phenomenon is the interferences produced within the SiO$_2$ layer with thickness $d_1$, grown onto silicon and containing the sublayer with the embedded nanocrystals.

**IV. SIMULATION OF OPTICAL MODULATION EFFECTS ON SI-NC PL SPECTRA**

We have been able to simulate the effect of interferences and then reproduce the PL spectra of Si-ncs in thermal oxide layers, by taking as a reference the spectra of Si-ncs in the corresponding silica samples, which show no interferences. These simulations provide a correction to the modulation superimposed to the spectra and thus allow obtaining of the actual spectra of Si-nc without distortions.

We have represented in Fig. 6 a sketch of the structure of the TO samples, for which we have developed the calculations. Starting from the outer surface, it contains a 50-nm-thick SiO$_2$ cladding layer, a 300-nm-thick Si-nc-rich SiO$_2$ layer, and a 1650-nm-thick SiO$_2$ buried layer on a Si substrate. This sequence well reproduces the structural characteristics of these samples, as obtained by the aforementioned characterization. The modeling of the PL emission has been performed in two steps: (i) calculating the continuous laser excitation and the profile of the related electromagnetic field inside the multilayered structure and (ii) the emission pattern, which depends on the excitation intensity and the location of the emitting Si-ncs.

![Layer sequence used for the calculation of the resulting PL emission and the multiple reflections within the structure.](image)

**FIG. 6.** Layer sequence used for the calculation of the resulting PL emission and the multiple reflections within the structure.

In the first step, for the optical study of excitation in multilayered materials we have used the approach of Harbecke,$^{20}$ which we have extensively developed and previously used in the study of vibrational spectra of multilayered systems.$^{21}$ This method allows the calculation of the complex-amplitude reflection and transmission coefficients ($r$ and $t$) for a stack of planar absorbent layers. The transformation of fields across the interface between two layers and the propagation inside each layer are obtained from the well-known matrix formalism developed by Abeles and Gittleman.$^{22}$ One of the main features of this procedure is its ability to disregard the extremely narrow interference fringes of thick layers, then considering an incoherent propagation of light in the thick silica substrate. We have adapted the simulator to this particular work to obtain the amplitude of the excitation electric field as a function of depth $z$,

$$a(z) = \frac{t_{ag}}{1 - r_{gb}r_{gb}^*} \{ \varphi_g(z) + r_{gb} \varphi_b(d_g - z) \},$$  

(2)

where $d_g$ is the thickness of layer $g$, and $\varphi$ stands for the propagation factor forward and backward within the active layer. The subindex of $r$ and $t$ indicates the transmission or reflection, respectively, of the whole stack from the top layer (subindex $a$) to the active layer ($g$) and from the active layer to the buried SiO$_2$ (subindex $b$). The angular dependence with $\theta$ is included in the coefficients $r$ and $t$.

**FIG. 7.** Amplitude of the electric field of the excitation radiation (325 nm) along the oxide multilayer structure, as calculated by the simulator. The pattern has been normalized to the incident field.

FIG. 5. Photoluminescence spectra from fused silica and thermal oxide samples with a 7% silicon excess for different detection angles.
layer \((d_e \leq \lambda/2n_0)\) or would present a very irregular density profile, then the excitation intensity across it would show a dramatic dependence on the excitation wavelength, thickness, and refractive index of the active layer and cladding or substrate.

The simulator must be fed with the optical constants of all the layers at the excitation wavelength. The refractive index \(n_{\text{nc}}\) of the layer consisting of Si-ncs embedded in SiO\(_2\) has been obtained from spectroscopic ellipsometry and lines and modeled using the Bruggeman effective-medium approximation. A detailed study concerning the complex refractive index of Si-ncs is not among the scopes of this paper and has been previously published by several authors. With only the Si excess volume fraction as input, the complex refractive index of a Si–SiO\(_2\) system can be easily calculated. Reflective index and extinction coefficient at the excitation wavelength (325 nm) and emitting wavelength (700 nm) are given in Table II for the three Si excesses studied here.

As far as the emission from the Si-nc ensemble is concerned, the light generated at each depth \(z\) within the active layer is considered, giving rise to forward and backward propagating waves and their interference patterns. The results of the calculation provide a modulation function, which has to be superimposed to the actual Gaussian spectra of the Si-ncs (that of the FS samples) to reproduce the experimental spectra for any given situation. This modulation function depends on the optical constants, oxide thickness, Si-nc region thickness, Si excess, incident and detection angles, and other geometrical parameters. For the calculation of the modulation functions, we have taken the complex refractive indices given in Table II for the composite layers in the 700–900 nm range, where the Si-ncs emit. It is worth to point out that throughout this range the variation of the refractive index (less than 0.01%) and the absolute value of the extinction coefficient (less than 0.002) of Si-ncs are negligible.

The basic procedure followed in this second step is described briefly hereafter. We have considered that a plane at depth \(z\) emits in all directions (see Fig. 6). Only reflections between the air-oxide and oxide-silicon substrate interfaces are considered, because the contrast of the refractive index between Si-nc-rich layer and pure SiO\(_2\) is low enough to consider that light suffers little deviation when crossing the interface among these two media. For a specific observation angle, two contributions to the final intensity are present, noted as the forward and the backward one, respectively. For each of them the geometric sum has to be made for all the succeeding reflections, taking into account the reflection and transmission coefficients and the phase-delay \(\delta(z)\) = \(4\pi n_1 \cos(\varepsilon')/\lambda\), where \(n_1\) is an average value of the refractive index over the whole SiO\(_2\) stack, \(n_1[d_p n_{\text{nc}}+(d_1-d_p)n_0]/d_1\), while \(\varepsilon'\) is obtained from the Snell law, \(n_0 \sin(\varepsilon')=\sin(\varepsilon)\). The complex coefficient is calculated and then the intensity factor, is integrated over the whole Si-nc emitting layer, considering that the emission of each \(dz\) is incoherent with all the others. For nonpolarized light, the modulation function \(M(E)\) is the average of both \(s\) (parallel polarization) and \(p\) (perpendicular polarization) waves. Then, the PL spectra can be written as the product of the modulation function and the Gaussian emission shape (centered at energy \(E_c\) and with full width \(w\)), which has been independently determined in FS samples,

\[
I_{\text{PL}}(E) = \frac{1}{w \sqrt{\pi/2}} e^{-2(E-E_c)^2/w} M(E),
\]
and the modulation function is given by

\[
M(E) = \int_{d_0}^{d_p+d_e} \frac{d^2(z)}{1+r_s^2+2r_s \sin \delta (d_1-z)} dz,
\]
where \(a(z)\) is the excitation profile amplitude calculated in Eq. (2), \(t_0\) and \(r_0\) are the transmission and reflection coefficients of the air-oxide interface, \(r_s\) is the reflection coefficient of the oxide-substrate interface, and \(\delta\) are the phase delays of the forward and backward contributions of the emission originated at depth \(z\), which include the dependence on the collection angle \(\varepsilon\).

V. SIMULATION RESULTS AND DISCUSSION

We have taken the emission shape of the samples synthesized in fused silica as the actual PL of the Si-ncs, free of any distortion. The Gaussian fitting following Eq. (2) gives \(E_c=1.571 \pm 0.001\) eV and \(w=0.248 \pm 0.001\) eV for the emission spectrum of FS sample with 7% of Si excess. This spectrum is the one already shown on top of Fig. 5. By calculating the modulation function for the corresponding TO sample and multiplying the result by the Gaussian fit, we have been able to reproduce with high accuracy its measured emission spectrum as a function of the detection angle, without making use of any free parameter (apart from an intensity normalization factor). The modulation function \(M(E)\) is shown in Fig. 8 for a normal incidence/detection configura-
tion and the results of the simulation for this sample are shown in Fig. 9, superimposed to the experimental spectra for detection angles of 6°, 26°, 36°, and 46°.

Figure 9 demonstrates that it is possible to generate the spectral shape of the PL emission for any multilayer arrangement or experimental configuration by using the procedure stated above and starting with the undistorted spectrum of the Si-ncs. This demonstrates that many shifts, shoulders, and splits found in the emission spectra of Si-ncs can be only optical-geometrical artifacts. Ignoring these effects can give rise to an erroneous attribution of bands and average size determination of the Si-ncs. The explained procedure can be summarized as

$$I_{PL}^{exp}(E) = M(E)I_{PL}^{Si-nc}(E),$$

where $I_{PL}^{Si-nc}(E)$ indicates the undistorted PL emission from Si-ncs. The procedure can be easily reversed and then, once the modulation function $M(E)$ has been calculated, it is straightforward to obtain the “neat” spectrum from the experimental one regardless of the multilayer configuration or experimental arrangement.

Interferences and spectral distortions such as those shown above have been observed in other thermal oxide samples with different Si excesses, active layer thicknesses, and oxide thicknesses. Distortions appear at any detection angle, even at normal incidence, and this implies that one has to routinely correct the spectrum to get the true spectral shape of the Si-nc emission. Distortions would be significant and more detrimental when the thickness of the sample is such to give interferences with maxima separation of the same order of magnitude than the width of the emission band. Interferences and distortions would remain unnoticed for very thin oxides (<0.3 μm) and for very thick ones (>200 μm).

To demonstrate further the impact of distortions on the aforementioned PL band shifts, and the large errors which can arise by not taking into account these effects, we have used the simulator to predict the apparent, so-called “experimental,” peak position of the PL emission from samples with different Si excesses, measured at normal incidence. Two different oxide thicknesses have been considered for simulation, 730 nm for a mild distortion and 2 μm, as before, for a relatively strong one. As shown in Fig. 10, there is a very good agreement between the predicted values and the ones found experimentally, in the case of the thick SiO₂ layers. More interestingly, the simulation shows that just by changing some experimental conditions, in this case the thickness of the SiO₂ layer underneath the region enriched with Si nanocrystals, dramatic changes in the variation of the peak position take place: From a PL band which looks rather insensitive to the size of the Si-nc population (<2 μm TO layer), up to an overestimation of the redshift (730-nm-thick TO layer) with respect to the true behavior, as measured in the silica samples.

VI. CONCLUSIONS

We have demonstrated by experiments and simulations that optical-geometrical effects can be at the origin of pronounced shifts, spectral distortions, and even band splitting of the emission of Si-ncs embedded in SiO₂. Ignoring these effects can give rise to an erroneous attribution of bands and average size determination of the Si-ncs. We have also developed a procedure to calculate a modulation function which depends upon the multilayer configuration and the experimental arrangement and can be used to correct the experimental photoluminescence spectra and get the true peak position and emission shape of the Si-ncs. The simulations have been compared with experimental data and reproduce the spectral shifts and distortions that are observed when changing the thickness of the samples, the Si excess, and the detection angle. Only Si-ncs embedded in either thick
samples, such as fused silica substrates or very thin oxides (less than 300 nm), are free from spectral distortions due to interferences.

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