Nonlinear optical properties of Si nanocrystals embedded in SiO_2 prepared by a cosputtering method

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Nonlinear optical properties of Si nanocrystals (Si-ncs) doped SiO_2 prepared by a cosputtering method were studied by *z*-scan technique in a femtosecond regime at around 1.6 eV. The nonlinear refractive index (n_2) and nonlinear absorption coefficient (β) were strongly enhanced compared to those of bulk Si and found to be about $\sim 2 \times 10^{-13}$ cm^2/W and ~ 0.8 cm/GW, respectively. In the photon energy region from 1.48 to 1.65 eV, the n_2 and β spectra followed the absorption spectra and no enhancement was observed in the band-edge photoluminescence region. In the diameter range of 2.7–5.4 nm, the size dependence of n_2 coincided well with that calculated by a pseudopotential approach, suggesting that the discrete energy states of Si-ncs are responsible for the observed enhanced optical nonlinearity. © *2009 American Institute of Physics*. [DOI: 10.1063/1.3125446]

I. INTRODUCTION

Silicon nanocrystal (Si-nc) is a topic of great interest in view of potential application in the field of optoelectronics.[1,2] The most interesting feature of this material is the strong photoluminescence (PL). Motivated by the potential for Si based light emitting devices, the origin of the PL has extensively been studied and often assigned to spatially confined carriers within Si-ncs.[3–5] To clarify the energy band structure and optical responses of Si-ncs, detailed theoretical calculations have been performed;[5–8] it has been shown that, in addition to the size, the surface passivation[8] and the shape distortion[7] significantly affect the band-gap energy of Si-ncs. Si-nc is also a promising material toward the application of optical switching devices. Nonlinear optical refractive index (n_2) of Si-ncs has been reported to be enhanced more than two orders of magnitudes than that of bulk Si in a relatively transparent energy range.[9–12] The elucidation of the origin of the large nonlinear optical response of Si-ncs has been attracting a great interest today.

There have been many reports on nonlinear optical response of various forms of Si-ncs. For example, a strong enhancement of the nonlinear optical response has been observed in chemically etched porous Si,[13,14] Si-ncs doped SiO_xN_y deposited by plasma enhanced chemical vapor deposition (PECVD),[15,16] Si-ncs doped SiO_2 prepared by ion implantation,[17] laser ablated Si-ncs deposited on quarts substrate,[18,19] Si-ncs doped SiO_2 prepared by a sol-gel method,[20] and so on. The nonlinear optical properties of Si-ncs depend strongly on the preparation method. The reported values of n_2 varied from $\sim 10^{-7}$ to $\sim 10^{-13}$ cm^2/W, and both positive and negative n_2 were reported for various forms of Si-ncs.[13–20] This implies that the origin of the enhancement may differ depending on the preparation methods and conditions. Therefore, further systematic studies for well-defined Si-nc samples are needed to elucidate the mechanism of large optical nonlinearity.

In this work, the nonlinear optical response of Si-ncs doped SiO_2 is studied for samples prepared by a conventional cosputtering method. Our samples are well-defined Si-ncs-doped SiO_2, where the simple quantum size effect can explain its linear optical properties. For example, they show strong PL above the bandgap of bulk Si (at around 1.4 eV). The origin of the PL is the band edge luminescence due to the recombination of quantum confined electron-hole pairs within Si-ncs, evidenced by the resonantly excited PL spectra,[21] the temperature dependence of the PL lifetime,[4] and resonant quenching of PL bands by energy transfer to nearby materials.[22] Since the quantum efficiency of the PL is rather high,[23] the PL peak energy well coincides with the theoretically predicated bandgap energy of Si-ncs.[4] The Si-nc samples are thus considered to be suitable for the systematic study to elucidate the mechanism of the nonlinear optical response of Si-ncs. In this paper, the size and photon-energy dependence of the nonlinear optical response of Si-ncs doped SiO_2 are discussed. These studies lead us to better understanding of the origin of the enhanced optical nonlinearity of Si-ncs.

II. EXPERIMENTAL PROCEDURE

Si-ncs doped SiO_2 was prepared by a cosputtering method. Si and SiO_2 were simultaneously sputter deposited in Ar gas on a quartz substrate and then the deposited SiO_x films were annealed in a N_2 gas (99.999%) atmosphere for 30 min above 1100 °C to grow nanocrystals in the films. In this method, the size of Si-ncs can be controlled by changing the concentration of Si in the films or changing the annealing temperature (Ta). The size of Si-ncs was estimated by cross-sectional transmission electron microscopic observations. The average diameter (D) was from 2.7 to 5.5 nm. The concentration of excess Si($C_{ex\ Si}$) ranges from 3 to 11 vol %, estimated by electron probe microscope analysis. The thick-

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ness of the samples was estimated by a physical-contact-type surface roughness measurement and to be about 8 μm. PL spectra were measured by using a single grating monochromator and an InGaAs near-infrared diode array. The spectral response of the detection system was calibrated with the aid of a reference spectrum of a standard tungsten lamp.

For the measurement of the nonlinear optical properties of Si-ncs, a z-scan method was used. Details of the z-scan method is found elsewhere.[24] Briefly, in the z-scan method, the tight focusing Gaussian beam is vertically irradiated onto a sample and the sample is moved along the direction of the beam propagation (z-axis). The transmitted light intensity is recorded as a function of the distance from the focal point (z). When whole transmitted light is detected (open aperture), the transmittance $[T_{\mathrm{op}}(z)]$ is determined by the nonlinear absorption coefficient (β), and its dependence on z is

$$T_{\mathrm{op}}(z) = 1 + \frac{\beta I_0 L}{1 + (z/z_0)^2}, \tag{1}$$

where I_0, L, and z_0 are the peak intensity of the beam, sample thickness, and the diffraction length of the beam, respectively. Note that T_{op} does not depend on the nonlinear refractive index (n_2) but does only on β, thus open aperture measurement provides the information on β.

When a small aperture is placed in front of the detector to cut peripheral region of the transmitted light (closed aperture), the transmittance (T_{cl}) depends both on n_2 and β. The information on n_2 is extracted by the division of T_{cl} by T_{op},

$$T_{\mathrm{cl}}/T_{\mathrm{op}}(z) = 1 + \frac{4\Delta\phi}{[(z/z_0)^2 + 9][(z/z_0)^2 + 1]}, \tag{2}$$

where $\Delta\phi$ is the nonlinear phase change. n_2 is obtained from $\Delta\phi$ as,

$$n_2 = \frac{\lambda\alpha\Delta\phi}{2\pi I_0(1 - e^{-\alpha L})}, \tag{3}$$

where α and λ are the linear absorption coefficient and the wavelength of the beam, respectively.

For the Gaussian beam, we used the mode-locked Ti-:sapphire femtosecond laser with the pulse width of 70 fs and the repetition frequency of 82 MHz. The photon energy was changed from 1.48 to 1.65 eV, which overlaps PL emission energy bands of Si-ncs. The incident beam was focused on a sample by a lens with the focus length of 100 mm, and the beam waist and diffraction length determined by a knife edge method were 18 μm and 1.1 mm, respectively. The peak intensity of the beam was typically 10 GW/cm^2. No notable change of nonlinear optical properties was observed in the intensity range of 0.5–20 GW/cm^2, suggesting that thermal effect is negligible in this measurement condition.[15,16] The validity of the obtained data was checked by measuring a fused quarts plate as a reference.

III. RESULTS AND DISCUSSION

Figure 1 shows PL (left-hand axis) and absorption spectra (right-hand axis) of a typical sample ($C_{\mathrm{ex\ Si}}$=3.3 vol %, Ta=1100 °C, D=2.7 nm). A broad PL peak at around 1.4 eV is assigned to the recombination of electron-hole pairs within Si-ncs. The optical bandgap (E_g) estimated from the absorption spectrum by using the $\sqrt{\alpha h\nu} \propto (h\nu - E_g)$ relation is around 2.4 eV. Therefore, in the energy range investigated in this paper (at around 1.6 eV), the samples are highly transparent.

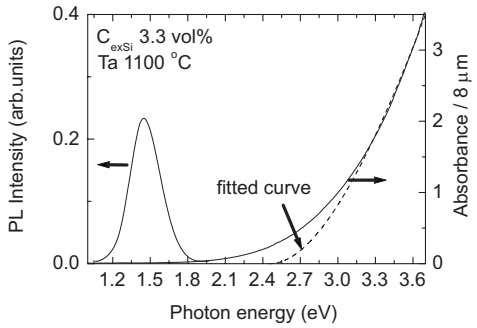

FIG. 1. PL (left axis) and absorption (right axis) spectra of a typical sample ($C_{\mathrm{ex\ Si}}$=3.3 vol %, Ta=1100 °C, D=2.7 nm). The dashed line is a result of fitting by assuming an indirect bandgap semiconductor $[\sqrt{\alpha h\nu} \propto (h\nu - E_g)]$. The optical bandgap is estimated to be 2.4 eV.

Figure 2 shows typical results of z-scan measurements for (a) a closed aperture (T_{cl}), (b) an open aperture (T_{op}), and (c) the ratio ($T_{\mathrm{cl}}/T_{\mathrm{op}}$). Open squares and solid curves represent experimental data and fitted results, respectively. In Figs. 2(b) and 2(c), Eqs. (1) and (2), respectively, are used for the fittings. The solid curve in Fig. 2(a) is generated by using the parameters obtained by the fittings of Figs. 2(b) and 2(c). The agreement between the experimental data and the fitted curves is very good and the diffraction length estimated from the fitting well coincides with those measured by a knife edge method. The clear symmetrical valley to peak

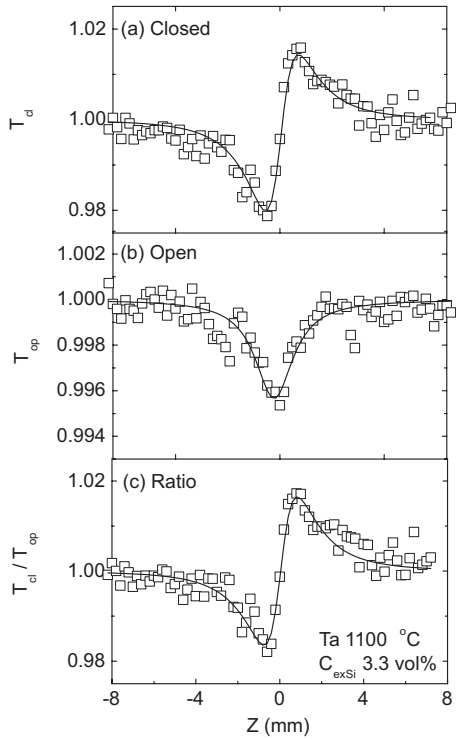

FIG. 2. Results of z-scan measurements for (a) a closed aperture (T_{cl}), (b) an open aperture (T_{op}), and (c) the ratio of the two results ($T_{\mathrm{cl}}/T_{\mathrm{op}}$). The squares are experimental results and the solid curves are results of fittings.

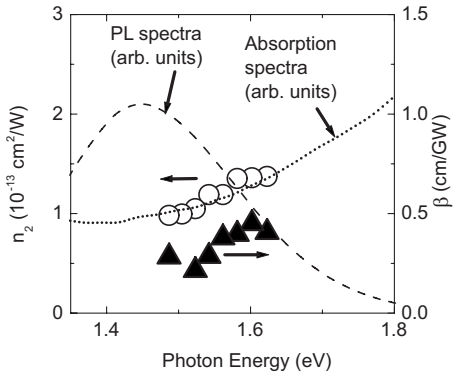

FIG. 3. n_2 (left axis) and β (right axis) spectra of a typical sample ($C_{ex\ Si}$ =3.3 vol %, Ta=1100 °C, D=2.7 nm). The dashed and dotted curves are PL and absorption spectra, respectively.

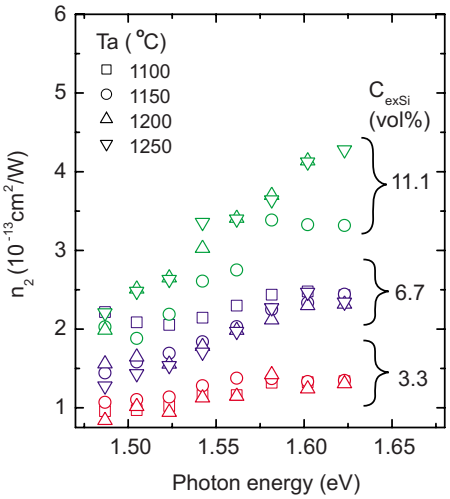

FIG. 4. (Color online) n_2 spectra of samples with different excess Si concentrations ($C_{ex\ Si}$) and different annealing temperatures (Ta). The same colored (or shaped) symbols are used for the same $C_{ex\ Si}$ (or Ta) samples.

trace in Fig. 2(a) suggests that the sign of n_2 is positive and that the nonlinearity is mainly refractive. The analysis of the z-scan traces by Eqs. (1)–(3) reveals that n_2 is $\sim 1.4 \times 10^{-13}$ cm^2/W and β is ~ 0.5 cm/GW, being similar values to previous reports.[9,15]

The observed n_2 is one order of magnitude larger than the bulk-Si value and three orders of magnitude than the amorphous SiO_2 value. In other words, addition of 3.3 vol % Si-ncs into SiO_2 results in as much as three orders of magnitudes enhancement of n_2 compared to SiO_2. It is worth noting that the linear refractive index of Si-ncs doped SiO_2 is explained simply by the volume ratio of Si and SiO_2 and is less than 1.6 in the present case, being very close to that of amorphous SiO_2. The small linear refractive index allows us to minimize the optical coupling loss with a conventional SiO_2 fiber. Therefore, both the linear and nonlinear refractive indices of Si-nc's doped SiO_2 are suitable for optical switching devices.

In order to elucidate the origin of the enhancement of n_2, the photon-energy dependence of n_2 and β is investigated. In Fig. 3, n_2 (left-hand axis) and β (right-hand axis) are plotted as a function of photon energy. n_2 increases from 0.98×10^{-13} to 1.4×10^{-13} cm^2/W with increasing the photon energy from 1.48 to 1.65 eV. Similarly, β increases from 0.3 to 0.5 cm/GW in the same range. The dashed and dotted lines are the PL and absorption spectra, respectively. A larger nonlinear optical response is obtained in a larger absorbance region, but not in a larger PL region. In the case of direct-bandgap semiconductors such as CuCl and CuBr, the nonlinear refractive index is the highest at the energy of the lowest level exciton absorption peak.[25] On the other hand, in absorption spectra of Si-ncs, clear features due to exciton absorption are not observed and the spectra are almost featureless.[26] The energy dependence of n_2 in Fig. 3 seems to follow the broad absorption spectra. Similar results were reported by Kanemitsu *et al.*[14] for porous Si. They found by third harmonic generation measurements that third order nonlinear susceptibility ($\chi^{(3)}$) shows no enhancement in the PL spectrum region, and pointed out two possible mechanisms. First, phonon-assisted optical transitions and resultant small oscillator strength due to indirect bandgap nature of porous Si result in the small enhancement in the PL spectrum region. Second, the PL from porous Si may come not from the band edge but from surface or defect levels with small density of states. If the actual band-edge energy region is far from the PL spectrum region, the enhancement is not expected in the PL spectrum region.

Basically, the same discussion can be applied to understand the data in Fig. 3. However, in the present samples, as mentioned in the introduction, PL from Si-ncs can be assigned to the band-edge luminescence due to the recombination of confined electron-hole pairs as evidenced by some kinds of experiments.[4,21,22] Thus the second hypothesis can be excluded. Probably, small oscillator strength due to the indirect bandgap nature causes the small enhancement at the PL energy.

The systematic control of the size and the concentration of Si-ncs provide further information to understand the mechanism of the large nonlinear response. In Fig. 4, the photon-energy dependence of n_2 for samples with different $C_{ex\ Si}$ and different Ta are shown. The same colored (or shaped) symbols are used for the samples with the same $C_{ex\ Si}$ (or Ta). For all the samples, n_2 is larger in higher photon energy. It is also interesting to note that n_2 strongly depends on $C_{ex\ Si}$, while hardly does on Ta. In Fig. 5, $C_{ex\ Si}$ dependence of n_2 is shown. n_2 is nearly proportional to $C_{ex\ Si}$, suggesting that $C_{ex\ Si}$ is a dominant factor to determine the nonlinear optical response of the samples.

From the experimentally obtained macroscopic nonlinear refractive index of the Si and SiO_2 composites, the microscopic nonlinear refractive index of Si-ncs ($\bar{n}_2$) is estimated by the relation $n_2 = pf^4\bar{n}_2$, where p is the Si volume fraction ($C_{ex\ Si}/100$) and f is the local field correction factor[27] given by $3\epsilon_{SiO_2}/(\epsilon_{SiO_2}+2\epsilon_{Si})$, where ϵ_{SiO_2} and ϵ_{Si} are the real part of the dielectric constant of SiO_2 and Si. The estimated values of $\bar{n}_2$ are $2-3 \times 10^{-10}$ cm^2/W for all the samples and to be four orders of magnitudes larger than the bulk-Si value. In the inset of Fig. 5, the obtained $\bar{n}_2$ is plotted as a function of the diameter of Si-ncs. $\bar{n}_2$ increases slightly with decreasing the size of Si-ncs. This result is qualitatively consistent with other reports, where the modification of the band structure by the quantum confinement effect is sug-

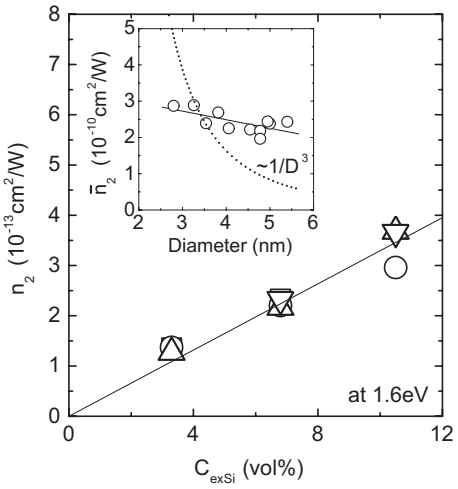

FIG. 5. Si concentration dependence of n_2. The inset shows the size dependence of the microscopic nonlinear refractive index of Si-ncs. The dotted line is a $1/D^3$ dependence predicted by a simple quantum confinement model.

gested to be the origin of the enhancement of optical nonlinear response of Si-ncs.[15,16] On the other hands, the result cannot quantitatively be explained by a simple quantum confinement model, which predicts the enhancement with the $1/D^3$ dependence (D is the diameter of Si-ncs)[29] as shown by a dotted line in the inset of Fig. 5. While the model predicts as much as eight times enhancement by the size reduction from 5.4 to 2.7 nm, the data show the enhancement of only about 30% in the size range.

To our knowledge, quantitative discussion on the size dependence of nonlinear optical response of Si-ncs has been made only in limited number of papers.[15,16] Vijaya Prakash *et al.*[16] reported nonlinear optical response of Si-ncs doped SiO_xN_y prepared by PECVD and demonstrated a strong enhancement of $\chi^{(3)}$ when the size of Si-ncs was very small (less than 1 nm). However, as they mentioned in the paper, the data were strongly scattered and thus the size dependence in the range of the present work was not very clear. Furthermore, the effect of Si concentration was not taken into account in the analysis. On the other hand, Hemandez *et al.*[15] discussed size dependence of $\chi^{(3)}$ by taking into account the Si concentration for similar Si-nc samples. They observed $\sim 1/D^3$ dependence of $\chi^{(3)}$ and suggested that the modification of the band structure by the quantum confinement effect is responsible for the enhanced nonlinear optical response of Si-ncs. However, the discussion, i.e., the fitting of data points by the $\sim 1/D^3$ dependence, was based only on three experimental points, and thus the dependency is not very convincing.

The results of Fig. 5 show for the first time detailed size dependence of the nonlinear optical response of Si-ncs. The observed deviation from $\sim 1/D^3$ dependence indicates that the nonlinear optical response of Si-ncs cannot be fully explained by the simple quantum confinement model. Recently, some investigations have revealed that the size dependence of the nonlinear optical response of semiconductor nanocrystals differs depending on the size range.[28,29] In a strong confinement regime where the size is much smaller than that of the exciton Bohr radius (a_B), i.e., $D \ll 2a_B$, the nonlinear optical response increases with decreasing the size, while in a weak confinement regime ($D \gg 2a_B$), it increases with increasing the size.[28–32] The above mentioned simple quantum confinement model is the case of the strong confinement regime. Since the size range of Si-ncs studied in this work is comparable to the exciton Bohr radius of bulk Si, the competition of the two different size dependences may result in the observed small size dependence.

Very recently, Yildirim and Bulutay[33] calculated the nonlinear optical properties of Si-ncs and Ge nanocrystals (Ge-ncs) in SiO_2 by an atomistic pseudopotential approach. Their calculations focused on the third-order nonlinearities of Si-ncs by assuming no interface defects, no strain and thermal effects, and no compounding contribution of the excited carriers. They showed that $\chi^{(3)}$ of Si-ncs and Ge-ncs hardly depend on the size in the size range above 2.5 nm, while they significantly increase with decreasing the size in the size range below 2.5 nm. Since the size range of this work is 2.7–5.4 nm, the predicted small size dependence above 2.5 nm agree qualitatively with the present results. Furthermore, they predicted that $\chi^{(3)}$ of Si-ncs is about 4–5 orders of magnitudes enhanced compared to that of bulk Si when the size is around 3.0 nm. This enhancement factor also coincides well with experimentally obtained ones (four orders of magnitudes). These agreement may allow us to conclude that the discrete energy states of Si-ncs is responsible for the observed enhanced optical nonlinearity.

It should be mentioned here that defects within nanocrystals or at the surface can also cause the enhancement of the optical nonlinear response.[34] Takagahara and Hanamura[34] reported that localization of excitons at disorders or impurities enhances its oscillator strength, and hence the nonlinear optical properties. They pointed out that sometimes this effect is more important than the dimensional confinement effect. In the model, the size dependence of the nonlinear optical response is expected to be very small. However, this model is considered to be not suitable to explain the observed enhanced nonlinear optical response of Si-ncs. As mentioned above, the nonlinear optical response of the present samples is hardly modified by annealing conditions. On the other hand, the quality, i.e., the defect density, of Si-ncs depends strongly on the annealing condition.[23] This inconsistency rules out the possibility of defect-related enhancement of the nonlinear response.

As mentioned in the introduction, the shape distortion of Si-ncs significantly modifies the electronic band structure and may also modify the nonlinear optical response. However, the present results give no information on the effect of shape distortion.

IV. CONCLUSION

Nonlinear optical response of Si-ncs doped SiO_2 prepared by a cosputtering method is studied by z-scan technique in a femtosecond regime at around 1.6 eV. An addition of a very small amount of Si-ncs into SiO_2 results in as much as three orders of magnitudes enhancement of n_2 compared to SiO_2 and one order of magnitude to bulk Si, while the

linear refractive index is kept low (less than 1.6). The small index mismatch with a conventional SiO_2 fiber and the large nonlinear response make Si-ncs doped SiO_2 an ideal candidate for realizing Si-based optical switching devices. In order to elucidate the origin of the enhanced optical response, the size dependence of n_2 is studied in detail in the diameter range of 2.7–5.4 nm. We show that the size dependence is very small in the range, although small increase in n_2 with decreasing the size is observed. The observed size dependence agrees very well with that calculated by pseudopotential approach, suggesting that the discrete energy states of Si-ncs is responsible for the observed enhanced optical nonlinearity.

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