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Color control of white photoluminescence from carbon-incorporated silicon oxide

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Color control of the white photoluminescence (PL) from carbon-incorporated silicon oxide is demonstrated. The carbon-incorporated silicon oxide was fabricated by carbonization of porous silicon in acetylene flow (at 650 and 850 °C) followed by wet oxidation (at 650 and 800 °C). It was shown that PL color can be controlled in the range of blue-white and yellow-white by selecting the porosity of starting porous silicon as well as the carbonization and oxidation temperatures. Low-temperature oxidation resulted in bluish light emission in lower porosity series, while high-temperature oxidation promoted yellow-white light emission. The maximal integral intensity of PL was observed after oxidation at 800 °C. It was shown that white PL from carbon-incorporated silicon oxide has blue and yellow-white PL bands originating from different light-emitting centers. The origin of blue PL is attributed to defects in silicon dioxide. Some trap levels at the interface of the carbon clusters and silicon oxide are suggested to be the origin of the yellow-white light emission. *© 2008 American Institute of Physics*. [DOI: 10.1063/1.3003079]

I. INTRODUCTION

White-light-emitting diodes (LEDs) attract considerable attention as the applicable field of lighting and display technologies are broadened by improvements in luminous efficiency, luminescence intensity, and energy consumption. The spectrum of this "white light" is not similar to that of the sunlight but it is composed of several colors from different light sources, which make the human eye recognize it as white. As examples, white light is composed of blue light from blue LEDs and yellow light from fluorescence materials, and also blue, green, and red light from fluorescence. This hampers the expansion of the application of white LEDs owing to low color rendering properties. This problem can be solved using materials that emit white light that can be tuned close to the spectral distribution of the sunlight. Development of materials that emit colored light similar to the sunlight is one of the most effective solutions to the problem regarding white LED.

From the viewpoint of broad-band white light, carbon-incorporated silicon oxide seems to be one of the promising candidates. It was shown that its photoluminescence (PL) covers the entire visible range of 350–800 nm. Light-emitting SiO_2:C layers have been fabricated by rf magnetron sputtering deposition,[1] C^+ implantation in SiO_2,[2–4] and chemical vapor deposition techniques.[5] Recently, a strong white PL from carbon-enriched SiO_2:C layers fabricated by successive carbonization/oxidation treatment of porous silicon (por-Si) has been reported.[6,7] This technique is relatively simple and based on the visible light-emitting por-Si technology.[8] In the present paper, we report about the effects of porosity of starting por-Si substrate as well as carbonization and oxidation temperatures on the spectral distribution of the light emission of SiO_2:C fabricated by carbonization/oxidation of por-Si.

II. EXPERIMENT

The starting por-Si samples were prepared by anodizing p^+-type Si(100) wafers (0.015–0.025 Ω cm) in a hydrogen fluoride (40%):ethanol mixture (1:1). High-porosity (HP) por-Si samples were prepared by anodization at 80 mA/cm^2 for 40 s, and low-porosity (LP) por-Si samples were prepared by anodization at 30 mA/cm^2 for 70 s. The por-Si samples were carbonized in N_2 (1.5 l/min)/C_2H_2[(1 l/min)] flow at 650 and 850 °C for 30 min. Details of the carbonization procedure can be found in Ref. 9. For convenience, the LP por-Si samples carbonized at 650 °C are indicated as LP650, others as idem quod. Owing to its specific morphology, por-Si serves as a nanoscale porous template for carbon deposited inside the pores by the decomposition of acetylene. As a result, a nanostructured por-Si:C layer is formed after carbonization.

The next sample preparation step was thermal treatment at atmospheric pressure in moisturized pure argon (Ar/H_2O) flow for 3 h at 650 and 800 °C. This treatment, particularly at a higher temperature of 800 °C, permits the preferential oxidation of the nanocrystalline silicon skeleton of such a structure and converts por-Si:C into carbon clusters incorporating the silicon oxide-based material SiO_2:C.[6] The flow rate of argon was 0.9 l/min and the temperature of the water

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TABLE I. Fabrication conditions of starting substrate, carbonization temperature, and wet oxidation temperature are shown by sample notations.

Substrate	Carbonization temperature (°C)	Oxidation temperature (°C)	Notation
Low porosity (LP series)	650	650	LP650-650
		800	LP650-800
	850	650	LP850-650
		800	LP850-800
High porosity (HP series)	650	650	HP650-650
		800	HP650-800
	850	650	HP850-650
		800	HP850-800

in the bubbler was 85 °C. The list of the samples as well as the temperatures of the treatments and the corresponding notations of the samples is presented in Table I.

The internal surface area of the por-Si layer, porosity, and the average size of the pores were examined by gravimetry, Brunauer–Emmett–Teller (BET), and Barrett–Joyner–Halenda (BJH) standard methods, respectively.

PL measurement was performed using a conventional rocking system with 351 nm Ar^+ laser excitation. PL in the pulse regime was measured with a 337 nm line of a N_2 laser. The natural duration of nitrogen laser pulses was about 10 ns; however, for some measurements, we reduced the pulse duration below 0.7 ns. The samples were also examined by Raman scattering (RS) spectroscopy and transmission electron microscopy (TEM). Cross-sectional TEM observations were performed using JEOL-JEM200CX equipped with a Gatan ENFINA 1000 spectrometer and a JEM3200FSK Ω-type energy-filtering setup.

III. EXPERIMENTAL RESULTS

A. Characterization of starting porous silicon substrate

The porosities of LP and HP por-Si substrates were about 50% and 70%, respectively. The surface area and average pore diameter of the LP substrate series were estimated to be 231 m^2/g and 7.9 nm, respectively. The HP series exhibited a 307 m^2/g surface area and an 11.8 nm average pore diameter. These values agreed well with the images obtained by cross-sectional TEM. Figures 1(a) and 1(b) show the images of LP and HP por-Si substrates, respectively. The images were formed using the energy-filtered Si-$L_{2,3}$ line of electron energy loss spectra, and the bright region indicates silicon. The average diameter of the pores in the HP series is significantly larger than that in the LP series, but the thickness of Si walls in the HP series, which is below 10–20 nm, is comparable to that in the LP series.

Carbonization of por-Si layers in acetylene flow at high temperatures increased the weight and decreased the surface area. Figure 2 shows the dependence of the weight increase and the surface area of the porous layer on the carbonization temperature. It should be noted that the weight is that of the por-Si layer. The amount of carbon in the por-Si layer increased with increasing carbonization temperature owing to the enhancement of acetylene decomposition inside the pores. Such weight increase was observed in both HP and LP samples. Simultaneously, the surface area of the por-Si layer decreased with increasing carbonization temperature. It is suggested that the surface area decrease is caused by the sealing of the small pores due to the carbon deposition.

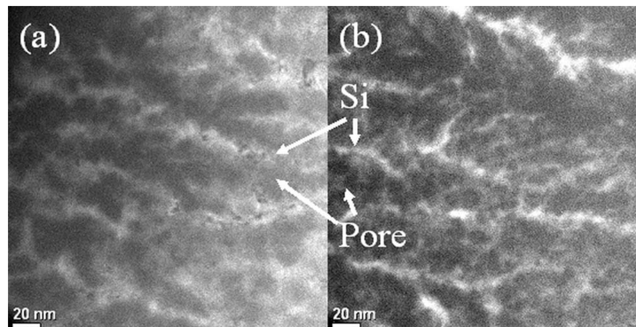

FIG. 1. Cross-sectional TEM images of (a) HP and (b) LP por-Si substrates. These images were obtained using the energy-filtered Si-L23 line of the electron energy loss spectra.

To understand the geometrical features of the carbonized por-Si layer, we represent the simplified morphology of the layers with different porosities after carbonization processes in Fig. 3. It is obvious that the thickness of the deposited carbon layer is proportional to the ratio of weight increase (in grams) per total surface area. According to such calculations, the thickness of the deposited carbon layer in the LP series was estimated to be 1.7 times lager than that in the HP series at the same carbonization temperature, and the carbon layer thickness increased 1.7 times with increasing carbonization temperature from 650 to 800 °C. Therefore, it can be concluded that carbon layer thickness increases in the following sequence: HP650, HP850 and LP650, LP850. The thickness of the carbon layer is approximately the same in HP850 and LP650.

B. Photoluminescence

Figure 4(a) shows the normalized spectra of SiO_2:C, which were recorded from the HP series at room temperature

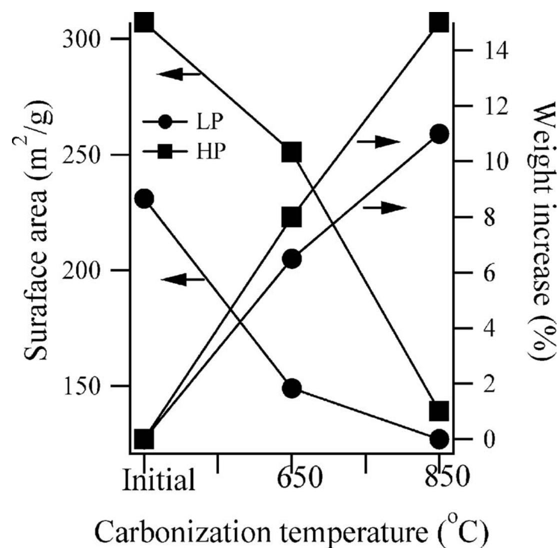

FIG. 2. Variations in weight and surface area of HP and LP Si layers depending on carbonization temperature using acetylene.

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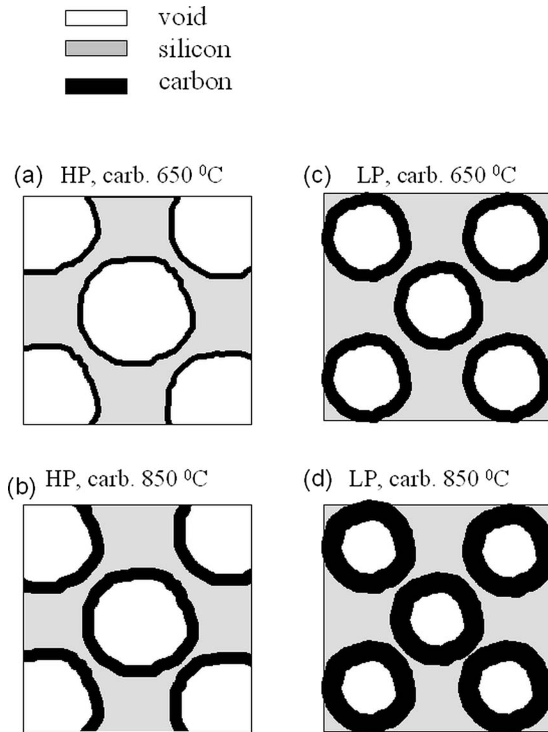

FIG. 3. Simplified morphology of carbonized por-Si layers. Adsorbed carbon layer thickness depended on the porosity of the por-Si substrate and carbonization temperature.

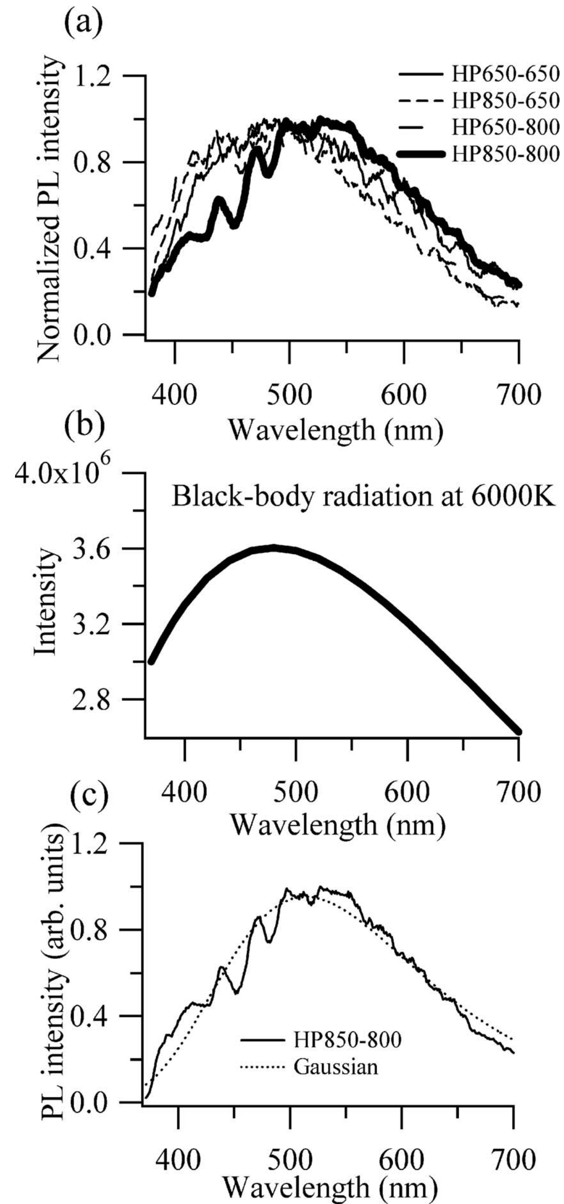

FIG. 4. (a) Normalized PL spectra of SiO_2:C prepared from HP por-Si substrate. The measurement was performed under 70 Hz chopped 351 nm Ar^+ laser light excitation at RT. (b) Spectrum from black body radiation at 6000 K. (c) PL spectrum of HP850–800 and fitted Gaussian profile. The fitted Gaussian has a peak at 2.27 eV and a FWHM of 0.54 eV.

(RT). The radiation spectrum from the black body at 6000 K is shown in Fig. 4(b). In general, the spectrum in Fig. 4(a) is similar to that presented in Fig. 4(b) in the visible light region. Therefore, the color of PL of SiO_2:C is close to that of the sunlight as the surface temperature of the sun is almost 6000 K. The clear modulation of the PL spectra is due to interference effects at the interface of the SiO_2:C layer and Si substrate. The spectrum shape of the HP650-650 sample (HP substrate, carbonization at 650 °C, and oxidation at 650 °C) is similar to that of the HP850-650 samples (HP substrate, carbonization at 850 °C, and oxidation at 650 °C). PL spectra of SiO_2:C layers after wet oxidation at 650 °C are represented by a broad featureless band with a maximum intensity of about 500 nm, and the color of PL is blue white. After oxidation at 800 °C, the integral PL intensity is more than one order of magnitude higher than that of the samples oxidized at 650 °C. The color and spectrum shape of the HP650 sample are not changed by 800 °C oxidation. However, the relative intensity of the light emission in the "blue" spectral range of 400–500 nm is significantly reduced and the PL color becomes yellowish white in the PL spectrum of the HP850–800 sample. This spectrum was well fitted to the Gaussian peak at 2.27 eV (546 nm) with a 0.56eV full width at half maximum (FWHM), as shown in Fig. 4(c). The detailed analysis of the effects of oxidation temperature on PL intensity and SiO_2:C layer structure is discussed below.

The effects of excitation conditions on PL were determined using a nitrogen laser that can be operated in an ordinary regime (pulse duration of 10–15 ns) and in a regime with ultrafast pulses (pulse duration below 0.7 ns). Figure 5(a) shows typical PL spectra of SiO_2:C layers (samples of the HP850-800 series) recorded in ordinary (10 ns) and ultrafast (0.7 ns) excitation regimes at RT. One can see that excitation by ultrafast pulses reduces significantly the blue shoulder of the PL spectrum. The spectrum excited by ultrafast pulses was fitted to the Gaussian peak at 2.27 eV (546 nm) with a 0.46 eV FWHM, as shown in Fig. 5(a).

In Fig. 5(b), we show the corresponding spectra recorded under the same conditions but at a temperature of 5 K. The spectrum recorded in the ordinary regime is similar to that recorded at RT. In contrast, the spectrum measured in the ultrafast regime excited only the blue component, and the yellow-white band disappeared. These observations indicate that white PL from SiO_2:C is composed of at least two emission bands, namely, a blue emission band (centered at about

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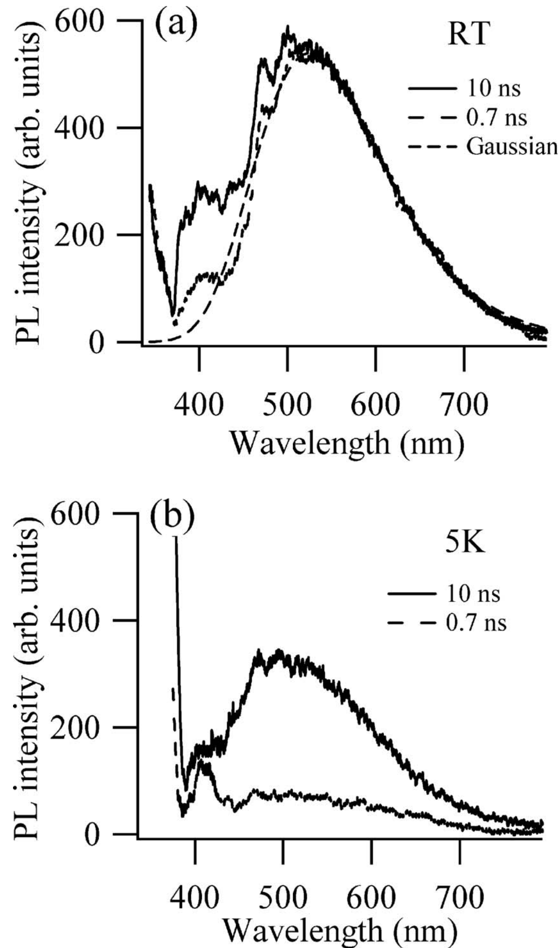

FIG. 5. PL spectra excited ordinary (10 ns) and ultrashort (0.7 ns) regimes of HP850–800. The measurement was performed under 337 nm N^+ laser light excitation at (a) RT and (b) 5 K. They represent spectral variations depending on the excitation time period. The fitted Gaussian to the spectrum of the ultrashort excitation regime of HP850-800 is indicated in (a). The fitted Gaussian has a peak at 2.27 eV and a FWHM of 0.46 eV.

410 nm) and a much broader yellow-white emission band (centered at about 546 nm). As the excitation time of the electronic states corresponding to these bands and the effect of measurement temperature are different, we conclude that the origin of these emission bands is associated with different light-emitting centers. Light-emitting mechanisms of these bands will be discussed in another report. The main conclusion from these observations is that controlling the relative intensity of the blue and yellow-white emission components makes it possible in principle to tune the color of light emission to natural white.

Figure 6 shows the PL spectra of SiO_2:C prepared from LP por-Si substrates (LP series). The spectra are normalized by the intensity (amplitude) at a 550 nm emission wavelength. This treatment seems to be reasonable for estimating the ratio of the blue component to the yellow-white component as the spectrum profile in the region of 500–700 nm does not contain the blue component, as can be seen in Figs. 4(a) and 5(b). By subtracting the Gaussian profile from PL spectra, the integrated intensity of blue component can be estimated and we found that the intensity ratios of the blue component to the yellow-white component I(B)/I(Y) are

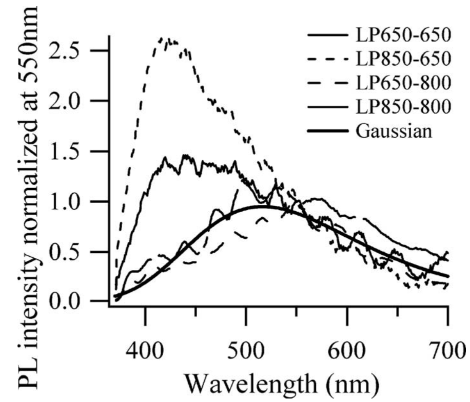

FIG. 6. Normalized PL spectra of SiO_2:C prepared from LP por-Si substrate with intensity at 550 nm and Gaussian profile with a peak at 2.27 eV and a FWHM of 0.54 eV, which was fitted to HP850-800 spectrum in Fig. 4(c). The measurement was performed under 70 Hz chopped 351 nm Ar^+ laser light excitation at RT.

LP850-650 (1.4), LP650-650 (0.7), LP850-800 (0), and LP650-800 (0), as described in Table II. The fractions of blue PL after wet oxidation at 650 °C are larger than those at 800 °C. Carbonization temperature also affects the ratio of the blue component to the yellow-white component. A high carbonization temperature results in a large ratio I(B)/I(Y) at the same oxidation temperature of 650 °C. The wet oxidation (800 °C) of LP samples after carbonization at 650 and 800 °C results in a similar spectrum, and the spectra almost coincide with that of HP850-800.

Summarized data on integral PL intensity in the region of 400–700 nm as a function of oxidation temperature are presented in Fig. 7. One can see that increasing the oxidation temperature up to 800 °C markedly increased the integral intensity of the light emission. However, further increase in wet oxidation temperature up to 950 °C resulted in the disappearance of PL, which is in good agreement with our previous results.[6]

C. Raman scattering measurements

Typical silicon-related RS spectra of the LP650 and HP 650 series are shown in Figs. 8(a) and 8(b). Spectra of all as-carbonized samples are represented by a broad band centered at around 500 cm^{-1} that can be unambiguously attributed to the nanocrystalline silicon of the porous layer. Oxidation at 650 °C does not change significantly the RS spectra. The small RS band broadening observed after wet

TABLE II. The prepared samples were classified on the basis of the ratio of the integrated blue PL intensity to the yellow-white PL intensity (I(B)/I(Y)).

Sample name	I(B)/I(Y)
LP650-800	
LP850-800	0
HP850-800	
HP650-800	
HP650-650	0.5
HP850-650	
LP650-650	0.7
LP850-650	1.4

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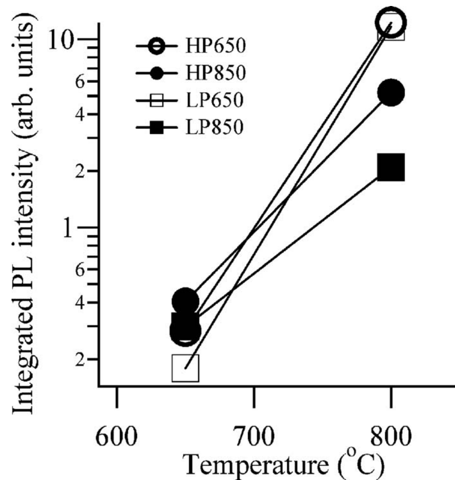

FIG. 7. Integrated PL intensities in 400–700 nm range depending on oxidation temperature.

oxidation at 650 °C [Figs. 8(a) and 8(b)] can be attributed to the decrease in Si particle diameter.[9] No broad band from nanocrystalline silicon was observed after wet oxidation at 800 °C, but a narrow peak at 518 cm^{-1} from the crystalline substrate was detected [Figs. 8(a) and 8(b)]. The appearance of the 518 cm^{-1} peak of the Si substrate indicates that the por-Si layer was completely converted into a transparent silicon oxide layer. This conclusion is well consistent with TEM and electron energy loss spectroscopy (EELS) measurements.[10] The effect of oxidation temperature on the evolution of silicon-related RS spectra was found to be the same in the LP850 and HP850 series.

The RS spectra of the LP650 and HP650 series after wet oxidation at 800 °C are represented by a very broad band with a maximum intensity of about 1360 cm^{-1} and a high-frequency shoulder at 1560 cm^{-1} [Fig. 8(a)]. These spectra can be interpreted as the superposition of *D*-band ("disordered," 1370 cm^{-1}) and *G*-band ("graphitic," 1560 cm^{-1}) that are inherent to the disordered graphitelike structure and associated with the out-of-plane breathing mode A_{1g} (Ref. 11) and in-plane stretching vibration mode E_{2g} of the defective graphite structure.[11,12] The very large width of the bands and the large relative intensity of the *D*-band indicate the presence of very small (below 1–2 nm) and highly disordered sp^2-coordinated carbon clusters.

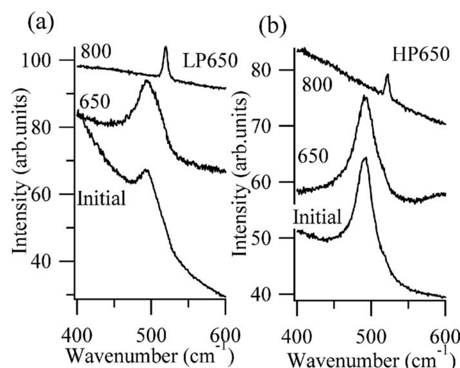

FIG. 8. RS spectra of Si depending on wet oxidation temperature. (a) LP (LP650) and (b) HP (HP650). Si substrates ware carbonized at 650 °C.

The spectra of samples carbonized at 850 °C were independent of porosity and represented by relatively narrow *D*- and *G*-bands [Fig. 8(b)]. Narrower *D*- and *G*-bands indicate a larger and more ordered graphitelike structure.

IV. DISCUSSION

There are many reports on the blue emission from SiO_2-based materials. Blue PL spectra were observed from Si^+- or Ar^+-implanted SiO_2 as well as from oxidized bulk and por-Si and silicon-rich SiO_x thin layers deposited by several techniques (see review in Ref. 13). It is commonly accepted that this blue PL originates from defect states related to silicon enrichment (Si–Si bonds) or siloxane groups. The origin of the yellow-white PL cannot be simply associated with a pure SiO_2 matrix as the light emission was not detected from SiO_2, which was fabricated by the wet oxidization of the por-Si substrate at 650 and 800 °C without carbonization pretreatment.[6] On the other hand, a similar yellow-white light emission was observed in silicon oxide layers incorporated with carbon.[1,3,5,14] Therefore, we can attribute the origin of yellow-white light emission to carbon incorporation.

Wet oxidation temperature has a strong effect on the oxidation state of silicon in the porous layer. RS spectra indicate that wet oxidation at 800 °C fully oxidized the por-Si layer, while wet oxidation at 650 °C resulted in only a partially oxidized por-Si layer. Fourier transform-IR measurements in the reflection mode (not presented here) confirmed the weak oxidation at 650 °C. As shown in Figs. 4 and 6, the partially oxidized samples (oxidation at 650 °C) emit blue-white (HP series) and blue (LP series) light. The fraction of the blue the component (see Table II) decreases in the samples in the following sequence: LP850-650 [I(B)/I(Y)=1.4], LP650-650 [I(B)/I(Y)=0.7], HP850-650 I(B)/I(Y) 0.5, and HP650-650 [I(B)/I(Y)=0.5]. This sequence is well consistent with that for the oxidation rate of silicon under a carbon layer in as-carbonized samples that increases in the following sequence of LP850 [Fig. 3(d)], LP650 [Fig. 3(c)], HP850 [Fig. 3(b)], and HP650 [Fig. 3(a)] [see Sec. IIA]. A thinner carbon layer and a larger surface area promote higher Si oxidation rates under the carbon layer, resulting in the lower contribution of the blue component.

The color of white PL after low-temperature oxidation (650 °C) can be tuned by controlling the porosity of the starting por-Si substrate and carbonization temperature. Selecting a LP por-Si substrate and high-temperature carbonization enhance blue-white PL. Yellow-white PL can be enhanced using a HP por-Si substrate and low-temperature carbonization.

After treatment in wet Ar at 800 °C, the full oxidation of the por-Si skeleton occurs, increasing the integral PL intensity by about one order of magnitude in all series (Fig. 7). The Gaussian spectra of HP850-800, LP650-800, and LP850-800 indicate that PL mostly consists of yellow-white component and contains a very small fraction of blue component. However, a large fraction of blue component was observed in the PL of HP650-800. This indicates that the density of light-emitting centers related to silicon oxide de-

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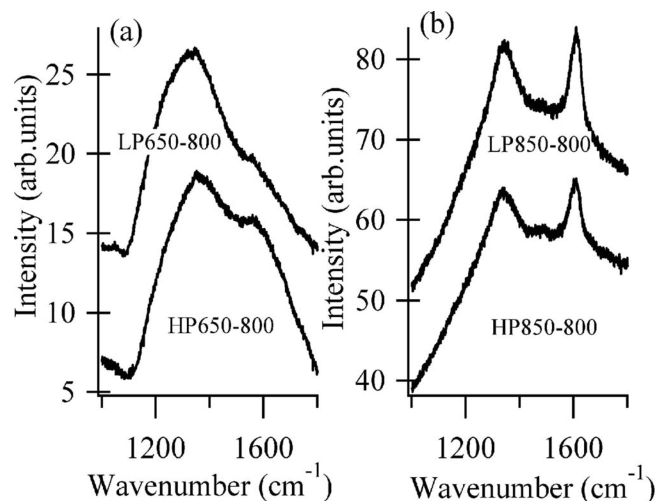

FIG. 9. RS spectra of C after wet oxidation at 800 °C. Samples carbonized (a) at 850 and (b) 650 °C.

fects is higher in HP650-800 than in HP850-800 (idem quod LP650-800 and LP850-800). Currently, we have no ready explanation for such an effect. It is possible that the higher defect density in HP650-800 is caused by overoxidation effects, i.e., the diffusion and reactions of carbon species in the silicon oxide matrix.[15]

In the case of high-temperature oxidation (800 °C), tuning the PL color from yellow white to blue white can be performed by selecting a HP por-Si substrate and carbonization at a lower temperature.

It is clear that a yellow-white light emission center is related to carbon incorporation in SiO_2. The obvious difference between the carbon-related Raman spectra of the LP650-800 and LP850-800 samples (width, peak position, and intensity ratio of the *G*-band to the *D*-band, see Fig. 9) indicates that structural (and hence electronic) properties of the carbon clusters in these samples are different, but their PL spectra are similar (see Fig. 6). By considering these findings, it can be attributed that the origin of the yellow-white component is not isolated carbon clusters. Electronic states at the interface between carbon clusters and silicon oxide and/or carbon-related defects in the SiO_2 matrix (for example, the substitution of oxygen atoms by carbon atoms) can be considered as yellow-white light-emitting sites. To clarify the origin of yellow-white PL, we require further analysis including electronic paramagnetic resonance, EELS, and photoluminescence excitation measurement.

V. CONCLUSIONS

Carbon-incorporated silicon oxide layers (a-SiO_2:C), which emit white PL, were fabricated by successive carbonization/oxidation thermal treatments of por-Si. The effects of porosity of starting substrates as well as carbonization and oxidation temperatures on the PL of SiO_2:C layers were studied. The spectrum of white PL is similar to that of the sunlight. It was shown that PL spectra of a-SiO_2:C layers are composed of blue and yellow-white components originating from different light-emitting centers. The blue PL originates from defects in a partially oxidized silicon layer. The yellow-white PL is related to carbon incorporation. Interface states of the carbon clusters and silicon oxide and/or carbon-related defects in the SiO_2 matrix are suggested to be yellow-white-light-emitting centers.

It was demonstrated that the color of PL can be controlled by varying the relative intensities of the blue and yellow-white components. Such variation can be performed by adjusting the porosity of starting por-Si substrates and carbonization and oxidation temperatures.

ACKNOWLEDGMENTS

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