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Effect of hydrogen passivation on the photoluminescence of Tb ions in silicon rich silicon oxide films

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In this work, silicon-rich silicon oxide films containing terbium were prepared by means of plasma enhanced chemical vapor deposition. The influence of hydrogen passivation on defects-mediated non-radiative recombination of excited Tb\(^{3+}\) ions was investigated by photoluminescence, photoluminescence excitation, and photoluminescence decay measurements. Passivation was found to have no effect on shape and spectral position of the excitation spectra. In contrast, a gradual increase in photoluminescence intensity and photoluminescence decay time was observed upon passivation for the main \(^{5}D_4-^{7}F_3\) transition of Tb\(^{3+}\) ions. This observation was attributed to passivation of non-radiative recombination defects centers with hydrogen. It was found that the number of emitted photons increases upon passivation as a result of two effects: (1) longer Tb\(^{3+}\) lifetime in the \(^{5}D_4\) excited state and (2) optical activation of new Tb\(^{3+}\) emitters.

Moreover, efficient light emission from Er-doped SRSN thin films has been recently demonstrated.\(^ 1\) As in the case of SRSO, the main motivation for exploring these new host materials is the improvement of the electrical properties, which is important from the point of view of application in electrically driven light sources.\(^ 1\)

Despite different approaches and materials which have been used for fabrication of Si-based light sources, there are some general physical problems limiting their emission efficiency. One such problem is related to the presence of defects in the host-matrix,\(^ 1\) which act as non-radiative de-excitation paths for RE ions. Since for most of the trivalent RE ions, the useful emission arises from transitions within the 4f shell of the ions, which have very long radiative recombination lifetimes, such defects-related non-radiative recombination seriously competes with photon emission, which has a very negative impact on the optical properties. Given the fact that RE doping always introduces additional defects to the host-matrix, it is a serious problem, especially when we take into account that RE ion concentrations useful for practical purposes are generally high. To take one example, it has been shown for Er-doped amorphous Si (a-Si) that although as much as 0.27 at. % of Er can be doped into the film, the generation of defects by Er-doping limits practical Er concentrations to 0.04 at. % as a result of strong non-radiative recombination.\(^ 1\) Furthermore, it has been shown that while using SRSO or SRSN matrices generally improves electrical properties in comparison with a SiO\(_2\) matrix, it also increases defects density, leading to a stronger contribution of non-radiative de-excitation channels.\(^ 1\)

When analyzed from this perspective, it is interesting and important to investigate the effect of defect passivation by hydrogen on the optical properties of Si-based thin films doped by RE.
ions, especially at higher dopant concentrations. At the moment, this problem has been given at most moderate attention, focusing only on Er-doped hydrogenated SRSO films. To our knowledge, no systematic experimental study exists which would address this issue for other RE dopants. This is rather surprising, especially when we take into account that hydrogenation has been shown to have beneficial effects on the optical properties of undoped a-Si thin films or SRSO structures containing silicon nanocrystals.

In this work, we experimentally study the effect of hydrogen passivation on the optical properties of SRSO films doped by high concentration of Tb atoms (0.76 at. %). We also discuss the physical mechanism responsible for the observed photoluminescence (PL) enhancement of Tb ions after hydrogenation. Finally, we compare our results with the available data on Er-doped hydrogenated SRSO films, to draw more general conclusions on the problem of defect-related non-radiative recombination in Si-based structures doped by RE.

II. EXPERIMENTAL DETAILS

Tb-doped SRSO thin film was grown on Si wafer by electron cyclotron resonance plasma enhanced chemical vapor deposition (ECR-PECVD). The silicon and oxygen precursors were silane (30% SiH4 in Ar) and O2 (10% O2 in Ar), respectively. The silane and oxygen flow rates were 2.5 sccm and 7 sccm, respectively. The volatile metal organic compound Terbium (TbIII) (tris(2,2,6,6-tetramethyl-3,5-heptanedionato) (Tb(tmhd)) was used as the Tb precursor. The terbium diffusion cell was heated up to 200 °C within a manifold to sublime the precursor and introduce it into the chamber alongside the silane, using Ar as carrier gas. Argon carrier gas flow rate was set constant at 10 sccm. During the deposition process, the substrate stage was heated to a temperature of 300 °C, resulting in a surface temperature of the sample of 121 °C. Substrate rotation was used to achieve uniformity and homogeneity of the film. Further details of the deposition system and film growth have been discussed previously.21

The composition of the as-deposited film was determined by Rutherford Backscattering Spectrometry (RBS) in conjunction with the SIMNRA software used to fit the measured RBS spectrum. The measurements were conducted in the Tandetron Accelerator Laboratory at Western University (London, Ontario, Canada), using 1.6 MeV 4He ions with an incident angle of 75°. The SRSO film was found to contain 3 at. % of excess Si and 0.76 at. % of Tb.

In the post-deposition process, the sample was cleaved into smaller pieces which were then all annealed for 1 h at a temperature of 1100 °C in a quartz tube furnace under flowing N2 gas at ambient pressure. As demonstrated in Ref. 8, annealing at 1100 °C activates strong Tb photoluminescence and improves the structural quality of SRSO films. Next, using the same quartz tube furnace, the samples were passivated by hydrogen for 1 h in flowing forming gas (N2:H2 = 95%-5%) at different passivation temperatures ranging from 400 to 750 °C. For comparison, one unpassivated reference sample was included in all measurements presented in this paper, and the results have been added to the corresponding graphs at a passivation temperature (Tpass) of 20 °C. Further details of the passivation method used in this study have been discussed in details elsewhere.22 Importantly, to make sure that the observations described in this paper are caused by hydrogen passivation, a control sample was annealed for 1 h at 750 °C in N2 atmosphere instead of the forming gas atmosphere. Since the optical properties determined for this sample (not reported here, for clarity reasons) were comparable with the results obtained for an unpassivated sample, we could exclude the possibility that the effects described in this paper are due to the annealing temperature itself.

Here, it is worth mentioning that hydrogenation of SRSO films was investigated by our group in a separate Elastic Recoil Detection (ERD) experiment, conducted in the Tandetron Accelerator Laboratory at Western University, using 1.6 MeV 4He ions with an incident angle of 75°. First, those measurements showed that upon annealing at 1100 °C in N2 atmosphere, the concentration of hydrogen remaining in SRSO film after PECVD deposition is reduced below the detection threshold of ERD. What is more important, ERD results confirmed that annealing in a forming gas atmosphere successfully introduces hydrogen into SRSO films. Importantly, the amount of hydrogen introduced to the film during passivation is rather low (single at. %) and depends on Tpass. Since the above-mentioned results were obtained for another SRSO samples, having lower excess Si content and lower Tb concentration, they were not included in the present paper.

Finally, the optical properties of investigated samples were determined by means of PL, photoluminescence excitation (PLE), and PL decay measurements. For PL and PLE measurements, a continuous wave (cw) xenon lamp connected to a monochromator (Jobin Yvon TRIAX 180) was used as the tunable excitation source. The signal emitted from the sample was then collected and detected by a Si CCD camera (HR4000 Ocean Optics). The excitation wavelength used for cw-PL measurements was set to 266 nm. In the case of PLE experiment, the measured signal was corrected for the xenon lamp spectral intensity profile. For PL decay measurements, we used the pulsed excitation line (λEXC = 266 nm) of an actively Q-switched diode pumped solid state laser (Elforlight Ltd.), together with a multichannel detection system equipped with a monochromator and a PMT detector. The time resolution in all measurements presented in this study was better than 1 μs.

III. RESULTS

The room-temperature PL spectra of SRSO:Tb films are shown in Fig. 1 for the unpassivated reference sample, denoted as Tpass = 20 °C, and for the sample passivated at Tpass = 750 °C. For each sample, four main PL bands can be seen that originate from radiative transitions within the 4f2 shell of Tb3+ ions. The observed transitions are 5D4 → 7F5 (489 nm), 5D4 → 7F4 (545 nm), 5D4 → 7F3 (589 nm), and 5D4 → 5G4 (623 nm). The most intense 5E4 → 5G4 band was chosen to represent the emission intensity of Tb3+ ions. For this...
band, the integrated emission intensity is shown in the inset to Fig. 1 as a function of $T_{\text{pass}}$. It can be seen that passivation at temperatures up to 750 °C results in a gradual increase in the Tb$^{3+}$ ions emission intensity by up to a factor of 1.54 compared to the unpassivated sample. Here, it is worth mentioning that this increase in the Tb$^{3+}$ emission after passivation is very similar to the observations reported for passivated SRSO:Er films, where an increase in the emission intensity was observed for the near-infrared $^4I_{13/2} \rightarrow ^4I_{15/2}$ transition of Er$^{3+}$ at 1535 nm.$^{18,19}$

Figure 2(a) shows the map of emission spectra measured at different excitation wavelengths for the sample passivated at $T_{\text{pass}} = 750$ °C. For clarity, only the main $^5D_4 \rightarrow ^7F_5$ transition has been shown in Fig. 2(a). In the next step, such maps were used to calculate PLE spectra. This was achieved by integrating the main $^5D_4 \rightarrow ^7F_5$ emission band of Tb$^{3+}$ separately for each excitation wavelength. In general, this method allows one to obtain a better signal-to-noise ratio in comparison with the traditional PLE measurements, where the emission intensity is only monitored at a fixed wavelength.

Figure 2(b) shows the PLE spectra obtained for the unpassivated sample ($T_{\text{pass}} = 20$ °C) and for the sample passivated at $T_{\text{pass}} = 750$ °C. For both samples, Fig. 2(b) reveals broad absorption bands with line shapes resembling Gaussians. In general, the absorption band is centered at around 233 nm (5.3 eV), and it does not seem to shift with $T_{\text{pass}}$ within the limits of experimental accuracy. Also, the full width at half maximum (FWHM) appears not to depend on $T_{\text{pass}}$ with a constant value of approximately 88 nm ($\sim 2$ eV). Importantly, since the absorption band does not shift with $T_{\text{pass}}$, it is justified to measure PL spectra using the same excitation wavelength for all samples. It should also be mentioned that at first sight, the absorption band maximum at 5.3 eV suggests a direct excitation mechanism of Tb$^{3+}$ ions, since this value is close to typical values reported for $4f \rightarrow 5d$ transition of Tb$^{3+}$ ions in different host materials.$^{23}$ On the other hand, the obtained FWHM is 4–5 times larger than the typical FWHM of $f \rightarrow d$ transitions in RE ions.$^{24,25}$

suggesting another excitation mechanism or the presence of several excitation channels. Since there are many different defect centers in SRSO,$^{26}$ with absorption bands located in the UV range, an indirect defects-mediated excitation of Tb$^{3+}$ is also possible. As a matter of fact, our earlier research$^{26}$ on the excitation mechanism of Tb$^{3+}$ ions in SRSO has shown that a broad absorption band at around 240 nm ($\sim 5.2$ eV) is present even for undoped SRSO films. This result strongly indicates that the main excitation mechanism of Tb$^{3+}$ is indirect, e.g., Tb$^{3+}$ ions could be excited in a non-radiative energy transfer process, being coupled to some kind of optically active defect centers. Interestingly, since the Tb$^{3+}$ concentration in the investigated samples is high, it is also possible that direct and indirect excitations occur simultaneously, but even then the indirect excitation mechanism appears to be dominant. If this is the case, it seems that the particular type of defect centers responsible for indirect excitation of Tb$^{3+}$ is not being passivated by hydrogen since neither peak position nor FWHM of the absorption band changes as a function of $T_{\text{pass}}$. A more detailed discussion of the excitation mechanism was given in our previous paper,$^{26}$ where we suggested that there are at least two types of Tb-coupled defect centers in SRSO, one of them being responsible for indirect excitation of Tb$^{3+}$ and the other one responsible for non-radiative recombination. Since later in this paper we focus only on non-radiative recombination, a more detailed knowledge of
the excitation mechanism is not important. Nevertheless, for clarity of discussion, these two types of defect centers should be differentiated.

Figure 3 shows the PL decay curves obtained for the unpassivated sample ($T_{\text{pass}} = 20^\circ\text{C}$) and for the sample passivated at $T_{\text{pass}} = 750^\circ\text{C}$. In this study, all decay traces were measured at the emission wavelength of 545 nm, corresponding to the main $^5\text{D}_4 \rightarrow ^7\text{F}_5$ transition of Tb$^{3+}$ ions. For the purpose of sample comparison, the decay curves shown in Fig. 3 were normalized to the same initial intensity. Here, it should be emphasized that the initial intensity of PL decay was found to depend on $T_{\text{pass}}$, but this important observation will be discussed later.

As it can be seen in Fig. 3, the obtained decay curves are significantly non-exponential. It is noteworthy that this kind of decay is characteristic for disordered systems with a complicated relaxation mechanism, such as non-radiative relaxation of an individual luminescence center via a number of parallel channels. In this case, the average PL decay time ($\tau_{\text{PL}}$) was calculated for each sample using the following equation:

$$\tau_{\text{PL}} = \int I_{\text{PL}}(t) dt / I_{\text{PL}}(t = 0),$$

where $I_{\text{PL}}(t)$ denotes PL intensity measured at time $t$. Here, it is worth mentioning that according to Eq. (1), $\tau_{\text{PL}}$ is the average amount of time an emitter spends in the excited state. The inset of Fig. 3 displays the calculated PL decay times as a function of $T_{\text{pass}}$. Clearly, the PL decay time gradually increases with $T_{\text{pass}}$ by up to a factor of 1.4, as compared to the unpassivated sample. It is worth mentioning that this result is substantially different from the observations on Er-doped SRSO films, wherein no change in PL decay time was observed upon passivation for the Er$^{3+}$ emission at 1535 nm.

**IV. DISCUSSION**

In Sec. III, it was shown for Tb$^{3+}$ ions that both PL intensity and PL decay time increase as a function of $T_{\text{pass}}$. These experimental observations can be explained by the saturation of non-radiative recombination defect centers with hydrogen upon passivation at elevated temperatures. Indeed, in the case of SRSO:Er films, electron spin resonance measurements have shown that annealing in an atmosphere containing hydrogen is an effective way of passivating, e.g., P$_5^-$type defects (silicon dangling bonds). Since these defects can act as non-radiative de-excitation channels for RE ions, passivation of SRSO reduces non-radiative recombination, thereby enhancing emission intensity and PL decay time simultaneously.

Based on the experimental results presented in this paper, it is possible to discuss in more detail the effect of passivation on the non-radiative recombination process. First, let us emphasize that the radiative lifetime of the $^5\text{D}_4$ state of Tb$^{3+}$ ion is very long, at least of the order of several milliseconds for most materials. The non-radiative de-excitation process occurs on a shorter time scale depending on the defect structure of the material. Now, if the excited $^5\text{D}_4$ state of Tb$^{3+}$ ion interacts with non-radiative recombination centers, the non-radiative de-excitation process competes with the emission of photons. In the case of strong coupling to defects, the rate of non-radiative recombination is much higher than the rate of radiative recombination, rendering Tb$^{3+}$ optically “dark” (undetectable in PL spectra or PL decay measurements). On the other hand, if the non-radiative coupling between Tb$^{3+}$ ions and defects is intermediate/weak, the Tb$^{3+}$ emission takes place but with a decreased QE. Notably, there are different opinions concerning the actual strength of non-radiative coupling between defects and Er$^{3+}$ ions in SRSO. First, it was suggested by Shin et al. that hydrogenation of SRSO:Er films enhances the Er$^{3+}$ photoluminescence intensity by increasing the number of optically active ("bright") Er$^{3+}$ ions. In other words, according to Shin, a fraction of Er$^{3+}$ ions was optically “dark” before passivation, which implies strong non-radiative coupling to defects. Second, it was proposed by Savchyn et al. that the non-radiative coupling between Er$^{3+}$ and defects affects excited states of Er$^{3+}$ above the $^4\text{I}_{13/2}$ level and is accompanied by a reduced excitation rate of the emitting $^4\text{I}_{15/2}$ level. In this situation, Er$^{3+}$ ions remain optically active, emitting photons with a decreased QE, which indicates intermediate/weak non-radiative coupling. It should also be underlined that in both of the above mentioned studies, it was observed that the $^4\text{I}_{13/2} \rightarrow ^4\text{I}_{15/2}$ emission intensity increases upon passivation without a simultaneous change of the PL decay time, which is most probably a result of non-radiative recombination taking place above the emitting $^4\text{I}_{13/2}$ level. From this point of view, Tb$^{3+}$ is a better candidate to study the non-radiative process, since the energy of the $^5\text{D}_4 \rightarrow ^7\text{F}_5$ emission is much higher (2.27 eV) in comparison with the $^4\text{I}_{13/2} \rightarrow ^4\text{I}_{15/2}$ emission of Er$^{3+}$ (0.81 eV), allowing one to directly observe the non-radiative interaction between the emitting $^5\text{D}_4$ level and defect states.

In general, it is possible to define whether the non-radiative coupling is strong or intermediate/weak. First of
all, let us consider the simplest case of single-exponential PL decay

$$I_{PL}(t) = CN^*k_{RAD}\exp(-t/\tau_{PL}),$$

(2)

where C is a constant determined by the collection efficiency of the detection system, \(N^*\) is the number of excited emitters at \(t=0\), \(k_{RAD}\) is the radiative recombination rate, and \(\tau_{PL}\) is the PL decay time. The initial intensity of such PL decay is simply given by

$$I_{PL}(t = 0) = CN^*k_{RAD}.$$  

(3)

This equation implies that in the case of strong non-radiative coupling, where emitters are either “dark” (coupled) or “bright” (not coupled, unaffected), the initial intensity of the PL decay curve increases with the number of emitting species \(N^*\), under the assumption that \(k_{RAD}\) does not change. Furthermore, in the case of PL decay given by Eq. (2), the number of emitted photons is given by

$$\int_0^\infty I_{PL}(t)dt = CN^*k_{RAD}\tau_{PL}.$$  

(4)

Here, the non-radiative processes are reflected in \(\tau_{PL}\) because \(\tau_{PL} = 1/(k_{NR} + k_{RAD})\), where \(k_{NR}\) denotes the non-radiative recombination rate. Based on Eq. (4), the non-radiative coupling to defect states can be regarded as intermediate/weak when the number of emitted photons increases linearly as a function of \(\tau_{PL}\), assuming that \(N^*\) and \(k_{RAD}\) do not change. In this way, the strong non-radiative coupling is manifested solely in the initial intensity of PL decay, which is proportional to the number of active emitters \(N^*\), whereas the intermediate/weak non-radiative coupling reflects solely in \(\tau_{PL}\), which is related to the emission QE (QE = \(k_{RAD}\tau_{PL}\)).

Figure 4 shows the average PL decay time of the \(5D_4 \rightarrow 7F_3\) transition as a function of the integrated PL decay. The solid arrow in Fig. 4 points in the direction of increasing \(T_{pass}\). It can be seen in Fig. 4 that upon passivation the average PL decay time increases with the number of emitted photons. This result directly proves that passivation reduces the non-radiative recombination rate, resulting in a longer Tb\(^{3+}\) lifetime in the \(5D_4\) excited state and enhanced emission of photons from those Tb\(^{3+}\) ions which were optically active before passivation. Apparently, some fraction of Tb\(^{3+}\) ions is intermediate/weakly coupled to non-radiative defects, and a competition between radiative and non-radiative recombination is possible for these emitters. For such Tb\(^{3+}\) ions, passivation causes an increase in the Tb\(^{3+}\) emission QE and more photons are emitted. Importantly, according to Eq. (4), the number of emitted photons should increase proportionally to \(\tau_{PL}\), assuming that \(N^*\) and \(k_{RAD}\) do not change. However, according to Fig. 4, the number of emitted photons increases by a factor of 1.90 upon passivation, while \(\tau_{PL}\) increases by only a factor of 1.40. Apparently, the observed increase of the number of emitted photons is only partially caused by the increase of \(\tau_{PL}\).

Next, we consider the effect of passivation on the number of optically active Tb\(^{3+}\) emitters. The inset of Fig. 4 shows the initial intensity of PL decay as a function of \(T_{pass}\). It can be seen that the initial intensity of PL decay increases with \(T_{pass}\) by up to a factor of 1.36, as compared to the unpassivated sample. Based on Eq. (3), this effect can be attributed to the increasing number of optically active Tb\(^{3+}\) emitters upon passivation. Apparently, the saturation of defects with hydrogen activates a fraction of those Tb\(^{3+}\) ions which were optically “dark” before passivation. Here, we deal with strong non-radiative coupling, where \(k_{NR} \gg k_{RAD}\). Importantly, we assumed that the radiative recombination rate does not change upon passivation. This assumption is fully justified since hydrogenation is a way of passivating defects without causing undue structural changes in the material itself.

As discussed in our previous paper, a more substantial modification of the SRSO structure is required to change the radiative recombination rate of Tb\(^{3+}\) in SRSO. It is also worth mentioning that according to a recent study of SRSO:Er films, the fraction of optically active Er\(^{3+}\) ions in SRSO does not exceed 10%. Also from this point of view, optical activation of “dark” ions is the most probable explanation of our observations.

So far, it has been shown that the number of emitted photons increases upon passivation as a result of reduced non-radiative recombination, which is manifested by two effects: (1) longer Tb\(^{3+}\) lifetime in the \(5D_4\) excited state and (2) optical activation of “dark” ions. According to our results, \(\tau_{PL}\) and \(N^*\) increase upon passivation by up to a factor of 1.40 and 1.36, respectively. Based on Eq. (4), we expect that the number of emitted photons should increase by up to a factor of \(1.40 \times 1.36 \approx 1.90\) upon passivation. Indeed, according to Fig. 4, the latter number (1.90) was obtained experimentally within the limits of experimental accuracy. Clearly, the two effects listed above have almost the
same contribution to the observed increase of the number of emitted photons.

Finally, based on the experimental results presented here, as well as those presented in the literature, we suggest that the physical interaction responsible for the non-radiative coupling between Tb$^{3+}$ ions and defects is probably Förster energy transfer. For such interaction, the rate of non-radiative recombination $k_{NR}$ depends on the distance $R$ between the excited Tb$^{3+}$ ion and a defect, e.g., $k_{NR}(R) \propto R^{-6}$ for a dipole-dipole Förster mechanism. Since this is a strong dependency, we expect that a fraction of Tb$^{3+}$ ions located very close to defects will be optically “dark.” On the other hand, we also expect that another fraction of Tb$^{3+}$ ions, located farther away from the defects, will be optically active. As shown previously, these two conditions are satisfied in the present case. Moreover, since Förster energy transfer is a resonant process, we also expect that the non-radiative coupling should involve only some particular energy levels of RE$^{3+}$ ions. In other words, a threshold should be observed for the lowest emitting energy state, below which the non-radiative interaction is not present. Indeed, as discussed by Savchyn et al., non-radiative coupling does not involve the Er$^{3+}$ first excited state $|5I_{13/2}\rangle$. This is in contrast to our results, where the Tb$^{3+}$ first excited state $|5D_{4}\rangle$ was affected by non-radiative interaction. The resonant nature of non-radiative coupling seems to be a probable explanation for these observations. More detailed research is necessary to validate this hypothesis.

V. CONCLUSIONS

In this work, terbium-doped silicon-rich silicon oxide films were obtained by means of plasma enhanced chemical vapor deposition. The effect of hydrogen passivation at different temperatures on non-radiative recombination was investigated for the first Tb$^{3+}$ excited state. We have found that passivation enhances the photoluminescence intensity and increases the photoluminescence decay time for the main $5D_{4} \rightarrow 7F_{2}$ transition of Tb$^{3+}$ ions. These experimental observations were explained by saturation of non-radiative recombination defect centers with hydrogen upon passivation at elevated temperatures. Moreover, it has been shown that the number of emitted photons increases upon passivation as a result of two effects: (1) longer Tb$^{3+}$ lifetime in the $5D_{4}$ excited state and (2) increasing number of optically active Tb$^{3+}$ emitters. Finally, it has been shown that the contribution of these two effects is equally important from the point of view of emission properties.

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