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Microscopic evidence for role of oxygen in luminescence of Er^{3+} ions in Si: Two-color and pump-probe spectroscopy

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Influence of oxygen on optical activity and thermal stability of the 1.5 μm emission of Er^{3+} ions in Si is well documented. Here we support this predominantly phenomenological notion with microscopic evidence. Using two-color and pump-probe spectroscopy with a free-electron laser we establish a direct link between the 1.5 μm emission from Er^{3+} ions and the 9 μm vibrational band of interstitial oxygen in Si. We show that: i) intensity of the 1.5 μm emission quenches when the 9 μm oxygen vibration is resonantly excited, and ii) the lifetime of the 9 μm vibrational band is affected by Er presence. Both findings evidence microscopic relation of interstitial oxygen and Er and their close proximity in Si matrix.

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I. INTRODUCTION

Among many new approaches to silicon photonics, optical doping with Er continues to draw attention as a way to improve optical properties of crystalline Si (*c*-Si). This follows from the conceptual simplicity as well as from the fact that doping with Er, realized by implantation or during growth, is fully complementary metal-oxide-superconductor (CMOS) compatible. This distinguishes then the "Er approach" from other possible routes toward enhancement of optical activity of Si, as enabled by, e.g., introduction of Si nanocrystals[1] or formation of Er silicides, among others. Since the idea has originally been introduced,[2] large progress was made in both material development and in understanding of physical mechanisms governing excitation and deexcitation of Er^{3+} ions embedded in crystalline Si matrix.[3–9] Also, practical devices have been demonstrated.[10] Recently, preferential formation of a particular type of Er-related optical center has been shown:[11] its homogeneous and ultranarrow linewidth renewed hopes for realization of optical gain in *c*-Si:Er. Nevertheless, important problems remain, with the most prominent one being the poor thermal stability of Er emission. In spite of the fact that a large energy difference exists between the emitted photon energy and the Si bandgap—in excess of 300 meV—this appears insufficient to assure intense emission at room temperature; intensity of photo- and electroluminescence of *c*-Si:Er exhibits strong thermal quenching already above liquid-nitrogen temperature. Theoretical explanation of this effect has been given in terms of thermally activated, phonon-assisted excitation reversal—the so-called *back-transfer* process[12] whose efficiency is enhanced due to the long radiative lifetime of the excited state of Er^{3+} ion. Also lower efficiency of Er excitation process at higher temperatures, due to Auger process of energy transfer to free carriers thermally liberated from shallow traps and dissociation of bound exciton state mediating the excitation, have been pointed out as possible reasons for thermal quenching. It is clear that the problem of thermal stability of Er emission has to be solved in order to enable efficient room-temperature photonics based on *c*-Si:Er. For that, comprehensive understanding of energy-transfer processes is necessary.

It is well established that thermal stability and the level of optical activity (i.e., percentage of dopants contributing to photon emission) of Er in *c*-Si can be considerably enhanced by codoping with oxygen.[3] While microscopic origin of the oxygen influence is not understood in detail, an experimentally developed "rule of thumb" states that the best optical properties are obtained for materials where concentration of oxygen is by an order of magnitude higher than that of Er. Microscopic models of optically-active Er-related centers in Si postulate multiple O atoms in the direct surrounding of Er^{3+} ions. However, evidence to support that comes only from indirect measurements. In particular, the model presented in Ref. 13 featured up to eight oxygen atoms in the usual puckered bond-centered sites surrounding an Er^{3+} ion occupying a high-symmetry interstitial position. In that case, participation of O was concluded from secondary ion mass spectroscopy (SIMS) measurements[14] which revealed oxygen concentration in the Er-doped layer to be almost an order of magnitude higher than the 10^{18} cm^{-3} level characteristic for Czochralski-grown Si. Also, extended x-ray absorption fine structures (EXAFS) experiments on Er-doped oxygen-rich Si reported that oxygen atoms are located in direct vicinity of Er—on average six per one Er^{3+} ion.[15] However, it is only fair to point out that EXAFS measurement cannot distinguish between optically-active and nonactive Er dopants. Since usually only a minor part, of an order of 1%, of Er content contributes to photon emission, EXAFS results cannot provide conclusive evidence on the role of oxygen in optical properties of Er. Such information is obtained in the current study, where we show direct relation between the 1.5 μm emission due to ${}^4I_{13/2} \rightarrow {}^4I_{15/2}$ radiative recombination of Er^{3+} and the 9 μm vibrational mode of interstitial oxygen. In that way we establish a microscopic link between optical activity of Er and presence of O.

II. EXPERIMENTAL

Investigations have been conducted for a Si/Si:Er nanolayer structure of 400 alternating layers of Si and Si:Er, of

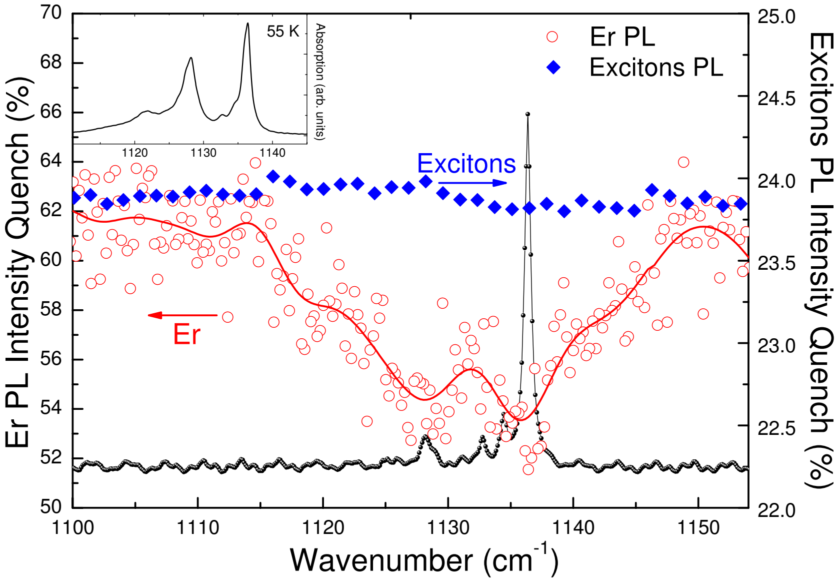

FIG. 1. (Color online) Results of 2C experiment for Er- and exciton-related PL. For comparison, the IR absorption spectrum at $T=4$ K (black curve) and at $T=55$ K (in the inset) are given.

1.7 and 2.3 nm thickness, respectively, stacked along the ⟨100⟩ growth direction. The multinanolayer structure was grown by sublimation molecular beam epitaxy (MBE) (at 560 °C) on a p-type Cz-Si substrate. Er and O concentrations have been determined by SIMS as 3.5×10^{18} and $1.5-4\times10^{19}$ cm^{-3}, respectively. After the growth procedure, the sample has been annealed at 800 °C for 30 min for optical activation of Er^{3+} ions. Previous research has shown that in that way one type of Er-related optical center is preferentially formed.[11] Therefore the multilayer material is particularly suitable for investigations of energy-transfer mechanisms, as inhomogeneous broadening of energy levels determining optical and electrical activity of Er is very significantly reduced.[16] Absorption measurements have been performed with a Fourier-Transform Infrared (FT-IR) spectrometer, at resolution of 0.12 cm^{-1}. Two-color (2C) experiments and pump-probe measurements have been conducted at the FELIX free-electron laser (FEL) facility in Nieuwegein, The Netherlands. For 2C spectroscopy, a pulsed Nd:YAG source operating at 532 nm (80 ps pulse duration and 5 Hz repetition frequency) and a FEL (~7 μs macropulses, each featuring a 1 GHz train of ~ps micropulses) operating in the midinfrared were used. The two sources were mutually synchronized so that the time delay between them could be set at will. The luminescence was resolved with a TRIAX 320 equipped with a 900 grooves/mm grating blazed at 2 μm and detected by an infrared Hamamatsu photomultiplier with a 30 μs response time. Lifetimes of the vibrational modes of oxygen were estimated by pump-probe experiments with FEL (the micropulse repetition rate operating at 25 MHz).[17] In this experimental configuration, following the pump-probe sequence also a third pulse, identical to the probe, was used as a reference. The transmitted probe beam was monitored by a MCT-22-1.0 Bfi Optilas detector.[18] All experiments were conducted with a sample placed in a cold finger helium cryostat.

III. EXPERIMENTAL RESULTS

In the first experiment we looked at 2C excitation of the structure. Following the goal of the project—the microscopic relation between optically-active Er^{3+} and oxygen—we investigated behavior of the 1.5 μm Er-related emission band upon activation of vibrational mode of interstitial oxygen. In this case, the investigated structure is excited by a band-to-band pump pulse from the Nd:YAG laser, and then, with a tunable delay, by a midinfrared FEL pulse, whose wavelength is scanned across the 9 μm vibrational band of oxygen. This characteristic absorption band (1136 cm^{-1}) is due to Si-O-Si vibrational mode and is induced by interstitial oxygen atoms present in the sample—in the Er-doped multinanolayer structure and in the substrate. The result of the 2C experiment is depicted in Fig. 1. On the vertical axis we plot the quenching ratio Q of Er-related PL intensity induced by the pump pulse with and without the subsequent FEL pulse: $Q=I_{\mathrm{FEL}}/I_0$. The PL intensity is measured at the most intense line of the Er-related emission spectrum. Figure 1 shows the quenching ratio Q as a function of the FEL wavelength. We note that in addition to an overall reduction of the Er emission for the whole depicted spectral range of FEL, a clear resonant feature is observed for FEL wavelength around 8.8 μm. This additional quenching clearly coincides with the oxygen-related vibrational absorption band—for an easy comparison, the absorption spectrum of the sample recorded in a separate experiment is also shown (black curve). We point out that by its very nature, the 2C experiment monitoring emission from Er^{3+} ions is selectively sensitive only to the optically-active fraction of Er dopants, regardless of the fact that these constitute only a small percentage of the total Er content.[19]

The coincidence between the absorption and 2C spectra becomes quite striking if we consider temperature dependence of the 9 μm vibrational band of interstitial oxygen. Vibrational modes of oxygen in silicon feature three different modes: symmetric ν_1, transverse ν_2, and antisymmetric mode ν_3.[20,21] The 9 μm band presents three main peaks at 1136 cm^{-1} (antisymmetric mode ν_3), 1128 and 1121.5 cm^{-1} (nonlinear combination of transverse ν_2 and antisymmetric ν_3 modes), with their mutual intensity ratio being strongly temperature dependent. At $T=4$ K the 1136 cm^{-1} band dominates the spectrum, with very small amplitude of the

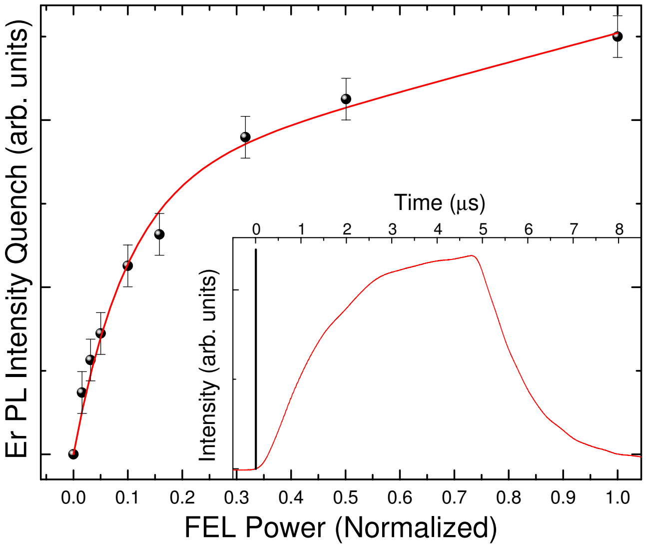

FIG. 2. (Color online) Photon flux dependence of the Er PL intensity quench for the FEL wavelength of $\lambda_{FEL}=8.8$ μm. The line is a guide to the eye. In the inset, the pulse sequence for delay time $\Delta t=0$ is depicted.

1128 cm^{-1} band and the 1121.5 cm^{-1} being practically absent. The FTIR spectrum depicted in Fig. 1 has been taken around $T\approx 4$ K; consequently the 1128 cm^{-1} mode is very small. The relative intensities of the low energy bands with respect to the 1136 cm^{-1} band increase with temperature, and for $T>20$ K, all three bands are present. Inset to Fig. 1 shows the FTIR spectrum of the oxygen vibrational band for $T=55$ K. When we take into account that the linewidth of the FEL used in the 2C experiment is much larger than the resolution of the FTIR spectrum, we conclude that the spectral dependence of Er PL quenching obtained in the 2C experiment reflects indeed the 9 μm vibrational mode at a higher temperature. This implies that the actual temperature of the sample in the 2C experiment is in the 20–40 K range. This significant rise of the temperature of the sample when FELIX beam is employed is independently confirmed by quenching of excitonic PL band at 1162 nm—see Fig. 1. In this case, the result differs from that obtained for Er PL: while PL intensity due to exciton recombination is also reduced upon FEL illumination, this effect is independent of FEL wavelength in the investigated range. We therefore conclude that illumination with FEL raises temperature of the sample, which leads to reduction of PL bands related to Er and excitons. On top of this background effect, strong reduction of Er-related PL appears upon activation of local vibrational modes of interstitial oxygen. In that way, "resonant" quenching of the 1.5 μm Er-related emission band takes place, whose spectral dependence reflects the vibrational band of interstitial oxygen.

Further insight into the mechanism of this resonant quenching is provided by investigation of its dependence on the photon flux. Figure 2 shows the magnitude of the resonant quenching effect Q_{FEL} as a function of FEL photon flux ϕ, with the wavelength of the mid-IR beam set to resonance with the oxygen band ($\lambda_{FEL}=8.80$ μm). The saturating character of the flux dependence reflects the limited number/density of available phonon modes as will be further confirmed by the pump-probe measurements discussed later.

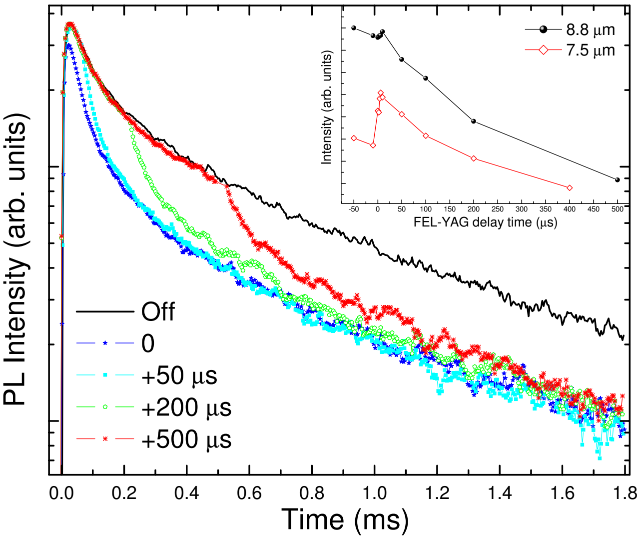

FIG. 3. (Color online) Delay time dependence of the resonant quenching of Er-related PL. In the inset, the delay-time dependence of the quenching effect $(1-I_{FEL}/I_0)$ for the large range is given for two FEL wavelengths of 7.5 and 8.8 μm.

Figure 3 illustrates temporal characteristics of the resonant quenching of Er PL induced by activation of Si-O vibrational mode. It shows the magnitude of the resonant quench of Er PL, with FEL set to $\lambda=8.80$ μm, as a function of delay time Δt between the pump (Nd:YAG) and the probe (FEL) pulses. We note two important characteristics of the quenching effect. First, the reduction of PL intensity commences simultaneously with the onset of the FEL pulse. Second, the quenching level is independent of the delay time, being determined exclusively by the number of IR photons in the FEL pulse. This confirms that FEL introduces a PL quenching process with the specific rate, and therefore the quench magnitude is directly proportional to number (concentration) of excited Er^{3+} ions available at that particular moment. The experimentally recorded decrease rate ($\tau_Q \approx 60$ μs) is clearly longer than the detector response time of $\tau_{PMT}\approx 30$ μs and therefore reflects the additional nonradiative decay of excited Er induced upon FEL illumination.

IV. DISCUSSION

Now we consider the mechanism for erbium luminescence quenching. As discussed in the past,[5] reduction of Er luminescence can be accomplished in two ways—by promoting nonradiative recombination of excited Er^{3+} ions (e.g., by Auger energy transfer to carriers) and/or by reducing efficiency of Er excitation (e.g., Auger recombination of the intermediate excitation stage). In the current situation, as depicted in Fig. 3, the FEL "quenching" pulse is applied with the delay time $\Delta t \geq 0$, and, as mentioned in the previous paragraph, identical reduction of Er excitation is observed. Therefore we conclude that only one of the two possible quenching mechanisms is realized, namely by inducing nonradiative recombination of excited Er^{3+}. Moreover, since in case of the smallest delay $\Delta t=0$ both pulses are fired at the same moment—see inset to Fig. 2 for schematic illustration of the $\Delta t=0$ pulse sequence—this implies that the Er excitation proceeds rapidly and is completed before FEL flux reaches a substantial level. In that way the current experiment provides the upper limit for the excitation time of Er in Si: $\tau_{\text{exc}} \lesssim 1 \ \mu\text{s}$.

In order to describe the quenching process, we note that the timescale of the luminescence decay is significantly larger than the duration of a FEL pulse; therefore we can regard this pulse as a δ-shaped source of quenching. In result, the PL decay will be described by the expression:

$$I_{\text{PL}} \sim \exp(-t/\tau)[1 - A\Theta(t-t_0)], \tag{1}$$

where $\Theta(t-t_0)$ is the step function, switched on at the moment t_0, and τ is the lifetime of Er in the excited state. A resonant absorption of FEL power occurs due to generation of oxygen vibrations. If we treat it in the two-level model, considering only the ground and the first-excited state of oxygen vibrations, we get for a number of phonons in the excited state

$$N_p = N_p^0 + \alpha N_0 I_{\text{FEL}} (2\alpha N_0 I_{\text{FEL}} + \tau_p^{-1})^{-1}, \tag{2}$$

where N_p is the population of oxygen phonons, N_p^0 its equilibrium value at the temperature of experiment T_0 (Planck function), τ_p is the lifetime of an oxygen phonon, α is the resonance absorption coefficient of light at the frequency of oxygen vibrations, N_0 the concentration of erbium-oxygen clusters, and I_{FEL} is the intensity of the FEL emission. (The factor 2 takes account of stimulated emission of phonons from the upper level). Absorption of mid-IR radiation leads to strongly nonequilibrium distribution of oxygen phonons: at sufficiently high FEL power the population of upper and lower levels become equal. This is just what can be expected at optical pumping of two-level system. Though, in general, the phonon distribution should not be necessarily described by phonon temperature, we will introduce here an "effective" temperature to simplify the further treatment of the quenching process. According to formula (2) we get

$$T_{\text{eff}} \approx T_0 + \frac{\hbar\omega}{k} \ln \frac{2\alpha I_{\text{FEL}} + \tau_p^{-1}}{\alpha I_{\text{FEL}} + 1}, \tag{3}$$

where ω is the frequency of the oxygen phonon and k is the Boltzmann constant. In Ref. 12, temperature quenching as a result of back-transfer process was quantitatively studied. Using the data of Fig. 5 from this reference we see that the temperature rise from 4 to 150 K (the Debye temperature of the oxygen phonon) can increase the probability of the back-transfer process by a factor ~100. However, at low temperature the lifetime τ is determined primarily by reverse Auger process (de-excitation of Er^{3+} ions by free electrons) and the real quenching ratio will be significantly less: it can be estimated from the temperature dependence of erbium luminescence intensity. Therefore, the quenching of ~20% as observed in this study upon activation of Si-O-Si vibrational mode is a quite reasonable value.

Consequently, we postulate to attribute the observed reduction of Er-related PL to "thermal quenching"(being a combination of thermally induced back-transfer process[12] and optically-induced dissociation of excitons when pump and probe pulses overlap in time) appearing due to the heating effect induced very locally, in direct vicinity of Er emitters. We also point out that contribution of other mechanisms, and in particular of optically-induced back transfer and optical ionization of Er-related donor level, can be excluded on basis of experimental evidence at hand. For clarity, this is shortly discussed below.

1) *The optically-induced excitation reversal "the back-transfer" process*: Some time ago we have demonstrated that the back transfer process can be observed in Yb-doped InP upon mid-IR illumination with FEL.[22] In this case the effect could be induced for photon energies higher than a threshold value of 140 meV, implying that there is a certain minimal energy necessary to transfer the energy from the RE ion back to the matrix. Therefore the observed wavelength dependence of the optically-induced PL quenching had a steplike character. This is clearly different from the resonant quenching process reported in the current study.

2) *Direct optical ionization of the Er-related donor*: Ionization of the Er donor level can lower the Er-related emission in two ways—(i) by disrupting the excitation channel and (ii) by providing free carriers and inducing Auger effect. The first of these possibilities has already been dismissed in the earlier discussion due to the fact that coincidence of pump and probe pulses is not the necessary condition for the observed PL quenching. In addition, we note here that also the latter possibility—the Auger quenching due to optical ionization of the Er donor—can be ruled out. This is because (i) in the Si/Si:Er multinanolayer sample this level has been established as $E_D = 218 \ \text{meV}$,[16] and (ii) ionization of an electrical level in the band gap should have a characteristic spectral dependence, with a sharp onset at $h\nu = E_D$, and long tail extending to higher photon energies, again in contrast with the experimental data.

The thermal origin of the observed PL intensity reduction finds further confirmation from the dependence of the quench magnitude on delay between the pump (Nd:YAG) and probe (FEL) pulses. This is illustrated in the inset to Fig. 3 for two wavelengths of FEL: $\lambda_{\text{FEL}} = 8.8 \ \mu\text{m}$, "on resonance," and $\lambda_{\text{FEL}} = 7.5 \ \mu\text{m}$, "off resonance" with the oxygen vibrational band. As can be seen, although the quenching magnitude clearly increases for the probe wavelength resonant with the oxygen vibrational mode, its delay-time dependence is similar for both wavelengths: it appears already for the FEL pulse applied long before the pump $\Delta t < 0$ (up to ~ms range, not shown), then grows as the two pulses approach each other $0 < \Delta t \lesssim 10 \ \mu\text{s}$, shows additional increase when both pulses overlap, and then remains constant for the situation when the band-to-band excitation precedes the FEL pulse $\Delta t > 0$ (i.e., disappears with the decay constant equal to lifetime of Er in the excited state). The fact that a FEL pulse applied before Er excitation can reduce Er PL intensity provides a clear indication as to the thermal origin of this effect. The gradual decrease of the quench, as the mid-IR pulse is applied longer before the Er excitation, illustrates cooling of the sample to the original bath temperature. The resonant effect, appearing for the FEL wavelength corresponding to oxygen vibrational band translates then into the higher amplitude of the heating effect, due to enhanced absorption of photons at that wavelength. At the same time, the fact that the resonant quenching effect appears for Er-related PL and not for exciton PL indicates the very local character of this optically-induced heating, in close vicinity of Er centers. Based on the above discussed evidence and reasoning, we propose that the FEL pulse applied to the sample gives rise to a rapid [~ps (Ref. 23)] increase of temperature of the illuminated layer. This leads to thermal changes (quenching) of emission from all the centers present in that volume—specifically, excitons and Er ions. When the FEL wavelength is in resonance with oxygen vibrational mode, additional energy is absorbed. This increases the local temperature, resulting in additional quench of Er PL, but negligibly increases the temperature of the whole layer, thus leaving the exciton-related PL practically unaltered—see Fig. 1. In that way, the resonant quenching of Er PL upon activation of oxygen vibrational modes evidences the spatial correlation of both dopants.

Interestingly, we point out that the thermally induced quenching of Er PL with the activation energy of ~150 meV (≈9 μm) investigated here is well discussed in the literature and commonly assigned to the back-transfer. In the back-transfer process,[12] de-excitation of an Er^{3+} ion promotes an electron from the valence band to the Er-related donor level. The energy needed to complete this process is $\Delta E = E_g - \epsilon_D - E_{Er}$, where E_g, ϵ_D, and E_{Er} correspond to the Si band gap, donor ionization, and Er excitation energies, respectively. Since in the Si/Si:Er multinanolayer structure, Er^{3+} ions introduce a donor level at 218 meV below the conduction band,[16] then the minimum energy necessary for activation of the back-transfer is $\Delta E \approx 150$ meV. While at high temperature this energy is provided by multiphonon absorption, the direct activation of the antisymmetric vibrational mode of Si-O-Si at low temperature could supply exactly the right amount of energy needed for that process, and being directly in vicinity of Er enhances the probability. A simple argument seems to rule out the process: the characteristic time of the back-transfer process is on the order of μs,[12] while the decay of vibrational modes into lattice modes proceeds with a time of a few picoseconds. The back transfer is then unlikely to take place in view of the difference by orders of magnitude in the probabilities of the two processes. However, the back-transfer process is strongly temperature dependent and its probability enhances dramatically at high temperature. As stated before, generation of vibrational modes in the surrounding of Er ions increases the effective temperature around Er. This could shorten the lifetime of the back transfer process down to the same order of magnitude as the decay of vibrational modes into lattice modes, making the two processes concurrent.

Based on the results if this study, we conclude that oxygen atoms are located in direct vicinity of Er^{3+} ions. Therefore one can expect that the presence of Er will also influence vibrational properties of oxygen. This could manifest itself as a shift (or more likely a broadening) of the 9 μm absorption band and/or a change of its lifetime. In the past, vibrational modes of oxygen have been studied and their lifetime accurately determined. In particular, the lifetime of 11 ps has been found for the 1136 cm^{-1} band.[24] As shown by Kohli *et al.*,[25] this lifetime is extremely sensitive to masses of vibrating atoms, showing strong variation with the isotopic composition of Si. In fact, the energy of the vibrational modes shifts to a lower value as the masses of the atoms involved in the oscillation increase. The lifetime of the vibrational modes in presence of heavier masses of the species increases because their energies fall outside the one-phonon density of states and decay must proceed via multiple phonon emission. Analogously, one could speculate that the presence of heavier Er ions in proximity of the Si-O-Si "oscillator" will reduce its vibrational energy, thus affecting the lifetime. This has not been confirmed in the present study, where we did not observe any energy shift of the oxygen vibrational band. This is most probably due to the fact that Er^{3+} ions take interstitial positions in the Si matrix, and thus do not bond with O atoms, leaving the vibrating Si-O-Si "molecule" unchanged. Their mutual spatial vicinity, though, should influence the time decay of the modes. In order to test that we have measured the decay time of the 9 μm vibrational mode of oxygen in our sample. We note that nonlinearity of absorption due to phonon modes is a required condition to perform pump-probe measurements. Such saturating behavior has indeed been seen already for the earlier described 2C experiment—see Fig. 2. Figure 4 shows a comparison between the lifetime of Si-O-Si modes in investigated multinanolayers structure and in Er-free oxygen-rich Si. As can be concluded, a clear difference appears, with the lifetime of the 1136 cm^{-1} mode increasing to ~17 ps, against the 11 ps for the Er-free material. (We point out that in the present case the measured vibrational lifetime represents superposed contribution of all O dopants present in the whole sample, i.e., in the Er-doped layer but also in the substrate.)

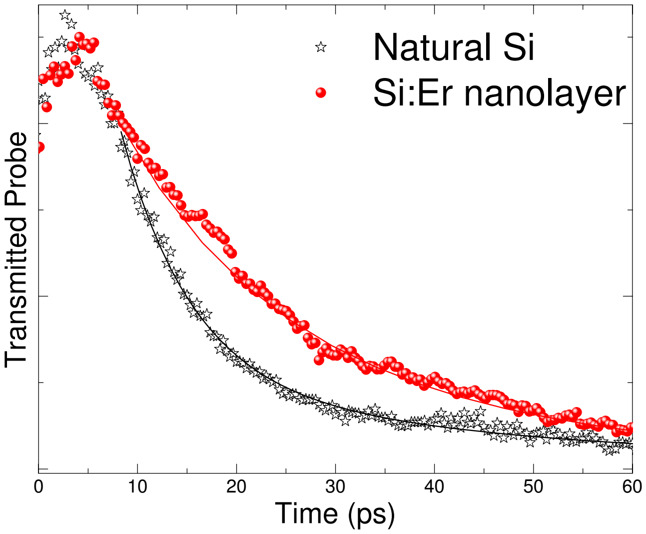

FIG. 4. (Color online) Pump-probe experiment comparing dynamics of the Si-O-Si local vibrational mode in the investigated Si:Er structure and in Er-free O-rich c-Si.

V. CONCLUSIONS

The current research establishes a direct microscopic link between intensity and thermal stability of emission of Er^{3+} in

Si and oxygen doping. Based on the combined results obtained in two-color spectroscopy and IR absorption, we postulate that the ~150 meV activation energy, commonly observed to govern thermal stability of Er emission, corresponds to the Si-O-Si vibrational mode whose activation increases the effective temperature of the excited Er^{3+} ions promoting in this way its nonradiative recombination by the back-transfer process. The close proximity of Er and O dopants in Si matrix is further confirmed by the perturbation of the lifetime of the 9 μm vibrational mode of interstitial oxygen in Si:Er.

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