

1.534 μm Er electroluminescence in the RF magnetron deposited $\text{In}_2\text{O}_3 : \text{Er}$ films on a Si substrate

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Abstract

$\text{In}_2\text{O}_3 : \text{Er}$ films were synthesized on silicon substrates by the RF magnetron sputter deposition. The solid solution $((\text{In}_{1-x}\text{Er}_x)_2\text{O}_3)$ is formed here. The 1.534 μm erbium electroluminescence is observed by the current through the investigated heterostructure substrate- $n\text{-Si}/\text{In}_2\text{O}_3 : \text{Er}$ -film/ITO-contact. The Er excitation model by the electron-hole recombination is proposed as follow. The electron current flows at the indium oxide conduction band. And the hole current flows at the channel in the middle of the $\text{In}_2\text{O}_3 : \text{Er}$ bandgap. The hole channel is formed by the defect state density spreading from the valence band edge into the bandgap. Therefore the electron-hole recombination energy is lower than the indium oxide bandgap and equals to the 1.56 eV. Then the electron-hole recombination excites in resonance the third excited state of $\text{Er}^{3+} {}^4I_{9/2}$ (1.53 eV). Then the nonradiative relaxation to the first excited state ${}^4I_{13/2}$ (0.81 eV) occurs. And finally the 1.534 μm radiative emission into ground state ${}^4I_{15/2}$ occurs.

Keywords

silicon, indium oxide, erbium, thin films, heterostructure, band structure, electrons, holes, electroluminescence

1. Introduction

The inter- and intro-chip data transfer by the optical interconnections is the ongoing perspective step in the development of computing systems [1, 2]. The key challenge here is the integration within chips, i.e. on the silicon (Si) substrate, of the Light Emitting Diodes (LEDs) with the 1.5 μm emission wave length appropriate for minimal optical fiber losses.

A small review of the main approaches to and features of LED on silicon integration is presented in paper [3]. The short list is presented here as follows. The mainstream approach is to transfer of the well-known $A^{\text{III}}B^{\text{V}}$ ($\text{In}_x\text{Ga}_{1-x}\text{As}$) films on a silicon substrate [4–10] either by the bonding technique [4, 5] or by the direct growth on Si by the molecular-beam epitaxy (MBE) technique [4, 6, 7].

The alternative approach is to use the Er^{3+} inneratomic optical transition $^4I_{13/2} \rightarrow ^4I_{15/2}$ with emission wavelength 1.54 μm [11, 12] well suited for minimal optical fiber losses. The straightforward doping of Si with erbium (Er) [13–17] gives inappropriate low quantum efficiency values [14, 16–21]. Nonetheless the Er optical fiber lasers are quite spread for now [22]. But the optical Er excitation by the same $A^{\text{III}}B^{\text{V}}$ LEDs is used in them. There arises a question: is it possible to reach an efficient Er excitation straightforward by the electrical current? The Er and other rare-earth metal electroluminescence (EL) was observed in silicon oxide [23, 24] and other dielectrics, such as Si_3N_4 [25] and TiO_2 [26]. But the Er excitation mechanism was realized by hot electrons in high electric fields. It unlikely gives the practically high quantum efficiency. The Er EL excited by the electron-hole recombination mechanism was demonstrated in some optical transparent conducting oxides, e.g., in ZnO [27] and TiO_2 [28] for example. But the ZnO has the defect related energy levels within the bandgap, which gives the luminescence in the visual range [27]. TiO_2 has the defect related energy levels within the bandgap as well. And Er excites the high levels through it. It gives the Er luminescence in the visual range [28]. The defect related energy levels above are the reason for the low efficiency of the main infrared (IR) emission at the 1.54 μm wavelength. In the previous [3, 29], In_2O_3 :Er is proposed to obtain the Er EL. The Er photoluminescence (PL) in the In_2O_3 was observed earlier [29–31]. And the relative ITO (In_2O_3 : SnO_2) is well known as an optically transparent conducting oxide for electrical contacts [32, 33]. That were the reasons for considering In_2O_3 :Er.

In the previous papers, the barriers for the current injection from the Si substrate into the In_2O_3 film were found [3, 29]. They are equal the 0.14 eV for electrons and 0.3 eV for holes. Together with the Si bandgap $E_{\text{gSi}} = 1.12$ eV [34] the electron-hole recombination energy should be the 1.56 eV. It is quite lower than the In_2O_3 bandgap $E_{\text{gIn}_2\text{O}_3} = 2.69\text{--}2.93$ eV [35, 36]. In [3] it is described by the suggestion of the hole transport channel

related to defect states within the In_2O_3 :Er bandgap. Whether the found current barriers capable to provide injection and transport of electrons and holes toward each other and get Er EL? The aim of this work is to check the hypothesis above.

2. Materials and methods

The In_2O_3 :Er films were deposited on the *n*-type $6 \cdot 10^{14} \text{ cm}^{-3}$ phosphorus-doped Cz-Si (100) wafers with resistivity 7.5 $\Omega \cdot \text{cm}$. The wafers were back-implanted with As^+ 100 keV, $1 \cdot 10^{15} \text{ cm}^{-2}$ and annealed at 1000 $^\circ\text{C}$ during 1 h in the Ar ambient in order to get the back-contact doping.

The Si wafer surface was cleaned by the RCA process [37] before the film deposition. The film deposition was performed by means of the RF magnetron sputtering on the BOC Edwards AUTO 500 system using the In_2O_3 and Er_2O_3 powder compound magnetron target. The Er concentration in the deposited films was found by the X-ray photoelectron spectra (XPS) and equals 1 at.% [29]. The main deposition parameters were slightly different from [3, 29]:

- Ar flow rate 20 sccm, O_2 flow rate 2 sccm;
- working pressure $P = 6 \cdot 10^{-3}$ mbar;
- RF power $W_{\text{RF}} = 100$ Wt;
- supply power frequency 13.56 MHz;
- substrate temperature 100 $^\circ\text{C}$;
- deposition time $t = 5$ h.

The deposited film thickness was 1 μm .

The EL emission was observed through top electric ITO (Indium Tin Oxide [32, 33]) contacts deposited through a $0.7 \times 0.7 \text{ mm}^2$ mask. The back-side contact was produced by the metallic In deposition over the whole back surface area.

The I–V curves for the structures Si/ In_2O_3 :Er/Contact on *n*- and *p*-type conductivity silicon substrates have rectifying patterns as shown in former works [3, 29]. The EL emission was measured in the forward positive bias at the top ITO-contact. The stationary forward current +6 mA, i.e. the $+1.2 \text{ A/cm}^2$ current density, and the voltage drop at the sample +6 V were mainly used for EL measurements.

The EL emission was measured at the Fourier-transform (FT) spectrometer Bruker Vertex 70. The spectrometer is equipped with the near-infrared range Raman module and liquid nitrogen-cooled germanium detector sensitive up to 1.7 μm including 1.54 μm required. The spectral resolution of the FTIR spectrometer is 4 cm^{-1} or 4 Å. The measurements were performed at room temperature. First, the Raman FTIR spectrum from the sample was measured excited with the apparatus neodymium laser at the 1064 nm wavelength and power 10 mWt. Focusing into $300 \times 300 \mu\text{m}^2$ area it gives power density of 14 Wt/ cm^2 . The Stocks part of Raman FTIR spectrum was measured. Second, the current was put through the sample and the EL spectrum was measured.

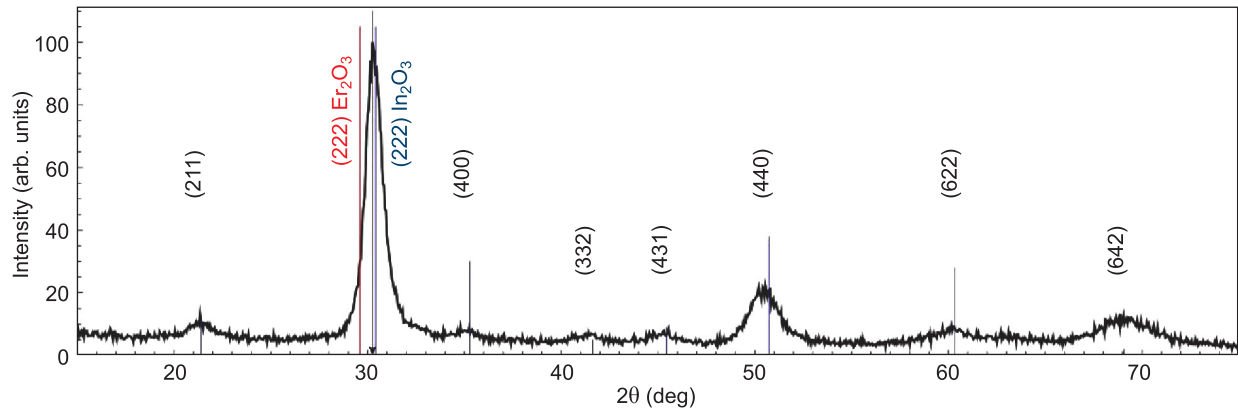


Figure 1. XRD of the $\text{In}_2\text{O}_3:\text{Er}$ film

In addition to the microscopy data of [29] the X-ray diffraction (XRD) measurements were performed here using the X-ray powder diffractometer ARL X'tra (ThermoFisher, Switzerland, Basel) with $\text{Cu-K}\alpha$ source ($\lambda = 0.15418 \text{ nm}$).

3. Experimental results

3.1. X-ray diffraction

As it was shown in [29], the microstructure of the RF magnetron-deposited $\text{Si}/\text{In}_2\text{O}_3:\text{Er}$ films represents an array of $\sim 7\text{--}10 \text{ nm}$ diam. nanowires densely grouped into bunches of discrete $50\text{--}100 \text{ nm}$ diam. nanocolumns. They are spread from the substrate to the film surface at the same small angle to the surface normal. All the nanowires have a single-crystalline body-centered cubic (*bcc*) lattice of typical bixbyite In_2O_3 . But each nanowire has an individual lattice orientation within [29]. In addition to the microscopy analysis above, the XRD analysis is performed here. The films for XRD were deposited with the same parameters as in [3, 29], i.e., with the next difference from the main parameters above: Ar flow 8 sccm, O_2 flow 2 sccm, RF power $W_{\text{RF}} = 120 \text{ W}$. Despite the slight difference of deposition parameters, the film microstructure is the same as above, i.e., nanowires bunched into nanocolumns.

Figure 1 presents the XRD of the sample. The XRD pattern is typical of a nanocrystalline film. There are reflections of only one phase on the pattern. The XRD pattern peaks are close to the *bcc* In_2O_3 *Ia-3* space group (PDF-2 No. 6-416). However the $\text{In}_2\text{O}_3:\text{Er}$ phase peaks are shifted slightly to the left from the In_2O_3 ones. Therefore, the lattice constant of $\text{In}_2\text{O}_3:\text{Er}$ is slightly bigger than that of In_2O_3 . There are the In_2O_3 (222) peak position presented by the blue line and that of Er_2O_3 (PDF-2 No. 8-50) shown by the red line in Fig. 1 for visual perception. Both indium oxide and erbium oxide have the same crystalline structure except the lattice constants are different: the 1.0118 nm for In_2O_3 against the 1.0548 nm for Er_2O_3 . The peaks at the XRD pattern are quite wide

but still single ones, and there are no the double peaks in the XRD pattern. Therefore, the obtained $\text{In}_2\text{O}_3:\text{Er}$ films consist of the solid solution $(\text{In}_{1-x}\text{Er}_x)_2\text{O}_3$. There is no content of impurity phases like In, Er or pure oxides Er_2O_3 , In_2O_3 observed in the XRD pattern.

The lattice constant was found from the experimental XRD pattern in Fig. 1 by the Polycrystal software package [38]. The obtained $\text{In}_2\text{O}_3:\text{Er}$ film has a lattice constant $1.0225 \pm 0.0004 \text{ nm}$. It is a bit larger than the one for In_2O_3 (1.0118 nm), but smaller than the one for Er_2O_3 (1.0548 nm). Since the solid solution $(\text{In}_{1-x}\text{Er}_x)_2\text{O}_3$ is obtained, then the lattice constant should linearly depend on the content. For example, the solid solution $(\text{In}_{0.5}\text{Er}_{0.5})_2\text{O}_3$ (PDF-2 No. 25-1145) has lattice constant 1.0338 nm exactly between In_2O_3 and Er_2O_3 . Other solid solutions $(\text{In}_{1-x}\text{Er}_x)_2\text{O}_3$ with the lattice parameter 1.01213 nm in accordance with Er content 1 at.% are observed in [39]. The linear extrapolation of the lattice constant obtained here presumes the Er_2O_3 content of 25 at.%. It obviously exceeds the Er content in the magnetron target. Moreover, the Er content of 1 at.% was checked for the same films in [29] by the XPS method. Therefore, the oversized lattice constant here is not related to the Er content. One possible reason could be the lattice vacancy defects. They could weaken the interatomic bonds in the lattice, increase the lattice constant and therefore shift the XRD pattern peaks. The other possible reason could be the residual extension strains in the crystalline lattice. They could shift the crystallographic plane distances and, as a result, the XRD pattern peaks. The lattice residual strain is often observed in the different magnetron deposited films [40]. An additional research is required to clarify the question of the oversized lattice constant here.

The blue lines at Fig. 1 indicate the strongest peaks of the theoretical In_2O_3 powder XRD pattern. They correspond, in their position, to the obtained experimental XRD pattern. Except for the maximal peak 222, there is some insignificant disagreement in peak amplitudes between the theoretical and experimental XRD pattern. Therefore, in addition to the complex film morphology (nanowires bunched into nanocolumns [29]), the crystallographic plane orientation in nanowires (texture) is similar mainly to disordered nanocrystals (powders).

However, some experimental reflections, such as 400, 440, 622 and 642, have significantly different amplitude than the theoretical XRD pattern for powders, probably because of the some texture in the obtained films.

Another feature of the $\text{In}_2\text{O}_3:\text{Er}$ film experimental XRD pattern is large width of peaks. It evidences a small coherent scattering area (CSA). CSA could depend of the film nanocrystal size. The CSA size was estimated with the Sheerer formula [41]:

$$D_{hkl} = \frac{n\lambda}{\beta \cos \theta}, \quad (1)$$

where D_{hkl} is the CSA size (in angstroms) perpendicular to the hkl plain; $n \approx 1$ is the nanocrystal shape coefficient; $\lambda = 0.15418 \text{ nm}$ is the apparatus wave length used; θ (in radians) is the scattering angle; β (in radians on 2θ scale) is the peak full width at a half maximum (FWHM). The estimation was performed for each key XRD peak and is presented in the Table 1. The obtained CSA size 6–10 nm fits the nanowire diameter obtained by the SEM [29]. It supports the nanocrystalline structure of the films.

Table 1. The reflexes parameters and CSA size

Reflex	Position (deg)	FWHM (deg)	CSA (nm)
211	21.375	1.629	5.8
222	30.32	1.008	9.8
400	35.08	1.692	5.7
440	50.49	1.788	5.7
642	69.12	3.369	3.3

3.2. Optical measurements

In Fig. 2 is the total combined spectrum of Raman, PL and EL obtained in different excitation conditions: 1) only the apparatus laser with the 1064 nm wavelength and 10 mWt power (black dashed line), 2) the laser together with the direct 6 mA current flow (green solid line),

3) only current without laser (red dashed line). The X -axis is the wavelength in nm units. The Y -axis is the emission intensity in arbitrary units. The upper X -axis gives some key points in energy units eV.

The black dashed line shows the typical silicon spectra obtained under laser excitation only. Since both the deposited $\text{In}_2\text{O}_3:\text{Er}$ film and the top ITO-contact are optically transparent under the exciting laser, then there is strong and narrow Raman peak out of silicon substrate observed at the 1126.3 nm. This Raman peak is due to the inelastic scattering by silicon optical phonons with wave vector $k = 520 \text{ cm}^{-1}$ and energy 64.5 meV [34, 42, 43]. Since the Stocks part of the Raman FTIR spectrum was measured at the energy less than that of the excitation laser, then the observed $k = 520 \text{ cm}^{-1}$ Raman peak is related to optical phonon emission.

Since the excitation laser energy ($\lambda = 1064 \text{ nm}$, $E = 1.17 \text{ eV}$) exceeds the silicon bandgap ($E_g = 1.12 \text{ eV}$ at $T = 300 \text{ K}$ [34]), then there is a wide peak of the silicon PL observed at the combined spectrum. This peak was also observed both for the silicon PL and EL in the literature [44, 45]. The observed silicon PL peak consists of the main peak and a secondary peak. The secondary peak is about one order amplitude less than the main one and is slightly shifted to the infrared direction. Therefore, it looks like a tail out of the main peak toward the IR region (Fig. 2). Since the silicon has the indirect bandgap, then the electron-hole recombination and photon emission requires the participation of one or two phonons for the main and secondary PL peak, accordingly [34]. The first one is the optical phonon with the wave vector within the X -valley of Brillouin zone. The electron conducting valley minimum in silicon is there [34]. So that the first phonon provides the impulse conservation during the electron-hole recombination. The first short-wavelength optical phonon has the energy of 58 meV [34, 42, 43]. Then the main PL peak edge is shifted toward IR at the $E_g - 58 \text{ meV}$ (see Fig. 2). The secondary PL peak edge is shifted toward IR at the $E_g - 58 - 64 \text{ meV}$ (see Fig. 2). The second phonon here is the above-mentioned Raman

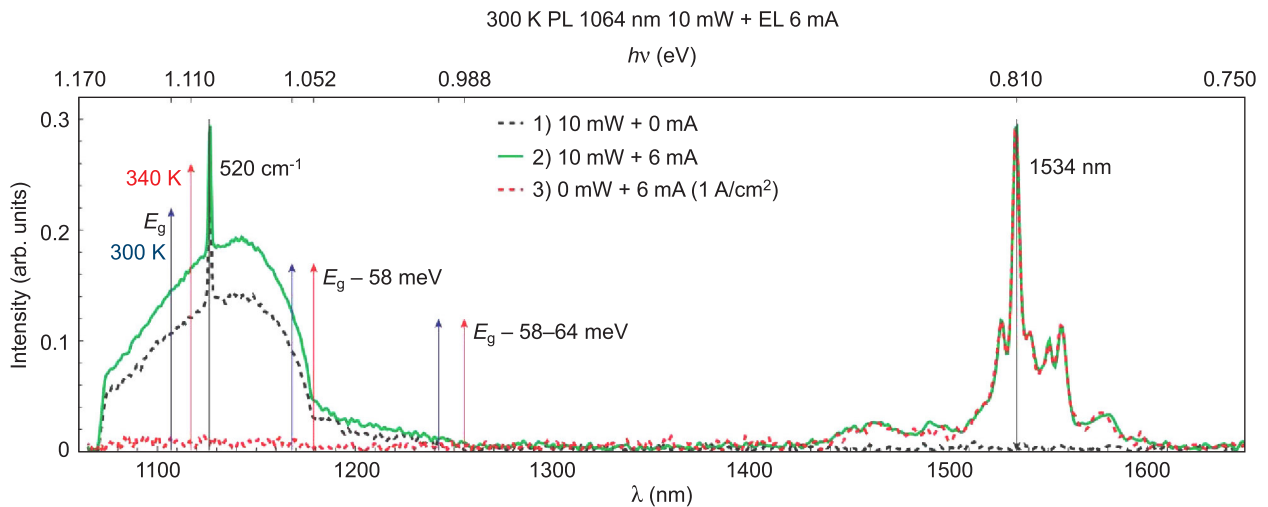


Figure 2. Combined spectrum of Raman, silicon PL and Er EL

long-wavelength optical phonon within the Γ -valley with $k = 520 \text{ cm}^{-1}$ and energy 64.5 meV. The two-phonon process probability and, consequently, secondary PL peak amplitude are about an order of magnitude less than those of one-phonon ones. The silicon bandgap $E_g = 1.12 \text{ eV}$ edge at 300 K and that shifted by one- and two-phonon positions, in comparison with the silicon PL edges, are shown with blue vertical lines in Fig. 2. The observed discrepancy could be accounted for with the sample heating and consequent bandgap reduction [34]. The assumption of the sample temperature $T = 340 \text{ K}$ gives the band gap $E_g = 1.11 \text{ eV}$, and one- and two-phonon shifted positions (Fig. 2, red vertical lines) coincide well with the experimental main and second PL edges. The such heating under the laser excitation used looks quite possible.

Under the stationary direct current flow through the sample the multiplet peak at the 1534 nm wavelength appears at the combined spectrum (see Fig. 2, green solid line). It corresponds to the Er^{3+} ion radiative transition from the first excited state to the ground one: ${}^4I_{13/2} \rightarrow {}^4I_{15/2}$ [11, 12]. The multiplet shape of peak is explained by the Stark splitting of Er^{3+} energy levels in the electric fields of nearby atoms of the In_2O_3 crystal (Fig. 4). The control measurements under the current flow but without excitation laser, were performed as well. Here the multiplet Er EL peak is observed, but Raman and silicon PL peaks are absent (see Fig. 2, red dashed line).

As it is shown at Fig. 2 by a black dashed line and a green solid line, when the direct current is turned on,

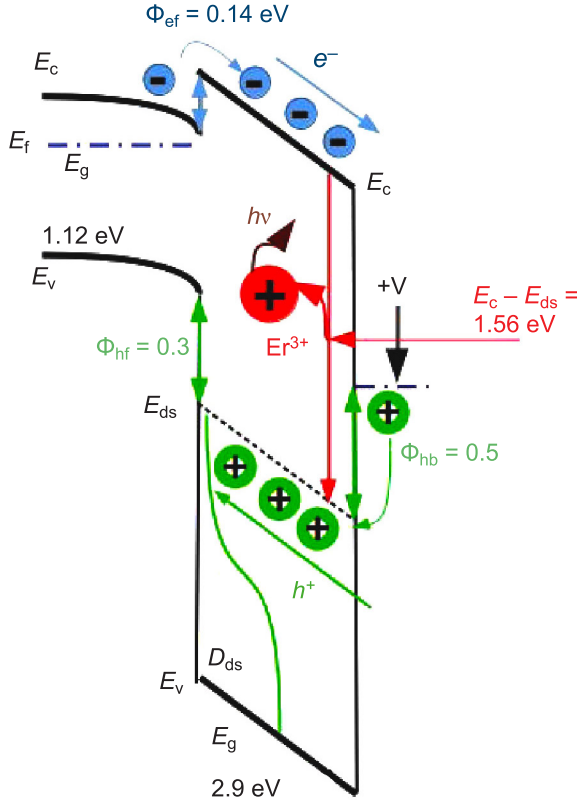


Figure 3. Schematic band diagram, injection barriers, current transport and Er^{3+} excitation mechanism in heterostructure $n\text{-Si}/\text{In}_2\text{O}_3:\text{Er}$ at forward bias $+V$ at the top contact

then the silicon PL peak at the E_g region is increased. As well the backward current reduces the silicon PL. This is not shown at Fig. 2 since the most interesting Er EL was not found at the backward currents in the samples. The silicon PL dependence on the current dependence is explained by the space charge region (SCR) at the silicon surface. In the SCR, the inner electric field separates the electrons and holes, prevents them from recombination and reduces the silicon PL. In the case of direct current flow, the SCR is reduced and the silicon PL is increased. In the case of backward current flow, vice versa, the SCR is increased and the silicon PL is reduced.

As it is shown at Fig. 2, the Er EL peak amplitude is compared with that of the silicon PL one. The silicon PL is excited by the 10 mWt laser. The Er EL is excited by the 6 mA current and about a 6 V voltage drop at the sample, i.e. the 36 mWt electric excitation power is used. The silicon has the indirect bandgap and a small probability of the phonon-assisted photon emission. Therefore, despite the Er EL is observed at room temperature, the Er EL efficiency is still low in the obtained samples.

4. Discussion: the Er EL excitation mechanism

The barriers for the currents injection from the silicon substrate into the RF-magnetron deposited $\text{In}_2\text{O}_3:\text{Er}$ films were obtained in [3, 29] by the temperature dependence measurements of forward and backward currents. In Fig. 3 is the schematic band diagram of the investigated heterostructure in the forward bias ($+V$ at the top contact) together with the electron and hole barriers found in [3, 29] put on it. Here the E_c and E_v is, accordingly, the conduction and valence bands, individual for silicon and erbium-doped indium oxide ($\text{In}_2\text{O}_3:\text{Er}$). E_f is the Fermi level and E_g is the bandgap: $E_{g\text{Si}} = 1.12 \text{ eV}$ for silicon [34] and $E_{g\text{In}_2\text{O}_3} = 2.69\text{--}2.93 \text{ eV}$ for In_2O_3 [35, 36]. The forward barrier for the electron injection from the conduction band of n-silicon into the conduction band of indium oxide was found $\Phi_{ef} = 0.14 \text{ eV}$ (see Fig. 3) [3, 29]. The forward barrier for the hole injection from the valence band of p-Si into indium oxide was also found $\Phi_{hf} = 0.3 \text{ eV}$ [3]. This is a lot less than the heterostructure valence band discontinuity (E_v in Fig. 3). This is explained in [3] by the assumption of the defect state density (D_{ds}) spreading from the indium oxide valence band to the bandgap. This defect state density is formed by the defects of nano-crystalline indium oxide structure and creates the hole current channel inside the bandgap (E_{ds} in Fig. 3) [3].

In this case the electron-hole pairs ($e^- - h^+$) are formed in indium oxide, when the electron is located in the conduction band (E_c) and the hole is in the defect channel (E_{ds}) inside the bandgap. The energy of such an electron-hole pair equals the sum of forward barrier for

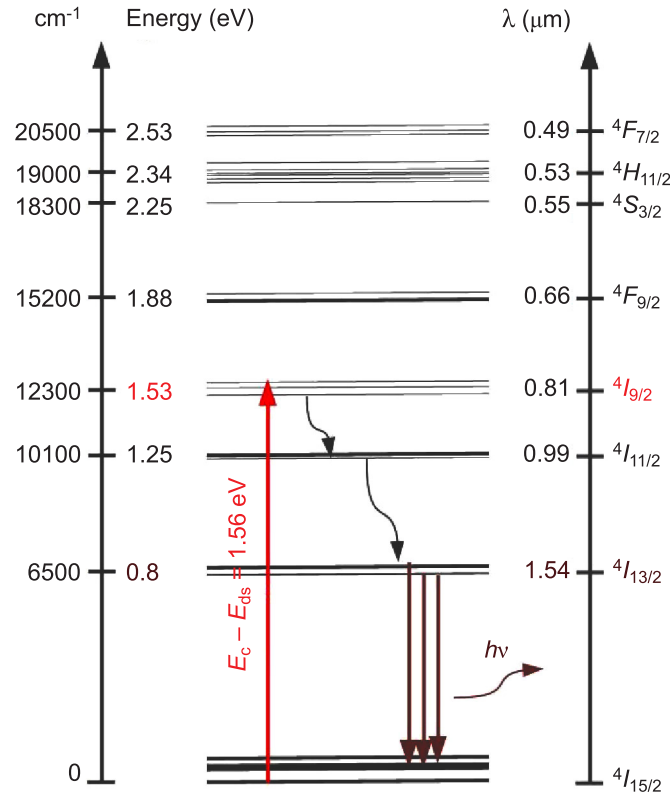


Figure 4. Excited states of Er^{3+} ion in accordance with the literature data [11, 12]. The Er excitation and emission mechanism

electrons, one for holes and the silicon bandgap ($\Phi_{\text{ef}} + \Phi_{\text{hf}} + E_{\text{g}}(\text{Si})$) which gives $E_{\text{c}} - E_{\text{ds}} = 0.14 + 0.3 + 1.12 = 1.56 \text{ eV}$ (see Fig. 3, [3]). This electron-hole pair recombination energy excites the nearby Er ion. The $h\nu$ designation is the IR photon emitted by excited erbium. The IR photon energy equals 0.81 eV (see Fig. 2), which is less than the energy transferred to excite the Er (1.56 eV, Fig. 3). This discrepancy will be discussed further. Thus, the small forward barrier for the electron injection from silicon into the $\text{In}_2\text{O}_3:\text{Er}$ film ($\Phi_{\text{ef}} = 0.14 \text{ eV}$, Fig. 3) and a relatively small barrier for the hole injection from the surface contact into the film ($\Phi_{\text{hb}} = 0.5 \text{ eV}$, Fig. 3, [3]) support the Er excitation mechanism as follows. The electrons and holes are injected over the barriers into the $\text{In}_2\text{O}_3:\text{Er}$ film at the forward bias, flow in the electric field toward each other, recombine with each other and excite the Er^{3+} ions (see Fig. 3).

As it was mentioned above, the Er^{3+} ions are excited by electron-hole pairs recombination energy $E_{\text{c}} - E_{\text{ds}} = 1.56 \text{ eV}$ (see Fig. 3). But the erbium photonic emission has the energy 0.81 eV (see Fig. 2). The erbium excitation mechanism is proposed as follows. In Fig. 4 are the Er^{3+} excited states obtained from the literature data [11, 12]. As it is seen, the electron-hole recombination energy $E_{\text{c}} - E_{\text{ds}} = 1.56 \text{ eV}$ (see Fig. 3) coincide in resonance with the third excited Er^{3+} state $^4I_{9/2}$ 1.53 eV (Fig. 4). Therefore, the electron-hole recombination excites this Er^{3+} state. Then the nonradiative relaxation without photon emission to the second $^4I_{11/2}$ and first $^4I_{13/2}$ excited Er^{3+} states occurs. And, finally, the radiative transition

from the first excited state $^4I_{13/2}$ (0.81 eV) to the ground state $^4I_{15/2}$ occurs with the photon emission with the 1.54 μm wavelength.

Thus, the observed 1.534 μm Er EL supports the proposed Er excitation mechanism by means of the electron-hole recombination. The strong confirmation of the proposed mechanism requires further investigations.

Conclusion

The magnetron sputtered $\text{In}_2\text{O}_3:\text{Er}$ films were found to be the solid solution $((\text{In}_{1-x}\text{Er}_x)_2\text{O}_3)$. The obtained $((\text{In}_{1-x}\text{Er}_x)_2\text{O}_3)$ solid solution has the bcc In_2O_3 *Ia-3* space group. The lattice constant of the $((\text{In}_{1-x}\text{Er}_x)_2\text{O}_3)$ solid solution was found equal to $a = 1.0225 \pm 0.0004 \text{ nm}$. The observed lattice constant exceeds the one for the given Er concentration 1 at.% in the film. This discrepancy could be related to the vacancy type defects or to the tensile lattice strain.

The EL at room temperature was observed for the $\text{In}_2\text{O}_3:\text{Er}$ films on the *n*-Si substrate with the top ITO-contact at the forward bias applied. The observed EL spectrum demonstrates the radiative transition from the first excited state $\text{Er}^{3+} ^4I_{13/2}$ to the ground state $^4I_{15/2}$ with the photon emission at the 1.534 μm wavelength.

The Er excitation mechanism proposed by the electron-hole recombination in the indium oxide and the electron-hole pair energy (1.56 eV) transfer to excite in resonance the third excited Er^{3+} state $^4I_{13/2}$ (1.53 eV), and by

a subsequent nonradiative relaxation to the first excited state $^4I_{13/2}$ (0.81 eV) followed by the radiative transfer to the ground state $^4I_{15/2}$ with the photon emission.

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