

Single crystal erbium compound nanowires as high gain material for on-chip light source applications*

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Abstract Integrated photonics requires high gain optical materials in the telecom wavelength range for optical amplifiers and coherent light sources. Erbium (Er) containing materials are ideal candidates due to the 1.5 μm emission from Er^{3+} ions. However, the Er density in typical Er-doped materials is less than 10^{20} cm^{-3} , thus limiting the maximum optical gain to a few dB/cm, too small to be useful for integrated photonic applications. Er compounds could potentially solve this problem since they contain much higher Er^{3+} density. So far the existing Er compounds suffer from short lifetime and strong up-conversion effects, mainly due to poor crystal qualities. Recently, we explore a new Er compound: erbium chloride silicate (ECS, $\text{Er}_3(\text{SiO}_4)_2\text{Cl}$) in the form of nanowire, which facilitates the growth of high quality single crystal with relatively large Er^{3+} density ($1.62 \times 10^{22} \text{ cm}^{-3}$). Previous optical results show that the high crystal quality of ECS material leads to a long lifetime up to 1 ms. The Er lifetime-density product was found to be the largest among all the Er containing materials. Pump-probe experiments demonstrated a 644 dB/cm signal enhancement and 30 dB/cm net gain per unit length from a single ECS wire. As a result, such high-gain ECS nanowires can be potentially fabricated into ultra-compact lasers. Even though a single ECS nanowire naturally serves as good waveguide, additional feedback mechanism is needed to form an ultra-compact laser. In this work, we demonstrate the direct fabrication of 1D photonic crystal (PhC) air hole array structure on a single ECS nanowire using focused ion beam (FIB). Transmission measurement shows polarization-dependent stop-band behavior. For transverse

electric (TE) polarization, we observed stop-band suppression as much as 12 dB with a 9 μm long airholed structure. Through numerical simulation, we showed that Q -factor as high as 11000 can be achieved at 1.53 μm for a 1D PhC micro-cavity on an ECS nanowire. Such a high Q cavity combined with the high material gain of ECS nanowires provides an attractive solution for ultra-compact lasers, an important goal of this research.

Keywords nanomaterials, rare-earth-doped materials, lasers, optical amplifiers, nanostructure fabrication, micro-cavity devices

1 Introduction

Among all the silicon photonics related components, coherent sources and amplifiers are of the most challenging tasks due to the lack of Si-compatible high gain materials. One important approach to integrate gain materials on Si is to introduce rare earth ions as impurities into Si [1]. Erbium (Er) ions, whose main features of emission are relatively insensitive to the host environment, have been intensively studied as dopants in different materials to activate optical transitions at telecommunication wavelengths. Erbium doped fiber amplifier [2] (EDFA) and erbium doped fiber laser [3] (EDFL) have achieved great success with sizes on the meter scale. To achieve device miniaturization for integrated on-chip photonic applications, Er-doped waveguide structures were proposed. Er-doped waveguide amplifiers [4–6] (EDWAs) with 5.3 dB/cm internal optical gain and Er-doped waveguide lasers [7] (EDWLs) have been demonstrated. These achievements opened the era of active Er-based devices with sizes on the centimeter scale. However, further size reduction into millimeter range or even smaller is needed for

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* Dedicated to Prof. Bingkun Zhou on the occasion of his 80th birthday. Prof. Zhou has made many important contributions in the areas of fiber optics and semiconductor-laser-pumped rare-earth-doped solid state lasers. It is therefore fitting to dedicate this paper on single crystal Erbium compound to him

integrated Si photonics. Given that the maximal Er density in Er-doped materials is around 10^{20} cm^{-3} , the highest internal net gain is only a few dB/cm [8]. The concentration of Er ions cannot be increased beyond a certain limit in Er-doped materials. Otherwise other detrimental phenomena occur, rendering the excited Er ions optically inactive, or worse, even decreasing optical gain.

On the other hand, Er compounds, instead of doped materials, have come into focus in the last decade for the high-gain amplifiers and ultra-compact lasers applications. Er compounds are the single-crystalline or polycrystalline compounds with Er-concentration higher than 10^{22} cm^{-3} , including erbium oxide (Er_2O_3) [9,10], erbium silicates (Er_2SiO_5 , $\text{Er}_2\text{Si}_2\text{O}_7$) [10,11] and erbium yttrium/ytterbium silicate alloy (EYS, EYbS) [12–14]. Due to the relative high Er-concentration, the potential gain of Er compounds as high as 30 dB/cm was predicted [9], much higher than the demonstrated gain in Er-doped materials. However, the material crystal qualities of these compounds are not good enough, which results in very short carrier lifetimes and strong upconversion effect, making it difficult to achieve even higher optical gain.

Recently, we reported the synthesis of a new Er-compound material: erbium chloride silicate (ECS), in the form of single crystal nanowire [15]. Due to the high crystal quality, large Er^{3+} concentration ($1.62 \times 10^{22} \text{ cm}^{-3}$), and relatively long lifetime ($> 0.5 \text{ ms}$) [16], more than 30 dB/cm net material gain and 644 dB/cm signal enhancement were demonstrated in a single ECS nanowire [17,18], much larger than reported values in all other Er-containing materials [19,20]. Such high-gain ECS nanowires are expected to have potential applications in silicon-compatible photonic integration as light sources or amplifiers. In this work, we report on the fabrication and characterization of 1D photonic crystal (PhC) structure on a single ECS nanowire by focused ion beam (FIB) etching. We observed the clear polarization-dependent stop-band behavior from the transmission spectra of the PhC with large transverse electric (TE) suppression. The refractive index of ECS material was determined by fitting the transmission spectra to the 3D finite-difference time-domain (FDTD) simulation. The 1D PhC micro-cavity with Q -factor over 10^5 with total length of only $24 \mu\text{m}$ is designed, which is sufficiently high for a single ECS wire lasing at $1.53 \mu\text{m}$. We believe the design of ultra-compact and easily transferable ECS nanowire laser has great potential for silicon-compatible integrated photonics.

2 Experiment

2.1 Growth and optical properties of ECS nanowire

Single crystal ECS nanowires are synthesized using Au-assisted chemical vapor deposition (CVD) method. In comparison with the thin film Er materials, Er containing

nanomaterials have fewer limitations on the substrate selection and often can be produced with higher crystal quality. In a typical procedure, silicon powder (Alfa Aesar, 99.99%) in a ceramic boat was placed at the center of the quartz tube. The silicon or quartz substrate pre-sputtered with a 10 nm-thick Au film was positioned downstream at a distance of 17 cm from the center of the furnace for the deposition. Anhydrous ErCl_3 micro beads (Alfa Aesar, 99.9%, diameter $\sim 1 \text{ mm}$) in another ceramic boat were loaded upstream from the substrate. The tube chamber was evacuated to a pressure below 100 mTorr and a constant flow of 50 sccm Ar- H_2 5% mixed gas was introduced as a carrier gas through the quartz tube. The pressure inside the quartz tube was adjusted to 400 mTorr. The center of the furnace was then heated to $1080 \text{ }^\circ\text{C}$, and maintained at this temperature for 180 min. The measured temperature was $\sim 600 \text{ }^\circ\text{C}$ for the growth substrate and $\sim 650 \text{ }^\circ\text{C}$ for the ErCl_3 micro beads. After the growth, the furnace was naturally cooled to room temperature.

The length of the nanowires can be controlled by the growth time, which is usually tens of microns. The diameter of the nanowire is determined by the size of the Au catalyst. Since the Au particles were formed from the sputtered Au film during the heating process, the size of the Au nanoparticles can be mostly controlled by the thickness of the Au film. The ECS nanowires have typically a square cross-section. From scanning electron microscope (SEM) images of the as-grown nanowires, more than 50% of the nanowires have the diameter between 700 nm and $1 \mu\text{m}$, while the thin nanowires with diameter around 100 nm also exist. Since we focus on the applications of the nanowires as the optical amplifiers and lasers, the large-diameter nanowires are preferred because they can support the propagation mode at $1.53 \mu\text{m}$. Single nanowires were selected from the original growth substrate and transferred onto a silicon substrate covered with Au film for optical measurements, by using a homemade tapered fiber with typical taper diameter of $1\text{--}2 \mu\text{m}$.

Knowing the optical processes of ECS nanowires is crucial for understanding how to leverage them for optoelectronic applications. The photoluminescence (PL) spectra are obtained under the excitation of a Ti:Sapphire laser at 800 nm. The full PL spectrum from visible to near infrared (NIR) range of ECS at room temperature is shown in Fig. 1. The PL intensity is normalized in four different wavelength ranges: 510–680, 780–880, 950–1050 and 1440–1600 nm. No PL features are found in the wavelength gaps between these four ranges. The PL spectrum from 780 to 880 nm is obtained under 667 nm excitation. Er^{3+} ions are first pumped to $^4\text{F}_{9/2}$ state due to the ground state absorption (GSA) and then relax back to lower states. The emission bands centered at 800 nm and 850 nm are originated from the $^4\text{I}_{9/2} \rightarrow ^4\text{I}_{15/2}$ and $^4\text{S}_{3/2} \rightarrow ^4\text{I}_{13/2}$ transitions, respectively. As shown in Fig. 2, Er^{3+} ions are first pumped to $^4\text{I}_{9/2}$ state. The downward transitions from $^4\text{I}_{11/2}$ and $^4\text{I}_{13/2}$ to the ground state $^4\text{I}_{15/2}$

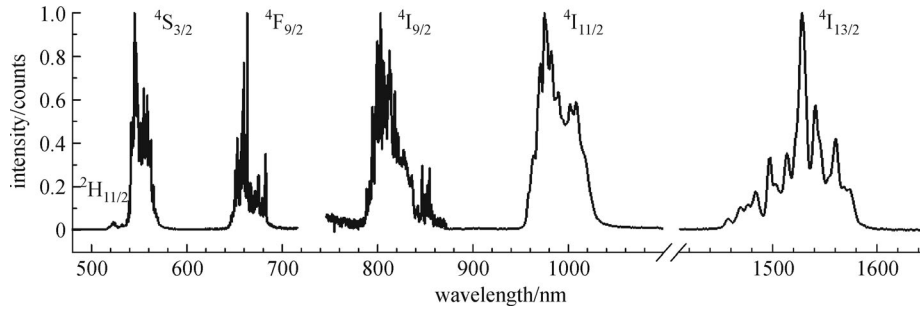


Fig. 1 PL spectrum of ECS across visible to NIR. The spectra are normalized at each emission band

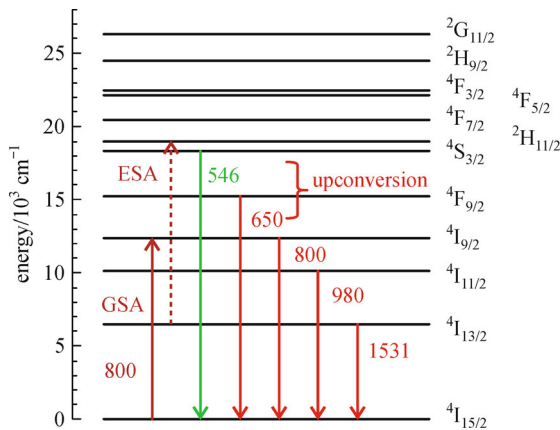


Fig. 2 Absorption and emission transitions with 800 nm laser pumping. The numbers at each emission transition represent the emission wavelengths, with the unit in nm

generate photon emission at the 980 nm band and the 1530 nm band. The visible emission under 800 nm excitation is due to the excited state absorption (ESA). The excited Er^{3+} ions on $^4\text{I}_{13/2}$ state can absorb another 800 nm pump photon and promote to $^2\text{H}_{11/2}$ state. The downward transition from $^2\text{H}_{11/2}$, $^4\text{S}_{3/2}$ and $^4\text{F}_{9/2}$ states to the ground state result in the emission bands centered at 520, 550 and 650 nm. Among all emission bands, the 1.5 μm transition from the first excited state to the ground state is of the most importance because this wavelength falls within the minimum loss band of fiber communications. One of the most remarkable features in the PL spectrum of ECS is that the spectrum consists of atomic like sharp emission lines, which reflects the high crystal quality.

2.2 Fabrication of 1D PhC on ECS nanowire

Due to the high crystal quality, more than 30 dB/cm net optical gain and 644 dB/cm signal enhancement at 1.53 μm in a single ECS nanowire have been demonstrated [17,18]. The relative low gain (compared to semiconductors) in ECS, is not high enough to compensate the mirror loss of the end-facets. A simple estimation shows that for a 100

μm -long ECS nanowire, the required minimum threshold gain to achieve lasing is 1200 dB/cm, which is impossible for ECS nanowires. To reduce the lasing threshold, it is necessary to fabricate resonator with high quality factor (Q -factor) resonator on the nanowire. Due to the large index contrast between the air-holes and the dielectric waveguide, the strength of the PhC as a total reflector is much stronger than distributed Bragg reflector (DFB) grating. Therefore, we performed FIB on a single ECS nanowire to fabricate PhC structures. An ECS nanowire of 590 nm in side length of cross-section was transferred on a silicon wafer for FIB etching. To demonstrate the 1D PhC fabrication and determine the refractive index of this ECS material, we first etched a uniform hole array of 14 periods on a single ECS nanowire, as shown in Fig. 3. The diameter of the air holes is 240 nm. The total length of the fabricated 1D PhC structure is 9 μm with a periodicity of 700 nm.

To determine the stop band of this 1D PhC reflector, transmission spectroscopy was performed. The etched ECS nanowire was transferred onto another silicon substrate pre-etched with 1 μm -depth trench, such that the segment with PhC structure was suspended in air while the two ends were placed on the silicon, as shown in Figs. 4 (a) and 4(b). The suspended PhC region is in the symmetric refractive-index environment, which can maximize the strength of the PhC reflector. A tunable laser at 1.35–1.63 μm was coupled into the nanowire through a tapered fiber. The polarization of the input signal was precisely controlled by a motorized polarization controller. The transmitted signal was delivered to the power detector through another tapered fiber.

The strength of the PhC reflector is sensitive to the polarization of the signal. Experimentally, it is difficult to calibrate the polarization at the output of the tapered fiber because the polarization is always shifting with even a slight change of the fiber curvature. So the TE and transverse magnetic (TM) polarization was determined by the minimum and maximum transmission at the center wavelength of the stop band. To examine the propagation properties for wavelengths within the stop band, we injected signal at stop-band wavelength 1.525 μm into the

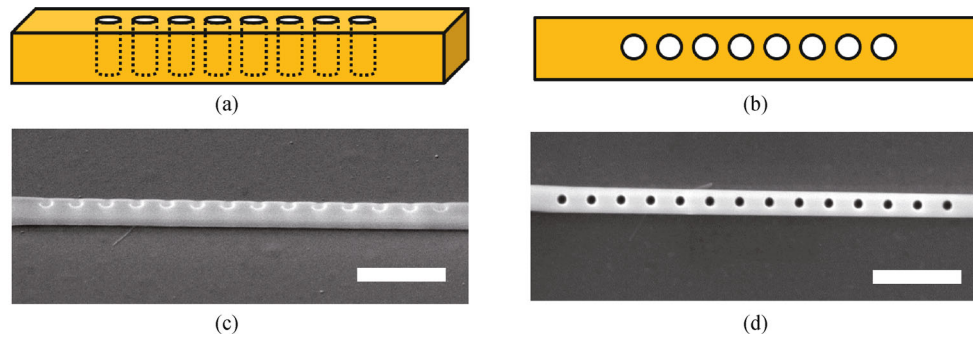


Fig. 3 (a) and (b) Illustration of the 1D PhC structure in angled view and top view; (c) and (d) SEM images of the 14-period PhC grating in single ECS nanowire. Scale bar is 2 μm

nanowire, and monitored the green upconversion decay along the wire, which represents the decay of intensity of the injected signal [18]. It is clear that the injected signal of TE polarization decays rapidly once entered into the PhC region, while corresponding decay for TM-polarization is much less obvious, as shown in the FDTD simulation results in Figs. 4(c) and 4(d). Figures 4(e) and 4(f) show the upconversion emission images under TE and TM injection, respectively. Clearly the green upconversion decays much quicker in TE polarization than in TM polarization, which agrees very well with the FDTD result in both polarizations. The transmission spectra under TE and TM polarization are shown in Fig. 4(g). Clear stop-band behavior with more than 12 dB suppression was observed at 1.525 μm for the TE polarization, while the suppression is much lower in the TM polarization. The refractive index of ECS is determined as $n = 1.64$, which is close to the reported value of erbium silicate film [21]. It is important

to point out that the green upconversion emission at the PhC region does not show significant degradation, indicating that the emission efficiency of ECS nanowire is not significantly degraded after the ion beam etching, unlike semiconductors and organic materials which are sensitive to ion damaging and impurity injection.

3 Discussion

To further explore the potential optoelectronic applications based on these high-gain ECS nanowires, we explore the possibility of high Q PhC microcavity structure toward laser applications. The microcavity is confined by two PhC gratings with uniform hole size as the Bragg mirrors. Although the PhC gratings can be treated as the total reflector, there is still considerable scattering loss from cavity to PhC grating due to the mismatch of the

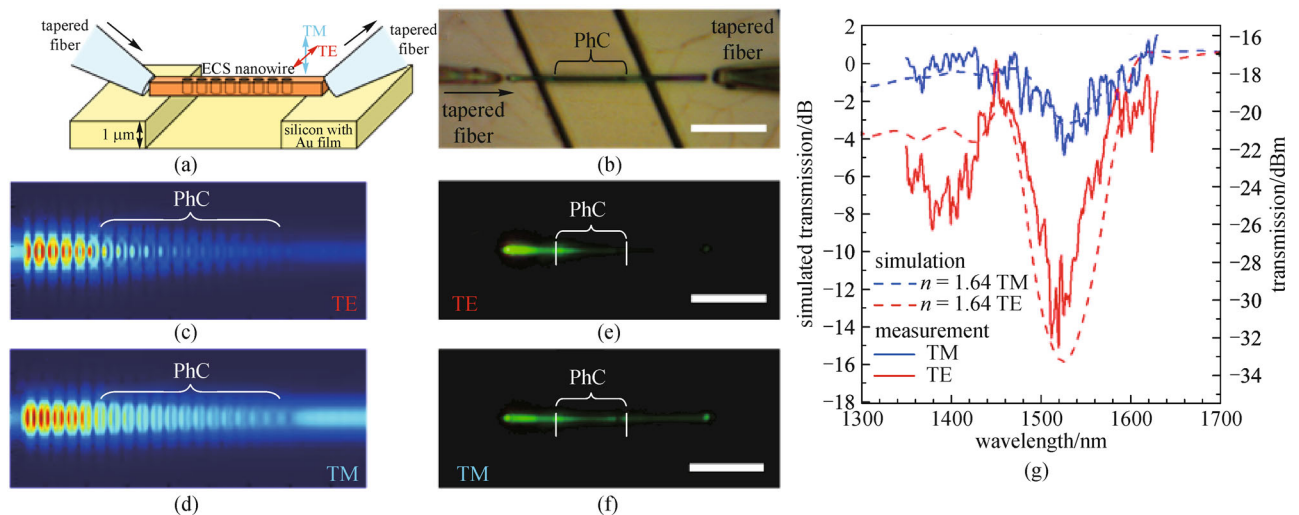


Fig. 4 (a) Illustration of the fiber-nanowire-fiber coupling system; (b) microscope image of the coupling system from the top view; (c) and (d) FDTD simulation of the E-field pattern under TE- and TM- polarization injection; (e) and (f) real-color images of the upconversion along the nanowire in (b) under TE- and TM- polarization injection at 1531 nm; (g) experimental (solid) and simulated transmission spectra under TE- (red) and TM- (blue) polarization injection. Scale bar is 10 μm

impedance between the waveguide mode in cavity and Bloch mode in PhC gratings [22–24]. One effective way to reduce the scattering loss is tapering both hole size and hole spacing toward the cavity [25–27]. It is demonstrated that 4–7 periods of linear tapering profile can significantly reduce the loss and enhance the Q -factor. The quadratic tapering profile is reported to have better performance than the linear tapering profile [24,28]. To simplify the calculation, the following PhC micro-cavity design uses the linear tapering profile. To further minimize the cavity loss, the cavity length is also optimized. It is reported that the optimized cavity length which is the distance between the two PhC gratings needs to be zero [22,28].

According to the above discussion, the high- Q PhC microcavity is designed on a ECS nanowire with width of the square cross-section to be 1 μm , the structure parameters are listed in Table 1. The microcavity consists of 15 pairs of uniform air holes as mirrors on both ends and 4 pairs of holes in the middle with linearly decreasing diameter, as shown in Fig. 5(a). The total length of PhC cavity is only 24 μm . Figure 5(b) shows the FDTD simulation of the E-field profile of the fundamental mode at 1.53 μm , which is the peak-gain wavelength [17,18]. The Q -factor of this mode is 11000, one order of magnitude higher than the required Q for the ECS nanowire laser with 30 dB/cm net gain. Such a high Q -factor makes it possible to achieve lasing on a single ECS nanowire, even

considering some inevitable fabrication imperfection of the PhC structure. Therefore, the high-gain ECS nanowire with such high- Q PhC microcavity offers an important potential alternative as an ultra-compact laser for the integrated and silicon-compatible photonics.

4 Conclusions

ECS nanowires have been established as a new Er-compound material for high-gain applications in the telecom wavelength range. This material features high Er density, long lifetime and high optical gain. Therefore, ECS nanowires are expected to be potential laser sources for silicon-based photonic integrated circuits. To achieve lasing, the optical mode must be confined in a low-threshold cavity. In this work, simulation and fabrication toward a single ECS nanowire laser are investigated. We report on the fabrication of 1D PhC as a feasible high- Q structure on a single ECS nanowire by FIB etching. The transmission spectroscopy of the ECS nanowire etched with uniform 1D PhC grating demonstrates the strong grating reflection, low scattering loss, and feasibility of the FIB fabrication. The exact refractive index of ECS is determined by FDTD simulation of the transmission spectra for the first time. Finally, a PhC micro-cavity structure with Q -factor as high as 11000 is designed based

Table 1 Structure parameters of the designed 1D PhC micro-cavity on an ECS nanowire. The Q -factor of the fundamental mode in this microcavity is 1.1×10^5

structure parameter	value	structure parameter	value
width (w)	800 nm	height (h)	800 nm
L	586 nm	n^*	15
r_0	182 nm	a	650 nm
r_1	179 nm	d_1	641 nm
r_2	177 nm	d_2	633 nm
r_3	175 nm	d_3	625 nm
r_4	173 nm	d_4	617 nm

* n is the number of air holes in the mirror region

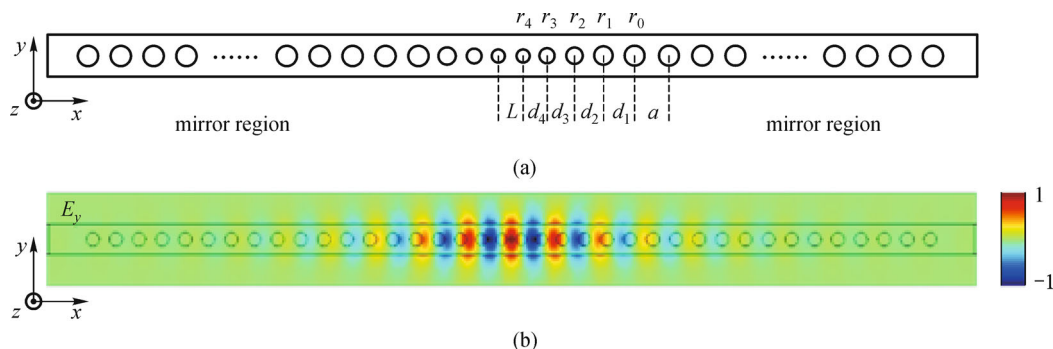


Fig. 5 (a) Design of 1D PhC micro-cavity on ECS nanowire; (b) E-field profile of the fundamental mode at 1.53 μm

on the optical and structural properties of the ECS nanowire. The total length of the PhC is less than 24 μm , demonstrating the prospect of the ultra-compact and silicon-compatible laser devices for photonic integrated circuits.

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