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Research Article

Optical Amplification at 1525 nm in $BaYF_5$: 20% Yb^{3+}, 2% Er^{3+} Nanocrystals Doped SU-8 Polymer Waveguide

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We demonstrated optical amplification in $BaYF_5$: 20% Yb^{3+}, 2% Er^{3+} (BYF) nanocrystals doped polymer waveguide. BYF nanocrystals with an average size of ~13 nm were synthesized by a high-boiling solvent process. Intense 1.53 μm fluorescence was obtained in the nanocrystals under excitation at 980 nm. An optical polymer waveguide was fabricated by using BYF nanocrystals doped SU-8 polymer as the core material. A relative optical gain of ~10.4 dB at 1525 nm was achieved in a 1.1 cm long waveguide for an input signal power of ~0.09 mW and a pump power of ~212 mW.

1. Introduction

Erbium doped fiber amplifier (EDFA) is considered as the most deployed fiber amplifier as its amplification window coincides with low loss telecommunication windows at 1550 nm. However, EDFAs are incompatible with miniature and integrated optical devices in access and home network applications [1, 2]. Compared to EDFAs, erbium doped waveguide amplifier (EDWA) provides a higher gain in a much smaller size waveguide rather than several meters of fiber and can be integrated with other photonic devices potentially, such as switches, couplers, and splitters [3–8]. Usually, inorganic host materials are used to fabricate EDWA. However, the use of polymeric hosts in the fabrication of EDWA gives many advantages, including low fabrication costs, simplified processing steps, and compatibility with processing techniques for patterning [8–12]. In particular, Er^{3+}-doped inorganic nanocrystals can be dispersed into polymer matrices and used to construct polymer-based EDWAs [8, 9, 13, 14]. However until recently there are only a few reports on obtained optical gains based on inorganic nanocrystals doped polymeric optical waveguide amplifiers. Zhang et al. synthesized LaF_3: Er^{3+}, Yb^{3+} nanocrystals and constructed a polymer-based EDWA with an optical gain of ~5 dB [8]. Zhai et al. demonstrated a polymer-based EDWA based on $NaYF_4$: Er^{3+}, Yb^{3+}, and Ce^{3+} nanocrystals and its maximum gain was about 4 dB [15]. Very recently, sub-10 nm $BaYF_5$: Yb^{3+}, Er^{3+} nanocrystals were synthesized and used to construct a polymer-based EDWA with an optical gain of ~6.3 dB [16]. The large gain in this device was attributed to the good dispersibility in polymer matrices and highly efficient 1.55 μm emission of $BaYF_5$: Yb^{3+}, Er^{3+} nanocrystals. Although smaller nanocrystals provide a good redispersibility, their luminescence quantum yield is generally low due to nonradiative energy losses caused by the surface defects as well as vibrational deactivation ascribed to solvent molecules and ligands absorbed on the nanocrystals. So we consider that there exists a trade-off between the luminescence quantum yield and the dispersibility in polymer matrices, and the gain performance of the polymer-based EDWA might be improved by slightly increasing the size of $BaYF_5$: Yb^{3+}, Er^{3+} nanocrystals owing to the reduction of surface quenching effects of the nanocrystals.

In this paper, $BaYF_5$: Yb^{3+}, Er^{3+} nanocrystals with an average size of ~13 nm were synthesized by a high-boiling

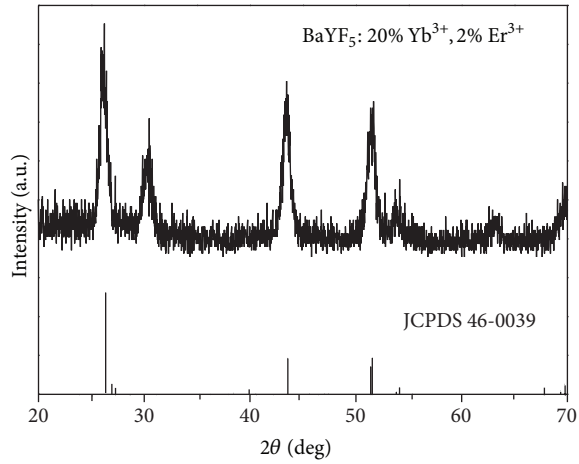


Figure 1: XRD pattern of the as-synthesized $BaYF_5$: Yb^{3+}, Er^{3+} samples.

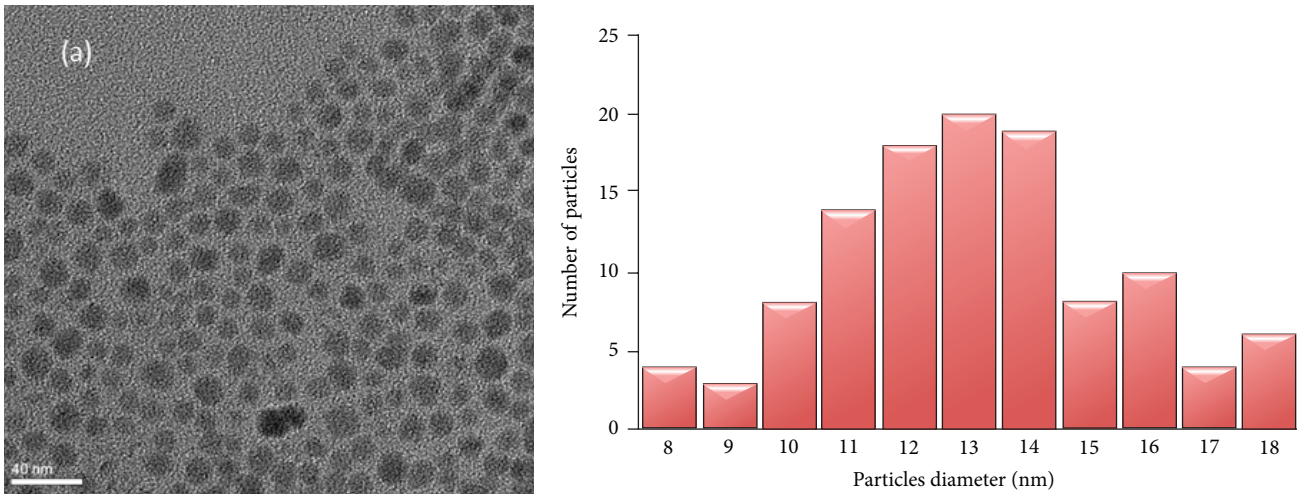
solvent process. The photoluminescence spectra were characterized. An optical polymer waveguide was fabricated by using $BaYF_5$: Yb^{3+}, Er^{3+} nanocrystals doped SU-8 polymer as the core layer and PMMA as the coating layer. A relative optical gain of ~10.4 dB at 1525 nm was achieved in a 1.1 cm long waveguide for an input signal power of ~0.09 mW and a pump power of ~212 mW. To the best of our knowledge, such an optical gain is the highest one in polymer-based EDWAs with erbium doped inorganic nanocrystals ever reported.

2. Results and Discussion

$BaYF_5$: Yb^{3+}, Er^{3+} nanocrystals were synthesized by a high-boiling solvent process. 0.5 mmol preprepared rare-earth stearate was added along with 0.5 mmol barium stearate to a 100 mL three neck round-bottom flask containing 5 mL 1-octadecene and 15 mL oleic acid. The solution was stirred magnetically and heated slowly to 100°C under vacuum for 30 min to remove residual water or oxygen. After 30 min, the reaction was brought under argon atmosphere and cooled to room temperature. A solution of NH_4F (3 mmol, 111 mg) in 10 mL of methanol was added, and then the solution was kept at 50°C for 30 min. After methanol was evaporated, the reaction was then heated to 300°C as soon as possible under an argon atmosphere, kept for 1 h, and then cooled to room temperature. The nanocrystals were precipitated with ethanol, collected after centrifugation, and redispersed in cyclohexane. The phase structure of the products was first examined by X-ray diffraction (XRD) (Model Rigaku Ru-200b), using a nickel-filtered Cu Kα radiation ($\lambda = 1.5406$ Å) in the range of $10^\circ \leq 2\theta \leq 70^\circ$. Figure 1 shows the XRD pattern of the as-synthesized $BaYF_5$: Yb^{3+}, Er^{3+} samples. The diffraction peaks of the sample can be indexed as a tetragonal-phase $BaYF_5$; the locations and relative intensity of the diffraction peaks coincide well with the literature values (JCPDS number 46-0039, space group P-421m). The size of the samples was further characterized by a JEM-2100F electron microscope at 200 kV. Figures 2(a) and 2(b) show the transmission electron microscope (TEM) image of the as-synthesized samples and the corresponding histogram of size distribution, respectively. The as-synthesized $BaYF_5$: Yb^{3+}, Er^{3+} samples have an average size of ~13 nm and a good uniformity of size.

Figure 3 shows infrared emission spectrum of $BaYF_5$: Yb^{3+}, Er^{3+} nanocrystals at room temperature under a 980 nm laser excitation. The emission peaked at 1535 nm is assigned to the $^4I_{13/2} \rightarrow {}^4I_{15/2}$ transition of Er^{3+}. The full width at half maximum of the emission is about 54 nm, which indicates a relatively wide bandwidth of the gain spectrum.

The fabrication procedure of a polymer-based waveguide is shown in Figure 4. SU-8 2005 polymer was used as the polymeric host for the fabrication of the optical waveguide. 0.005 g $BaYF_5$: Yb^{3+}, Er^{3+} nanocrystals were dissolved into 1.8 g toluene; the solution was ultrasonically agitated for 3 h and then was mixed into SU-8 2005 polymer, and the concentration of nanocrystals in SU-8 2005 polymer was about 0.12 wt%. Waveguides of dimensions, 4 μm height by 8 μm width, were fabricated by standard photolithography and wet etching technology of a thin silicon dioxide layer. A 5 μm thick film of BYF nanocrystals doped SU-8 2005 was spin-coated onto a 2 μm thick silicon dioxide layer based on silicon substrates and prebaked at 90°C for 20 min to remove any traces of the solvent. The pattern exposure was performed at a wavelength of 365 nm using the 350 mW Hg lamp power through a contact chromium mask. The exposure time was 180 s. After postbaking, the resist was developed into propylene glycol-monomethyl ether-acetate (PGMEA) for 40 s, rinsed in isopropyl alcohol and then deionized water, and was subsequently blow-dried. A 5 μm thick P(MMA-GMA) film is spin-coated as the upper cladding layer. The refractive indices of the core and upper cladding were measured using ellipsometry method (J. A. Woollam., Co. M2000) to be 1.578 and 1.495 at 1535 nm wavelength, respectively. Figure 5 is a SEM image of the $BaYF_5$: Yb^{3+}, Er^{3+} nanocrystals dispersed polymer rectangular waveguide amplifier (without top cladding), indicating that the size



(a) (b)

Figure 2: (a) A TEM image of nanocrystals, the average crystals size is about 13 nm. (b) The corresponding histogram of size distribution.

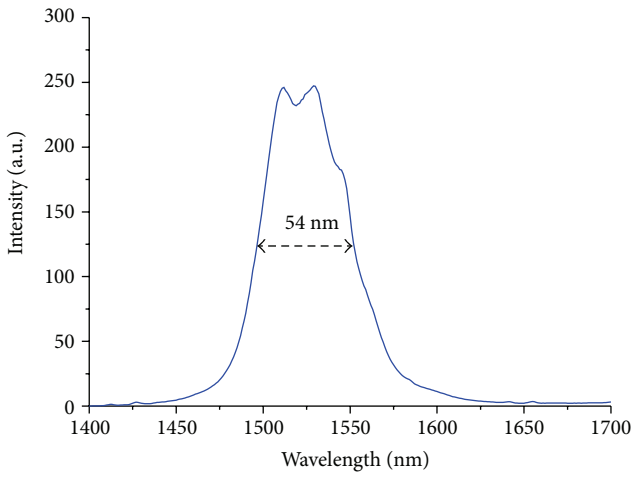


Figure 3: Room temperature fluorescence spectra of the nanocrystals excited under 980 nm in 0.01 M cyclohexane solutions.

of the waveguide amplifier is 8 μm high and 4 μm wide. Figure 6 is the output near-field profile from a 16 mm long waveguide at 1535 nm wavelength measured by using an infrared camera (Electrophysics model 7290A). The input signal power is 0.2 mW. The background loss of the waveguide with nanocrystals was also measured by using a cutback method and was about 3.1 dB/cm, which was higher than that (~1 dB/cm) of the waveguide without nanocrystals.

Then, we constructed a waveguide amplifier and measured their performances. Figure 7 shows the schematics of the experimental setup for the optical gain measurement. Relative gain of the waveguide amplifiers was carried out by using a tunable laser with a working wavelength range of 1510–1590 nm (Santec TSL-210) as the signal source and a 976 nm continuous wave laser diode as the pump source. Both the pump and signal sources were coupled to the channel waveguides through a 980/1550 nm wavelength-division multiplexing fiber coupler. Output light from the device was collected and coupled to an optical spectrum analyzer (OSA, Ando AQ-6315A). The relative gain was calculated using the formula 10 $\log[(P_{\text{out}} - P_{\text{ASE}})/P_{\text{in}}]$ dB, where P_{out} was the output signal power amplified by the waveguide, P_{ASE} was the amplified spontaneous emission power, and P_{in} was the input signal power. Figure 8 shows the measured gain as a function of pump power at 976 nm in a 16 mm long rectangular waveguide. The gain gradually increases with the increment of pump power. A maximum relative optical gain of ~10.4 dB at 1525 nm was achieved in a 1.1 cm long waveguide for an input signal power of ~ 0.09 mW and a pump power of ~212 mW. Such an optical gain is the highest one in polymer-based EDWAs with erbium doped inorganic nanocrystals ever reported, to the best of our knowledge. The gain coefficient of our device was also calculated based on the above measured results and was about 9.5 dB/cm [17]. Such a value is higher than that in previous published results [8, 15, 16]. We considered that such an improvement compared to previous results was obtained by investigating the trade-off between the luminescence quantum yield and the dispersibility in polymer matrices for the nanocrystals and further solving it with the optimized nanocrystals.

In addition, we measured the long time stability of the device performance under the irradiation of pump laser and found that the gain value was almost kept stable and unchanged for more than two hours. And also, we remeasured the performance of our device after two months since the fabrication of the device. The measured results showed

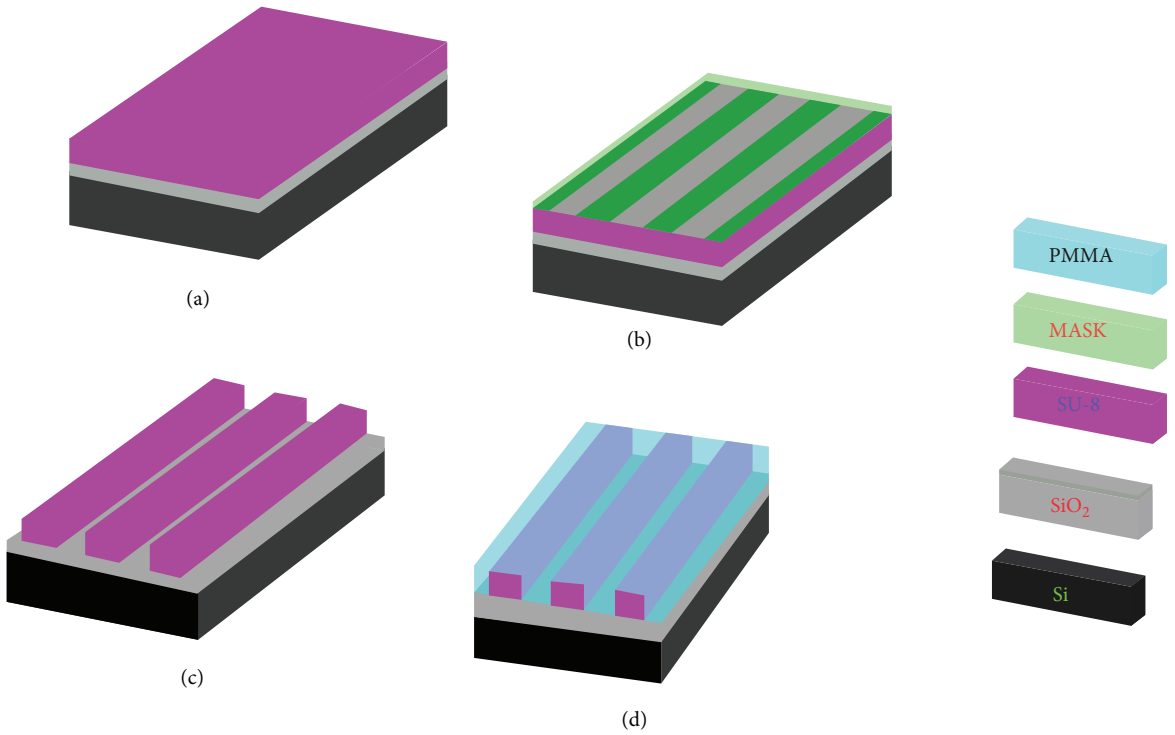


Figure 4: (a) Spin coating and soft bake. (b) Exposure. (c) Development and postexposure bake. (d) Spin coating top cladding and cure.

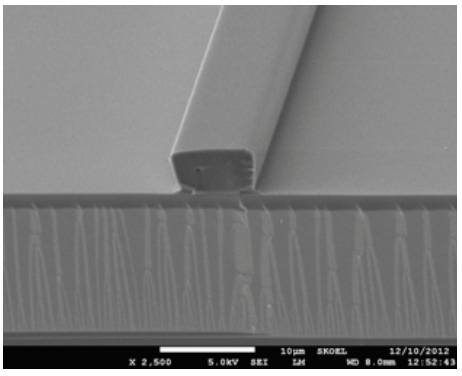


Figure 5: A SEM micrograph of a waveguide amplifier cross section without top cladding indicating that the size of the waveguide is 8 μm wide and 4 μm high.

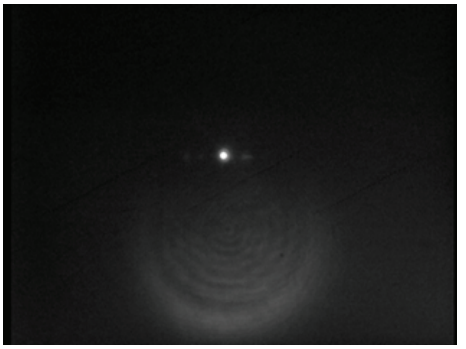


Figure 6: Output near-field profile from a 1.1 cm long waveguide at 1525 nm. The input signal power is 0.09 mW.

that the optical gain was not changed compared to the previous measured value.

3. Conclusions

In conclusion, we demonstrated a $BaYF_5$: 20% Yb^{3+}, 2% Er^{3+} (BYF) nanocrystals doped polymer waveguide amplifier. BYF nanocrystals with an average size of ~13 nm were synthesized by a high-boiling solvent process. An optical polymer waveguide was fabricated by using $BaYF_5$: Yb^{3+}, Er^{3+} nanocrystals doped SU-8 polymer as the core layer and PMMA as the coating layer. A relative optical gain of ~10.4 dB at 1525 nm was achieved in a 1.1 cm long waveguide for an input signal power of ~0.09 mW and a pump power of ~ 212 mW. Our results show that $BaYF_5$: Yb^{3+}, Er^{3+} nanocrystals are promising candidates for building high gain polymer-based EDWAs.

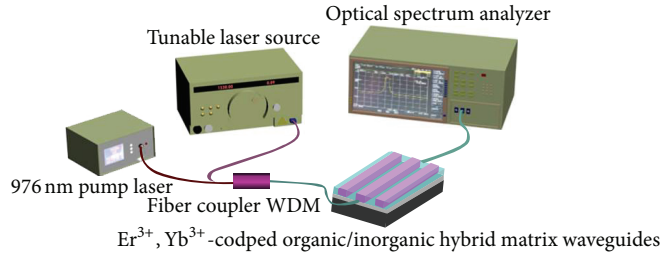


Figure 7: The schematics of the experimental setup for the optical gain measurement.

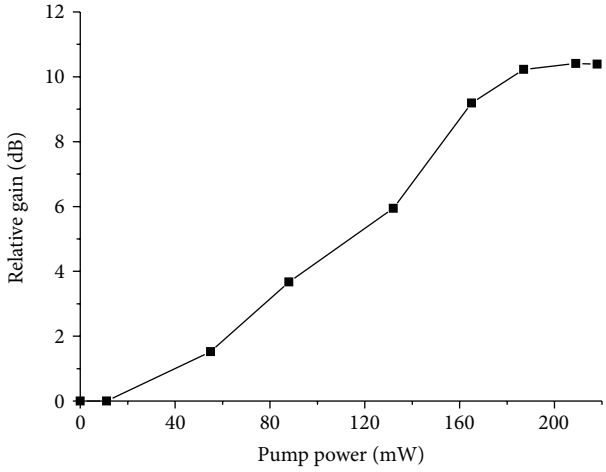


Figure 8: A 10.4 dB optical relative gain at 1525 wavelength was observed in a 1.1 cm long device with a cross section of $8 \times 4\ \mu m^2$.

Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

Acknowledgments

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References

[1] L. H. Slooff, A. van Blaaderen, A. Polman et al., "Rare-earth doped polymers for planar optical amplifiers," *Journal of Applied Physics*, vol. 91, no. 7, pp. 3955–3980, 2002.

[2] A. Polman and F. C. J. M. van Veggel, "Broadband sensitizers for erbium-doped planar optical amplifiers: review," *Journal of the Optical Society of America B: Optical Physics*, vol. 21, no. 5, pp. 871–892, 2004.

[3] R. N. Ghosh, J. Shmulovich, C. F. Kane et al., "8-mW threshold Er^{3+}-doped planar waveguide amplifier," *IEEE Photonics Technology Letters*, vol. 8, no. 4, pp. 518–520, 1996.

[4] Y. Wei, F. Lu, X. Zhang, and D. Chen, "Polyol-mediated synthesis of water-soluble LaF_3:Yb,Er upconversion fluorescent nanocrystals," *Materials Letters*, vol. 61, no. 6, pp. 1337–1340, 2007.

[5] D. Zhang, C. Chen, D. Zheng, T. Li, L. Song, and Z. Zhen, "Erbium-ytterbium codoped waveguide amplifier fabricated with solution-processable complex," *Applied Physics Letters*, vol. 94, no. 4, Article ID 041119, 2009.

[6] C. Chen, D. Zhang, T. Li, D. Zhang, L. Song, and Z. Zhen, "Demonstration of Optical Gain at 1550 nm in erbium-ytterbium co-doped polymer waveguide amplifier," *Journal of Nanoscience and Nanotechnology*, vol. 10, no. 3, pp. 1947–1950, 2010.

[7] K. Vu and S. Madden, "Tellurium dioxide Erbium doped planar rib waveguide amplifiers with net gain and 2.8 dB/cm internal gain," *Optics Express*, vol. 18, no. 18, pp. 19192–19200, 2010.

[8] D. Zhang, C. Chen, C. Chen et al., "Optical gain at 1535 nm in LaF_3:Er,Yb nanoparticle-doped organic-inorganic hybrid material waveguide," *Applied Physics Letters*, vol. 91, no. 16, Article ID 161109, 2007.

[9] S. H. Bo, J. Hu, Z. Chen et al., "Core-shell LaF_3:Er,Yb nanocrystal doped sol-gel materials as waveguide amplifiers," *Applied Physics B: Lasers and Optics*, vol. 97, no. 3, pp. 665–669, 2009.

[10] S. Bo, J. Wang, H. Zhao et al., "LaF_3:Er,Yb doped sol-gel polymeric optical waveguide amplifiers," *Applied Physics B: Lasers and Optics*, vol. 91, no. 1, pp. 79–83, 2008.

[11] J. Wang, J. Hu, D. Tang, X. Liu, and Z. Zhen, "Oleic acid (OA)-modified LaF_3:Er,Yb nanocrystals and their polymer hybrid materials for potential optical-amplification applications," *Journal of Materials Chemistry*, vol. 17, no. 16, pp. 1597–1601, 2007.

[12] W. H. Wong, E. Y. B. Pun, and K. S. Chan, "Er^{3+}-Yb^{3+} codoped polymeric optical waveguide amplifiers," *Applied Physics Letters*, vol. 84, no. 2, pp. 176–178, 2004.

[13] G. A. Kumar, C. W. Chen, J. Ballato, and R. E. Riman, "Optical characterization of infrared emitting rare-earth-doped fluoride nanocrystals and their transparent nanocomposites," *Chemistry of Materials*, vol. 19, no. 6, pp. 1523–1528, 2007.

[14] G. A. Kumar, C. W. Chen, R. Riman, S. Chen, D. Smith, and J. Ballato, "Optical properties of a transparent CaF_2:Er^{3+} fluoropolymer nanocomposite," *Applied Physics Letters*, vol. 86, no. 24, Article ID 241105, pp. 1–3, 2005.

[15] X. Zhai, J. Li, S. Liu et al., "Enhancement of 1.53 μm emission band in $NaYF_4$:Er^{3+},Yb^{3+},Ce^{3+} nanocrystals for polymer-based optical waveguide amplifiers," *Optical Materials Express*, vol. 3, no. 2, pp. 270–277, 2013.

[16] X. Zhai, S. Liu, X. Liu et al., "Sub-10 nm $BaYF_5$:Yb^{3+},Er^{3+} core-shell nanoparticles with intense 1.53 μm fluorescence for polymer-based waveguide amplifiers," *Journal of Materials Chemistry C*, vol. 1, no. 7, pp. 1525–1530, 2013.

[17] D. Dai, Y. Shi, S. He, L. Wosinski, and L. Thylen, "Gain enhancement in a hybrid plasmonic nano-waveguide with a low-index or high-index gain medium," *Optics Express*, vol. 19, no. 14, pp. 12925–12936, 2011.