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Spectroscopy of low-phonon-energy Er:La₂O₂S oxysulfide

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Abstract. Lanthanum oxysulfide powders doped with Erbium ions were fabricated by the combustion method followed by sulfurization at 1000 °C under H₂S + N₂ atmosphere. X-ray diffraction confirms their single-phase nature (sp. gr. *P-3m1*). Raman spectroscopy reveals a maximum phonon energy of 389 cm⁻¹. The effect of Er³⁺ doping level (0.5 – 7 mol%) on the visible and mid-infrared emission properties of Er:La₂O₂S was studied.

1 Introduction

Mixed-anion materials like lanthanide oxysulfides attract attention for doping with trivalent rare-earth ions for phosphors with good chemical and thermal stability and high luminescence quantum yield. Lanthanum oxysulfide, La₂O₂S, also attracts attention due to its low-phonon energy behavior ($h\nu_{ph} \sim 389 \text{ cm}^{-1}$) unlocking mid-infrared emission. Recently, Le Coz *et al.* reported on the first infrared transparent La₂O₂S ceramic prepared by hot-pressing powders obtained by combustion synthesis and a sulfurization treatment [1]. This result highlights the interest in further studying the mid-infrared (MIR) emission properties of rare-earth ions in La₂O₂S. This work presents an investigation of the structural and spectroscopic properties of Er:La₂O₂S oxysulfide with various rare-earth doping levels seen as the first step towards the fabrication of infrared transparent ceramics emitting at $\sim 2.8 \mu\text{m}$.

2 Results and discussion

The crystalline Er:La₂O₂S powders were prepared by the solid combustion method followed by sulfurization. The starting reagents were lanthanum nitrate La(NO₃)₃, Er(NO₃)₃, and thioacetamide C₂H₅NS (dissolved in distilled water). The nominal Er³⁺ doping level was 0.5 – 7 mol%. The precursor powder was obtained by combustion synthesis at 500 °C during 30 min. Then, the precursors were subjected to sulfurization at 1000 °C during 30 min under an atmosphere of H₂S and N₂ gases, taken in the ratio of 1:9.

The X-ray powder diffraction (XRD) revealed the single-phase nature of powders (hexagonal class, sp. gr. *D_{3d}^{3d} - P-3m1*) being isostructural to La₂O₂S, Fig. 1(a). The Raman spectra of Er:La₂O₂S are shown in Fig. 1(b). In the structure of La₂O₂S, the La site is coordinated by

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three oxygens and four sulfurs in the nearest neighbor having a symmetry of C_{3v} . All four Raman-active modes are assigned in the spectra, at 99 (E_g), 191 (A_{1g}), 358 (E_g) and 389 (A_{1g}) cm^{-1} . This low phonon energy behavior is beneficial for targeting mid-infrared applications as it is expected to reduce the non-radiative relaxation from Er^{3+} multiplets.

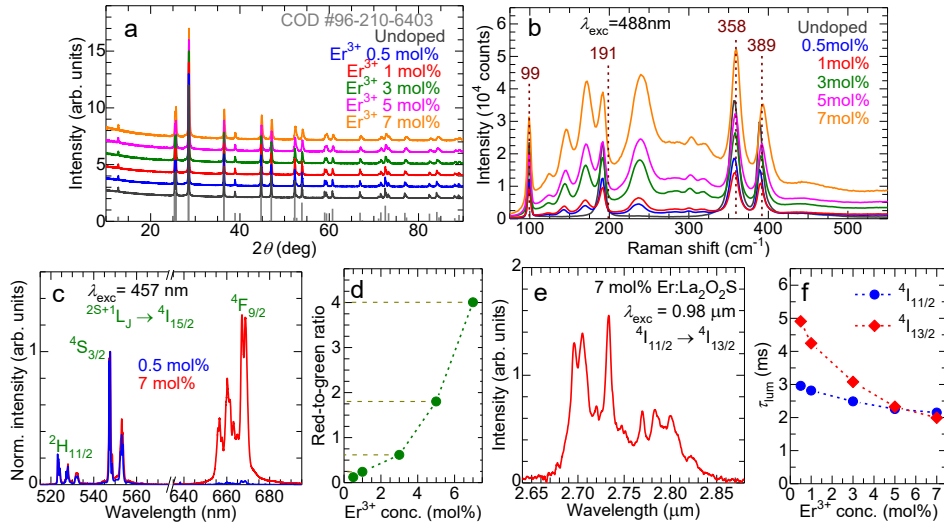


Fig. 1. Structure, vibronic and spectroscopic properties of $\text{Er}:\text{La}_2\text{O}_2\text{S}$ powders: (a) XRD patterns; (b) Raman spectra; (c-f) visible and infrared emission of Er^{3+} ions; (c) visible luminescence spectra, (d) red-to-green intensity ratio vs. Er^{3+} doping level; (e) MIR luminescence at 2.8 μm ; (f) luminescence lifetimes, τ_{lum} , of the $^4I_{11/2}$ and $^4I_{13/2}$ levels vs. Er^{3+} doping level. λ_{exc} – excitation wavelength.

The visible luminescence spectra are shown in Fig. 1(c). The emission bands in the green and red are assigned to the $^2H_{11/2} + ^4S_{3/2} \rightarrow ^4I_{15/2}$ and $^4F_{9/2} \rightarrow ^4I_{15/2}$ transitions, respectively. With the Er^{3+} doping, the relative emission intensity in the red (~ 660 nm) increases, as expressed by the red-to-green ratio which depends quadratically on the doping level, Fig. 1(d). This parameter is an indicator of the energy transfer efficiency between Er^{3+} ions, leading to self-quenching of the green emission. The observed behavior is favorable for the design of color tunable (green-to-red) visible powder and ceramic phosphors based on $\text{Er}:\text{La}_2\text{O}_2\text{S}$.

The MIR luminescence related to the $^4I_{11/2} \rightarrow ^4I_{13/2}$ transition spans from 2.65 μm to 2.85 μm , Fig. 1(e). The luminescence lifetimes τ_{lum} of the $^4I_{11/2}$ and $^4I_{13/2}$ states of Er^{3+} ion $\text{La}_2\text{O}_2\text{S}$ powders are plotted as a function of the doping level in Fig. 1(f). For 0.5 mol% Er^{3+} doping, they amount to 2.96 ms and 4.91 ms, respectively, while already at 7 mol% Er^{3+} doping, the lifetime ratio ($\tau_{\text{lum}}(^4I_{11/2}) > \tau_{\text{lum}}(^4I_{13/2})$) becomes favorable for the mid-infrared emission.

3 Conclusion

To conclude, heavily-doped 7 mol% $\text{Er}:\text{La}_2\text{O}_2\text{S}$ powders demonstrate broadband emission properties in the mid-infrared, around 2.8 μm , and a favorable ratio of the $^4I_{11/2}$ to $^4I_{13/2}$ luminescence lifetimes. Further work will focus on the fabrication of infrared transparent ceramics based on these materials.

References

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