

Broad NIR and visible emission from rare earth doped SiO₂-Nb₂O₅ nanostructured materials prepared by an alternative sol-gel route

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Among the preparation methods, the Sol-Gel process has been successfully used for obtaining a wide range of rare earth doped materials. Ordinarily, the sol-gel route employs metal alkoxide precursors, such as niobium ethoxide.¹ Nevertheless; these precursors normally are very sensitive to moisture. In view of that, this study focuses on the preparation and structural and spectroscopic characterization of Er³⁺-, Yb³⁺-, and Eu³⁺-doped and co-doped (100-x)SiO₂-xNb₂O₅, using optical grade Nb₂O₅ as an alternative precursor instead of niobium ethoxide. The Nb₂O₅ was dissolved in HCl, followed by drying with addition of anhydrous ethanol and rare earth ions. TEOS, anhydrous ethanol, and HCl were prepared in a separate container. The Nb₂O₅ and SiO₂ precursors were mixed and the resulting sols were maintained at room temperature for 60 days. Lastly, the xerogels were tritured to powder and annealed at 700 °C, 900 °C, and 1100 °C. The Nb₂O₅ crystallization was depended on the annealing temperature and Nb content, with the lanthanide ions distributed in the Nb₂O₅-rich environment preferentially, which affected the luminescent properties. The effect of the annealing temperature on the Nb₂O₅ crystallization process was assessed for the Er³⁺/Yb³⁺ co-doped 70Si:30Nb nanocomposites. An amorphous material emerged at 700 °C, whereas the orthorhombic (T-phase) and monoclinic (M-phase) Nb₂O₅ phases arose at 900 °C and 1100 °C. The NIR emission spectra of the samples showed a broadband with a maximum around 1530 nm corresponding to ⁴I_{13/2} → ⁴I_{15/2} transition of Er³⁺ ions when excited at 980 nm radiation. Full width at half maximum (FWHM) of 52 and 67 nm and ⁴I_{13/2} lifetime values of 5.6 and 5.4 ms were verified at 1.5 μm for 700 °C and 900 °C, respectively.² For the Er³⁺/Yb³⁺ co-doped (100-x)SiO₂-xNb₂O₅ nanocomposites annealed at 900 °C the FWHM and the ⁴I_{13/2} lifetime values were as high as 70 nm and 5.2 ms. The visible emission from upconversion processes also depended on the Nb content. For lower Nb concentrations, emission occurred mostly in the green region, whereas cross-relaxation enhanced the red emission for higher Nb concentrations. For the Eu³⁺-doped nanocomposites annealed at 900 °C a quantum efficiency values up to 71 % was calculated, where the Eu³⁺ are found distributed in different symmetry sites. In conclusion, photonic materials with excellent optical properties were developed, which paves the way for the applications such as optical amplifiers at 1.5 μm, solid-state lasers, energy converters and phosphors.

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