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Synthesis and characterization of $HfO_2:Eu^{3+}$ phosphor nanoparticles by polyol process

M. Villanueva-Ibáñez[(1)*], M.A. Hernández-Pérez[(2)] and M.A. Flores-González[(1)]

(1) Laboratorio de Nanotecnología - Universidad Politécnica de Pachuca, Pachuca-Cd. Sahagún Km 20, ex-Hda. de Santa Bárbara 43830, Zempoala, Hidalgo. México, e-mail: villanueva@upp.edu.mx
(2) Escuela Superior de Ingeniería Química e Industrias Extractivas, Instituto Politécnico Nacional, CP 07738, Mexico D.F., México.
* Corresponding author.

Abstract – Sub-micrometer Eu-doped HfO_2 luminescent powders were prepared from chloride precursors using for the first time a direct oxide precipitation in high-boiling polyalcohol solution. Structural and optical characterization of powders is presented. The structural properties were studied by X-ray diffraction, transmission electron microscopy, and scanning electron microscopy. The powders obtained consist of aggregates composed of nanoparticles (from 15 to 20 nm). The fluorescence properties were studied as a function of the annealing temperature and of the Eu^{3+} concentration. The red luminescence relative to the $^5D_0 \rightarrow ^7F_J$ transitions was observed.

Hafnium dioxide presents interesting physical and chemical properties. This oxide is widely used in optical fields because its high refractive index (≈2), low optical losses and scatter in the near UV (below 300 nm) and IR (10 μm) regions. The high density of HfO_2 (9.68 g/cm^3, Hf atomic number of 72), also makes it an attractive host lattice activated by rare earths for heavy scintillator [1]. Nanoparticles are under extensive study particularly in the case of luminescent materials because of a quantum confinement effect which leads to novel optoelectronic properties.

Numerous methods have been already reported to prepare HfO_2, principally the sol-gel process is known as an efficient and low cost method for the fabrication of HfO_2 with good optical properties [2,3]. In this work, the elaboration of sub-micrometer Eu-doped HfO_2 luminescent powders is conducted for the first time by an original approach with the polyol route. This process is based on the direct precipitation of oxides in a high boiling alcohol: diethylene glycol (DEG) [4,5]. The powders were calcinated and the crystalline structure and the luminescence were studied as a function of the annealing temperature (from 300 to 700ºC). The samples present structural changes depending on Eu^{3+} concentration (from 1 to 25 mol%). The monoclinic stable phase appears at low doping concentrations (fig.1). The powders obtained consist of aggregates composed of nanoparticles (around 15 to 20 nm) (fig. 2). Upon UV excitation, the emission spectra exhibit the well-known $^5D_0 \rightarrow ^7F_J$ lines (J= 0, 1, 2, 3, 4) of the Eu^{3+} ion.

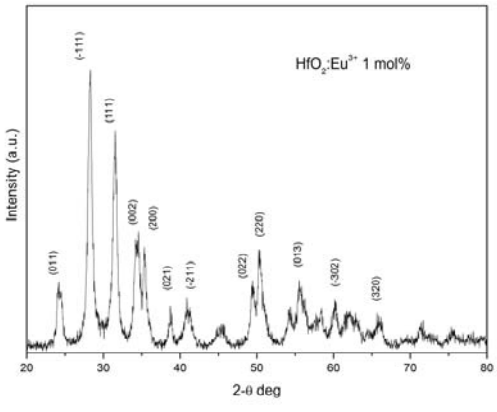

Figure 1: X-ray diffraction pattern of Eu-doped HfO_2 1 mol% heat-treated at 700ºC.

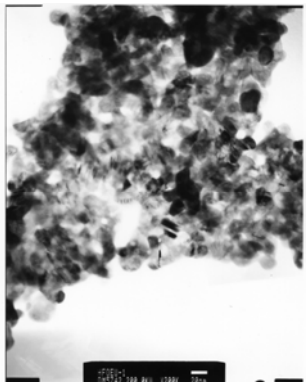

Figure 2: TEM observation conducted on Eu-doped HfO_2 1 mol% heat-treated at 700ºC.

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