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# Dual Emission of Dysprosium-Doped Hafnium Oxide Charecterization as a Function of the Sintering Temperature

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#### Abstract

The effect of the sintering temperature on the luminescence of trivalent dysprosium ion doped hafnium oxide compounds is studied. Temperature treatments of 400°C, 600°C, 800°C, and 1000°C over a period of 12 hours were used to prepare all the samples. Typical optical transitions of Dy<sup>3+</sup> ions are observed and properly labeled according to the <sup>2S+1</sup>LJ multiplets of this ion. Changes in luminescence behavior and the yellow/blue emissions ratio reveal that doping ions can occupy different sites in the host depending on the sintering temperature. The emission efficiency does not depend exclusively on the sintering temperature but also on the occupied site in the host matrix.

Keywords: Phosphors of metal oxides • Hafnium oxide compounds • Hydrothermal-microwave • Crystalline structures

### Introduction

Research on the luminescent properties of lanthanides has increased notably during the last decade. Phosphors of metal oxides doped with rare earth (RE) ions have been synthesized by various techniques, such as precipitation method [1], solid state reaction [2], hydrothermal-microwave [3], coprecipitation [4], ultrasonic pyrolytic spray [5,6], sputtering [7], polyethylene glycol [8], sol-gel [9-11], chemical vapor deposition (CVD) [12], atomic layer deposition (ALD) [13,14], solvent evaporation [15-17], metal organic chemical vapor deposition (MOCVD) [18,19], etc. On the other hand, Hafnium oxide is one of the materials that have been investigated for its mechanical, physical, chemical and electrical properties and with an interest due to its high dielectric constant, its high melting point, its wide band gap and excellent thermal stability [6-19]. The broader band gap of HfO<sub>2</sub> effectively suppresses luminescence thermal quenching effects, enhancing HfO<sub>2</sub> as an ideal material for a wide range of optoelectronic applications [20-23].

There are different crystalline structures in hafnium oxide such as: monoclinic, tetragonal and cubic, corresponding to the space groups P21/a, P42/nmc and Fm-3 m respectively. High temperatures are required to obtain the tetragonal and cubic phase; ~1700°C for the first and ~2200°C for the latter. These phases can be stabilized at temperatures below 1700°C, when the material is doped with different elements such as Magnesium, Barium, Lanthanum, Strontium, Neodymium, Calcium, Ytterbium, Yttrium, Samarium, Dysprosium, among others. [12,17,24-28].

Rare earth ion doped materials are widely used in the development of lasers, optical amplifiers, optical memory devices, medical lasers, flat panel displays, fluorescent lamps and white LED's, to give a few examples [29]. In this sense, Dysprosium is one of the most popular RE doping ions due to its characteristic transitions. Two of its dominant transitions in the emission spectrum are one in the Yellow (Y) region centered at 579 nm corresponding to the hypersensitive transition  ${}^{4}F_{9/2} \rightarrow {}^{6}H_{11/2}$  and a Blue (B) one at 485 nm corresponding to  ${}^{4}F_{7/2} \rightarrow {}^{6}H_{11/2}$  transition. These two emissions are very attractive for modern technology industry because of their potential to create an efficient white light source by simple adjustments of the Y/B ratio [30-34].

Obtaining phosphors by means of the solvent evaporation technique is one of the simplest to be carried out in the laboratory, because it is economical and versatile; the precursors can be dissolved in a suitable solvent; the solvent is evaporated at an appropriate temperature; and phosphors with excellent photoluminescent properties can be synthesized; using this technique, various concentrations of lanthanides can be incorporated during synthesis [35]. In this paper, an approach to improve the luminescence efficiency and optimize the emission ratio Y/B of Dy<sup>3+</sup> doped HfO<sub>2</sub> samples by controlling the sintering temperature is presented.

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The results show that for a given sintering temperature the blue emission can be optimized, and that the luminescence technique is a very effective one to distinguish between different crystal sites in one compound; these phosphors were synthesized by evaporation of solvents; were characterized by different techniques, such as X-ray diffraction (XRD), photoluminescence (PL), Energy dispersión spectroscopy of X-rays (EDS) and Raman.

# Method

For the sample preparation, typically 12 grams of HfCl, were dissolved in a solution of 20 ml of deionized water and 20 ml of methanol. Dysprosium doping was performed by adding dysprosium chloride (DyCl<sub>3</sub>) to the solution. The doping concentration for all the cases was 1 atomic percent. All the reagents were used without any further purification and the solution was vigorously stirred using a magnetic stirrer until a homogenous solution was formed. The obtained solution was continuously heated at 250°C for 20 minutes in order to evaporate solvents. The resulting white precipitate was completely dried at 350°C for ten minutes at ambient atmosphere and then cooled gradually to room temperature. The dried powders were sintered in air at 400, 600, 800 and 1000°C for 12 hours. Subsequently 1.2 cm diameter pellets were made by using a stainless-steel die statically pressing the compounds at about 100 MPa. The samples were characterized measuring the X-ray diffraction (XRD) pattern using a Siemens D-5000 diffractometer with Cu-Ka radiation and a Perkin Elmer LS55 fluorescence spectrometer was used to obtain the emission and excitation spectra; the (EDS) measurements were carried out using an Oxford Pentafet Si-Li detector integrated on a Leica-Cambridge Scanning Electron Microscope model Stereoscan 440 (SEM). Raman results were measured with a WITec combined Confocal Raman Imaging and Atomic Force Microscope System, highest sensitivity for 633-nm excitation wavelength.

### **Results and Discussion**

Figure 1a shows the X-ray diffraction spectra for the undoped  $HfO_2$  samples as a function of the sintering temperature.



**Figure 1.** (a) X-ray diffraction plots for HfO<sub>2</sub> samples for all the sinterization temperatures. The diffraction pattern can be associated with monoclinic

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structure of  $HfO_2$ . The blue arrow indicates a 30.3° diffraction peak associated to the orthorhombic structure. (b) This figure shows a comparison between a pure sample and a doped one, showing another peak related to the orthorhombic phase.

The diffraction curves correspond to samples treated at temperatures of 400, 600, 800 and 1000°C for periods of 12 hours, as mentioned before. All diffractograms were plotted with the same vertical scale and up-shifted to allow a clear comparison. The peak distribution of the XRD graphs can be associated nicely with the monoclinic structure of  $HfO_2$  (JCPDS 00-043-1017).

Similar diffraction spectra are observed for Dy<sup>3+</sup> doped samples. From Figure 1a, diffraction peaks centered at: 28.3°, 31.6°, 34.3°, 34.6°, and 35.4° can be matched respectively with the planes (111), (111), (002), (020), and (200), where the strongest diffraction is related to plane (111), central in HfO<sub>2</sub> monoclinic structure. The blue arrow indicates a diffraction peak at 30.3° of the plane (111) that is not part of the monoclinic structure but can be related with the orthorhombic structure according to HfO<sub>2</sub> (JCPDS 01-070-2832). Another orthorhombic phase diffraction peak can be found through an inspection and it is centered at 43.6° as shown in Figure 1b for undoped and Dy<sup>3+</sup> doped HfO<sub>2</sub> samples treated at 600°C. The introduction of Dy<sup>3+</sup> in HfO<sub>2</sub> does not show any structural change for 1% concentration.

Some important facts can be inferred from Figure 1. (a) The samples treated at sintering temperatures below 600°C do not present a well formed crystalline structure, (b) At higher temperatures, the crystalline formation is evident and it improves as the temperature increases, (c) The presence of diffraction peaks at 30.3° and 43.6°, that corresponds to an orthorhombic structure, reveals the formation of an additional crystalline structure for all temperatures above 400°C, and (d) the relative amplitude of the orthorhombic peak as compared to the rest of the diffraction peaks, indicates that as the temperature increases the monoclinic structure becomes dominant and orthorhombic structure diminishes. From Figure 1, in addition to the crystalline structure, it is possible to estimate the crystal sizes using the Scherrer's formula:

#### D=0.9λ/βcosθ

Where  $\lambda$ =1.5406Å is the X-ray wavelength,  $\beta$  is the full width at half maximum (FWHM) of the diffraction peak in the XRD patterns in radians, and  $\theta$  is the Bragg diffraction angle. The FWHM values of the peak using a pattern were adjusted to a Gaussian. The estimated sizes of the crystals are: 13.1 nm for 400°C, 18.6 nm for 600°C, 21.7 nm for 800°C, and 26.3 nm for 1000°C. All the estimations were performed using the strongest diffraction peak at 28.3°. As can be seen, the crystal size using the same composition increases with temperature as expected, which is why it has a greater crystallinity.

Figure 2 shows the Raman spectra of the undoped hafnium oxide phosphors and those doped with dysprosium, at different temperatures; the peaks observed at 109, 133, 147, 256, 381, 499, 578, and 672 cm<sup>-1</sup> can be assigned to  $A_g$  modes and the peaks found at 164, 242, 324, 335, 398, 521, 555, 639, and 775 cm<sup>-1</sup> correspond to the Bg modes of monoclinic HfO<sub>2</sub> [36-38]. The reflections and relative intensity of these peaks are in agreement with previous studies for the monoclinic structure of hafnium oxide [39]; the spectrum that corresponds to the dysprosium doped hafnium oxide shows that the monoclinic crystalline structure of hafnium oxide is not modified by the presence of this lanthanide; furthermore, it can be observed that the intensities of the peaks in both spectra show that the crystallinity of these phosphors increases according to the XRD results.

Another Raman peak that can be observed in undoped and Dy<sup>3+</sup> doped samples is the one centered at 445 cm<sup>-1</sup> this is reported to arise from orthorhombic HfO<sub>2</sub> [39,40]. The reason why the 800°C of the undoped samples presents an intense orthorhombic Raman scattering is not clear, because is not in agreement with XRD spectra, the possible inhomogeneous presence of orthorhombic crystals, the inherent characteristics of Raman spectroscopy technique and the randomly selected spot where the

laser incides could be the possible reasons for this result. A splitting or a shifting in the  $Dy^{3+}$  energy levels is not expected due to its 5s and 5p shells shielding, however, it is well known that crystal fields can influence on energy transitions probability, so that the presence of an orthorhombic phase could show an effect on  $Dy^{3+}$  luminescence as will be shown next.



**Figure 2.** Raman spectra for (a) undoped and b)  $Dy^{3+}$  doped  $HfO_2$  for all sinthering temperatures, showing the tipical characteristics of monoclinic phase, plus one peak centered at 445 cm-1 corresponding to orthorhombic phase.

Continuing the characterization, the excitation spectra for all the samples were measured using a selective emission from one of the main Dy<sup>3+</sup> transitions ( ${}^{4}F_{_{9/2}} - {}^{6}H_{_{13/2}}$ ) at 579 nm. Figure 3 shows excitation spectra for all sintering temperatures. The 800°C sample is nearly the same as the 1000°C sample, same intensity and form, so it was intentionally removed in order to have a clearest sight of the spectra. The same is applied for emission spectra.



Figure 3. Excitation spectra of Dy3+ doped HfO2 for all sinthering temperatures with emission centered at 579 nm.

Typical excitation wavelengths for Dy<sup>3+</sup> ion have been identified at the wavelengths of: 326, 353, 367, 393, 447 and 482 nm corresponding to transitions from the ground state to the excited energy levels:  $4M_{17/2}$ ,  $^{6}P_{7/2}$ ,  $^{6}P_{5/2}$ ,  $4I_{13/2}$ ,  $4I_{15/2}$  and  $^{4}F_{9/2}$  respectively. From Figure 3, it is possible to observe that the transition 4113/2 clearly shows a double energy level that is the contribution of  $^{4}F_{7/2}$  level. As can be noticed, the sample annealed at

#### 600°C shows the higher intensity.

Figure 4 shows the emission spectra at the excitation wavelength of 354 nm for sintering temperatures of 400°C, 600°C and 1000°C. It is important to notice that the spectrum for sample annealed at 400°C have similar luminescence characteristics of other samples even with its low crystallinity as expected from a lanthanide.



Figure 4. Emission spectra of Dy3+ dopes HfO2 for all sinthering temperatures with excitation at 354 nm corresponding to  ${}^6P_{_{7/2}}$  energy level.

From Figure 4, it is possible to observe a double emission characteristic for the transitions  ${}^{4}F_{_{9/2}}$  to  ${}^{6}H_{_{15/2}}$  and  ${}^{4}F_{_{9/2}}$  to  ${}^{6}H_{_{13/2}}$ , each of them with a pair of emission wavelengths at 487 nm and 492 nm and 580 nm and 592 nm, respectively and a low intensity red emission centered at 666 nm corresponding to  ${}^{4}F_{_{9/2}} \rightarrow ({}^{6}F_{_{11/2}} + {}^{6}H_{_{9/2}})$  transition.

Analyzing these spectra, it is easy to observe the dominance of the blue emission over yellow emission. The  ${}^4\text{F}_{_{9/2}}$   $\rightarrow$   ${}^6\text{H}_{_{15/2}}$  (blue) transition is magnetically allowed and hardly varies with the surrounding crystal field while the  ${}^{4}F_{u_{2}} \rightarrow {}^{6}H_{13/2}$  (yellow) transition is a forced electric-dipole transition, whose emission intensity is hyper sensitive to the local environment of the Dy3+ ions. The Judd-Ofelt parity law predicts that the mangnetic-dipole transition is allowed while the electric-dipole transition is forbidden, and the latter is allowed only on the condition that the Dy3+ ions occupy a low symmetry site without an inversion center, therefore the yellow and blue intensities ratio (Y/B) can be adjusted for a tunable emission so that the white emission can be reached by controlling the site symmetry. Figure 5 shows the CIE 1931 chromaticity diagram for the emission with excitation at 354 nm. The emission color can be tuned from green, orange and blue as can be seen, this shows the importance of the synthering temperature and local environment on the Dy3+ ion. The position of 600°C sample in yellow in the chromaticity diagram clearly shows the increase of the allowance of electric-dipole transition.



Figure 5. CIE 1931 chromaticity diagram of the  $Dy^{3+}$  doped  $HfO_2$  emission sintesized at different temperatures with excitation at 354 nm.

Taking into account that the 600°C sample presents the strongest excitation/ emission and considering that the XRD spectrum presents a minority contribution of an orthorhombic phase in the same sample, clearly shows that the presence of this phase increases the allowance of electric-dipole transition by distorting the local symmetry surrounding the Dy<sup>3+</sup> ions and the dominance of blue emission in 1000°C sample shows the stabilization of the monoclinic phase.

As can be seen, the blue emission diminishes when synthesis temperature increases beyond 600°C even when it's a magnetically allowed transition independent on crystal field, the observed reduction of the two emission peaks intensity is probably due to a quenching, which can occur as a result of: (1) loss of excitation energy from the emitting state due to cross-relaxation between the activators; (2) excitation migration owing the resonance between the activator ions and (3) paired activator ions acting as quenching center. The emission quenching mainly ocurrs from non-radiative energy transfer among Dy<sup>3+</sup> ions as exchange interactions, radiation reabsorption and multipolar interactions, because, as can be seen in Figures 3 and 4, there is an overlap between the emission and the  ${}^{4}F_{_{9/2}}$  energy level and appears that the quenching is accentuated when the local symmetry is mainly monoclinic. This is an interesting result that can be tested in a future work at different Dy3+ concentrations, synthesys temperatures and times. To present the observed transitions in a clearer form, an energy level diagram is introduced. Figure 6 shows the calculated energy levels for the free Dy<sup>3+</sup> ion [41]. The excitation wavelength is indicated by showing one solid upward arrow. The three solid downward arrows correspond to the discussed transitions in Figure 4, transition from  ${}^4\text{F}_{_{9/2}}$  to  ${}^6\text{H}_{_{15/2}}$  at 487 nm, transition from  ${}^{4}F_{_{9/2}}$  to  ${}^{6}H_{_{13/2}}$  at 580 nm and a transition from  ${}^{4}F_{_{9/2}}$  to  $({}^{6}F_{11/2} + {}^{6}H_{9/2})$  at 666 nm. The downward dashed arrow denotes the nonradiative transitions. The cross relaxation responsible for contribution of the non-radiative transitions could be attributed to  $({}^{4}F_{_{9/2}}, {}^{6}H_{_{15/2}}) \rightarrow ({}^{6}F_{_{11/2}} {}^{+6}H_{_{9/2}},$  ${}^6F_{_{3/2}})$  and  $({}^4F_{_{9/2}}, {}^6H_{_{15/2}}) \rightarrow ({}^6F_{_{3/2}}, {}^6F_{_{11/2}} + {}^6H_{_{9/2}})$  transitions [42,43]. Apart from the cross-relaxation channels, the resonant energy transfer between  ${}^4F_{_{9/2}}$ and <sup>6</sup>H<sub>15/2</sub> levels also causes the non-radiative relaxation.



**Figure 6.** Energy levels for the free  $Dy^{3+}$  ion calculated using the intermediate coupling theory and the non-radiative energy transfers between  $Dy^{3+}$  ions.

# Conclusion

Summarizing the important facts from the characterization we have: (1) the activation of  $Dy^{3*}$  ions in the HfO<sub>2</sub> matrix is clear from the intense emission spectra, (2) HfO<sub>2</sub> matrix presents two different phases and they are present for sintering temperatures above 400°C, (3)  $Dy^{3*}$ -doped samples show the existence of a sintering temperature threshold that triggers the dominance of the monoclinic structure and one temperature for the apparition of orthorhombic structure. The luminescent properties of  $Dy^{3*}$  ions in a matrix depend in general on the structure of the closest neighbors and the symmetry of the crystalline formation, (4) the XRD and Raman results indicate that the increase in the temperature of the synthesis of these phosphors, the crystallinity increases and the doping with dysprosium does not modify the Raman and the XRD spectra of the phosphors that are not doped.

Based on the addressed facts from the figures, it is possible to conclude that  $HFO_2$  matrix represents a convenient host for  $Dy^{3+}$  ions, giving the fact that for sinterization temperatures of 600°C the host matrix provides an adequate symmetry that acts as an active sensitizer giving outstanding conditions for the activation of  $Dy^{3+}$  ions and allowing a much more effective excitation process. Moreover,  $HFO_2$  matrix exhibits the formation of two phases that allows some tailoring of the standard and special transitions, in particular increasing the yellow over the blue intensity. It has been also shown that the luminescence technique is a very powerful one since it allows distinguishing the presence of different phases, no matter one of them is present in a small amount.

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