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Morphological and Optical Characterizations of $CaAl_2O_4$ Doped with Trivalent Praseodymium

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Abstract: This work was synthesized $CaAl_2O_4$ (calcium aluminate) doped with trivalent praseodymium ions. $CaAl_2O_4$ with praseodymium 1.0% and 0.5% were synthesized. The powders of aluminates were characterized by scanning electron microscopy and luminescence spectrofluorimetry. $CaAl_2O_4$ doped with praseodymium ion that showed less porosity in its grain structure and luminescent transitions that are dependent on the wavelengths of excitation and showing both types of transitions both 5d-4f and 4f-4f.

Key words: $CaAl_2O_4$, praseodymium ions, luminescence.

1. Introduction

The $CaAl_2O_4$ (calcium aluminate) may be applied in optical ceramics, catalyst supports, flame retardants and structural ceramics [1]. Luminescent applications of this material have been little studied by scientists. The synthesis of this aluminate is normally performed by routes such as gel process, which synthesizes fine and homogeneous powders [2]. This work was synthesized $CaAl_2O_4$ doped with praseodymium ions using gel route.

2. Experiments

2.1 Experimental Set Up

Synthesis of $CaAl_2O_4$:Pr^{3+} (1.0%) was made by dissolving ammonium hydroxide solution in EDTA (ethylene diaminetetraacetic acid) solution. Then, it was added solutions of aluminum nitrate and calcium nitrate. This mixture was heated at about 80 ℃ with continuous slow stirring for 2 h to evaporate water. After this time, it was added solution of $Pr(NO_3)_3$, heating at around 140 ℃ with continuous stirring to form a viscous solution and gelled. The precursor was pre-calcined at 200 ℃ in a muffle furnace, pulverized and calcined with temperature ramp of 20 ℃/min to 800 ℃ for 2 h. Synthesis of $CaAl_2O_3$:Pr^{3+} (0.5%) was performed in the same manner. For the characterization of powders of doped $CaAl_2O_4$, the authors used the techniques of SEM (scanning electron microscopy) and spectrofluorimetry.

3. Results and Discussions

3.1 MEV Analysis

The morphological characterization was made by SEM images were obtained to $CaAl_2O_4$:Pr^{3+} (1.0%) showing the morphology of doped $CaAl_2O_4$. Fig. 1(a) shows the shape of the particles is less varied in size and more irregular, presenting a more compact structure with less porosity and having a continuous distribution and defined as nano particulated material (Fig. 1(b)).

3.2 EDX Analysis

EDS (Energy Dispersive X-ray Spectrometry) was used to perform an elemental analysis of $CaAl_2O_4$:Pr^{3+}

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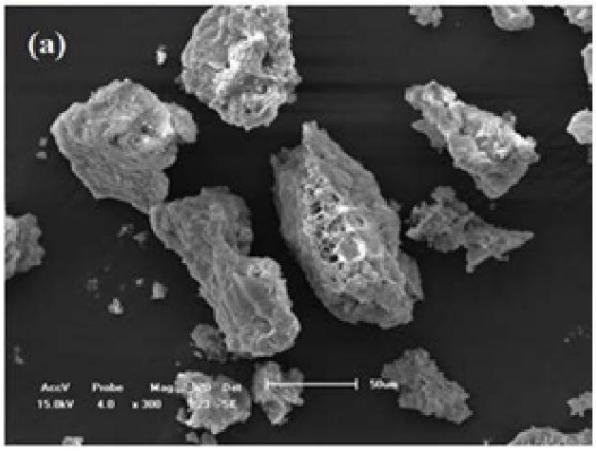

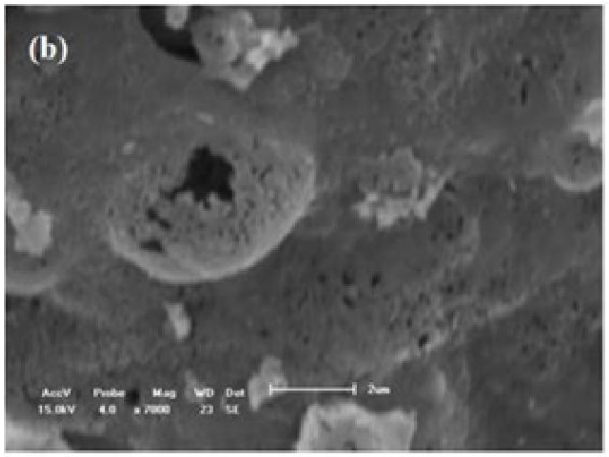

Fig. 1 SEM images for $CaAl_2O_4:Pr^{3+}$ (1.0%) with magnification of (a) 300X; (b) 7,000X.

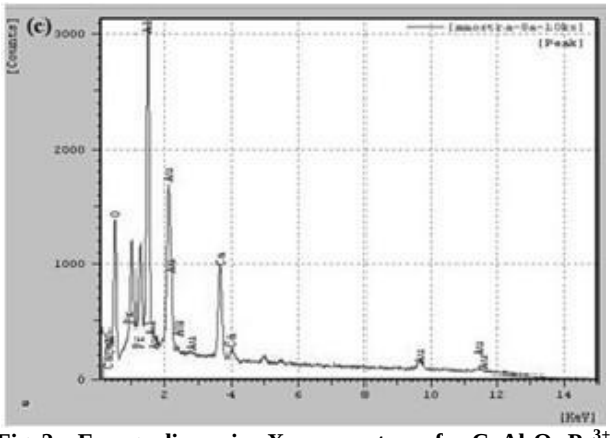

Fig. 2 Energy dispersive X-ray spectrum for $CaAl_2O_4:Pr^{3+}$ (1.0%).

(1.0%). Fig. 2 shows EDX spectrum, in which was observed the presence of Ca (calcium) in the indicated by the peaks of energy from 3.8 keV to 4.0 keV approximately. Others elements found were Al (aluminum), with one energy peak of approximately 1.7 keV, and Pr (praseodymium), with energy peaks at 1.0 keV and 1.1 keV approximately.

3.3 Luminescence Analysis

The characterization of luminescence was performed by spectrofluorimetry. The emission spectra of the samples doped with Pr^{3+} (1.0%) and (0.5%) are shown in Figs. 3 and 4, respectively. The spectra have been normalized for ease comparison. The wavelengths in the spectra were compared with the energy level diagrams in the literature [3, 4]. Fig. 3 shows emission spectrum with λ_{exc} = 280 nm having a broadband, related to an allowed d-f transition. For the emission spectrum with λ_{exc} = 465 nm, appeared a sharp band assigned to a f-f transition due to $^1D_2 \rightarrow {}^3H_5$. Fig. 3 (down) shows the emission spectrum with com λ_{exc} = 300 nm having a peak at 20,533 cm^{-1} (487 nm), which

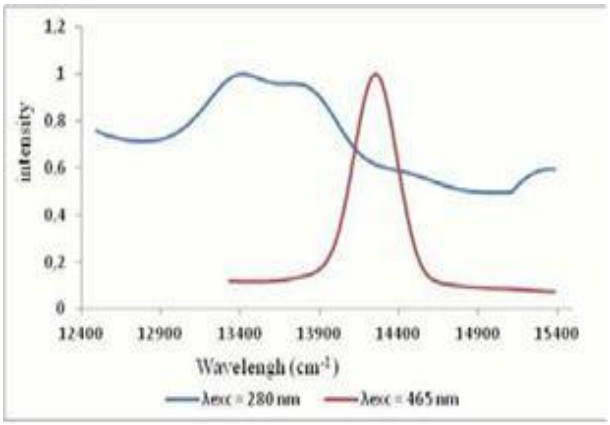

Fig. 3 Emission spectra for $CaAl_2O_4:Pr^{3+}$ (1.0%).

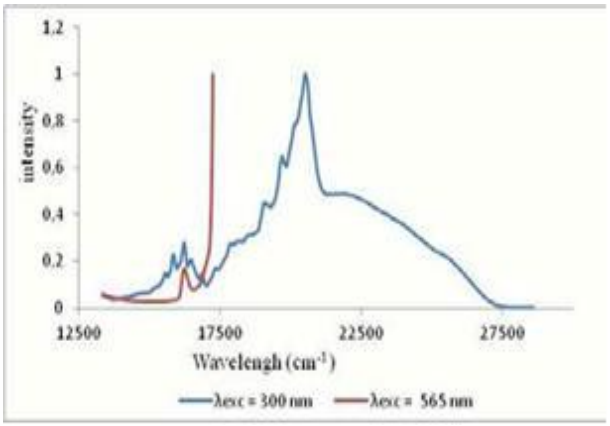

Fig. 4 Emission spectra for $CaAl_2O_4:Pr^{3+}$ (0.5%).

was attributed to $^3P_0 \rightarrow {}^3H_4$ transition and a less intense band at 16,181 cm^{-1} (618 nm) due to $^1D_2 \rightarrow {}^3H_5$ transition. In the emission spectrum with λ_{exc} = 565 nm there is an intense peak at 16,207 cm^{-1} (617 nm), which the transition is assigned to $^3P_0 \rightarrow {}^3H_6$. Those transitions were found for $CaAl_2O_4:Pr^{3+}$ (1.0%) and $CaAl_2O_4:Pr^{3+}$ (0.5%) and corroborated at Ronda et al. [5].

4. Conclusions

$CaAl_2O_4$ doped with praseodymium trivalent ion that showed less porosity in its grain structure were prepared. $CaAl_2O_4$ doped with praseodymium is luminescent with transitions that are dependent on the wavelengths of excitation and showing both types of transitions both 5d-4f and 4f-4f.

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References

[1] Mercury, J. M. R., Aza, A. H., and Pena, P. 2005. "Synthesis of $CaAl_2O_4$ from Powders: Particle Size Effect." *Journal of the European Ceramic Society* 25: 3269-79.

[2] Li, W., Zhuo, M. W., and Shi, J. L. 2004. "Synthesizing Nano $LaAlO_3$ Powders Via Co-precipitation Method." *Materials Letters* 58: 365-8.

[3] Chen, D., Wang, Y., and Hong, M. 2012. "Lanthanide Nanomaterials with Photon Management Characteristics for Photovoltaic Application." *Nano Energy* 1: 73-90.

[4] Hasegawa, Y., Wada, Y., and Yanagida, S. 2004. "Strategies for the Design of Luminescent Lanthanide (III) Complexes and their Photonic Applications." *Journal of Photochemistry and Photobiology C: Photochemistry Reviews* 5: 183-202.

[5] Ronda, C. R., Jüstel, T., and Nikol, H. 1998. "Rare Earth Phosphors: Fundamentals and Applications." *Journal of Alloys and Compounds* 275-7: 669-76.