A SYNTHESIS OF RARE EARTH ELEMENT DOPED \( \text{Ln}_2\text{O}_2\text{S} \) ( \( \text{Ln} = \text{Y}, \text{La}, \text{Gd} \) ) PHOSPHORS USING COMBUSTION REACTION

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Powder phosphors of \((\text{Y}_{1-x}\text{Re}_x)_2\text{O}_2\text{S}\), \((\text{La}_{1-x}\text{Re}_x)_2\text{O}_2\text{S}\), and \((\text{Gd}_{1-x}\text{Re}_x)_2\text{O}_2\text{S}\) where \(\text{Re} = \text{Eu}^{3+}, \text{Tb}^{3+}, \text{or Tm}^{3+}\), and \(x = 0.0005 ~ 0.032\) respectively, were prepared by combustion reaction from the mixed metal nitrate reactants and organic fuel such as dithiooxamide \((\text{CSNH}_2)_2\). In order to synthesize crystalline \(\text{La}_2\text{O}_2\text{S}\) powders, the reaction with dithiooxamide at \(\text{F/O} = 2.0\), defined below, was used.

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a \cdot [(1-x)\text{La(NO}_3)_3 + x \text{Eu(NO}_3)_3] + b \cdot [(\text{CSNH}_2)_2] \rightarrow a/2 \cdot [(\text{La}_{1-x}\text{Eu}_x)_2\text{O}_2\text{S}] + \text{by-products}
\]

where \(b/a = \text{fuel to oxidizer mole ratio (F/O)}\). The dehydrated reactants were fired at the ignition temperature \(T_i = 300 - 350^\circ\text{C}\). After reaction, the powder product was gently crushed with a mortar and pestle in the preparation for characterization.

As shown in fig.1, the XRD pattern of as-synthesized \(\text{La}_2\text{O}_2\text{S}:\text{Eu}^{3+}\) powder revealed that the \(\text{La}_2\text{O}_2\text{S}\) phase crystallized directly from the combustion reaction with dithiooxamide. No other phases were detected. The SEM image of as-synthesized \(\text{La}_2\text{O}_2\text{S}:\text{Eu}^{3+}\) powders showed that the morphology consisted of a foamy, porous agglomeration and a continuous three-dimensional network, presumably due to the rapid release of gaseous by-products. The agglomerates ranged in size between 10 \(\mu\text{m}\) and 30 \(\mu\text{m}\), while the primary particles ranged in size between 100nm and 200nm.

The phosphors prepared by combustion reaction were cathodoluminescent and photoluminescent exhibiting characteristic emission spectra of \(\text{Eu}^{3+}\), \(\text{Tb}^{3+}\), or \(\text{Tm}^{3+}\). Fig.2 shows the cathodoluminescent spectrum from as-synthesized \(\text{La}_2\text{O}_2\text{S}:\text{Eu}^{3+}\). The emission intensity from the \(^5\text{D}_0 \rightarrow ^7\text{F}_2\) electronic transition (at 626nm) exhibited a maximum at 0.5 mole % of \(\text{Eu}^{3+}\).

Finally, the effect of water on the combustion reaction, grinding, annealing and the oxidation stability of as-synthesized powder will be reported.

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Fig.1. X-ray diffraction pattern of as-synthesized \(\text{La}_2\text{O}_2\text{S}:\text{Eu}^{3+}\) (3.2mole%) powder prepared by combustion reaction with dithiooxamide (F/O = 2.0). (JCPDS # : 27-263 for \(\text{La}_2\text{O}_2\text{S}\))

Fig.2. Cathodoluminescence spectrum of \(\text{La}_2\text{O}_2\text{S}:\text{Eu}^{3+}\) (0.5mole%) measured at 2kV and 30\mu\text{A/cm}^2