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SYNTHESIS OF $SrF_2:Eu^{2+}$ UP CONVERSION PHOSPHORS

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Abstract- SrF_2:Eu upconversion luminescence phosphor synthesized via solid state metathesis route. The samples were characterized with X-ray diffraction (XRD) and upconversion emission spectra. A systematic study on the photoluminescence of Eu^{3+} doped SrF_2 samples with cubic shape showed that the optical properties of these products were strongly dependent on their morphologies and size. Though SrF_2 is formed by metathesis, luminescence centers were not formed in one step; as prepared samples did not show any luminescence. Sample reduced at1000^0C for 1hr exhibited very intense, characteristic emission. Activator concentration, and in turn, the intensity of luminescence depends on the partition coefficient of the activator in the host and the washable reaction products (KCl in the present case).In case of Europium, most of the activator is retained by SrF_2.
Keywords- SrF_2:Eu, solid state metathesis, up conversion, rare earths

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Introduction

Trivalent lanthanide(Ln^{3+}) doped upconversion materials can be utilized for short wavelength solid state lasers in both CW and pulsed modes without using any nonlinear frequency conversion, so they can provide in principle practical solutions for applications such as high density data storage, medical diagnosis and treatment, under water surveillance ,and full color all solid state displays [1–5]. A variety of methods are used for fabricating these anisotropic inorganic materials, most of which are synthesized in the presence of either hard templates or soft directing agents, including surfactants or polymers with complex functionalization patterns, which can direct the growth of inorganic crystals with controlled morphologies and architectures[6,7]. However, the addition of a template to the reaction system involves a complicated process and may result in impurity in the products. In contrast, a solid state metathesis process can overcome these difficulties and provide an attractive option for fabricating inorganic materials with diverse controllable morphologies or even special morphologies [8,9].

Rare-earth-ion-doped fluorides upconversion (UC) phosphors are of particular interest to be used in lasers, three-dimensional displays, light emitting devices, biological detection, and many others, due to their unique spectral property to transform the infrared (IR) light into visible and stable fluorescence[10-13]. As an important kind of fluoride, strontium fluoride (SrF_2) is dielectric and thus has great applications in micro electric and optoelectric devices[14,15] On the other hand, SrF_2 used as an attractive host for phosphors and activated with lanthanide ions (Ln^{3+}) have also been reported to display unique up/down-conversion luminescence properties[16,17]. However, to our knowledge, there are few reports on the synthesis SrF_2 upconversion luminescent behaviors of the products have been rarely investigated in detail.

Herein, a simple and one-step solid state metathesis synthesis was used to synthesize cubic-phase SrF_2: Eu^{3+} crystals. The photoluminescence

properties of SrF_2: Eu^{3+} were investigated. In this paper we report for the first time solid state metathesis route [18-20] for the preparation of SrF_2 with $CaCl_2$ as the starting material.

Experimental

SrF_2 with a molar composition was prepared by using starting material as 2KF and $SrCl_2$. All the reagents used were of the Analytical Reagent grade. $SrCl_2$:Eu powders were first prepared by dissolving $CaCO_3$ and Eu_2O_3 in concentrated HCl. Ratio of Sr:Eu was 99:1.The excess acid was distilled off in a closed assembly.$SrCl_2$:Eu powder so obtained was thoroughly mixed with anhydrous potassium fluoride in the proportion compatible with their action contents.

$2KF+SrCl_2= SrF_2$: Eu+2KCl

The resulting mixture was placed in a domestic microwave oven operating at 2.45GHz with 900W microwave power. After 05 min. the crucible containing the mixture was removed from the oven. The products obtained were washed with distilled water and ethanol several times to remove excess KF and KCl formed in the metathesis and then dried on a hot plate at 90^0C overnight. The insoluble matter was used for further experiments. For reducing Europium to divalent form, the $CaSrF_2$:Eu powder formed by the metathesis reaction was heated at 800^0C for1hr in a reducing atmosphere provided by the burning charcoal. X-ray diffraction patterns were recorded on a Philips PANalytical X'pert Pro diffractometer. Photoluminescence (PL) spectra in the spectral range220 –700nm were recorded on HitachiF-4000 spectro-fluorimeter with spectral slit width of 1.5nm.

Results and Discussion

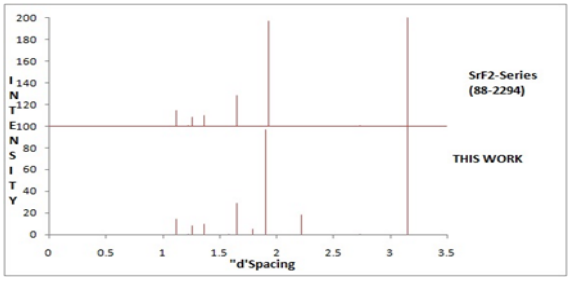

Fig. 1- Comparison of XRD Pattern of SrF_2 synthesized by solid state metathesis

Fig. 1 shows the XRD patterns of the as-prepared SrF_2 powder with the ICDD data file 88-2294. This crystallizes in BCC phase. An excellent match is seen. SrF_2 is thus successfully prepared by the solid state metathesis.

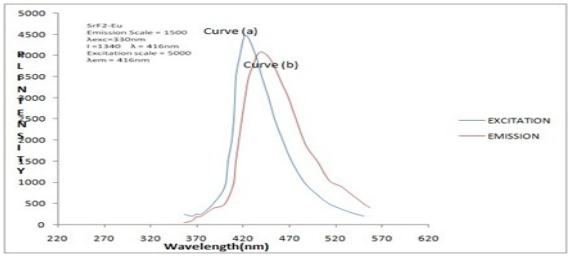

Fig. 2- Typical PL spectra of Eu^{2+} in SrF_2

SrF_2 is wide band gap material with a band gap of 11.4 eV. It is extensively studied for its ionic conductivity properties.[21].Pr^{3+} doped SrF_2 is studied for VUV spectroscopy.Tm^{3+} doped SrF_2 is a suitable up-conversion material for red to blue conversion[22] Fig.2 shows PL excitation(curve a) and emission (curve b) spectra of SrF_2:Eu^{2+}.Excitation spectra(fig.2a) of SrF_2 consist of 330nm.An intense emission(fig.2b) at 416nm is obtained in the sample prepared by metathesis. We have obtained similar results for CaF_2:RE and $CaSO_4$:Eu^{2+} samples prepared by the metathesis [23,24]

Conclusions

SrF_2:Eu is formed by solid state metathesis which is a fast method for synthesis. However, preparation of phosphors is not straight forward. The formation of SrF_2 was confirmed by XRD. Intense upconversion luminescence of the glass ceramics, including strong emission at 416nm.This demonstrates the potential application of the glass ceramics in the optoelectronic fields such as short wavelength solid state lasers.

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