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Solid state metathesis of (Ba,Sr)SO_4:Eu^{2+} phosphor by microwave assisted synthesis

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Abstract

A novel microwave initiated solid state metathesis synthesis of oxide is investigated previously in which the high lattice energy of NaCl drives the solid state metathesis reaction in forward direction to obtain the final product. (Ba,Sr)SO_4:Eu^{2+} could be prepared by solid state metathesis in few minutes using domestic microwave oven. Solid state metathesis of some double sufates like (Ba,Sr)SO_4 :Eu^{2+} can be performed which is quite attractive as it is fast and simple. The reaction gets completed within few minutes. The host was doped with activators like Eu and then was characterized using XRD and PL techniques.

Keywords: ***Solid state metathesis, Phosphors, Photolumenescence, (Ba,Sr)SO_4:Eu^{2+}.***

1.0 INTRODUCTION

The rare earth doped luminescent materials play integral role in modern life with tremendous applications ranging from color display, Scientillators, fluorescent lamps, intensifying screens, dosimentry of ionizing radiations and so on [1,2,3]. The optical properties of phosphor materials are influenced by chemical compositionand by the presence of dopants or impurities and its concentration. Metathesis (double exchange) reaction taking place in solid state are not quite common. However, these extremely fast, self energetic reactions yield crystalline materials in very short time intervals with unusual microstructures [4].

Hydrothermal methods where the reaction temperature is around 200^oC, in general follow complex preccedures in addition to prolonged reactiontimes of up to several days [5]. Therefore available procedures for the synthesis of phosphors increase the capital cost requirement ultimately increasing the production cost in addition to producing poor quality materials. A simpler procedure that can overcome the above disadvantage yet produce high purity materials in a cost – effective manner is needed.

In this paper, we described synthesis of (Ba,Sr)SO_4:Eu^{2+} using solid state metathesis. In solid state metathesis, exchange of bonds between the two reacting chemical takes place and results in creation of products having identical bonding affiliations.

1.1 EXPERIMENTAL

The chemicals used for the reaction were of Analytical Reagent grade. $SrCl_2$:Eu powder was first prepared by dissolving $SrCO_3$ and Eu_2O_3 in concentrated HCl in the stoichiometric ratio of 99:1. Simillarly $BaCl_2$ powder was

prepared by dissolving $BaCO_3$ in concentrated HCl solution. The excess acid while formation of $SrCl_2$:Eu and $BaCl_2$ powder was evaporated. Chlorides so formed were thouroughly mixed with Na_2SO_4 powder in the proportion compatible with the metathesis reaction.

$$BaCl_2 + SrCl_2{:}Eu + Na_2SO_4 \rightarrow (Ba,Sr)SO_4{:}Eu + 2NaCl$$

A china dish containing the resulting mixture was placed in a domestic microwave oven operating at 2.45 GHz and output power of 900W. The reaction mixture was irradiated in oven for nearly 10 minutes and then removed from microwave oven. The unreacted reagents used in reaction, like SrCl2:Eu, $BaCl_2$, Na_2SO_4 and NaCl formed in metathesis can be easily removed by repeatedly washing the products with highly pure distilled water. The final yield was then dried on a hot plate at 90°C overnight. The insoluble matter was used for further experiments [6,7,8].

X- ray diffraction patterns were recorded on a Philips PANalytical X'pert Pro diffractometer. Photolumenescence spectra were recorded on a Hitachi F- 4000 spectrofluorimeter with a special slit width of 1.5 nm in the spectral range 220 – 700nm.

1.1.1 RESULT AND DISCUSSIONS

The crystal structure of the resulting $(Ba,Sr)SO_4$ was investigated by XRD measurements. The XRD pattern displayed in Fig. 1 shows the comparison of XRD patterns of $(Ba,Sr)SO_4$ powder prepared by the metathesis method with the ICDD data file 39-1468. An excellent match was seen. No reflection peaks from other impurities were detected.

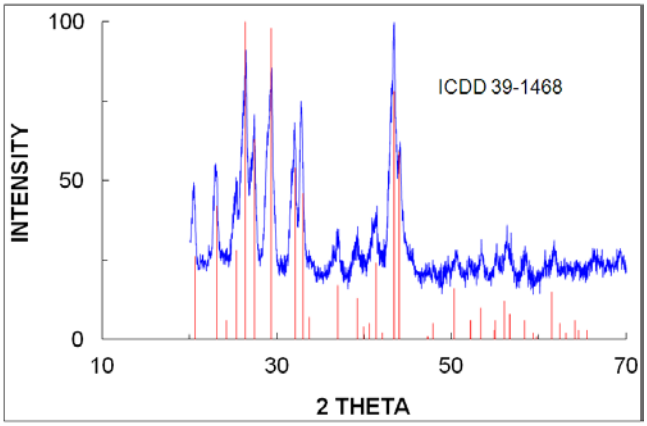

Fig. 1: XRD results for $(Ba,Sr)SO_4$. Stick pattern synthesized by solid state metathesis is compared with the ICDD file.

Fig. 2 shows the PL excitation (curve a) and emission (curve b) spectra of $(Ba,Sr)SO_4:Eu^{2+}$. When the sample is excited at 254nm, it shows a broad – band emission at 380nm. The band at 380nm can be assigned to transitions between the lowest band of the $4f^6 5d$ configuration and the ground state $^8S_{7/2}$ of the $4f^7$ configuration of Eu^{2+} [9].

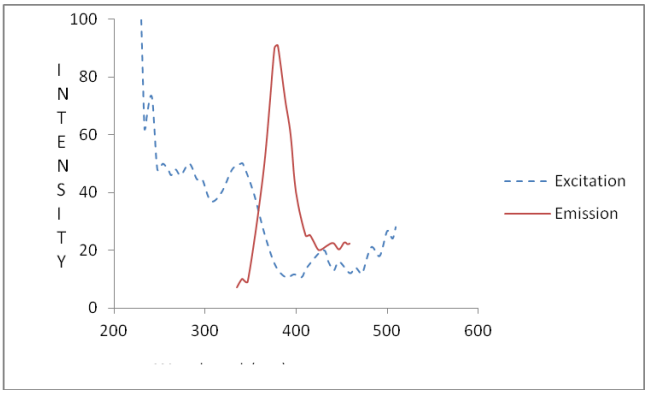

Fig. 2: PL spectra for $(Ba,Sr)SO_4:Eu^{2+}$. (a) Excitation spectra of $(Ba,Sr)SO_4:Eu^{2+}$. (b) Emission Spectra of $(Ba,Sr)SO_4:Eu^{2+}$.

2.0 CONCLUSION

The prepared $(Ba,Sr)SO_4:Eu^{2+}$ phosphor by microwave assisted solid state metathesis exhibits an orthorhombic structure and is confirmed by XRD analysis. The solid state metathesis is the feasible method for the preparation of mixed sulphates required relatively less time and less energy. The intense photoluminescence peak at 380 nm is suitable for the application of material in photocopying lamps as well as in sun tanning applications [10]. It can be again concluded that, this phosphor exhibits a high sensitivity; it might prove useful in TL dosimetry for low exposures of ionizing radiation. From literature survey, it can be concluded that the sensitivity of this phosphor is found to be at least 7 and 2.5 times more than those of $SrSO_4$:Eu and $BaSO_4$:Eu phosphors.

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