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The Purification Process On Scintillator Material (SrI₂: Eu) By Zone-refinement Technique

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Abstract. The thermal properties of Europium doped strontium iodide was analyzed through Thermogravimetric (TG) and differential thermal analyses (DTA). The melting point of europium doped strontium iodide is around 531°C. The hydrated and oxyhalide impurities were found before melting temperature. In order to remove these impurities we have done purification process by Zone-refinement technique. The effective output of purification of zone refining was also observed through the segregation of impurities.

Keywords: Scintillation detectors, Growth from melts; zone melting and refining, Impurities in crystals, Alkali and alkaline earth metals

PACS: 29.40.Mc; 81.10.Fq; 61.72.S; 71.20.Dg

INTRODUCTION

Metal halide materials (Br⁻, Cl⁻, and I⁻) have a large potential as scintillators and are interesting materials for luminescence and fluorescence. Gamma ray detection techniques are used in homeland security, medical imaging and many other applications. In last few years lot of work has been done on several scintillators materials like Tl:NaI, Ce:LaBr₃, Bi₄Ge₃O₁₂ and SrI₂:Eu [1]. Specifically, SrI₂:Eu single crystal has a high light yield (90,000 photons/MeV)[2], fast response (~1.3 µs), high energy resolution (~2.6% at 662 keV)[3], high density (4.55 g/cm³), emission range (430 nm) and transparency. But the growth of SrI₂:Eu is still challenging because of its strongly hygroscopic nature. Another problem is the purity of the raw materials and the material contact with ampoule wall induces thermal stress. Many research works were devoted to optimize the crystal growth process and improve the energy transfer processes in strontium iodide crystals.

In this work we have studied the thermal properties of SrI₂:Eu by using Thermogravimetric (TG) and Differential Thermal Analyses (DTA). And also we have reported the purification process of this material by using Zone-refinement technique.

EXPERIMENTAL

TG and DTA of the SrI₂:Eu material were carried out using Perkin Elmer thermal analyzer instrument. An alumina crucible was used for heating the sample and analyses were carried out in an atmosphere of dry high pure nitrogen at a heating rate of 10°C/min in the temperature range of 30°–650°C followed by cooling to 200°C. The results obtained from TG and DTA thermal studies are shown in Fig. 1.

FIGURE 1. TG/DTA of SrI₂:Eu

The homemade single-pass zone refinement process setup is shown in the Fig. 2. The starting materials used were iodide powders with ultra dry purity of 99.9% (EuI₂) and 99.99% (SrI₂) from Sigma Aldrich and Alfa Aesar respectively. The materials were weighed according to stoichiometric ratio of
SrI₂:5% Eu²⁺. The weighted materials were grounded in an agate mortar for homogeneous mixing. The grounded materials were loaded in a freshly cleaned and pre-backed quartz ampoule. This process is carried out under argon atmosphere and humidity maintained in a glove box was 10%. The ampoule was sealed under high vacuum of 2x10⁻⁶ torr and same time ampoule was heated in 200° C. The dimension of the ampoule is approximately 200 mm long with a diameter of 14 mm in the base configuration. The ampoule was transferred in to a horizontal zone refining system.

RESULTS AND DISCUSSIONS

Thermal Analysis

In the figure.1, first endothermic peak in the DTA curve is observed at 90° C and is assigned to the hexa water molecule evaporating on the material. The second endothermic peak around 193° C corresponds to the dehydrate water molecules which are evaporated.

FIGURE 2. Photograph of the zone-refinement experimental setup.

The furnace is heated at room temperature to 350° C in 10 hours and from 350° C to 550° C in 12 hours. The zone-refinement temperature profile is shown in the Fig. 3. The right end of the ampoule was fixed in maximum temperature of the furnace. The length of molten zone was ~2 cm and the furnace translation speed was kept constant at 0.66 mm/h by the controller of the drive motor. This process is repeated three times. Then it is slowly cooled to room temperature. The impurities are segregated from right to left corner of the ampoule which is shown in Fig. 4.

FIGURE 3. Temperature profile of Zone-refinement process.

The segregated materials are decomposed part of the starting material. The coloration is from escaping iodine gas.

FIGURE 4. The segregation of the impurities after zone refinement process.

The third endothermic peak at 323° C may be possibly due to evaporation of hydrate containing a lower number of water molecules. The last endothermic peak is observed at 531° C and is assigned to the melting point of the prepared material (SrI₂: Eu). The freezing point of the material is 473° C. There is a weight loss between 50° C and 330° C, which is due to impurities that are evaporating. There is no phase transition before the melting point.

CONCLUSIONS

The insoluble hydrate impurities from SrI2:Eu material was successfully removed by using Zone-refining process. It is expected that this purification will allow improving further crystal growth process of this material. From the TG/DTA melting point and freezing point of SrI₂:Eu were observed to be 531 °C and 473 °C respectively.

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REFERENCES