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Synthesis, Characterization and TL Properties of γ-ray Irradiated Copper doped Calcium/Strontium Tetraborate Inorganic Phosphor via Solution Combustion Method

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Abstract
The polycrystalline samples of copper doped CaB_4O_7 and SrB_4O_7 phosphors are synthesized by modified solution combustion technique. XRD proved confirmation while SEM provides particle sizes. Thermoluminescence properties (TL) of (Ca, Sr) B_4O_7: Cu phosphors are observed by exposing it by gamma-rays of suitable (about 5 Gy) dose. Thermoluminescence (TL) glow curve of the CaB_4O_7: Cu and SrB_4O_7: Cu phosphors show peaks at 154 ºC and 237 ºC sample.

Keywords: Borates, Thermoluminescence, Kinetic parameters.

1. Introduction
Low synthetic temperature, High Luminescent brightness and easy preparations etc., are the plus points of borates in comparison with the other investigated hosts [1-3]. The simplistic synthesis with quite cheap raw materials like boric acid and urea results in excellent chemical and thermal stabilized alkaline earth borate [4-5]. Following Daniels et al [6], lots of researchers have applied the thermoluminescence phenomenon on dosimetry purpose. Some of the investigations on the TL characteristics of borates are un-doped and Ce-doped BaB_4O_7 [7], Tb^{3+} doped $Ba_2Ca(BO_3)_2$ [8], un-doped and Cu and Mn doped $K_2B_4O_7$ [9], MgB_4O_7:Dy,Na [10], SrB_4O_7:Dy [11], rare-earth doped $Sr_2Mg(BO_3)_2$ [12], $Li_2B_4O_7$:Cu,In [13,14] and BaB_4O_7: Dy [15]. The studies for the TL phenomenon of borates were started by the work of Schulman et al [16]. Thermoluminescence dosimetry (TLD) has been widely applied in areas such as clinical, personal and environmental monitoring of ionizing radiation. Much research has been carried out to find better dosimetric materials and borates fulfilled the needs of sensitivity with near tissue equivalent absorption coefficient, to some extent [17-19]. The copper containing materials are supposed to be the most sensitive known thermoluminescence (TL) phosphors and proposed for dosimetry applications [20, 21]. We studied the TL characteristics of copper activated CaB_4O_7 and SrB_4O_7 under the irradiation of γ-rays, which was presented in this paper

2. Experimental
The (Ca,Sr) B_4O_7: Cu Phosphors was obtained by the combustion of aqueous solution containing stoichiometric amounts (using oxidizer/fuel ratio) of calcium/strontium nitrate, copper chloride, ammonium nitrate, urea and Ammonium Pentaborate as boron source. All the precursors (AR grade) were dissolved in a china dish using minimum amount of water. The dish containing the solution was introduced into a muffle furnace maintained at 823±10 K. The solution undergoes dehydration followed by decomposition with the evolution of large number of gases (oxides of nitrogen and ammonia) and ignited to burn with a flame yielding voluminous powder of SrB_4O_7: Cu This raw powder was sintered for 2 h at 1023 K and cooled to room temperature on aluminium plate and crushed into a fine powder. The same process is repeated for the different concentration of copper [22, 23]. The prepared

powder samples were then subjected to the powder XRD analysis. Samples were exposed to gamma rays from a ^{60}Co-source at room temperature. After the desired exposure, TL glow curves were recorded for samples at a heating rate of 5 K/s. The photoluminescence of as prepared sample of CaB_4O_7: Cu (0.007 mol) over 200–400 nm excitation range was taken on a HITACHI F-7000 fluorescence spectrophotometer.

Table-1: Balance reaction for the phosphor.

Product	Corresponding reaction with balance molar ratios of precursors
$Ca/Sr_{(1-x)}\ B_4O_7$: xCu	(Ca/Sr) $(NO_3)_2$ + **1.6** $NH_4B_5O_8$ + **10.2** $CO(NH_2)_2$ + **10.2** NH_4NO_3 + **x**$CuCl_2$ $\longrightarrow$ **$Ca/Sr_{(1-x)}\ B_4O_7$: xCu** + Gaseous (H_2O, NH_4 and NO_2 etc) [x = 0.001, 0.002, 0.005, 0.01 and 0.02]

3. Results and Discussion

3.1. X-ray Diffraction of CaB_4O_7: Cu

Figure-1 represents the XRD pattern for polycrystalline sample of CaB_4O_7: Cu and SrB_4O_7: Cu. The results are confirmed by comparing the observed XRD with standard ICDD file (00-031-0253) and (00-015-0801) for CaB_4O_7: Cu and SrB_4O_7: Cu respectively, which is in good agreement and show peak to peak matching.

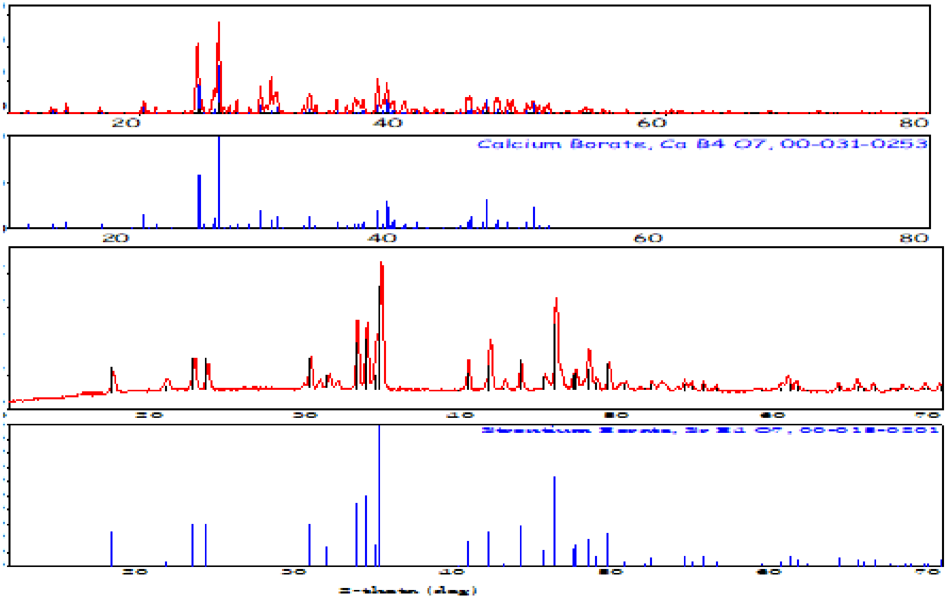

Fig. 1: XRD image of CaB_4O_7: Cu and SrB_6O_7: Cu.

3.2 SEM Analysis

SEM image of this phosphor CaB_4O_7: Cu and SrB_4O_7: Cu was taken from Synthetic and Art Silk Mills Research Association (SASMIRA), Mumbai (Fig. 2). The material shows irregular spherical as well cylindrical shape particles with stone like structure. The powder sample shows the sizes of particles ranging from 2 µm to 5 µm. This irregularity in masses may be caused due to the irregular mass flow during combustion process.

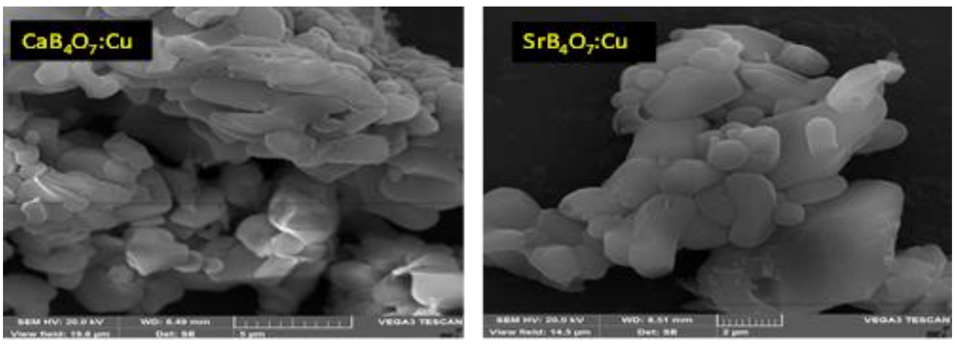

Fig. 2: SEM images of CaB_4O_7: Cu and SrB_4O_7: Cu.

3.3 Thermoluminescence Studies

The prepared sample CaB_4O_7: Cu and SrB_4O_7: Cu phosphor exposed to ^{60}Co gamma-ray radiation source at RTM Nagpur University with dose rate of 0.3712 kGy / hr. The TL glow curve of newly developed SrB_4O_7: Cu and CaB_4O_7: Cu for a test dose of 5 Gy is shown in the Fig. 3 obtained at heating rate of 5 °C /sec. Glow curve for CaB_4O_7: Cu and SrB_4O_7: Cu has the main TL peak at about 150-250 °C which is good symptom for this phosphor to be used as good TLD material. [15]

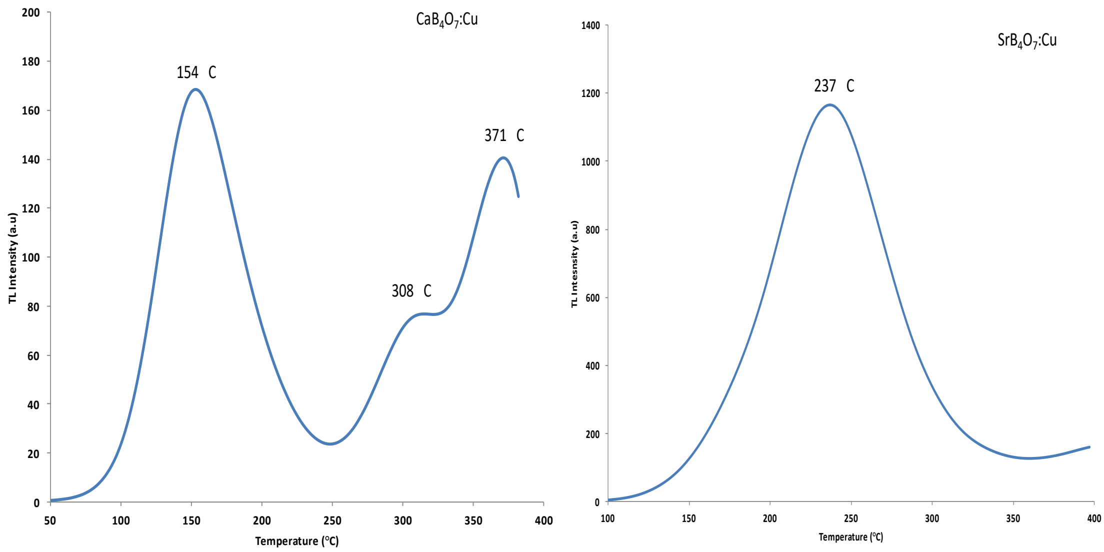

Fig. 3: TL glow curve of CaB_4O_7: Cu and SrB_4O_7: Cu phosphor.

4. Conclusions

In current report X-ray diffraction result supports the complete crystalline formation of CaB_4O_7: Cu and SrB_4O_7: Cu by combustion synthesis. Thermoluminescence studies of these two samples are compared and found to be in the range of 150-250 ℃. Again SrB_4O_7: Cu shows single peak while CaB_4O_7: Cu have additional shoulder peak at 308 ℃ and 370 ℃ respectively. Hence the phosphor CaB_4O_7: Cu and SrB_4O_7: Cu are said to be best for TLD phosphor.

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