COMBUSTION SYNTHESIS AND THERMOLUMINESCENCE STUDIES OF γ-ray IRRADIATED $K_2B_4O_7$: Cu PHOSPHOR

Z. S. Khan*[1], N. B. Ingale[2], S. K. Omanwar[3]
*[1]Department of Physics, The Institute of Science, Mumbai, Maharashtra, India
[2]Department of Applied Science, Prof Ram Meghe Institute of Technology and Research, Badnera, Maharashtra, India
[3]Department of Physics, SGB Amravati University, Amravati, Maharashtra, India

ABSTRACT
Copper doped K2B4O7 material is synthesized by modified solution combustion technique. X-ray diffraction (XRD) and Scanning Electron Microscope (SEM) characteristics are carried out. Exposing with gamma-rays of suitable (about 5 Gy) dose, thermoluminescence (TL) properties of K2B4O7: Cu phosphor is observed. Thermoluminescence (TL) glow curve of the K2B4O7: Cu phosphors show two peaks at 235 ˚C and 340 ˚C with a shoulder at 420 ˚C. The kinetic parameters are calculated using peak shape method. The effect of dose variation on K2B4O7: Cu phosphor is also studied
Keywords: Borates, Thermoluminescence, Activation Energy, Frequency Factor

I. 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]. The simplistic synthesis with quite cheap raw materials like boric acid and urea results in excellent chemical and thermal stabilized alkaline earth borate [2,3]. Following Daniels et al (1953) [4] 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 [5], un-doped and Cu- and Mn-doped $K_2B_4O_7$ [6], MgB_4O_7: Dy, Na [7], SrB_4O_7: Dy [8], $Li_2B_4O_7$: Cu, In [9] and BaB_4O_7: Dy [10]. The studies for the TL phenomenon of borates were started by the work of Schulman et al (1967) [11] 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 [12]. The copper containing materials are among the most sensitive known thermoluminescence (TL) phosphors [13]. These phosphors are suggested to be used for dosimetry applications [14]. We studied the TL characteristics and some dosimetric properties of copper activated $K_2B_4O_7$ under the irradiation of γ-rays, which was presented in this paper.

II. METHODS AND MATERIAL

The $K_2B_4O_7$: Cu Phosphor was obtained by the combustion of aqueous solution containing stoichiometric amounts (using oxidizer/fuel ratio) of potassium nitrate, copper chloride, ammonium nitrate, urea and Ammonium Pentaborate as boron source [15, 16]. 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 amount of gases (oxides of nitrogen and ammonia) and ignited to burn with a flame yielding voluminous powder of $K_2B_4O_7$: 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. 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 $K_2B_4O_7$: Cu (0.007 mol) over 200–400 nm excitation range was taken on a HITACHI F-7000 fluorescence spectrophotometer.

TABLE I

BALANCED REACTION FOR $K_2B_4O_7$: CU

Product	Corresponding reaction with balance molar ratios of precursors
$K_2B_4O_7$: Cu	$KNO_3 + H_3BO_3 + 5\ CO(NH_2)_2 + 7.5\ NH_4NO_3 + x\ CuCl_2 \longrightarrow K_2B_4O_7$: Cu + Gaseous ($H_2O$, NH_4 and NO_2 etc)

III. RESULTS AND DISCUSSION

Figure-1 represents the XRD pattern for polycrystalline sample of $K_2B_4O_7$: Cu. It is found to be Monoclinic with space group P21/n (14) and lattice parameter a = 12.26 A.U, b = 9.895 A.U, c = 7.796 A.U. The results are confirmed by comparing the observed XRD with standard ICDD file (00-031-0253) which is in good agreement and show peak to peak matching.

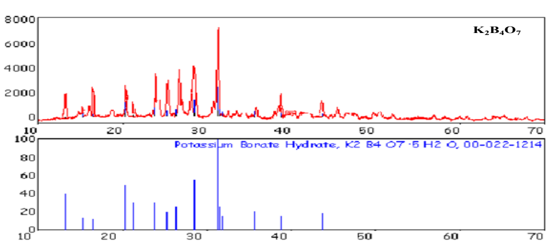

Fig. 1 XRD pattern of $K_2B_4O_7$: Cu

SEM image is represented in Fig. 2 for solution combustion synthesized $K_2B_4O_7$: Cu material. The material shows irregular shape expanded particle structure. It shows the sizes of particles from 0.5 µm to 5 µm range. The irregularity may be caused due to the irregular mass flow during combustion process.

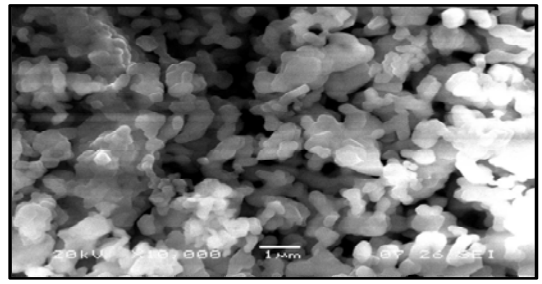

Fig. 2 SEM image of $K_2B_4O_7$: Cu

The TL glow curve of newly developed $LiBaBO_3$ with different concentration of Eu^{3+} for a test dose of 1 Gy is shown in the Fig. 3 obtained at heating rate is 5 °C /sec. The glow curve consists of two peaks P_1 and P_2 at 170 ^{0}C and 266 ^{0}C respectively. This range (175-225 ˚C) is favorable for the desired dosimetry purposes [17]. Here 0.002 mole of Eu^{3+} shows optimum thermoluminescence (TL) intensity for this phosphor. The TL glow curves for $LiBaBO_3:Eu^{3+}$ phosphors were deconvoluted by using the PeakFit software [18] as shown in Fig. 3. We employed the peak shape method to analyze the activation energy of $LiBaBO_3:Eu^{3+}$ phosphors using formula used by Mckeever [19].

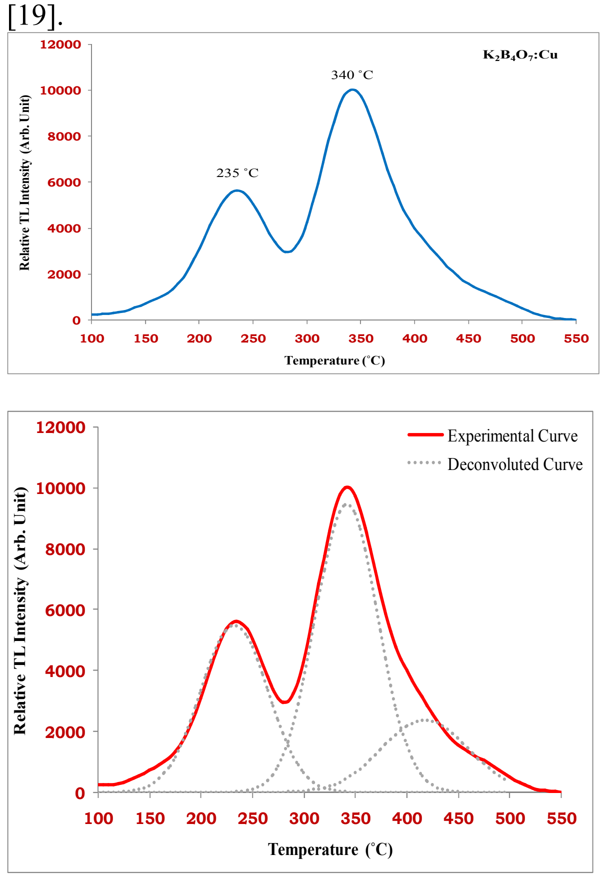

Fig. 3 TL glow peak & deconvoluted TL glow curve for $K_2B_4O_7$: Cu

TABLE III
Kinetic parameters for $K_2B_4O_7$: Cu

	Peak	T_m (˚C)	μ_g	E (eV)	S (s^{-1})
$K_2B_4O_7$: Cu	**P_1**	235 ˚C	0.47	0.961	7.24 X 10^8
	P_2	340 ˚C	0.53	1.425	1.12 X 10^{11}
	P_3	420 ˚C	0.50	1.341	8.97 X 10^8

We have decided 5 Gy to 25 Gy dose for irradiations. After about five hours, TL reading was taken with TL readout heating rate of 5 °C/sec on TL 1009I reader designed by Nucleonix system with the temperature range of integration of the TL signal from 40 °C to 400 °C. The linearity was observed for the first peak in the range from 5 Gy to 25 Gy. The relationship between the TL response of the high intensity peak and the absorbed dose for $K_2B_4O_7$: Cu phosphor was shown in Fig. 4 which was found to be linear.

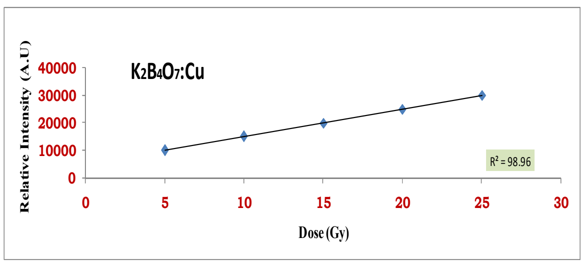

Fig. 4 dose response for $K_2B_4O_7$: Cu

IV. CONCLUSION

Using combustion method, $K_2B_4O_7$: Cu was successfully prepared. TL and PL properties of $K_2B_4O_7$: Cu is reported with X-ray Diffraction. It witnessed maximum TL at 0.007 mole of copper irradiated with gamma dose for 15 sec. Besides a good candidate for lamp phosphor and display application, this phosphor may be applicable for environmental monitoring applications.

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