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Kinetic parameters and TL mechanism in cadmium tetra borate phosphor

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ABSTRACT

Polycrystalline powder samples of cadmium tetra borate were synthesized by a simple solid state sintering technique and gamma irradiated sample showed a simple Thermoluminescence (TL) glow peak around 460 K. The TL kinetic parameters of gamma irradiated phosphor were determined by initial rise (IR), isothermal decay (ID), peak shape (PS), variable heating rate (VHR) and glow curve de-convolution method. The kinetic parameters such as activation energy (E), frequency factor (s) and order of kinetics (b) were calculated by IR, ID, PS and VHR methods are in the order of ~1.05 eV, 10^9–10^{12} s^{-1} and 1.58, respectively. From the results of TL and PL emission studies carried out on the phosphor revealed that the defect centers related to TL is different from that for PL. EPR measurements were carried out to identify the defect centers formed in cadmium tetra borate phosphor on gamma irradiation. Based on EPR studies the mechanism for TL process in cadmium tetra borate is proposed in this paper

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1. Introduction

Borate based materials have been studied for thermoluminescent dosimetry application since 1967 [1]. Among the borates studied so far lithium and magnesium has attracted more attention because of their near tissue equivalence [2–10] and their neutron response. Since cadmium has high capture cross-section (~10^4 barn) for neutrons, cadmium based borates can be good material for neutron dosimetry. Cadmium tetra borate (CdB_4O_7) has not been studied earlier from the point of view of thermoluminescence. The Mn activated cadmium borate ($Cd_2B_2O_5$) has been reported to exhibit four distinct peaks at 110 K, 215 K, 370 K and 405 K upon UV irradiation and low temperature measurements [6]. Hence we have tried to synthesize a cadmium borate material with a stable TL peak around 473 K. We have recently successfully synthesized few cadmium tetra borate phosphors and studied its TL properties for the dosimetry application. Undoped cadmium tetra borate showed a simple TL peak around 458 K and gadolinium doping has enhanced its TL sensitivity with formation of additional new main TL peak at 563 K. A detailed study on the TL properties of the cadmium tetra borate is communicated for publication [11].

The dosimetric properties of TL materials mainly depend on the kinetic parameters of its glow peak. Kinetic parameters give valuable information about mechanism responsible for the TL emission in material. This paper discusses the kinetic parameters of cadmium tetra borate phosphor synthesized by the high temperature solid state diffusion technique. Electron paramagnetic resonance (EPR) is a technique that can be employed to investigate the presence of paramagnetic species (trapped electron or a hole). In the case of TL materials, EPR offers a non-destructive method of obtaining the information on the charge traps in the crystal lattice. The information about the charge traps though vital to explain the TL process, cannot be obtained from the TL measurements alone. The TL emission spectrum provides information on the wavelength of the emission center but cannot give information about the nature of electron and hole traps. In TL applications, the main requirement is the identification of radiation induced defects. This paper presents a correlation between TL and EPR signals and thereby elucidates a mechanism for TL cadmium tetra borate phosphor.

2. Experimental

2.1. Synthesis

Cadmium tetra borate phosphor was prepared by solid-state reaction at high temperature using analytical grade raw materials cadmium carbonate and boric acid according to the following chemical equations.

$$CdCO_3 + 4H_3BO_3 \rightarrow CdB_4O_7 + 6H_2O + CO_2$$

Required quantities of the starting materials were mixed homogenously using distilled water and the resultant mixture is dried at around 373 K. The dried powder was ground and fired at a higher temperature (1173 K) slightly less than the melting point

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in order to avoid melting. It was then slowly cooled to 773 K inside the closed furnace and subsequently the alumina crucible was removed from the furnace and cooled to room temperature. The resulting phosphor was ground and used for all other characterization studies. The phase of the phosphor was confirmed by X-ray diffraction technique using Cu-$K\alpha$ radiation [11].

2.2. Measurement

Gamma irradiation of the phosphor was carried out at room temperature using a gamma chamber loaded with the Co-60 (gamma energies 1.173 MeV and 1.33 MeV) source of dose rate 270 Gy h^{-1}. Thermoluminescence measurements were done using RISO make TL/OSL reader at various heating rates. TL glow curves of gamma irradiated (12 mGy) phosphor are recorded at different heating rates (0.1 K s^{-1}, 0.3 K s^{-1}, 0.7 K s^{-1}, 1 K s^{-1}, 3 K s^{-1}, 7 K s^{-1} and 10 K s^{-1}). TL emission spectra of the irradiated phosphor powders at each glow peak temperatures were obtained by controlled heating of the samples in a Kanthal tray enclosed inside a light tight metallic cover. The light emitted from the irradiated sample during heating was collected through an optical cable placed over the sample. Final recording of the spectra were done by feeding the optical cable outlet to the emission monochromator of Jobin Yvon-Spex make Spectrofluorometer (Fluorolog version-3; Model FL3-11) with xenon lamp in switched off condition. PL excitation and emission spectra were acquired at room temperature with both excitation and emission slit width kept at 3 nm.

The most important information regarding the radiation induced defects in borates can be obtained from the electron paramagnetic resonance spectroscopy. EPR experiments reported in the paper were performed using a Bruker EMX X-band spectrometer with 100 kHz field modulation. The g-value was calibrated with standard DPPH (α,α'-diphenyl-β-picryl hydrazyl) and is used as a field marker. EPR measurements were carried out for samples which are unirradiated, irradiated and annealed at different temperatures after irradiation for identification of free radicals formed on irradiation. The microwave frequency used was ~9.4 GHz (power 3.196 mW).

3. Results and discussion

3.1. TL and PL emission spectra

TL emission spectrum of undoped cadmium tetraborate phosphor was recorded after irradiating the material to a dose of 10 Gy with Co-60 gamma source. Fig. 1 shows the TL and PL emission spectra of the cadmium tetra borate phosphors. Isothermal heating of gamma irradiated cadmium tetra borate powder at 453 K produced green light emission at a wavelength of about 515 nm corresponds for the main TL dosimetric peak of the material. The PL emission of the material on excitation with UV light of wavelength 258 nm is observed at 620 nm. A similar result was observed in $Cd_2B_2O_5$:Mn, PL spectrum consists of a broad emission band in the red spectral region with a maximum at 630 nm. At longer excitation wavelengths there appears a green emission band which is obviously caused by other borate components than $Cd_2B_2O_5$ [6]. From the results of the TL emission and PL studies, it was observed that the emission centers related to TL is different from that for PL.

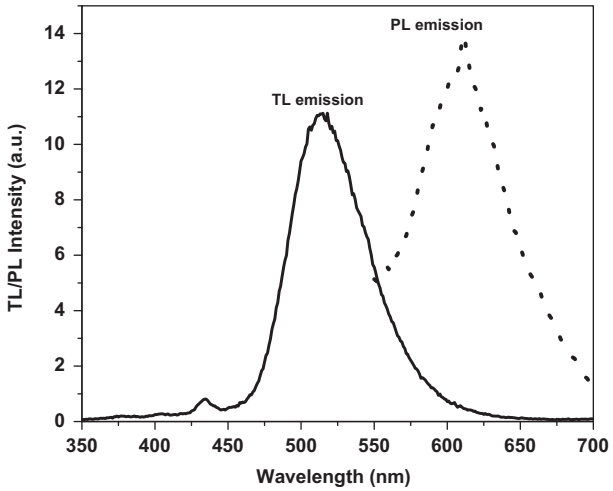

Fig. 1. TL and PL emission spectra of cadmium tetra borate.

3.2. Determination of TL kinetic parameters

In order to completely characterize the TL properties, one should have an idea of the kinetic parameters of the glow peak like activation energy (E), the order of kinetics (b) and frequency factor (s) of the traps responsible for TL. These parameters determine the stability of the traps. In general for a better stability of the traps, the activation energy (E) should be high which results in the higher glow peak temperature and hence less fading. For cadmium tetraborate phosphor, these parameters for the TL peak at 458 K are determined by different methods like variable heating rate, initial rise, peak shape, isothermal decay and glow curve deconvoultion methods.

3.2.1. Variable heating rate method

In this method TL glow curves of gamma irradiated (12 mGy) phosphor are recorded at different heating rates (0.1 K s^{-1}, 0.3 K s^{-1}, 0.7 K s^{-1}, 1 K s^{-1}, 3 K s^{-1}, 7 K s^{-1} and 10 K s^{-1}). No change in the glow curve structure has been observed at various heating rates. No change in the glow curve structure has been observed at various heating rates but glow peaks showed a systematic shift in peak positions with heating rate (Fig. 2). The glow peak position showed a shifting from 414 K to 493 K for the change of heating rate of 0.1 K s^{-1} to 10 K s^{-1}. The whole glow-peak is shifted to higher temperatures as heating rate increases in a manner depending on the half life and time spent at each temperature. Temperature lag is expected in thick samples at higher heating rates. We used very small amount (about 10 mg) of polycrystalline powder of size ~100 μm for each measurements

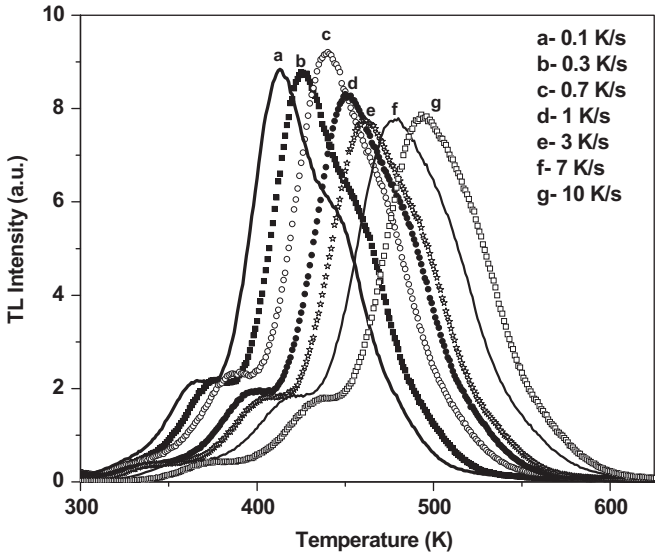

Fig. 2. Variation of TL glow peak position for different heating rates.

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