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PROCEEDINGS BOOK

Computer

Electrical and Electronics

Mechanical Engineering

November 23-25, 2016

Rome / ITALY

Production and Testing of TL Dosimeters as an Ionizing Radiation Sensor

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***Abstract*— In this study, the precipitation method has been used for preparation of $CaSO_4$ phosphor. The raw materials were used for preparation of this phosphor were $CaCl_2$, Na_2SO_4 and distilled deionized water. The structural characterization of the samples was obtained using X-ray diffraction (XRD) method and scanning electron microscopy (SEM) images. $CaSO_4$ pellets were made by cold pressing using obtained powder samples and TL measurements were carried out with an automated Risø TL/OSL DA-20 reader. TL emission was detected through a filter pack (Hoya U-340) transmitting between 290 and 370 nm. The activation energy (E), order of kinetics (b), and frequency factor (s) of the samples were determined using the various heating rate (VHR) and peak shape (PS) methods after being exposed in 1 Gy beta dose for 260°C TL peak.**

***Keywords*— Thermoluminescence, production of dosimeter, radiation sensor, activation energy, VHR, PS.**

I. INTRODUCTION

Thermoluminescence (TL) is the emission of light from an insulator or a semiconductor following the previous absorption. The phenomenon of TL may have been known as early as 1663 as empirically defined, when Sir Robert Boyle reported to the Royal Society about "experiments and considerations upon colours with observations on diamond that shines in the dark" (1663) and described how, upon warming of a diamond in contact with his body, he saw a luminescence in the dark [1]. Many natural and synthetic minerals exhibit TL properties and it is important for dosimetry studies [2, 3].

Thermoluminescent dosimeter (TLD) is a radiation dosimeter that measures ionizing radiation (e.g. ultraviolet radiation, x-rays, gamma rays, alpha, beta and neutrons) exposure by measuring the intensity of visible light emitted from a crystal in the detector when the crystal is heated [4, 5]. This heating process for crystals is performed using TL measurements and it gives some information related to defects and impurities in solids. These solids are insulator or semiconductor that have a lot of defects and these defects are called electron traps. In the development of a new TLD material, these defects are very significant and it represents a new and fast evolving application of research in dosimetry. Processing synthesis is critical in order to control the final properties of the crystal defects [6].

Earlier contributions on the studies of defect structures in both synthetic and natural $CaSO_4$ include a systematic study due to the irradiation of different rare-earth ions-doped $CaSO_4$ [7, 8]. Firstly, Nambi (1975) studied an experimental procedure to determine absolute TL emission spectra and presents results for the cases of $CaSO_4$:Dy phosphors which are widely used in radiation dosimetry. Dysprosium-doped $CaSO_4$ known as TLD 900 is excellent TL phosphor for dosimetry of ionizing radiations due to its high sensitivity.

The dosimetric characteristics of TL materials mainly depend on the kinetic parameters quantitatively describing the trapping–emitting centres responsible for the TL emission. The glow curves are influenced by changes in location, size and shape of the glow curves due to changes in the heating rate [9-11]. For this purpose we investigated TL properties of $CaSO_4$ using the various heating rate (VHR) and peak shape (PS) methods. The shape of a TL glow-peak is the basis of important and convenient methods for calculating the trapping parameters of distinct energy levels within the crystal [12]. In this work, $CaSO_4$ phosphor was synthesized and the order of kinetics (b), the trap depth (or activation energy (E)) and frequency factor (s) were calculated using VHR and PS methods for 260°C TL peak and then these values were compared each other.

II. EXPERIMENTAL PROCEDURE

The precipitation method has been used for preparation of $CaSO_4$ phosphor. The raw materials used for preparation of $CaSO_4$ were $CaCl_2$ 99.99% (Aldrich), Na_2SO_4 99.00 % (Sigma) and distilled deionized water. $CaSO_4$ was prepared in three stages as follows:

(a) 4.390 g of $CaCl_2$ and 5.614 g of Na_2SO_4 was dissolved in 50 ml distilled deionized water and stirred for 20 minutes.

(b) The solution was heated up to 100°C using hotplate and stirred for 15 minutes for following chemical reaction:

$$Na_2SO_4 + CaCl_2 \rightarrow CaSO_4 + 2NaCl$$

(c) The solution was filtered (Fig. 1). The filtered material was dissolved in water and heated up to 100°C and filtered again. These processes were repeated five times in order to remove sodium chloride (NaCl) with water.

The obtained powder was pelletized under the cold pressure of 800 kg and then the pellets were sintered in furnace at 700°C for 8 hours (Fig. 2).

Fig. 1 $CaSO_4$ sample preparation using precipitation method.

The crystalline structures of the samples were confirmed through X-ray diffraction studies using Rigaku Miniflex II diffractometer at 30 kV (scanning rate: 2°/min, 15 mA with λ=1.5406 Å Cu-Kα radiation).

Fig. 2 Pelletizing and sintering of $CaSO_4$ samples.

The morphological form of samples was studied using scanning electron microscopy (SEM) with Zeiss Supra 55.

All TL measurements were carried out using an automatic Risø TL-OSL-DA-20 reader system (Fig. 3) with an EMI 9235 QA photomultiplier tube (PMT) and attached to filter pack consisting of a Hoya U-340 (290–370 nm) filter, which has an 80% minimum peak transmittance. Beta (β)-irradiation was performed using a 1.48 GBq (40 mCi) $^{90}Sr/^{90}Y$ beta source (dose rate: 6.689 Gy/min) with a maximum energy of 2.27 MeV [13, 14]. In order to determine TL kinetic parameters of 260ºC peak, three $CaSO_4$ pellets were irradiated with ~1 Gy β dose and then TL glow curves were recorded from room temperature to 350ºC using different linear heating rates from 1 to 6ºC/s and then obtained TL glow curves were used for calculations.

Fig. 3 Risø TL-OSL-DA-20 reader system.

III. Results and Discussion

The prepared $CaSO_4$ powders were confirmed through X-ray diffraction (XRD) studies using Rigaku Miniflex II diffractometer. X-ray source operation at 30 kV and 15 mA for samples. The XRD spectra of samples are shown in Fig. 4.

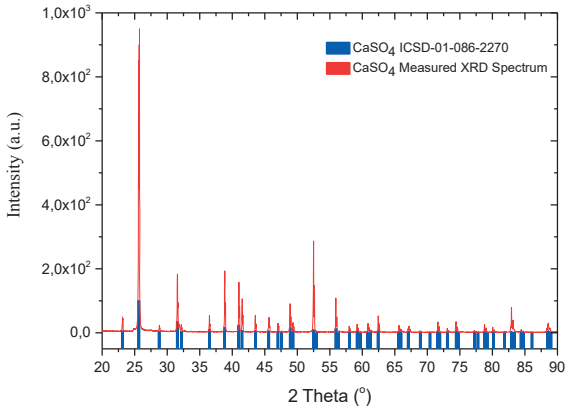

Fig. 4 XRD pattern of $CaSO_4$ sample.

XRD patterns of $CaSO_4$ samples show a lot of peaks were observed in various degrees. An unknown crystalline component was not detected in the samples and measured XRD spectrum is compatible with ICSD-01-086-2270 $CaSO_4$ card.

The particle size can significantly affect luminescence intensity of phosphors. The sample with larger particle size presents higher luminescence intensity [15]. The structural form of the prepared $CaSO_4$ samples were studied using a scanning electron microscopy (SEM) with Zeiss Supra 55. The SEM images of $CaSO_4$ powder samples are given in Fig. 5.

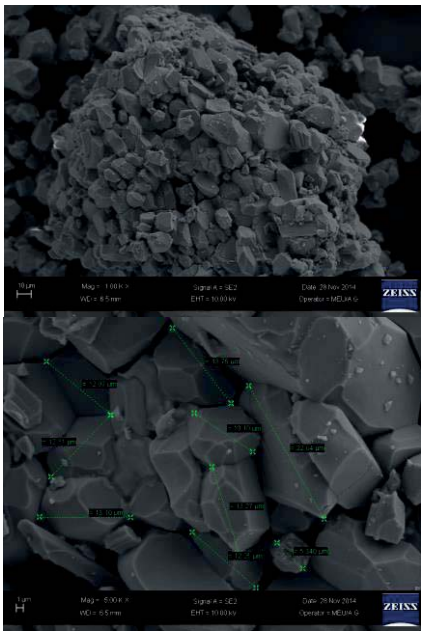

Fig. 5 SEM images of prepared $CaSO_4$ samples with different resolution.

As seen in Fig. 5, the synthesized $CaSO_4$ powder samples show good morphology and connectivity with grains, but the particle sizes are inhomogeneous and it was observed that the particle sizes vary from 5.34 µm to 22.04 µm.

TL glow curves of irradiated $CaSO_4$ samples were recorded at different linear heating rate values (Fig. 6). The heating rate is a fundamental experimental variable in TL measurements. Various heating rate methods are based on the shift of the glow peak temperature to higher temperatures with heating rate. This phenomenon has been explained to be due to thermal quenching [13, 16].

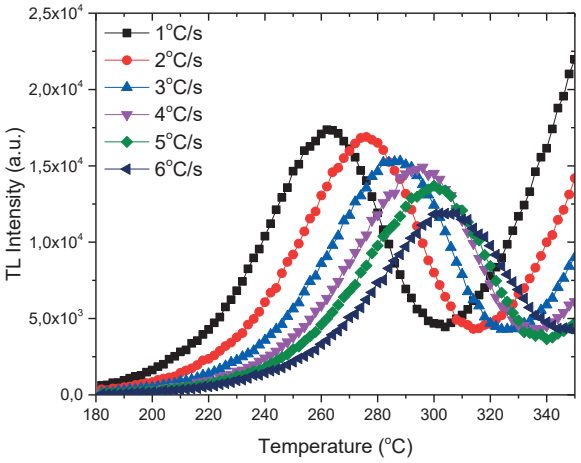

Fig. 6 TL glow curves of beta irradiated $CaSO_4$ pellets at various heating rates (beta dose – 1Gy).

Using Hoogenstraaten [17] equation and various heating rate values, activation energy (or trap depth, E) can be evaluate graphically, based on the position of T_M with heating rate β. A linear relation is obtained between $ln(T_M^2/\beta)$ and $1/kT_M$ as follows:

$$ln\left(\frac{T_M^2}{\beta}\right) = \left(\frac{E}{k}\right)\left(\frac{1}{T_M}\right) + ln\left(\frac{E}{sk}\right)$$

The plot of $ln(T_M^2/\beta)$ versus $1/T_M$ should give a straight line with slope E and intercept $ln(E/sk)$. Extrapolation to $1/T_M = 0$ gives $ln(E/sk)$ from which frequency factor (s) can be calculated using the value of E obtained from the slope.

In present study activation energy (E) and frequency factor (s) of peak at 260°C were calculated using various heating rate values. By graphical method E and s values are calculated from the linear plot of $\ln(T_M^2/\beta)$ versus $1/T_M$ (Fig. 7). From the slope of the linear graph one can obtain the activation energy. [18].

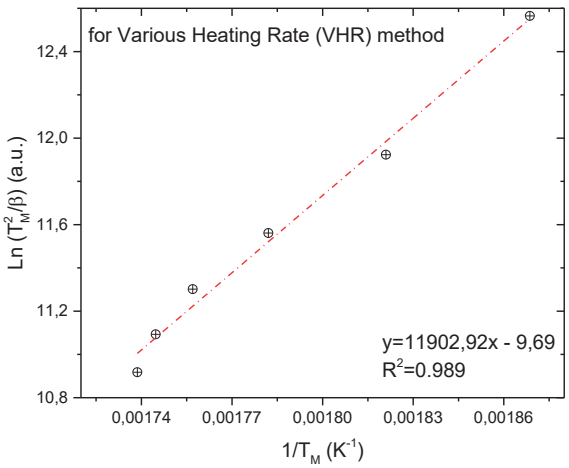

Fig. 7 The linear relation of $\ln(T_M^2/\beta)$ versus $1/T_M$ for 260°C TL peak.

Activation energy and frequency factor of 260°C peak estimated from Fig. 7 are found to be at 1.026±0.005 eV and 1.95×10^8 s^{-1} respectively (Table 1).

One way of determining TL kinetic parameters is by considering its geometrical properties with peak shape (PS) method [19]. These methods are based on measurements of a few points on the glow-peak, shown in Fig. 8.

The peak maximum temperature T_M and the temperatures at half maximum TL intensity T_1 and T_2 at the low and high temperature side of the glow-peak respectively. These quantities are used to define further the widths $\omega = T_1 - T_2$, $\delta = T_2 - T_m$ and $\tau = T_m - T_1$ as well as the symmetry factor of the glow-peak $\mu_g = \delta/\omega$. [20].

The kinetic parameters were calculated by using following expressions:

$$E = 1.51 \times \left[\frac{T_M \times T_1}{T_M - T_1}\right] \qquad s = \frac{\beta E}{kT_M^2} \times \exp\left(\frac{E}{k \times T_M}\right)$$

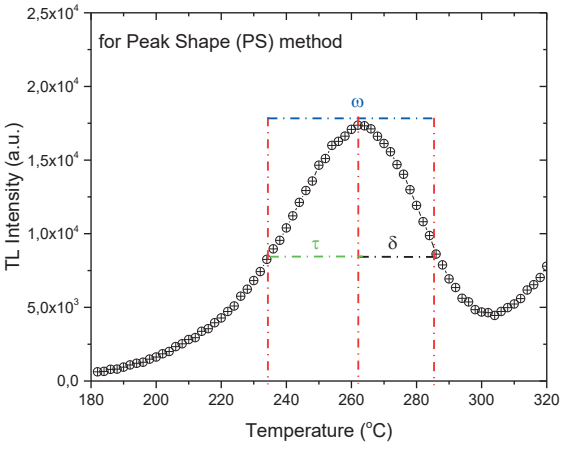

Fig. 8 Geometrical characteristics of 260°C TL peak.

E and s of 260°C TL peak are found to be at 1.024±0.006 eV and 1.75×10^{8} s^{-1} respectively for b=1 (Table 1).

TABLE I
Kinetic parameters of $CaSO_4$ phosphor.

Methods	Activation Energy (E) eV	Frequency Factor (s) s^{-1}	Order of Kinetics (b)
Various Heating Rate (VHR)	1.026±0.005	$1.95x10^{8}$	-
Peak Shape (PS)	1.024±0.006	$1.75x10^{8}$	1

IV. Conclusions

In this study, $CaSO_4$ phosphor was synthesized using precipitation method and the $CaSO_4$ powders were pelletized. The particle morphology and crystalline structures of the powder $CaSO_4$ samples were determined using XRD method and SEM images, respectively. TL kinetic parameters of $CaSO_4$ phosphors were calculated by using VHR and PS methods. The kinetic parameters (E and s) obtained with these methods for $CaSO_4$ phosphors are consistent with each other, but the b values does not compared. The activation energies (E) of $CaSO_4$ phosphors according to VHR and PS methods were determined as 1.026±0.005 and 1.024±0.006 eV, and the frequency factors (s) were determined as $1.95x10^{8}$ and $1.75x10^{8}$ s^{-1} for the 260°C TL peak, respectively.

Acknowledgement

This work was supported by Research Fund of Çukurova University (Project Number: **FED-2016-7668**). All authors would like to thank Research Fund of Çukurova University for financial support.

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