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RESEARCH ARTICLE

DETERMINATION OF TRAPPING PARAMETERS OF CALCINED ALUMINA BY VARIOUS HEATING RATE METHODS

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ABSTRACT

Trapping parameters of calcined alumina (alpha phase) has been determined by resorting to Various Heating Rate (VHR) method in the dose range from 5 to 100Gy. Peak finding by second derivative plot of the glow curve is used. It is observed that highly overlapped traps of charge (electron) could be observed by variation of radiation dose. As many as twelve traps could be located for the glow peaks in the range of temperature from 350 to 770 K. The traps are pretty close to each other and also showing frequency factor more or less constant with a value $4.8 \pm 1.4 \times 1011s^{-1}$.

INTRODUCTION

Alumina and alumina based phosphors have become attractive thermoluminescent materials (Mckeever *et al*., 1995; Mckeever, 1985), for its high sensitivity as well as low effective atomic number (Akselrod *et al*., 1990). Many studies have been done on the dosimetric properties of these materials (Akselrod *et al*., 1990; Pokorny and Ibarra, 1993). Analysis of thermoluminescence (TL) glow curve is one of the thermally stimulated process which gives information about defect centre in solid. It has been recognized that information about the trapping levels can be extracted from the TL glow curve (Kenny *et al*., 1967; Bos and Dielhof, 1991). Various Heating Rate (VHR) of Hoogenstraten (Hoogenstraaten, 1958) is one method which is a simple, rapid and reliable technique for extracting trapping parameter namely activation energy (E) and frequency factor (s) (Gartia *et al*., 2005). The information about the lifetime of trap charge (electron) is of fundamental importance in the use of TL as a technique in Dating and Dosimetry. This lifetime depends on the trapping parameter namely activation energy (E), frequency factor (s) and order of kinetics (b) and temperature of storage of the material. Thus the determination of traps of any system relevant to dating and dosimetry is of practical importance. In this paper we demonstrate the possibility to locate the spectrum of trap of the laboratory reagent grade Al_2O_3 by VHR method using different radiation dose level (5-100 Gy).

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MATERIALS AND METHODS

Calcined alumina (alpha phase) is used in the study. The sample was annealed at 500°C for one hour to erase natural TL. In each measurement 10 mg of sample was used. TL measurement of samples for doses of 5-100 Gy of beta irradiation (Sr-90 source) were carried out on the Riso TL/OSL (Model TL/OSL –DA-15) reader. The photomultiplier tube used is EMI 9635 in the operating voltage of 950 volts with optical filter Schott UG-11 in conjunction with Schott BG-39 for filtering out the unwanted radiation. The combination allows transmission in the wavelength range 300 ~ 400 nm. A second read out was also performed to record the background radiation, which include the black body radiation. The presented graphs are all with the background subtraction. All measurements were done in the flowing N2 atmosphere. All samples were measured within 10 minutes after irradiation. Riso TL/OSL reader is a commercial system used globally for dating and dosimetry (Bøtter-Jensen, 1997). It has in built beta irradiation (Sr-90) facility. The strength of the beta source at the time of irradiation was 0.083 Gy/sec^{-1}. The heating rates (β) used for the present analysis are 0.5, 1.0, 2.0 and 5.0 °C/s.

Method of Analysis

The VHR method was used to determine the kinetic parameters of TL glow peaks for the dose ranges of 5-100Gy.

This method is based on the shift of peak temperature (Tm) to higher temperature when the heating rate is increased. A plot of In (Tm/β) vs$(1/Tm)$ should yield a straight line of slope "E/k" (k is a constant) from which E can be found. Extrapolation to 1/Tm=0, a value for in (sk/E) is obtained, from which s can be calculated by the insertion of E/k found from the slope. Since the required data is taken at peak maximum (Im, Tm) which in the case of complex composite peaks, the positions of individual peaks are estimated from the analysis of second derivatives of the experimental glow curve (Singh, 2001). Another difficulty still exists in case of highly overlapping peaks where location of peak from shoulder region is uncertain. This problem may overcome by deconvolution of glow curve with computer programme. The calculation of E and s by VHR method was performed on the peaks which are identified by negative minima of the second derivative of the experimental glow curve. Certainty in the measurement of Tm within 1K is also considered by dividing the temperature scale less than 1K so that the accuracy in the estimation of E and s is optimum. Another very important point that has taken into consideration for the evaluation of kinetic parameters by VHR method is the Temperature Lag (TLA) between the heating element and the thermo luminescent sample during the TL readout in reader using contact heating. To avoid this problem, Kitis and Tuyn (1998) has developed a simple method for TLA correction to determine the exact peak temperature after different heating rates by using the equation

$$T_m^j = T_m^i - c\,ln(\beta_i / \beta_j)$$

Where T_m^j and T_m^i are the maximum temperatures of a glow peak heating rates β_j and β_i respectively and c is a constant which is usually evaluated by using two very low heating rates where the TLA can be considered as negligible (Yazici and Topaksu, 2003). In this work heating rates at 0.5°C/sec and 1.0°C/sec were used to determine the constant c and then the peak temperatures were corrected for TLA.

RESULTS AND DISCUSSION

A set of measured TL glow curves for different doses of 5, 25, 50 and 100Gy at heating rate of 1.0°C/s is shown in Figure 1 along with their second derivative plots. The minima of the second derivative plot correspond to the glow peaks of the TL glow curves were obtained and were used for evaluation of kinetic parameters of trap charges (Singh, 2001; Gartia *et al.*, 2005). But the identification of minima of the highly overlapped peaks i.e. when appears at the shoulder region of one minima, often give difficulty in the identification of minima and evaluation of the trapping parameter by VHR method. However this highly overlapped glow peaks may be identified through minima of second derivative plot at one radiation dose or other as shown in figure 1.The minima of the second derivative plots which are possible to evaluate their trapping parameters are marked by solid arrows. As many as 9 trap level and their corresponding kinetic parameters (E and s) for TL glow curve of dose 5Gy are possible to retrieve, 11 for 25Gy, 8 for 50Gy and 10 for 100Gy. The minima observed at higher temperature indicated by broken arrows are unable to evaluate their trapping parameters due to the non-appearance of the corresponding minima at higher heating rates. The results of the trapping parameter as obtained by the VHR method under different dose level are shown in Table 1.

Table 1. Trapping parameters as obtained using VHR of the glow curve for different doses (5-100Gy)

	5 Gy		25 Gy		50 Gy		100 Gy		**Average**	
Glow Peak	E (eV)	s $(s^{-1}) \times 10^{11}$	E (eV)	s $(s^{-1}) \times 10^{11}$	E (eV)	s $(s^{-1}) \times 10^{11}$	E (eV)	s $(s^{-1}) \times 10^{11}$	E (eV)	s $(s^{-1}) \times 10^{11}$
I	0.87	2.4	0.90	5.2	0.89	2.8	0.87	2.0	0.88 ± 0.02	3.1 ± 1.4
II	1.03	6.8	1.03	6.5	1.04	7.9	1.03	6.8	1.03 ± 0.01	7.0 ± 0. 6
III	1.08	1.1	1.08	1.2	1.11	2.2	1.12	2.9	1.10 ± 0.02	1.9 ± 0.9
IV	-	-	-	-	-	-	1.27	5.8	1.27 ± 0.00	5.8 ± 0.0
V	1.32	4.2	1.33	5.9	1.33	4.5	1.34	4.6	1.33 ± 0.01	4.8 ± 0.8
VI	-	-	1.37	3.4	-	-	1.37	4.8	1.37 ± 0.00	4.1± 1.0
VII	1.40	3.5	1.42	4.3	1.43	3.6	1.43	3.9	1.42 ± 0.01	3.8 ± 0.4
VIII	-	-	1.58	5.1	-	-	1.59	6.8	1.59 ± 0.01	6.0 ± 1.1
IX	1.63	6.2	1.64	5.9	1.62	6.7	-	-	1.63 ± 0.01	6.3 ± 0.4
X	1.74	4.9	1.72	5.4	-	-	-	-	1.73 ± 0.01	5.2 ± 0.4
XI	1.81	5.3	1.79	5.5	1.82	3.4	1.82	5.2	1.81 ± 0.01	4.9 ± 0.8
XII	1.85	4.1	1.89	6.0	1.88	3.1	1.90	6.1	1.88 ± 0.02	4.8 ± 1.3

It reveals as many as twelve (12) trap levels of this particular material in the temperature range of 350 – 770K. This is well agreement with earlier work on the analysis of traps of Al & O by Computerized Glow Curve Deconvolution (CGCD) method except for the traps around 400°C or above, a region where glow peak intensity is weak and CGCD method find problem to extract the trapping parameter even though peaks are indicated by second derivative plot (8,13).

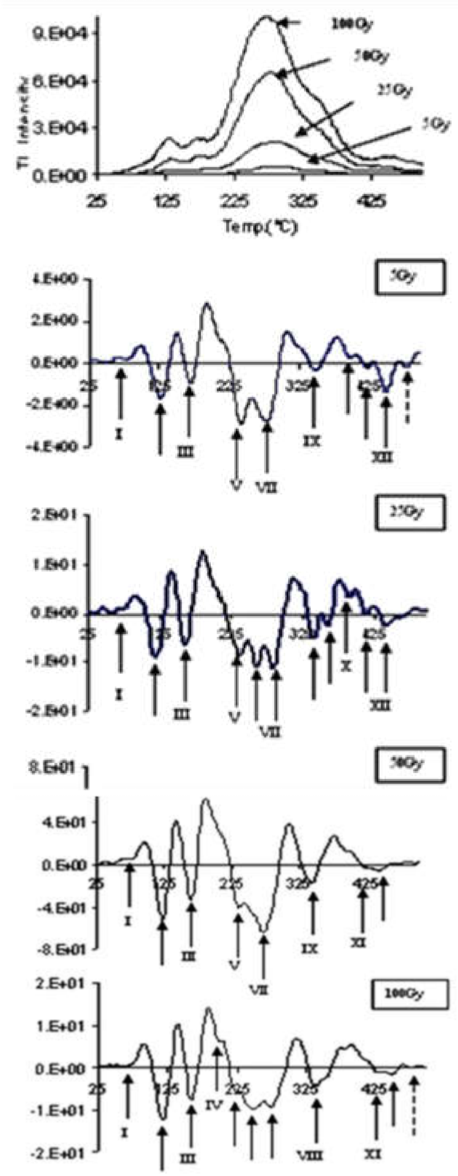

Figure 1. TL glow curves of Al_2O_3 (β=1°C/s) in the dose range from 5-100Gy. Below: 2nd derivative plots of 5, 25, 50 & 100 Gy TL curves

The trapping parameter for glow peak IV (as per our nomenclature: Table 1) is difficult to evaluate from TL glow curves of 5, 25, and 50 Gy by VHR using this second derivative plot technique. Similarly, glow peak VI and VIII is difficult to identify in 5 and 50 Gyglow curves; glow peak X is difficult in 50 and 100 Gy glow curves. Glow peak IX is again difficult to evaluate its trapping parameter in glow curve of 100 Gy.

Summary

The trap parameters of the laboratory reagent grade Al_2O_3as evaluated by Various Heating Rate method under different dose levels reveals as many as twelve different trap depths. The analysis enables one to show clearly that the traps are discrete with frequency factors $4.8 \pm 1.4 \times 1011s^{-1}$. This study also suggest that spectroscopy of traps of TL phosphor may be evaluated by various heating rate technique using TL glow curves for a range of radiation dose.

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