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TL Glow Curve and Kinetic Parameters of Amethyst Exposed to High Dose of Gamma Radiation

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Abstract*—*To know if a certain thermoluminescent (TL) peak is suitable for dosimetry, it is important to determine its lifetime. The mean lifetime may be calculated using the geometrical factor of the TL glow curve to determine the kinetic parameters such as activation energy and frequency factor of electrons in traps. The kinetic parameters to the peak at 202 ºC of a natural amethyst from Brazil, were obtained by using the peak shape methods applied on the TL glow curve, before and after deconvolution procedure. The peak at 202 °C was chosen because lies between 180 °C and 400 °C, a region of interest for dosimetric applications. Before deconvolution, the calculated lifetime for the peak at 202 °C was relatively short close to 35 days. However, the calculated lifetime after deconvolution was around 312 days, suggesting a longer lifetime. The results showed the importance of deconvolution for the calculation of the kinetic parameters of the TL peak of amethyst exposed to high dose of gamma radiation.

I. INTRODUCTION

Some studies bring an easier understanding introduction to the phenomena of thermoluminescence (TL) and its applications, but in general it is known that the TL materials when pre-excited by radiation (cosmic, ultraviolet,α, β, X or γrays) may retain energy within them [1, 2].

The principle of TL dosimetry is based on the statement that ionizing radiation produces free electrons, some of which can be trapped by point defects existing in the crystalline lattice of an insulating material. After being exposed to ionizing radiation, the TL material emits light while they are heated. The intensity of the emitted visible light is proportional to the number of trapped electrons and therefore to the amount of absorbed radiation [3, 4]. The emitted light can be detected by a photomultiplier Anda associated electronic equipment. In many cases, the detected light is proportional to the radiation absorbed and it is possible to use TL dosimetry for several applications [3,5].TL dosimetry is a well-established dosimetric technique with applications in areas such as personnel, environmental, archaeological, dating, retrospective and clinical dosimetry [3].

Many synthetic materials are produced with properties suitable for TL applications, among which are lithium fluoride (LiF), calcium fluoride (CaF_2), calcium sulfate ($CaSO_4$), beryl oxide (BeO) and aluminum oxide (Al_2O_3) [4,5]. On the other hand, there are natural minerals with interesting TL properties, such as zirconite ($ZrSiO_4$), microcline ($KAlSi_3O_8$), albite ($NaAlSi_3O_8$), calcite ($CaCO_3$), topaz $Al_2(F,OH)2SiO_4$ and quartz (SiO_2) [6-9].Among these, quartz is one of the most popular ones

and can be used in many TL dosimetry applications [3, 9-12].

Natural amethyst is a purple variety of α-quartz (SiO_2) and has received considerable attention from the point of view of technological applications with low and highγ dose radiation [13, 14, 17, 18]. Thus, these studies have published the thermoluminescence analysis, as well as suitability of amethyst as dosimeter.

In many industrial processes the high doses can be used to material sterilization, food tuber germination treatments, grain and seed growing, water purification, among other possibilities [19, 20]. Then, there is a possibility of using the amethyst to high dose dosimetry in similar applications.

Amethyst generally exhibits a TL glow curve with two or more main peaks. The TL glow curve is an intrinsic characteristic of each amethyst crystal and is associated to high concentration of electron or hole traps and high efficiency of light emission associated with recombination process between electron and hole [3]. However, the iron (Fe^{3+}), which exist as impurities into the amethyst lattice, is generally responsible for the concentration of electrons and holes traps, can vary from one deposit to the other affecting the intensity of TL glow curve [21-23].

To determine if the peaks on the TL glow curve are useful for dosimetry, it is important to know its lifetime. The lifetime may be calculated using the trap physical parameters as order of kinetic, activation energy and frequency factor [3, 4, 23].

In order to obtain these physical parameters, some procedures can be used, such as initial rise, different heating rates and peak shape methods [23-25]. However, the methods that use the peak shape are probably the simplest because activation energy can be determined by knowing only two or three characteristic temperatures of the peak and no additional measurements are needed.

Thus, the aim of this study was to calculate the kinetics order (*b*), activation energy (*E*)and frequency factor (*s*) of the peak at 202 °C for an amethyst taken from a deposit located in South of Brazil. By using peak shape methods was possible to estimate the life time of TL peak at 202 °C with the order of days at 25 °C of room temperature.

II. EXPERIMENTAL

2.1 Peak shape methods for general order

The geometrical shape of the peak on TL glow curve is an important characteristic for the material that will be used as a TL dosimeter. This characteristic can be evaluated using the peak shape methods, that is based on measurements of a few points of the isolated glow peak as shown in Figure 1 [4].In this figure, it is possible to define the following parameters: T_m = temperature at maximum intensity; $T_1$and T_2 = temperatures on either side of T_m, corresponding to half of the maximum intensity; $\tau = T_m - T_1$is the half-width of the low temperature side of the peak; $\delta = T_2 - T_m$ is the halfwidth at the high temperature side of the peak; $\omega = T_2 - T_1$is the total half-width and$\mu = \delta/\omega$ is the geometrical form factor or symmetry factor. The factor μ is adopted as 0.42 for first order kinetics peaks, 0.52 for the second order and for values other than 0.42 and 0.52 the general order peaks is adopted.

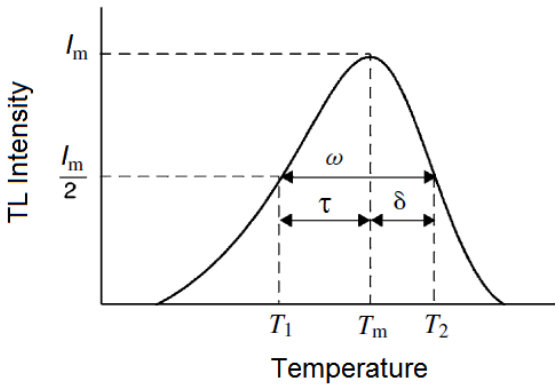

Fig.1: Geometrical parameters of the TL peak

By determining the form factor μ, the corresponding order of kinetic *b* can be predicted from the calibration curve shown in Figure 2. It is pertinent to mention that μ is practically independent of *E* in the range from 0.1 to 1.6 eV and strongly dependent on the kinetics order *b*, to the range $0.7 \leq b \leq 2.5$ [4].

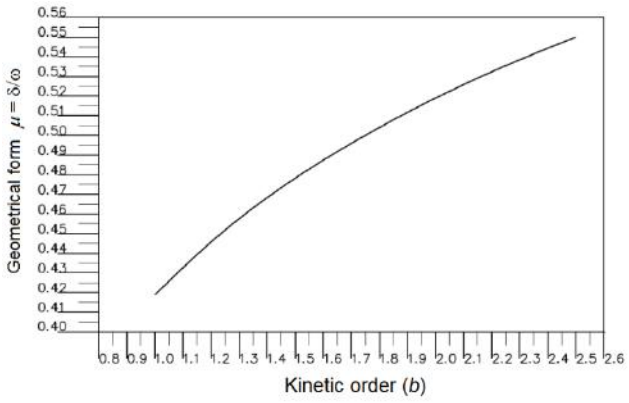

Fig.2: Variation of μ with order of kinetics b

In the general order peaks case, the energy *E* (eV) can be calculated using Equation 1, that require only knowledge of the parametersτ, δ, ω and μ [4].

$$E_\alpha = C_\alpha \frac{kT_m^2}{\alpha} - B_\alpha \,.\,(2kT_m) \qquad (1)$$

Where the index α stands for τ, δ, ω and *k* = Boltzmann constant (8.62 x 10^{-5} eV/K).

The values or C_α and B_α for the three methods are presented in the Equations 2, 3 and 4 [4].

$$C_\tau = 1.51 + 3(\mu - 0.42), B_\tau = 1.58 + 4.2(\mu - 0.42) \quad (2)$$
$$C_\delta = 0.976 + 7.3(\mu - 0.42), B_\delta = 0 \quad (3)$$
$$C_\omega = 2.52 + 10.2(\mu - 0.42), B_\omega = 1 \quad (4)$$

The frequency factor s (s^{-1}) for the general order kinetics peaks is calculated using the Equation 5 [25].

$$s = \left(\frac{\beta E}{kT_m^2}\right)\frac{1}{1 + (b-1)\left(\frac{2kT_m}{E}\right)} exp\left(\frac{E}{kT_m}\right) \quad (5)$$

Where b = value of kinetic order, β= heating rate (K/s).

2.2 Sample preparation

The natural amethyst used in the present study was a single crystal extracted from one deposit located in the district of Lajeado (Rio Grande do Sul State, South of Brazil). The crystal (Figure 3) was cleaned with acetone for 20 minutes using an ultrasonic bath and then were manually crushed using an agate mortar and pestle. After crushing, the grains were classified into 75 x 150 μm by using standard Tyler sieves. After that, the grains were submitted a heat treatment in order to guarantee the release of charge carriers from the trap levels. The heat-treatment were performed in a muffle furnace at 400 °C for 1hour and cooling at room temperature of 25 °C.

Fig.3: Amethyst crystal

2.3 TL measurements

To determine the TL glow curve of the amethyst exposed to high gamma doses, five powder aliquots with approximately 20 mg were used. The weight of each aliquot was determined with an analytical balance accurate to 0.1 mg. The aliquots were exposed to doses ranging from 1 to 50 kGy in a gamma-cell irradiator with a dose rate close to 10 kGy/h. The TL measurements were carried out at temperatures ranging from 50to 350 ºC with a heating rate of 5 ºC/s, using a TL Harshaw3500 reader equipment. After that, the TL glow curve obtained with intermediate dose of 25 kGy was deconvoluted to calculate the lifetime.

2.4 Deconvolution and TL peak lifetime

Another important subject in dosimetric studies is the stability of the stored signal at room temperature. Then, the TL glow curves were analyzed by using the peak shape methods before and after the deconvolution procedure with Origin program. The deconvolution is defined as a mathematical method to create a curve using some theoretically and experimentally determined parameters [26, 27]. Thus, it was possible to analyze the overlapping peaks in the region between 125 and 275 °C of TL glow curve and determine the kinetics parameters of an isolated peak for the lifetime (t) calculation by using Equation 6.

$$t = s^{-1}\, exp(E/kT) \quad (6)$$

Where T = room temperature (298 K).

The accuracy of the deconvolution method used for TL peaks determination is similar to that found with the first, second and general order TL kinetic equations [25, 28]. In this study, the method was validated by analyzing the Figure of Merit (FOM), which is the degree of similarity between the theoretical and experimental curves. In this case, an FOM value of less than 2.5% indicates a good result [27, 28]. Four peaks were considered for the deconvolution, being one of first order, another of second order and two of general order, with activation energies ranging between 0.83 and 1.41 eV.

2.5 XRD Analysis

To investigate the crystal structure of amethyst, standard X-ray diffraction (XRD) analyses were carried out. For that, XRD patterns were obtained with a 2θ diffractometer between 10° to 60° using Cu-Kα radiation. Then, the result was compared with XRD pattern ofα-quartz.

III. RESULTS AND DISCUSSION

Figure 4 shows the XRD patterns of powdered sample amethyst and α-quartz. By comparing the two diffractograms recorded between 10° and 60° (in 2θ scale), it can be seen that they have the same peak positions. There is an intense peak at 26.56°and several other diffraction peaks of moderate and low intensity between

20.78° and 59.88°. The peaks presented correspond to the crystalline planes of natural quartz and are in accordance with the XRD pattern found for α-quartz [29].

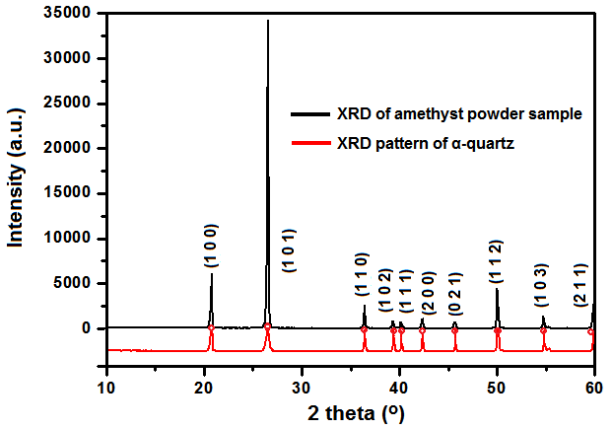

Fig.4: XRD of amethyst compared to pattern of α-quartz

Figure 5 shows the TL glow curve for amethyst powdered exposed to doses of 1, 5, 7, 10, 25 and 50 kGy of ^{60}Co. This result shows two overlapping glow peaks near to 125 and 202 °C, respectively. In the present study, the peak at 125 °C was not considered due to its unstable behavior at room temperature. On the other hand, the peak at 202 °C is located in the region of interest for TL dosimetry, which lies between 180 °C and 400 °C [3, 23].

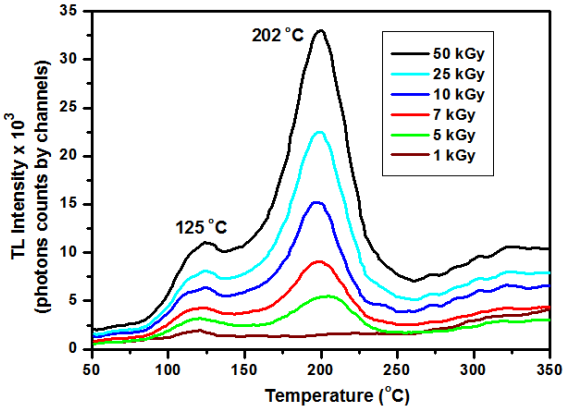

Fig.5: TL glow curve of amethyst exposed to high doses of gamma radiation.

The peak at 202 °C may exhibit different behaviors depending on the origin or shape of the sample investigated. For example, in samples powdered irradiated with doses in an interval of 0.89 – 21.36 Gy (^{90}Sr/^{90}Y), the peak at 202 °C may appears at 240 °C [18]. However, in amethyst sintered pellet irradiated with 1 kGy (^{60}Co) the glow peak is observed at 210 °C [17].

The TL intensity of the peaks at 125 and 202 °C increases within creasing dose from 1to 50 kGy, but only with 5 kGy the peak at 202 °Cis noticed. Some amethysts from Brazil show a very well defined dosimetric peak near to 210 °C that can be observed with 1 kGy of ^{60}Co or with lower doses [7, 17]. It is known that the TL intensity of quartz exposed to gamma radiation decreases with high content of OH defect centers, which varies in samples from one deposit to another [30]. It is pertinent to mention that others studies have reported the high OH concentrations in some amethysts from South of Brazil [21,31]. Thus, it is believed that the absence of the TL peak at 202 °C for dose of 1 kGy can be associated with the high content of OH centers present in the sample.

Figure 6 shows the TL glow curve of amethyst exposed to 25 kGy. The peak shape expressions for general order were used assuming that the peaks at 125 °C and 202 °C were sufficiently isolated. In this case, it was possible to calculate the following parameters:ω = 51.66, τ = 29.00, δ = 22.66 and μ = 0.44. After that, the value of μ = 0.44 was projected on the graph in Figure 2, which yielded one values for the kinetic order *b* = 1.15 for the peak at 202 °C.

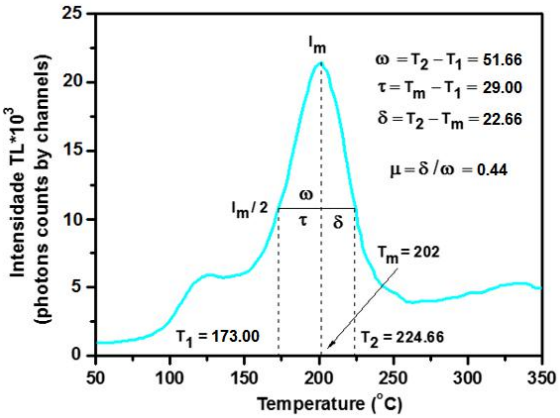

Fig.6: Peak shape methods in glow peak of amethyst exposed to 25 kGy.

Figure 7 shows the TL glow curve after deconvolution. This result suggests that the TL glow curve of amethyst exposed to 25 kGy consists of four overlapping peaks, three of them in the region between 125 °C and 275 °C, as shown in Figure 7(a). According to Figure 7(b), the Figure of Merit (FOM) presents a degree of similarity between the theoretical and experimental curves indicating a good result with FOM = 2.5%. The FOM values in the range 2.5% to 3.5% are acceptable and indicate only small flaws in the method [28]. The peak shape expressions for general order were used in the isolated main peak at 200 °C (Figure 8). Thus, were obtained the parameters:ω = 48.19, τ = 25.91, δ = 22.28 and μ = 0.46. The value of μ = 0.46 was projected on the graph in Figure 2for the kinetic order *b* = 1.32.

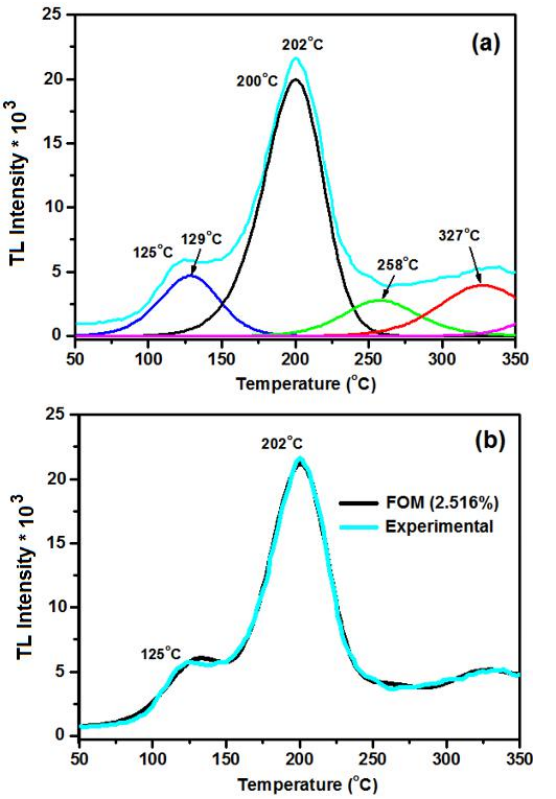

Fig.7: TL glow curve of amethyst: (a) after deconvolution and (b)degree of similarity between the theoretical and experimental curves

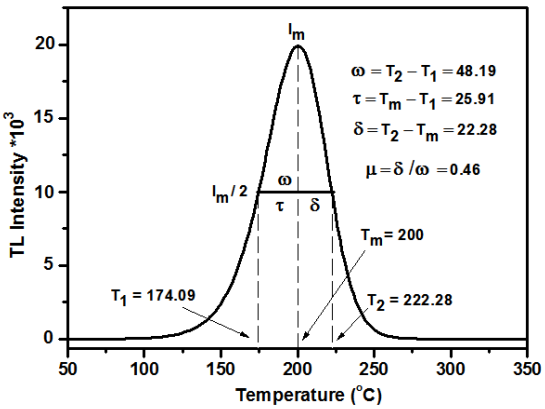

Fig.8: Peak shape methods in glow peak deconvoluted

Table 1 summarizes the values of the activation energy (E) to the glow peak before and after deconvolution. These results were obtained by using the Equations 1, 2, 3 and 4. The calculated values of the activation energy as a function of the geometric parameters (ω, τ and δ) showed only a small variation around 0.2 eV between the peaks at 202 °C and at 200 °C. The average activation energy (E_a) for the glow peak at 202 °C was equal to 0.93 ± 0.02 eV and 1.10 ± 0.02 eV for the peak at 200 °C.

Table 1 - Activation energy (E) values: peak at 202 °C and at 200 °C after deconvolution

T_m(°C)	E_ω(eV)	E_τ(eV)	E_δ(eV)	E_a (eV)
202	0.94	0.91	0.95	0.93 ± 0.02
200	1.10	1.08	1.11	1.10 ± 0.02

Table 2 presents the frequency factor (s) and lifetime (t) values considering general order kinetics. To calculate the frequency factor, the average activation energy (E_a) and kinetic order (b) were used in Equation 5. The mean lifetime was estimated at 25 °C (298 K) room temperature by using the Equation 6.

Table 2 - Frequency factor(s) and kinetic order b values

T_m (°C)	b	E_a (eV)	s (s^{-1})	t (days)
202(475 K)	1.15	0.93	1.72×10^9	35.45
200 (473 K)	1.32	1.10	1.47×10^{11}	311.76

The differences found in the activation energy (E_a) and kinetic order (b) values interfere significantly in the calculation of frequency factor (s). As a consequence, the calculated lifetime at room temperature for the glow peak of amethyst is also affected. Before the deconvolution procedure, the mean lifetime for the peak at 202 °C was approximately35 days. However, the calculated lifetime for the peak at 200 °C after deconvolution was around 312 days, suggesting a longer lifetime. The divergence in the results suggests that the peak at 202 °C may not be isolated enough for analysis with the peak shape methods.

In the powdered amethyst from Turkey (grains size 90x 140 μm), the main peak at 270 °C decay 14% after 7 days of stored at room temperature of 25°C (298 K) [14]. After that, no significant fading effect was observed around one month [14]. This result is more compatible with the lifetime calculated for the peak at 200 °C showed in Table 2.

In another study carried out in quartz from Japan, the parameters E = 1.07 eV and b = 1.32 for the TL peak at 192°C were observed after deconvolution [15]. These results are similar to the values of E and b for the peak at 200 °C shown in Table 2. On the other hand, in a natural quartz from Brazil, it was observed for the deconvoluted peak at 200 °C the parameters E = 1.28 eV and b = 1.38. In this case, a mean lifetime of 82 years was obtained at room temperature of 18 °C [16].

According to other authors, the fading observed after about one month of storage does not prevent the use of the amethyst for dosimetric applications [14]. In addition, the amethyst has the advantage that it can be reused without any substantial loss in sensitivity and various publications suggest the feasibility of utilizing the luminescence properties of amethyst for dosimetry purposes in high doses [14, 17, 18].

IV. CONCLUSION

By using the peak shape methods, it was observed a difference in the values of the kinetic parameters *b*, *E* and *s* before and after the deconvolution procedure. Different values of kinetic parameters directly interfere to calculated lifetime of the glow peak at 202 °C of amethyst investigated.

The mean lifetime found at room temperature of 25ºC, before and after deconvolution, were of the order of 35 and 312 days, respectively. These divergent results show the importance of the deconvolution to analysis of glow peaks that are not sufficiently separated.

It is important to highlight that no study has been found until now that investigated the kinetic parameters of the glow peak at 202 °C of amethyst from Lajeado exposed to high dose of gamma radiation. Therefore, further studies are still necessary that can affirm these preliminary results.

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