

STUDY OF THERMOLUMINESCENCE AND OPTICAL STIMULATED
LUMINESCENCE PROPERTIES QUARTZ CRYSTAL

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Abstract

Thermoluminescence (TL) and Optical Stimulated Luminescence (OSL) of natural quartz crystals from sediments have been studied in order to obtain the ages of the sediments deposition. The sediments were collected from an archaeological site, located in State of Mato Grosso do Sul, Brazil. In the present work the phototransfer TL process was observed in 394°C TL peak, that increased 10-20% in its intensity, when the sample was exposed to sunlight. As the samples did not provide a residual TL, a new methodology to evaluate the paleodose (P) was elaborated. Annual doses were evaluated based in uranium, thorium and potassium contents determined by neutron activation analysis. Preliminary ages were compared to those obtained by OSL and ages between (11 – 35) 10³ BP were obtained.

Keywords: Thermoluminescence, Optical Stimulated Luminescence, PTTL and Quartz

INTRODUCTION

TL dating using quartz crystal collected from dune sand, loess, colluvial sediments, etc, are often reported [1,2,3]. Usually its TL intensity decay up to residual one, when the crystal is exposed to sunlight, providing the "zero TL" of the sediments. However sometimes an occurrence of the Phototransfer TL (PTTL) process is verified, electron trapped in deep trap is evicted to shallow trap, when the crystal is stimulated by photon [4]. In this case traditional TL procedures [5,6,7] to evaluate P value can not be used. In the present work a new methodology to determine P was performed in sediments, that supply PTTL. Results of sediment ages are compared to the ones obtained by OSL.

EXPERIMENTAL

Six samples of sediments, from an archaeological site located in the State of Mato Grosso do Sul, Brazil (22°23'04''S and 52°52'08''W), were collected at different depths from 0.5 to 3.0m. Pure quartz grains with 88-180 μ m size were obtained after chemical separation with HF 20% (45min), HCl 20% (3 hours) and Heavy Liquid (SPT). Feldspar crystal was not verified during a test using OSL measurements with infrared stimulation.

TL glow curves were recorded with TL/OSL automated systems, model 1100-series Daybreak Inc, with optical filters Schott BG-39 (300-700nm) and the heating rate was 10⁰C/s.

For sunlight irradiation Hagen Incandescent heat lamp (150Watt) was used. OSL measurements were carried out with green light (590nm) and 10³second of stimulation. Irradiation was performed with a ⁶⁰Co source.

Annual dose values (Da) were determined assuming a cosmic ray contribution of 180 μ Gy/yr and the Bell's equations [8]. Uranium, thorium and potassium contents were determined by neutron activation analysis. About 100 mg of samples and standards of U, Th and K were irradiated in the swimming pool research reactor, IEA-R1m at a thermal neutron flux of about

$5 \times 10^{12} \text{ n cm}^{-2} \text{ s}^{-1}$ for 8h. Gamma spectra were obtained after 7 and 15 day decay time using a Ge hyperpure detector, model GX 2020 from Canberra, FWHM 1.9keV at 1332.5keV gamma peak of ^{60}Co and a 8192 channel S-100 Canberra MCA.

RESULTS AND DISCUSSION

Additive dose method with natural normalization (NN) protocol, using multiple-aliquot procedure was adopted to OSL measurement [8]. Figure 1a shows OSL shine-down decay curves for the sample P105, in which it was plotted the area below each shine-down versus additional doses. Exponential growths were observed (Figure 1b) and the equivalent dose (Q) was determined.

TL glow curves of samples supplied peaks at (173 ± 4) , (215 ± 5) , $(394 \pm 7) ^\circ\text{C}$ and also peaks above 400°C , as is shown in Figure 2a and their intensities increased with crescent radiation doses. In order to determine P the main assumption is based on the fact that before deposition the grain was exposed simultaneously to sunlight and ionizing radiation and after deposition just to ionizing radiation; growth curves of the 394°C TL peak intensities for both cases were obtained. It should be noted that the samples were previously heated at 480°C during 10 minutes.

Growth curves were plotted at the same figure and the intersection point simulates the TL intensity (I_0) of the grain at the deposition moment. An accumulated dose (D_0) related to this intensity I_0 was obtained and was subtracted from the total one (D) to obtain the point P. Figure 2b) shows TL growth curves obtained with the preceding procedure applied to P110 sample. In this case the $P = D - D_0$ is approximately 7.3Gy. It can be noted that both growths have a sub-linear behavior and after I_0 the sample exposed only to γ -irradiation grows faster than those exposed to sunlight and γ -irradiation.

All results obtained, i.e. uranium, thorium and potassium contents, Da, P, Q and ages determined by TL and by OSL are summarized in Table 1.

CONCLUSION

TL glow curves of quartz usually supply a variety of peaks and are related to different defects created by ionizing radiation and impurities that can be incorporated in the crystal net during the crystal formation. Many authors investigated the relationships between these defects and impurities with each TL peaks, but it is difficult to compare all the results, since there are a large specimens, impurities and formation conditions of the crystal in nature [9]. Matsuoka *et al.*, 1993 [10], studied TL and ESR of the quartz from Adamantina Formation, State of São Paulo, Brazil, according to the authors the $[\text{AlO}_4]^\circ$ center is related to peaks between 230 and 370°C and the E'_1 center with TL glow curve above 400°C. In the present work the PTTL effect was observed in samples that were heated at 480°C before irradiation and in natural samples exposed only to sunlight, thus the source of the electrons related to the PTTL process can be, probably, in temperatures higher than 480°C. Transfer of charges took place in all the peaks, including the two previously cited centers $[\text{AlO}_4]^\circ$ and E'_1 . Another conclusion is that there is a constant concentration of deep centers, because the rate of the growth, after I0, of the 394°C TL peak, irradiated just with γ -radiation increased faster than the one submitted to sunlight.

Within experimental error associated to both methods, the preliminary estimated ages seem to be in good agreement. Although the proposed method demands more caution in the experimental procedures and also in the analysis of the curves, it provides another way to date sample, which exhibit PTTL effects.

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FIGURES CAPTIONS

Figure 1.: **a)** OSL shine-down curves of quartz, natural and irradiated to crescent γ -radiation doses, using green light (590nm) and 10^3 second of stimulation; **b)** OSL growth curve for sample P105, Multiple-aliquot methods with natural normalization for quartz crystals from State of Mato Grosso do Sul, Brazil

Figure 2:**a)** TL Glow curves of quartz submitted to γ -radiation (dose 4Gy) and simultaneously γ -radiation (dose 4Gy) and sunlight; **b)** TL growth curves of 390°C TL peak, for sample P110 with γ -radiation doses, with and without sunlight exposition.

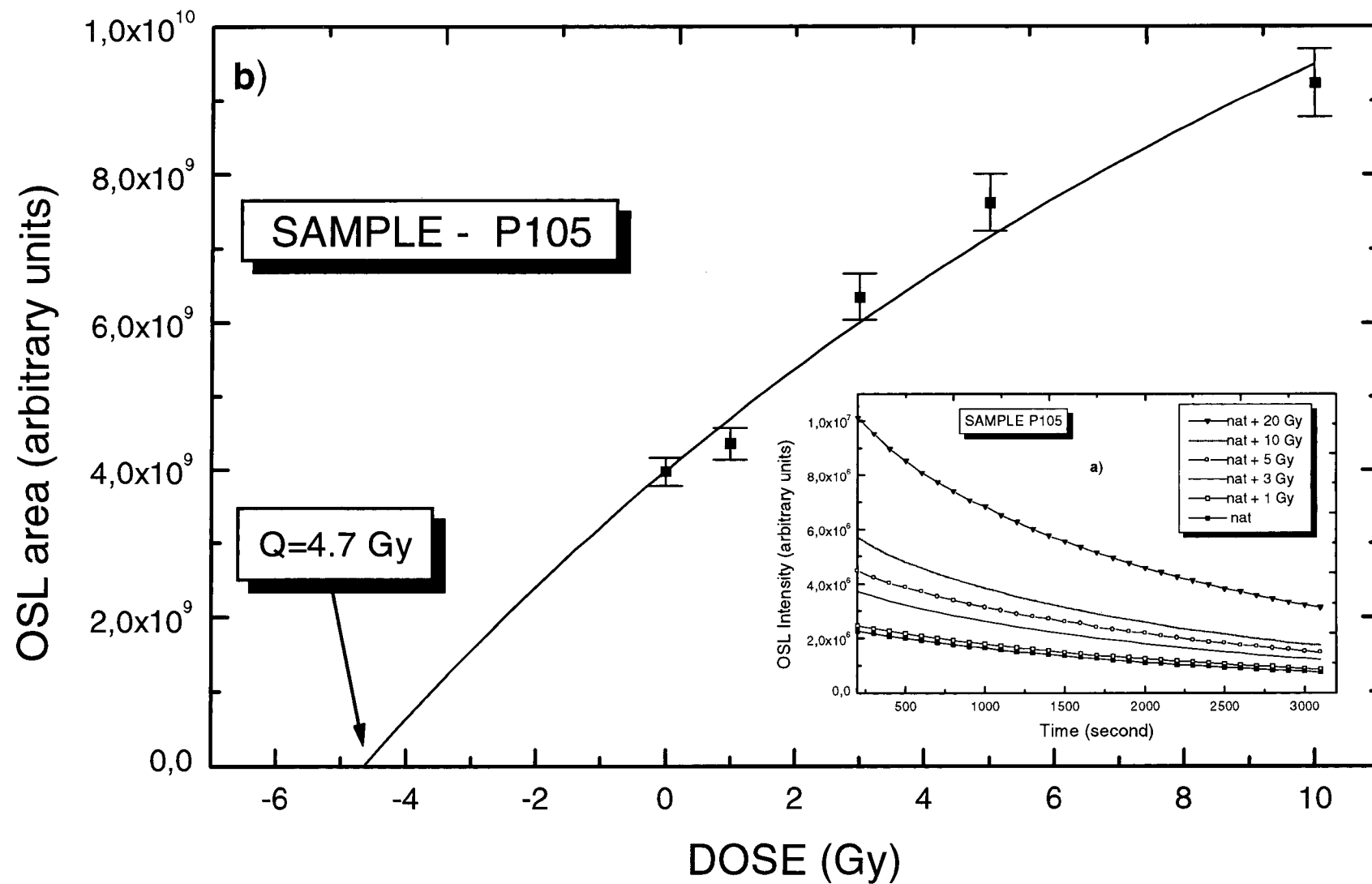


Figure 1

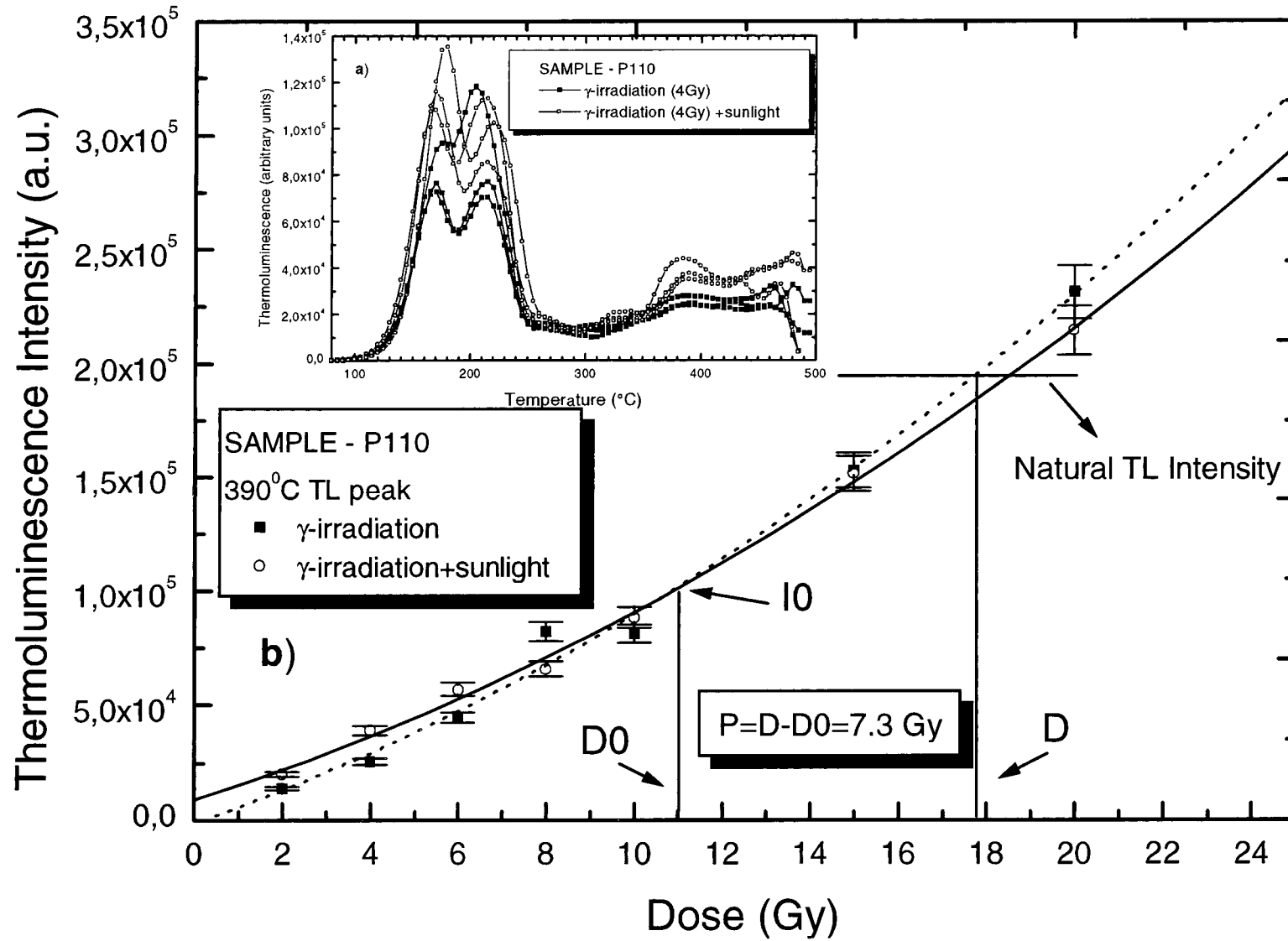


Figure 2

Table 1. Uranium, thorium and potassium contents, annual dose (Da), paleodose (P), equivalent dose (Q) and age determined by TL and by OSL.

| Sample Name | Depth (m) | U ²³⁵ +U ²³⁸ (ppm) | Th ²³² (ppm) | K ⁴⁰ (10 ⁻⁴ %) | Da (μGy/yr) | P (Gy) | Age (by TL) (10 ³ yr) | Q (Gy) | Age (by OSL) (10 ³ yr) |
|-------------|-----------|--|-------------------------|--------------------------------------|-------------|----------|----------------------------------|---------|-----------------------------------|
| P105 | 0.5 | 0.47±0.03 | 1.67±0.03 | 3.0±0.3 | 426±9 | 5.0±0.4 | 11.7±1.2 | 4.7±0.5 | 11.0±1.4 |
| P110 | 1.0 | 0.23±0.02 | 0.88±0.02 | 2.9±0.2 | 305±7 | 7.3±0.5 | 23.9±2.1 | 5.9±0.7 | 19.3±2.7 |
| P115 | 1.5 | 0.35±0.03 | 1.13±0.03 | 3.2±0.3 | 356±10 | 7.8±0.6 | 21.9±2.3 | 5.4±0.7 | 15.2±2.4 |
| P120 | 2.0 | 0.34±0.03 | 1.59±0.03 | 3.30±0.3 | 387±10 | 9.5±0.8 | 24.5±2.7 | 9.1±0.9 | 23.5±2.9 |
| P125 | 2.5 | 0.30±0.02 | 1.17±0.03 | 3.1±0.2 | 345±7 | 9.8±0.8 | 28.4±2.9 | 9.0±0.8 | 26.1±2.9 |
| P130 | 3.0 | 0.26±0.02 | 0.97±0.02 | 2.8±0.2 | 320±7 | 11.2±0.9 | 35.0±3.6 | 9.8±1.0 | 30.6±4.0 |

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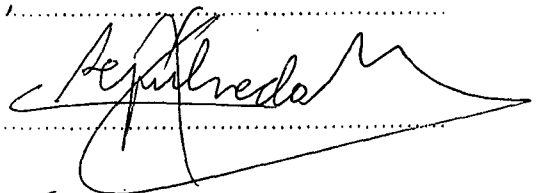
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