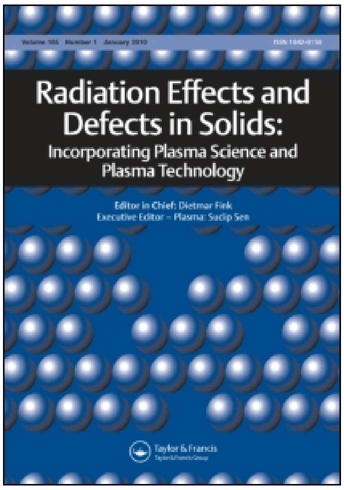

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Study of the Thermoluminescence and Optical Stimulated Luminescence properties of quartz crystal

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STUDY OF THE THERMOLUMINESCENCE AND OPTICAL STIMULATED LUMINESCENCE PROPERTIES OF QUARTZ CRYSTAL

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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 the 394°C TL peak, which 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 devised. Annual doses were evaluated based on uranium, thorium and potassium contents determined by neutron activation analysis. Preliminary ages were compared to those obtained by OSL and ages between (11–35) 10^3 BP were obtained.

Keywords: Thermoluminescence; Optical stimulated luminescence; PTTL; Quartz

1. INTRODUCTION

TL dating using quartz crystals collected from dune sand, loess, colluvial sediments, etc, are often reported [1–3]. Usually the TL intensity decays to a residual value, when the crystal is exposed to sunlight, providing the

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"zero TL" of the sediments. However sometimes an occurrence of the Phototransfer TL (PTTL) process is verified, where electrons trapped in a deep trap are evicted to a shallow trap when the crystal is stimulated by photons [4]. In this case traditional TL procedures [5–7] to evaluate the *P*-value cannot be used. In the present work a new methodology to determine *P* was performed on sediments that show PTTL. The results of sediment ages are compared to the ones obtained by OSL.

2. 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 3.0 m. Pure quartz grains with 88–180 μm size were obtained after chemical separation with HF 20% (45 min), HCl 20% (3 h) and Heavy Liquid (SPT). Feldspar crystals were 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 (300–700 nm) and kopp 7–59, and the heating rate was 10°C/s.

For sunlight irradiation a Hagen Incandescent heat lamps (150 Watt) was used. OSL measurements were carried out with green light (590 nm) and 10^3 s of stimulation. Gamma irradiation was performed with a ^{60}Co source.

Annual dose values (D_a) were determined assuming a cosmic ray contribution of 180 μGy/y and 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 5×10^{12} ncm^{-2}s^{-1} for 8 h. Gamma spectra were obtained after 7 and 15 day decay time using a Ge hyperpure detector, model GX 2020 from Canberra, FWHM 1.9 keV at the 1332.5 keV gamma peak of ^{60}Co, and an 8192 channel S-100 Canberra MCA.

3. RESULTS AND DISCUSSION

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

TL glow curves of the samples gave peaks at (173 ± 4), (215 ± 5), (394 ± 7)°C and also peaks above 400°C, as is shown in Figure 2(a), and their intensities increased with increasing radiation doses. In order to

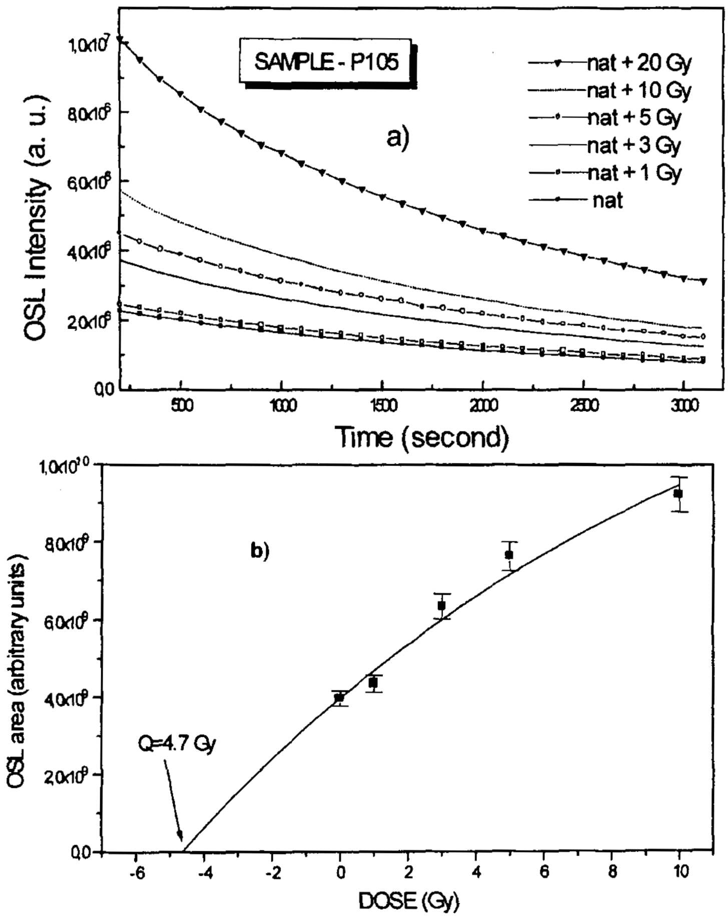

FIGURE 1 a) OSL shine-down curves of quartz, natural and irradiated to increasing γ-radiation doses, using green light (590 nm) and 10^3 s of stimulation; b) OSL growth curve for sample P105, Multiple-aliquot methods with natural normalization for quartz crystals from State of Mato Grosso do Sul, Brazil.

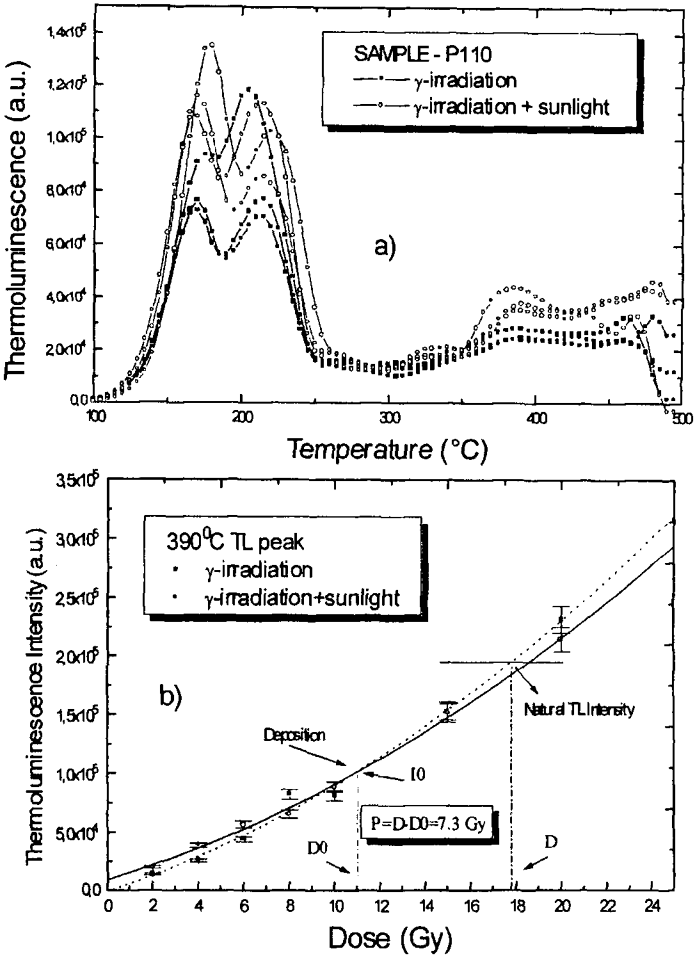

FIGURE 2 a) TL Glow curves of quartz submitted to γ-radiation (dose 4 Gy) and simultaneously γ-radiation (dose 4 Gy) and sunlight; b) TL growth curves of 390°C TL peak, for sample P110 with γ-radiation doses, with and without sunlight exposure.

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 to 480°C for 10 min.

Growth curves were plotted on 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 2(b) shows TL growth curves obtained with the preceding procedure applied to sample P110. In this case the $P = D - D_0$ is approximately 7.3 Gy. 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, D_a, P, Q and ages determined by TL and by OSL are summarized in Table I.

4. CONCLUSION

TL glow curves of quartz usually show a variety of peaks which are related to different defects created by ionizing radiation and impurities that can be incorporated in the crystal lattice during the crystal formation. Many authors have investigated the relationship between these defects or impurities and each TL peak, but it is difficult to compare all the results, since there are variety of specimens, impurities and formation conditions of the crystal in nature [9]. Matsuoka *et al*, [10], studied TL and ESR of the quartz from Adamantina Formation, State of São Paulo, Brazil; according to the authors the $[AlO_4]^0$ is related to peaks between 230 and 370°C and the E'_1 center with the TL glow curve above 400°C. In the present work the PTTL effect was observed in samples that were heated to 480°C before irradiation, and in natural samples exposed only to sunlight, thus the source of the electrons related to the PTTL process could be at temperatures higher than 480°C. Transfer of charges took place in all the peaks, including the two previously cited centers $[AlO_4]^0$ and E'_1. Another conclusion is that there is a constant concentration of deep centers, because the rate of the growth, after I_0, of the 394°C TL peak, irradiated just with γ-radiation increased faster than the one submitted to sunlight.

Within experimental error associated with 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

TABLE I Uranium, thorium and potassium contents, annual dose (d_a), paleodose (p), equivalent dose (q) and age determined by tl and by osl

Sample Name	*Depth (m)*	$^{235}U + ^{238}U$ *(ppm)*	^{232}Th *(ppm)*	K_2O *(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

the analysis of the curves, it provides another way to date samples which exhibit PTTL effects.

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