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ANNUAL DOSE MEASUREMENTS AND TL-DATING OF ANCIENT EGYPTIAN POTTERY

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Abstract—In the course of the dating of ancient Egyptian pottery, pottery sherds were collected from three archaeological tombs in Nazlet El Samman region, Giza zone (Egypt). The annual dose from natural background was measured by gamma spectroscopic technique as well as thermoluminescence (TL) measurements. The results of both methods are in good agreement with a consistency of 99.69%. The extracted quartz exhibited TL dating peaks at about (305 ± 5)°C. The TL dating shows an age of 4301 ± 100 years for the examined pottery which belongs to the "Fourth Dynasty" in the "Old Kingdom". The uncertainties in TL dating using the additive method are much lower than that of archaeologists.

1. INTRODUCTION

A summary of the dating ranges of various methods used in archaeological chronology is provided by Fleming (1976). The first researches for pottery dating have been investigated by many authors. Aitken (Aitken *et al.*, 1968; Aitken, 1985) and Mejdahl (1970) discussed the principles of TL dating. Fleming (1970, 1971) has developed two new techniques of TL dating for ancient pottery. The dating of pottery by TL has been investigated in the last twenty years in different laboratories over the world (Yanchon *et al.*, 1988; Nishimura and Horinouchi, 1989; Roosevelt *et al.*, 1991; Koul, 1992). In the present work, TL datings of old Egyptian artifacts are given. Our samples were extracted from Pharaonic tombs located at Giza Pyramids zone about 2 km south-east of the Sphinx. The objective of the dating project was to investigate the feasibility of TL dating for the pottery samples from this particular site and to find out whether the dates would help the excavating archaeologists to solve their puzzles. The old Egyptian sites usually are well dated by archaeological evidence but in cases where the only evidence is everyday pottery with stylistic features scarcely changing through the centuries, TL dating should be more useful.

2. GAMMA SPECTROSCOPY TECHNIQUE

The gamma spectrum of the soil samples was investigated using a 10% relative efficiency hyper-pure germanium (HPGe) gamma ray spectrometer with an energy resolution of about 2 keV at 1.33 MeV. The soil samples were stored for at least 24 days to ensure the existence of the radon daughters.

From the calibration curve of a HPGe spectrometer (El-Kamessy *et al.*, 1992), the energies of the photopeaks of γ-spectrum can be determined and the radioactive elements can be qualitatively measured. According to the fact that U and Th ores should be in series equilibrium with their daughter products, the most suitable characteristic γ-rays peaks are at 1.764 MeV due to ^{214}Bi and 2.615 MeV due to ^{208}Tl. These peaks are used to detect the presence of equivalent uranium and equivalent thorium, respectively, and to measure their concentrations. The concentrations in units of parts per million (ppm) were calculated using the general decay equations and the conversion factors given by Singhvi and Chawia (1986). The dose rates shared by α and β particles and γ-rays from each nuclear series were also calculated. The concentration of ^{40}K was estimated by using the flame photometry technique. Table 1 illustrates the concentrations of ^{238}U, ^{232}Th and ^{40}K in soil together with the corresponding annual doses. The total annual dose was found to be 3.5 ± 0.15 mGy/y.

3. THERMOLUMINESCENCE TECHNIQUE

The measurement of the external γ-dose rate at the excavation site was carried out using a thermoluminescence dosimeter type $CaSO_4$:Dy of dimensions $6 \times 6 \times 1$ mm^3 over two periods of time (1 and 2 months) and at 3 different locations on the site. Using 30 assorted and annealed dosimeter chips, the experimental and calculational procedures comprise the following steps for each time period:

(1) All chips were annealed under the same circumstances.
(2) Fifteen of these chips were irradiated with a predose (D_0), using a calibrated β-radiation source (^{90}Sr Harshaw model 200-DI of 6.2 mrads/rev).

Table 1. Annual dose from different types of radiation in soil (mGy/y)

Radioactive series	Concentration in soil	Annual dose (mGy/y)				
		α	β	γ	Sub-total	Total
^{232}Th	3.346 ± 0.134 ppm	2.47 ± 0.1	0.1 ± 0.004	0.17 ± 0.007	2.74 ± 0.11	0.68 ± 0.03
^{238}U	1.4876 ± 0.087 ppm	4.14 ± 0.24	0.22 ± 0.01	0.17 ± 0.01	4.53 ± 0.27	0.99 ± 0.06
^{40}K	(1.7 ± 0.06) %	—	1.4 ± 0.05	0.42 ± 0.015	1.84 ± 0.07	1.84 ± 0.07
Total annual dose						3.5 ± 0.15

(3) A set of three irradiated and three unirradiated TL-chips were buried in each of the three archaeological locations. The chips were packed in thin bags to avoid the effect of sunlight while all types of radiations can pass through.
(4) The remaining irradiated and unirradiated TL-dosimeters were kept as references and read out along with the buried dosimeters. The calibration factor (K) for the dosimeters was calculated using the following equation

$$K = (I_{irr} - I_{bg})/D_0 \quad (1)$$

where I_{irr} = TL of the reference pre-irradiated dosimeters, I_{bg} = TL of the reference unirradiated dosimeters (background) and D_0 (pre-irradiation dose) = 2 rads.
(5) After a burying period T, the buried dosimeters were recovered and read out. From the resulting data, the fading correction factor (F) can be calculated as follows:

$$F = (I_{irr} - I_{bg})/(I - I_u) \quad (2)$$

where I = TL of buried pre-irradiated dosimeters and I_u = TL of buried unirradiated dosimeters after the burying time T.
(6) Finally, using the calibration factor K and the fading correction factor F, the annual dose can be calculated using the following equation

$$D_s = (I_u - I_{bg})F/(KT) \quad (3)$$

The results are illustrated in Table 2. These results are in good agreement with those determined by the γ-spectroscopy method and they are in consistence within 92.57%.

4. SAMPLE PREPARATION AND MEASURING TECHNIQUE

The pottery samples which were extracted from different tombs (denoted by GSE 1903, GSE 1908 and GSE 1907) contain few quartz and feldspars inclusions. Some of the pottery pieces are red-brown in colour, with thick layer texture, while the others are brown-grey, with thin layer structure and comparatively hard. About a 2 mm layer from to pottery sample surface was removed using a sharp knife to eliminate the effect of light and β irradiation from the surrounding soil. The pottery sherds then were broken down by a gentle crushing in an agate mortar and larger crystal grains (about 200 μm) were removed by hand. TL dating measurement have been achieved using crystals in the range from 90 to 100 μm diameter (Fleming, 1970). The outside of the grain is etched with hydrofluoric acid (1 N) for 100 min (Fleming, 1970). This was originally performed to remove any minerals except quartz grains such as feldspars and calcite.

Table 2. Annual dose results using $CaSO_4$:Dy dosimeter

Measured parameters	Buried time	
	One month	Two months
TL_N	37.86 ± 0.1	45.73 ± 1.87
TL_{bg}	36.73 ± 0.49	43.43 ± 0.61
TL_{irr}	128.41 ± 0.88	135.11 ± 0.79
TL	126.9 ± 0.88	123.46 ± 2.01
Annual dose (rad/year)	0.3 ± 0.02	0.348 ± 0.03
Average annual dose	0.324 ± 0.005 (mGy/year)	

The thermoluminescence characteristics for each sample were measured on a set of different weights of 1–14 mg. The results show that the optimum weight for such quartz grains must be in the range 5–10 mg. The effect of heat on the quartz samples was also extensively studied. The optimum condition for the annealing process of the quartz grains was found to be 500°C for 2 h. The fading characteristic of the quartz samples extracted from the pottery sherds (for the 305°C glow peak) is illustrated in Fig. 1. It is clear that the TL-signal decreases to 96% from the original value after the first 5 days. The TL-signal is then stabilized up to 60 days. There is clearly good agreement that the sample under investigation

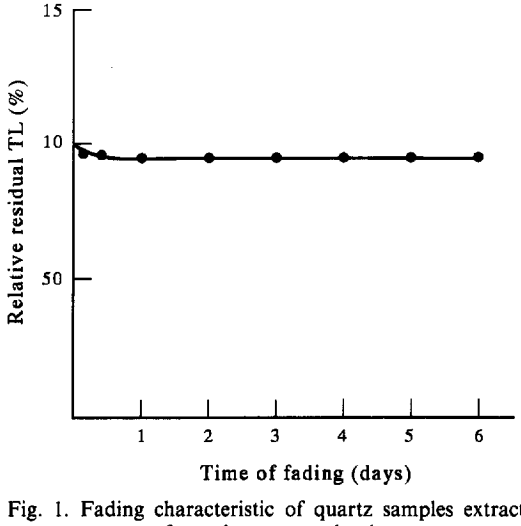

Fig. 1. Fading characteristic of quartz samples extracted from the pottery sherds.

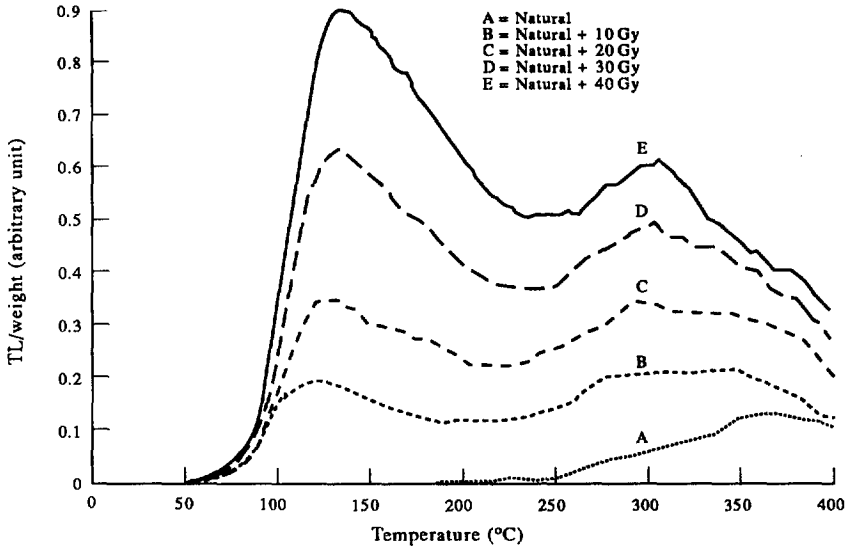

Fig. 2. The TL glow curves of quartz grains from pottery by the fixed additive dose procedure (without annealing).

(quartz grains) are not subjected to an anomalous fading. For archaeological dose measurements, a fixed additive dose procedure was followed using 25 weighed samples (5–10 mg). The "additive method" (Fleming, 1970) is used to overcome the problem of the change in the sensitivity of the sample after 500°C. The irradiation of the pottery sample was performed using the automatic ^{137}Cs source. To obtain the first glow growth curve, five of these samples were used for measuring the natural TL-signal, and the other 20 samples for the higher TL-levels by irradiation with additive doses ranged from 10 to 40 Gy in 10 Gy increments. The optimum heating rate is 2.5°C/s. The TL-glow curves are illustrated in Fig. 2. With respect to the second glow-growth curves, the samples were irradiated with the same doses as in the case of the first set of glow curves after annealing at 500°C for 2 h and left cooling to room temperature in oven. The results are illustrated in Fig. 3. The peak above 300°C was used for dating calculations. Figure 4 illustrates growth curves for quartz grain samples from the tomb, showing the excellent linearity and supralinearity which were commonly observed in the quartz samples. The equivalent dose ED as well as the correction term I were calculated by a linear extrapolation as shown in Table 3. The archaeological dose AD = ED + I. The TL ages for the samples were calculated and the results are illustrated in Table 3. The obtained ages give a mean value of 4301 ± 100 years.

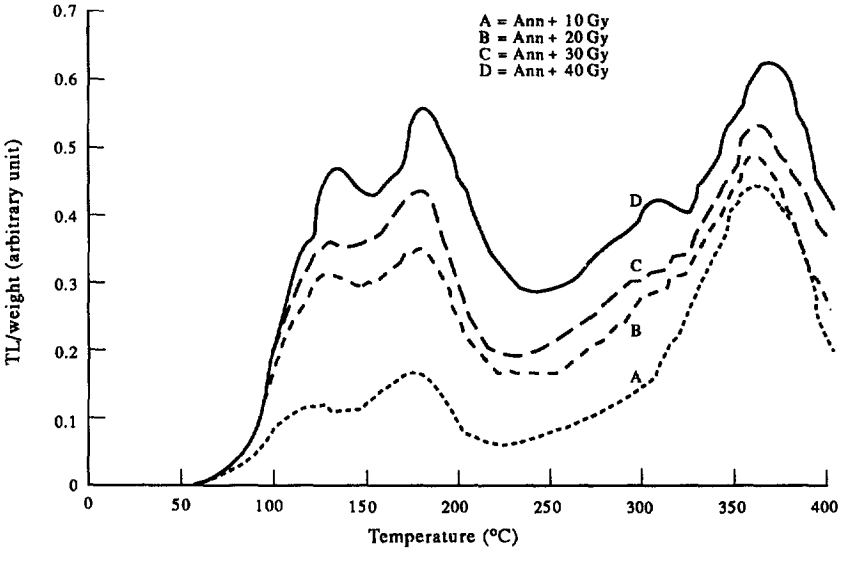

Fig. 3. The TL glow curves of quartz grains from pottery by the fixed additive dose procedure (after annealing at 500°C for 2 h).

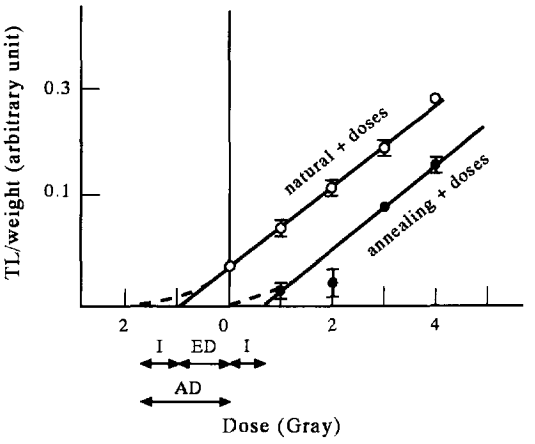

Fig. 4. Growth curves for quartz grain samples from the tomb.

Table 3. Age determinations for pottery samples extracted from Pharaonic tombs

Sample	Measured dose (Gy)	Intercept (I) (Gy)	Archaeological dose (AD) (Gy)	Annual dose (mGy/y)	TL age (year)	Mean TL age (year)	Archaeological age (year)
GSE 1903	9.16	4.556	13.716		4194.49		
GSE 1907	9.563	4.941	14.504	3.27 ± 0.07	4435.47	4301 ± 100	4218–4992
GSE 1908	10.164	3.812	13.9766		4274.19		

5. CONCLUSION

A comprehensive investigation has been made to measure the annual dose at an archaeological site, south west of Nazlet El Semman region, Giza, Egypt. The result obtained by the gamma spectroscopic analysis for the total annual dose was 3.5 ± 0.15 mGy/y and that by the TL method was 3.24 ± 0.05 mGy/y. The results of these two methods are in good agreement. One of the main contributions of this work lies in the positive new results obtained, which at least show that it is possible to use these methods to date other samples from sites in the nearby areas. As far as we are aware, the present work was the first measurement of the annual dose for TL dating of the ancient Egyptian pottery.

Then, the obtained results using the thermoluminescence technique for dating of ancient Egyptian pottery proved to be very valuable to the archaeologists. The age of the extracted pottery sherds is consistent with that estimated by the archaeologists using some special notations which attributed the tombs to the "Fourth Dynasty" in the "Old Kingdom". The obtained ages show that the uncertainties in dating using the TL technique are much lower than the age range of the archaeologists, and that extension of the programme to other sites will be of significant value for understanding the morphology of the ancient pottery samples.

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