

# **Report on the Optical Dating of Wong Tei Tung Archaeological Site**

Department of Earth Sciences, The University of Hong Kong

## **Summary**

Four samples taken from section of Wong Tei Tung Archaeological Site have been examined using the optically stimulated luminescence (OSL) from quartz sand grains extracted from the deposit. Each sample represents a sedimentary layer containing archaeological findings. OSL can be used to date the last time the sediment was exposed to light. This is usually during the last transport event or exposed to the surface prior to final deposition, if there was sufficient exposing to sunlight. OSL studies indicate that the sedimentary grains had been exposed uniformly to sunlight prior to deposition. Hence, the OSL ages give the maximum ages for the samples, because the 'OSL clock' was not reset at deposition. Results using this method give a most likely age of 0.57 thousand years (ka) for the top layer L1. This reliable ages for the layers, L3, L4 and L5 are 2.23, 2.76 and 10.3 ka, respectively.

## **總結**

本報告描述了香港西貢黃地峒考古遺址的光釋光測年研究。共研究 4 個光釋光樣品，每個樣品代表一個有考古發現的層位。光釋光方法是測量沉積物顆粒最後一次暴露太陽光以來的年齡。如果曝光的時間夠長，這一般是指沉積顆粒處於地表或最後一次搬運以來的年齡。我們研究發現，黃地峒樣品中顆粒的曝光不均勻。樣品在沉積時並沒有使光釋光「時鐘」完全回零。這樣，光釋光年齡只能認為是樣品的最老年齡。結果顯示，沉積層位 L1, L3, L4 和 L5 的最可靠年齡分別為 570 年, 2230 年, 2760 年和 10300 年。

## 1 INTRODUCTION

This report describes results from the optically stimulated luminescence (OSL) dating of Wong Tei Tung Archaeological site. OSL dating depends on the phenomenon of luminescence, where visible light is emitted by the sample if appropriately stimulated. This light is derived from electrons trapped in defects in the crystal structure of certain minerals, particularly quartz. The electrons are trapped when ionising rays (from natural radioactivity) pass through the mineral; for a constant environment, the number of trapped electrons is dependent on time. Electron traps can be emptied by exposure to daylight (a process known as bleaching), and so in sediments the trapped population of electrons begins to build up from the time of deposition. Thus the trapping process represents a ‘clock’ used to date the time since the grains last exposed to sunlight prior to deposition, which was when the ‘clock’ was reset.

The trapped electron population is measured by stimulating a sample of quartz using a blue light. This excites the electrons from the traps, and allows them to give up their stored energy during recombination. In the process they emit ultra-violet light, which is measured using a photomultiplier equipped with filters to reject the stimulating light. This signal is known as optically stimulated luminescence (OSL). To convert the OSL signal into a measure of the total energy absorbed by the crystal (the dose), a known radiation dose is given to the sample, and the OSL response derived from this known dose is measured, to give the sensitivity,  $\chi$ . The so-called equivalent dose,  $D_e$ , is given by dividing the OSL from the natural sample by  $\chi$ .  $D_e$  is the dose equivalent in unit of Gy to that which has been accumulated in the sample since burial. The age equation can then be expressed as

$$Age = \frac{D_e}{doserate} \quad (1)$$

The dose rate, in unit of Gy/ka, is calculated by measuring the concentrations of naturally occurring radioisotopes (U, Th and K), and converting these to dose rates using standard relationships (Aitken 1998). A contribution from cosmic radiations is included, and some allowance were made for the average water content of a sample, because the water can attenuate the effects of the radiations.

## 2 SAMPLES

The samples used in this study were collected on May, 19 2006 with assisting from Mr. K.T.W. Sun and Ms. C.K.M. Wong from the Antiquities and Monuments Office (AMO) of the Leisure and Cultural Services Department, HKSAR. During sampling, assistants were obtained from the experts of Dr Tracey Lu and Profs. Xia Zhengkai and Lu Zune from The Chinese University of Hong Kong and Peking University, respectively. 12 samples were collected with duplicates samples from each archaeological layer (2 from layer I, 3 from layer III and IV, 2 from layer V and VI), . Duplicate samples were collected by HKU and Dr Tracey Lu in the section. There is no suitable material for luminescence dating from layer II. The sampling positions are given in Figure 1.

All OSL samples were obtained by hammering stainless steel tubes into freshly cleaned vertical sections (Figure 1b). The sample cans were covered with a black plastic film soon after taking them from the section, then sealed inside plastic bags to ensure that the sediment retained its natural water content.

a.





b.



Figure 1a. Prof. Xia Zhengkai divided the soil layers, which form the stratigraphy of this report;  
Figure 1b. Photos of sampling positions in Wong Tei Tong site.

The archaeological section was described in details from a report from Hong Kong Archaeological Society given by AMO. The archaeological layers and positions are shown in Figure 2. The section is located in the root of a hill. The sediments are mainly poorly sorted colluvial sediments, consisting of gravels, clays and sands, transported in relatively short distance. On the top of the section, layers 1 and 2 were soils developed and showing grey, whereas the sediments below layer 2 are yellowish in colour.

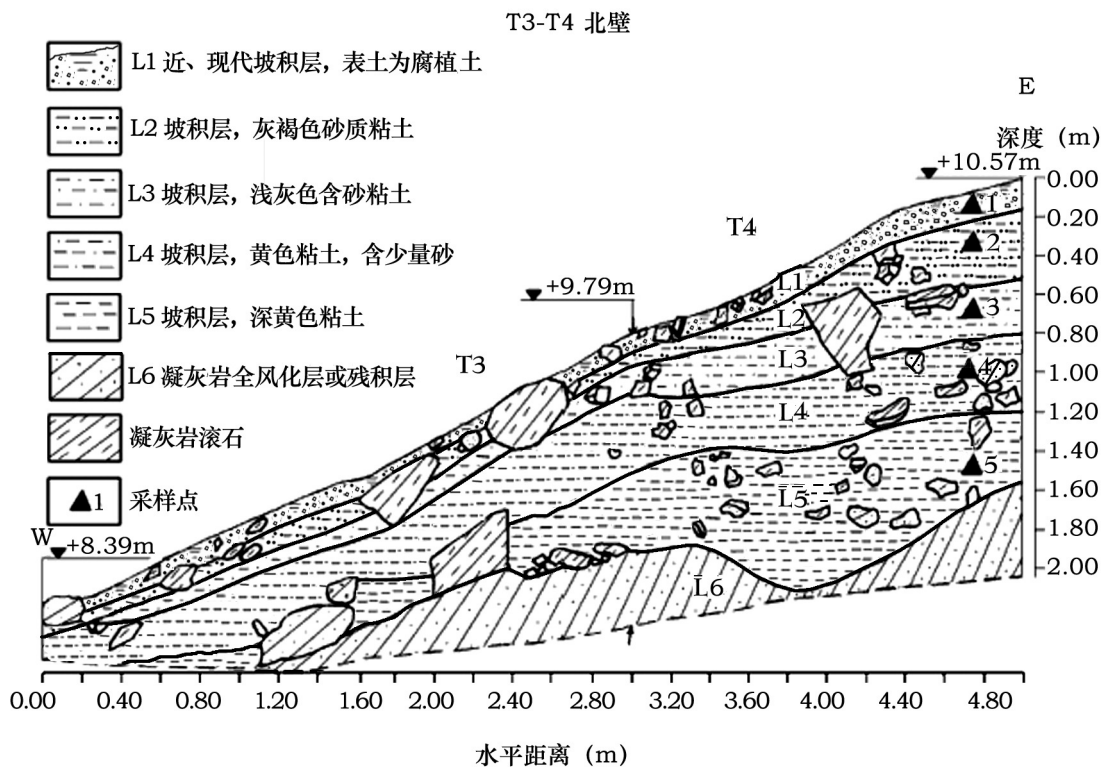


Figure 2. Schematic diagram showing the sedimentary layers in Wong Tei Tung site (redraw from a report from Hong Kong Archaeological Society given by AMO).

In the laboratory, the material at each end of the tube was scraped away and used for dose rate measurements. Quartz grains were prepared following procedures of sieving, heavy liquid separation and HF etching in subdued red safe light conditions. Raw samples were treated first with 20%  $H_2O_2$  and 10% HCl to remove organic materials and carbonates. Grains between 125-150  $\mu m$  were selected by mechanical dry sieving. Quartz grains (density between 2.62 and 2.70  $g/cm^3$ ) were separated using sodium polytungstate heavy liquid. The separated quartz grains were treated with 40% hydrofluoric acid for 1 hours to destroy any remaining feldspar. Separated quartz grains were mounted on 10 mm diameter aluminium discs with “Silkospay” silicone oil for measurement. The purity of the quartz grains was

tested by monitoring infrared bleaching of the OSL signal and by measuring the 110°C TL peak (Li et al., 2002).

Sand-sized (125-150µm) quartz grains were used for dating, because quartz is known to be very easily reset by daylight, and because it stores its trapped electron population for times much longer than the likely range of practical dating. Thus the trapped electron population which gives rise to the OSL signal is completely stable over the time period of interest.

### **3. Analytical facilities and experimental details**

All the OSL measurements reported here were carried out using an automated Risø TL/OSL reader with a blue light OSL attachment (Bøtter-Jensen, et al. 1999). The stimulating light source is an array of blue (470±30 nm) light emitting diodes filtered using a GG-420 filter to remove the short wavelength tail in the output spectrum. The intensity of light incident on the sample is about 16 mW cm<sup>-2</sup>.

The background signal recorded by the TL/OSL reader during blue light stimulation, measured using a blank disc is about 60 counts s<sup>-1</sup>. The OSL is measured through two 3 mm thick U-340 filters (Bøtter-Jensen et al., 1999). All measurements were run under the control of the software package TL/OSL for Windows. The reader is also equipped with a <sup>90</sup>Sr/<sup>90</sup>Y beta source delivering about 0.092 Gy s<sup>-1</sup> to quartz mounted on aluminum discs.

The equivalent doses ( $D_e$ ) were determined by the single-aliquot regenerative-dose (SAR) protocol (Murray and Wintle, 2000) for quartz aliquots. The stimulation conditions were 40 s of blue light. The preheat condition was 260°C for 10 s with a cut heat temperature of 220°C applied after the test dose. The OSL signals,  $L_n$  and  $L_x$ , integrated over the first 2 s and with subtraction of the equivalent average of OSL signals in the last 10 s, were taken as the OSL intensities used for  $D_e$  determination. Sensitivity monitoring was achieved by measuring the OSL signal,  $T_n$  and  $T_x$ , created by a test dose of typically 4.0 Gy. The  $D_e$  values were calculated using the sensitivity-corrected OSL intensities  $L_n/T_n$  and  $L_x/T_x$ . All samples were subjected to tests for recycling ratio and dose recovery. Aliquots with a recycling ratio falling outside the range of 1.0±0.1 and recovering a dose more than 10% away from the delivered dose were not used in the  $D_e$  calculation. The  $D_e$  values of each samples were shown in Table 1.

The environmental dose rate was measured using a variety of techniques. Thick source alpha counting (TSAC) was used to measure contributions from the U and Th decay chains. The alpha count rates were converted into alpha, beta and gamma dose rates according to Aitken (1998). The K content of the bulk sediments was measured by flame photometry (Zhao and Li 2006). The water content was calculated from the sample weights before and after drying at 105°C in an oven. The cosmic ray contribution to the dose rate was calculated from the burial depth and the altitude of the samples.

#### 4 RESULTS AND DISCUSSIONS

The samples information and dating results were summarized in table 1 and Table 2. Because the nature of samples is colluvial deposits, the grains only exposed shortly to sunlight prior deposition. The average  $D_e$  values and their ages are overestimated (Li 1994; Li 2001). This insufficient bleaching can be seen clearly in the large scatter shown by the samples and radial plots (Figure 3).

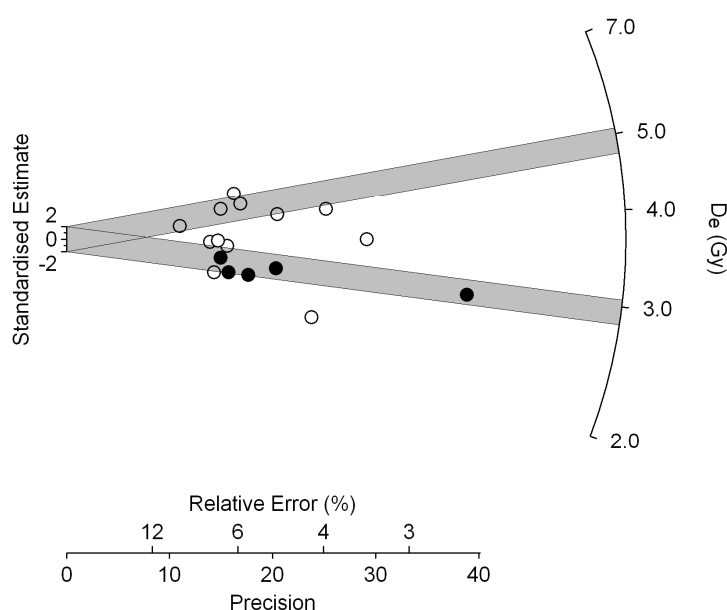


Figure 3. Radial plot of  $D_e$  values obtained from aliquots of sample WTT-1.

The scatter in values of  $D_e$  from all samples was unusually large. This was arisen because of incomplete bleaching, which would result in OSL ages that are systematically too old. This is a result of incompletely reset of the clock of some quartz grains and give rise to a



residual dose (or age) in the grains. It can also be the result of mixed grains with different ages. The appropriate De values must be analysed for these samples.

Table 1. Summary of samples and OSL dating results

HKU laboratory code	Sediment layer	Alpha counting rate c/ks	Water content %	K content %	Dose rate Gy/ka	De Gy	OSL age ka
WTT-1	L1	29.98	22.1	1.8	5.19	2.95	0.57
WTT-3	L3	32.26	20.1	1.71	5.39	12.0	2.23
WTT-4	L4	41.15	21.0	1.86	6.52	18.0	2.76
WTT-5	L5	45.57	24.2	1.81	6.81	70.0	10.3

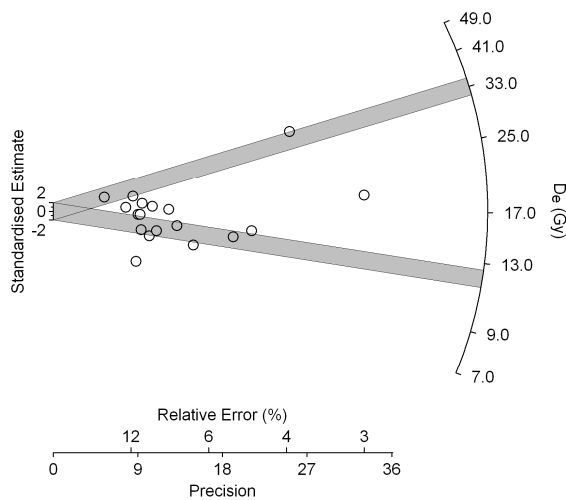
Table 2. Summary of De values obtained for samples from Wong Tei Tung

HKU laboratory code	Sediment layer	Average De value (Gy)	Average Age (Ka)	De value from Radial plot (Gy)	Likely age (ka)	Largest De from radial plot	Oldest ages (ka)
WTT-1	L1	3.7±1.0	0.71±0.49	2.95	0.57	4.90	0.94
WTT-3	L3	17.0±7.9	3.15±2.43	12.0	2.23	33.0	6.12
WTT-4	L4	23.9±11.8	3.66±2.12	18.0	2.76	45.0	6.90
WTT-5	L5	90.2±32.0	13.2±6.1	70.0	10.3	120	17.60

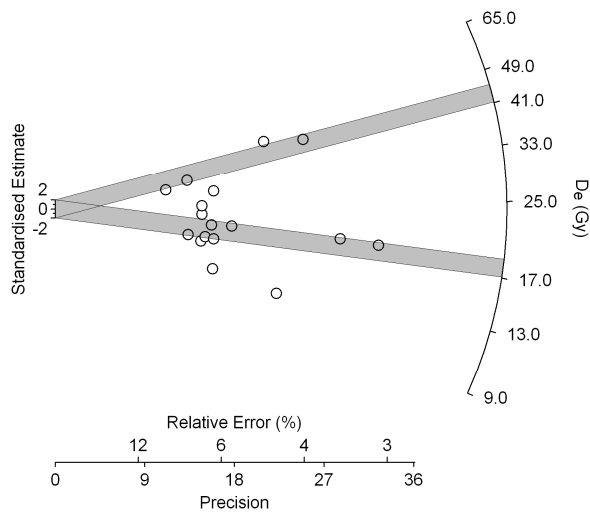
Note: sediment layer 2 has no suitable materials for OSL dating in the ditch.

Instead of using the average De values, we have analysed the De values in radial plots of the samples (Figure 4). Both a minimum and a maximum De values for each sample were obtained. Giving the nature of the samples, the minimum De values, and thus the ages, represent the most reliable De values and ages, because they may still contain some residual doses. The maximum ages were also given to give an indication on the likely age range of the samples.

a.



b.



c.

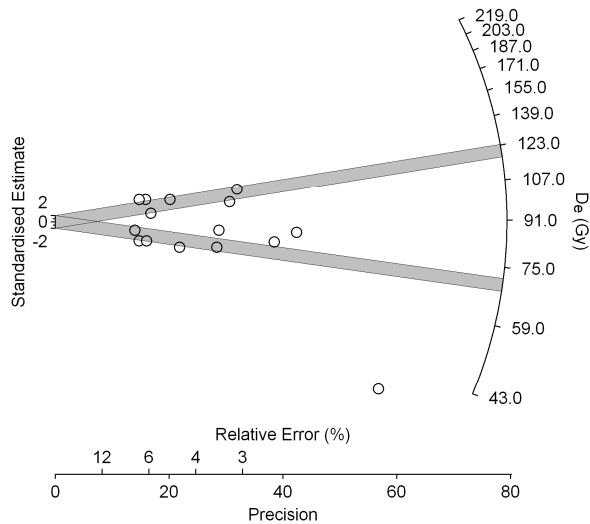


Figure 4. Radial plots of samples from Wong Tei Tung. a. WTT-3, b. WTT-4, c. WTT-5.

The reliable ages for the samples were summarized in Table 1. The errors for the ages are difficult to analysed precisely because of uncertainty in De values of each sample. However, it would not be larger than the error given in the average ages.

The results indicate that the Wong Tei Tung site was occupied by human as early as beginning of the Holocene (10.3 ka). Such age is supported by the sea-level changes in the past as suggested that the sea-level reached a level comparable to present level during the early part of the Holocene (Lambeck and Chappell, 2001; Yam and Huang ,2002). Thus, human preferred to residence in places where food from the sea was easily accessed. Human occupied the site is unlikely to be earlier than 18 ka ago because the see level was about 100 meters below current level (Lambeck and Chappell, 2001). Results also indicate that the upper part of the section formed in late part of the Holocene.

## 5 CONCLUSIONS

Four samples from Wong Tei Tung site presented some difficulties in optical dating, because of incompletely bleaching of the samples prior to deposition. Nevertheless, the OSL ages derived from analyzing the De values are considered reliable. Such occupying ages is supported by the sea-level changes during the later Quaternary period-Holocene. The deposits of the sedimentary layers formed not earlier than 18ka ago. In order to date the samples



precisely, Single Grain Dating system should be used. We recommend that the samples should be measured with single grain analyses.

#### Acknowledgements

We thank Mr. K.T.W. Sun and Ms. C.K.M. Wong for help in sampling, and comments on the initial version of this report, which make this in a better shape. Our thanks go to Mr. Li Bo and Miss Lee Ting for preparing the samples and doing laboratory measurements.

#### References:

- Aitken, M.J. 1998: An Introduction to Optical Dating. Oxford University Press, Oxford.
- Bøtter-Jensen, L., Duller, G. A. T., Murray, A. S., Banerjee, D., 1999. Blue light emitting diodes for optical stimulation of quartz in retrospective dosimetry and dating. *Radiation Protection Dosimetry* 84, 335-340.
- Lambeck K. and Chappell J. 2001. Sea level change through the last glacial cycle. *Science* 292, 679-686.
- Li S.-H. 1994. Optical dating: insufficiently bleached sediments. *Radiation Measurements* 23, 563-567.
- Li S.-H. 2001. Identification of well-bleached grains in the optical dating of quartz. *Quaternary Science Reviews* 20, 1365-1370.
- Li S.-H. Sun J.M. and Zhao H. 2002. Optical dating of dune sands in the northeastern deserts of China. *Palaeogeography, Palaeoclimatology, Palaeoecology*, **181**, 419-429.
- Murray, A.S. and Wintle, A.G. 2000. Luminescence dating of quartz using an improved single-aliquot regenerative-dose protocol. *Radiation Measurements* 32, 57-73.
- Yim W.W.S. and Huang G.Q. 2002. Middle Holocene higher sea-level indicators from the south China coast. *Marine Geology* 182, 225-230.
- Zhao H., Li S.-H. 2005. Internal dose rate to K-feldspar grains from radioactive elements other than potassium. *Radiation Measurements* **40**, 84-93.

#### Supplementary information from AMO

OSL dating report of Zhongshan University  
Sampling record for 2004-05 from archaeological survey  
Articles from 人類學報 and SCMP's news clipping.