OPTICALLY STIMULATED LUMINESCENCE (OSL) DATING OF ÇATALHÖYÜK SAMPLES

A THESIS SUBMITTED TO
THE GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES
OF
THE MIDDLE EAST TECHNICAL UNIVERSITY

BY

K. GÖZE AKOĞLU

IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF
MASTER OF SCIENCE
IN
THE PROGRAM OF ARCHAEOMETRY

JANUARY 2003

Approval of the Graduate School of Natural and Applied Sciences

Prof. Dr. Tayfur Öztürk
Director

I certify that this thesis satisfies all the requirements as a thesis for the degree of Master of Science .

Prof. Dr. Ay Melek Özer
Head of Department

This is to certify that we have read this thesis and that in our opinion it is fully adequate, in scope and quality, as a thesis for the degree of Master of Science.

Asst. Prof. Dr. Mustafa Özbakan
Supervisor

Examining Committee Members

Asst. Prof. Dr. Mustafa Özbakan

Prof. Dr. Ay Melek Özer

Prof. Dr. Şahinde Demirci

Prof. Dr. E. Hale Göktürk

Prof. Dr. Selahattin Özdemir

ABSTRACT

OPTICALLY STIMULATED LUMINESCENCE (OSL) DATING OF ÇATALHÖYÜK SAMPLES

Akoğlu, K. Göze

M.Sc., Department of Archaeometry

Supervisor: Asst. Prof. Dr. Mustafa Özbakan

January 2003, 55 pages

The objective of this study was to perform Optically Stimulated Luminescence (OSL) dating on mud brick samples from different levels of the Neolithic site Çatalhöyük using both feldspars and quartz in them.

At the beginning, a literature survey has been done about the dating system and the methodology of dating. The calibration of the system was done to carry out the experiments. The five mud brick samples, which had been sent to the METU (Middle East Technical University), Archaeometry Graduate Program in 1998 and 1999, were dated.

The mineral compositions of the samples were examined by X-ray Diffraction Analysis, which showed that all samples contain quartz and feldspars.

The equivalent dose was found by using Multiple Aliquot Additive Dose (MAAD) technique using both Infra Red diode array and Filtered Lamp

Module of the system that gave the Infra Red Stimulated Luminescence (IRSL) and Green Light Stimulated Luminescence (GLSL) ages for samples respectively.

Alpha counter specified the dose components of uranium and thorium contributions to the annual dose. The potassium was determined by Atomic Emission Spectrometry. The cosmic ray component of annual dose was evaluated by the Thermoluminescence Dosimeter (TLD) discs which have been placed and kept for 8 months in the site Çatalhöyük where samples were taken.

From data the IRSL and GLSL ages were calculated for five mud brick samples with unit numbers S4240, S5206, N3010, S2817 and S3707 with the help of the OSL system software. The IRSL ages for samples are (3.07 ± 0.23) ky, (7.77 ± 0.62) ky,(2.79 ± 0.27) ky, (8.54 ± 0.69) ky, (2.37 ± 0.23) ky respectively. The GLSL ages for samples are (5.68 ± 0.92) ky, (7.14 ± 0.57) ky, (6.34 ± 0.57) ky, (8.45 ± 0.73) ky and (2.80 ± 0.28) ky respectively.

Key Words: dating, Optically Stimulated Luminescence (OSL), Green Light Stimulated Luminescence (GLSL), Infra Red Stimulated Luminescence (IRSL), mud brick, annual dose, equivalent dose, Çatalhöyük, Multiple Aliquot Additive Dose (MAAD) technique.

ÖZ

ÇATALHÖYÜK ÖRNEKLERİNİN OPTİK UYARMALI LÜMİNESANS (OSL) TARİHLEMESİ

Akoğlu, K. Göze
Yüksek Lisans, Arkeometri Ana Bilim Dalı
Tez Yöneticisi: Yrd. Doç. Dr. Mustafa Özbakan

Ocak 2003, 55 sayfa

Bu çalışmada Çatalhöyük Neolitik yerleşiminin farklı seviyelerinden alınmış beş kerpiç tuğla örneği üzerinde, içerisinde bulunan feldsparlar ve kuvartzlar kullanılarak OSL tarihlemesinin yapılması amaçlanmıştır.

Çalışmaların başlangıcında cihaz ve metodoloji ile ilgili çalışmalar ve literatür araştırması yapılmıştır. Sistem kalibrasyonları yapıldıktan sonra, 1998 ve 1999 yıllarında ODTÜ, Arkeometri Laboratuvarlarına gönderilen beş kerpiç tuğla örnek tarihlenmiştir.

Örneklerin mineral içerikleri X Işınları Kırınım analizleriyle bulunmuş ve tüm örneklerin feldispar ve kuvartz içerdikleri görülmüştür.

Eşdeğer doz, Çok Örnekli Eklemeli Doz (Multiple Aliquot Additive Dose) yöntemi ile ve uyarma kaynağı olarak hem Kızıl Ötesi (Infra Red) Diyot Düzeneği hem de Filtrelenmiş Işık Kullanılarak tespit edilmiştir. Bu iki uyarma

kaynağı kullanılarak örneklerin Yeşil Işık Uyarmalı Lüminesans (GLSL) ve Kızıl Ötesi (Infra Red) Uyarmalı Lüminesans (IRSL) yaşları bulunmuştur.
Yıllık doz alfa, beta, gama ve kozmik ışıma doz hızlarını içerir. Alfa ve beta doz hızları, uranyum, toryum ve potasyum miktarlarıyla ölçülür. Uranyum ve toryumun yıllık doza katkısı alfa parçacık sayım yöntemiyle, potasyum katkısı Atomik Emisyon spektrometrisi ile bulunmuştur. Yıllık doza kozmik ışımanın etkisi ise kazı alanına gömülen ve 8 ay süre ile orada bırakılan Termolüminesans Dozimetre (TLD) diskleri ile bulunmuştur.

Her örnek için IRSL ve GLSL yaşları elde edilen bu verilerin ve OSL sisteminin yazılımı yardımıyla hesaplanmıştır. Beş kerpiç tuğla örneğinin ünite numaraları S4240, S5206, S3010, S2817 ve S3707 dir. Bu örnekler için IRSL yaşları sırasıyla (3.07 ± 0.23) ky, (7.77 ± 0.62) ky, (2.79 ± 0.27) ky, (8.54 ± 0.69) ky, (2.37 ± 0.23) ky ve GLSL yaşları ise (5.68 ± 0.92) ky, (7.14 ± 0.57) ky, (6.34 ± 0.57) ky, (8.45 ± 0.73) ky and (2.80 ± 0.28) ky olarak hesaplanmıştır..

Anahtar Sözcükler: tarihleme, Optik Uyarmalı Luminesans (OSL), Yeşil Işık Uyarmalı Lüminesans (GLSL), Kızılı Ötesi (Infra Red) Uyarmalı Lüminesans (IRSL), kerpiç tuğla, yıllık doz, eş değer doz, Çatalhöyük, Çok Örnekli Eklemeli Doz (MAAD) yöntemi.

ACKNOWLEDGEMENTS

I would like to thank to Prof. Dr. Ay Melek Özer, for her special guidance throughout my archaeometry education and her support.

I am grateful to my supervisor Asst. Prof. Dr. Mustafa Özbakan for his guidance and comprehension.

I also thank to Prof.Dr. Şahinde Demirci for her help in the analysis of potassium and other chemical determinations.

It is a very big pleasure for me to thank to Dr. Wendy Matthews, if I had never met her this work was not started.

I am really thankful to Çatalhöyük Excavation Team and especially to Shahina Farid who helped me during my visits to the site of Çatalhöyük for placing and taking back TLDs.

I also would like to thank to Dr. Esma Buluş-Kırıkkaya with whom we spent hours in the dark rooms of OSL laboratories.

I am grateful to Prof. Dr. Ali Çulfaz from METU Chemistry Engineering Department for his help in XRD analysis and Prof. Dr. Yavuz Ataman from Chemistry Department for his help in AES analysis.

It is also a pleasure for me to thank to Asst. Prof. Dr. Niyazi Meriç from Ankara University Physics Engeneering Department for his help in TL measurements.

I would like to thank to the technicians of METU Chemistry Department, Saliha Pirdoğan and Osman Yassıtaş for their help during the sample preparations for potassium analysis and my thanks also go to the asistants of METU Chemistry Department Süleyman Can and Deniz Karadeniz for their assistance in AES analysis.

I would also like to thank to Prof. Dr. Emine Caner Saltık who was always helpful in the METU Restoration Laboratory where the water content experiments were conducted.

It is a very big pleasure for me to thank to my parents, Birim and Ersin Akoğlu, without their valuable support all this work was not possible.

Special thanks go to my sister Özge and my friends, İrem, Evin, Gökçen and Murat who always shared my excitements and enthusiasms during my study.

TABLE OF CONTENTS

LIST OF FIGURES

FIGURE

LIST OF TABLES

TABLE

CHAPTER 1

INTRODUCTION

There are numerous dating methods, which can establish the time of formation of a wide range of organic and inorganic materials. Among these techniques radiocarbon dating has a very common usage in organic materials. For the direct dating of inorganic materials the methods are relatively few, e.g., luminescence methods for pottery, bricks and sediments etc., is the principal one and of these, optical dating is proving to be increasingly advantageous.

Various materials contain certain amount of radioactive impurities in very low concentration. These radioactive impurities radiate energy and this energy is trapped in the material. When the material is heated or illuminated the trapped energy is released as light. This process is known as luminescence. The luminescence resulted from heat stimulation is called thermoluminescence (TL) while the luminescence resulted from light is called optically stimulated luminescence (OSL). The variants of luminescence phenomena can be seen by naked eye, one of the most common of these is the bioluminescence and can be found as a reference to fireflies in the Chinese literature about three millenia ago (Harvey, 1957). It is also known that the majority of minerals exhibit luminescence.

In the context of luminescence dating, quartz and feldspar are the minerals primarily concerned and comprises the measurement of trapped charge within

the crystals of these minerals. The charges in these traps result from the decay of naturally occuring radioelements such as, potassium-40 , thorium-232 and uranium-238. Thus the luminescence dating is in the class of methods based on the stored energy emitted. For optical dating, the clock-resetting event is the last exposure to sunlight which sediment grains undergo during transport and the process of sedimentation. Optical dating is therefore suitable for the dating of a wide variety of unheated sedimentary deposits laid down within the last nearly 500,000 years. This may include silty and sandy waterlaid sediments, sand dunes, and loess deposits. It may also date certain soil horizons, colluvial deposits, and glacial varves. This technique is not suitable for sediments deposited with little or no exposure to light, such as gravels, turbidity flows, glacial tills, or sub glacially derived massive clays and silts. Because they have a high level of geological luminescence and this exceeds the upper dating limit of OSL.

In this work, experimental studies were carried out on five mud brick samples from a Neolithic site Çatalhöyük East Mound, near Konya collected in 1998 and 1999.

Çatalhöyük was first discovered by James Melaart in the late 1950's and excavated by him between 1961 and 1965. The site rapidly became famous internationally due to the size and dense occupation of the settlement, as well as the spectacular wall paintings and other arts that were uncovered inside the houses. Since 1993, an international team of archaeologists, led by Ian Hodder, has been carrying out new excavations and research.

Although first dating studies had been done in Melaart's time, dating studies has been still carried out by various scientists. Series of radiocarbon dating has been done before (Newton and Kuniholm, 1999, Cessford, 2001), lately Accelarator Mass spectrometry (AMS) applied to radiocarbon dating was used on charred seed samples of Çatalhöyük East Mound (Göktürk *et al.*, 2002),

Furthermore, a pilot study on OSL has been carried out by Parish (1996) for East Mound on two mud bricks from different levels (level XII, level III/IV) of Çatalhöyük.

Mud bricks are suitable for luminescence dating since they contain feldspars and quartz and their production process involves direct exposure to light. The main aim of this study is to perform OSL dating of mud brick samples from different levels of the Neolithic site, Çatalhöyük, using both feldspars and quartz in them. The OSL dating of mud bricks represents a new approach in Anatolian archaeology since it provides dating of the building materials directly.

In Chapter 1 a brief introduction about previous studies on dating of Çatalhöyük samples and OSL studies is given; in Chapter 2, principles of OSL dating are stated; in Chapter 3, experimental methods and measurements are explained, in Chapter 4, age results are given an discussed; in Chapter 5, the conclusions of the study is given.

CHAPTER 2

PRINCIPLES OF OSL AND OSL DATING

2.1 OSL Phenomena

Luminescence is the emission of light from non-conducting solids in addition to their black body radiation. It is caused by the stimulation of trapped electrons from metastable energy levels, which are related to their subsequent recombination under photon-emission. Luminescence dating exploits the fact that ionising radiation from natural radioactivity and cosmic rays produce electrons, which are partly stored in the crystal lattice. Since these charges accumulate with time, their amount and thus the intensity of the luminescence signal can be used for dating (Lang and Wagner, 1996).

Luminescence dating techniques, i.e. TL and OSL, have been widely used for dating a wide range of materials such as, pottery, bricks and sediments etc. In both techniques, luminescence emitted by constituent mineral grains, which is the dating signal, is measured by a highly sensitive photomultiplier tube. However, in thermoluminescence the dating signal is obtained by heating the sample while for optical dating the signal is obtained by shining a beam of light onto it (Aitken, 1998).

Dating of archaeological materials depends on the fact that, when mineral grains are isolated from daylight by burial, they begin to accumulate electrons in their traps. These electrons result from exposure to the ionising radiation emitted by the decay of naturally occurring radioisotopes K-40, Th-232 and U-

238. If the flux of ionising radiation is constant, then the burial time of the grains can simply be determined by dividing the total dose (burial dose, equivalent dose or paleodose) which have been accumulated during burial to the Dose-Rate (annual dose) i.e.,

Burial Time = Burial Dose / Dose-Rate (2.1)

or it can also be given as,

Age (years) = Equivalent Dose (Gy) / Annual Dose (Gy/year) (2.2)

The dose rate represents the yearly rate at which energy is absorbed from the flux of nuclear radiation provided from thorium, uranium and potassium-40 in the material, as well as by cosmic rays. The annual dose is assumed to be constant and evaluated by assessment of the radioactivity of the sediment carried out both in the laboratory and on-site by using dosimeters.

Although the process is much more complex the features of OSL dating can be studied in terms of a simple model, as shown in Figure 2.1. An ionic crystal is given in this figure to show some defects in crystals. The defects in the mineral crystals i.e, negative ion vacancy, negative-ion interstitial, and substitutional impurity centers behave as the trap for electrons. The ionizing radiation results from the decay of the naturally occuring radioisotopes interacts with a crystalline substance, freeing electrons from their normal atomic sites. Some of these electrons become trapped at defect sites and, if the trap depth is large enough, they will remain there indefinitely on a geological time scale. The number of these electrons is thus a measure of the radiation dose since some event in which all the traps were emptied. It is assumed that electrons caught in traps stay in there indefinitely. In fact, the lifetime of an electron in a trap is not infinite and has a value which depends on the type of the trap (Aitken, 1989).

Energy Level Diagram, Figure 2.2, is a convenient basis to discuss the luminescence mechanism. In this model a trap, T, is characterised by the energy E which an electron must acquire from the lattice vibrations in order to escape from it and diffuse around the crystal which can be described as being

in the conduction band. By heating or shining a light onto the sample, electrons are removed from their traps and some of these reach to the luminescence centers, L, resulting in emission of light. If the process is done by heating it is called thermoluminescence (TL) if the shining of light is used it is called as Optically Stimulated Luminescence(OSL).

The main difference between thermoluminescence and Optically Stimulated Luminescence is the stimulating source. Although the mechanisms are the same for both tecniques there are some advantages of OSL over TL . These can be summarised as,

- OSL is measured near or at room temperature hence it is less destructive and more sensitive method than TL.
- Parts of OSL signal can be measured many times on same sample however in TL this is impossible since the measurement involves the total erasure of the signal. Therefore, for normalization of aliquots short shines of OSL can be used.
- After measurement of OSL a TL signal can be measured on the same sample however the reverse is impossible.
- OSL measures the electrons held in traps, which are the most sensitive to light and is thus particularly important in dating geological sediment samples, which had been zeroed in the past by sun bleaching. Furthermore, in many cases, OSL has the same dose response as TL. (Botter-Jensen, 2000).

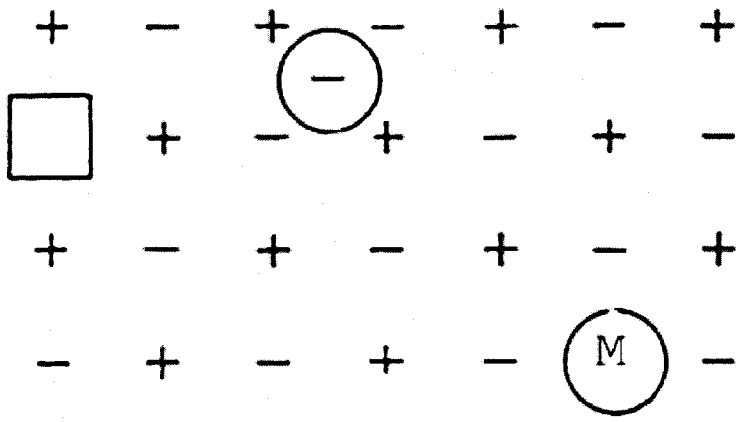
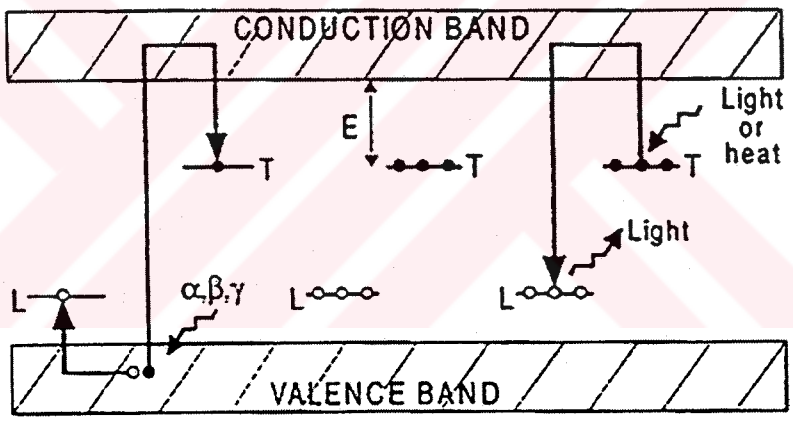


Figure 2.1 Simple types of defects in the lattice structure of an ionic crystal. From left to right: negative-ion vacancy, negative-ion interstitial, substitutional impurity center. Here 'M' refers to any suitable '+' ion. (Aitken, 1985).



(i) ionization **(ii) storage** **(iii) eviction**

Figure2.2 Energy-level representation of TL and OSL processes (based on Aitken 1998). (i) Ionization due to the exposure to nuclear radiation with trapping of electrons and holes at defects T and L, respectively. (ii) Storage of radiation energy during time; if leakage is negligible the lifetime of the electrons in the traps needs to be much longer than the storage time of the sample. This lifetime is dependent on the energy depth E of the trap below the conduction band. (iii) By heating or shining a light onto the sample, electrons are evictid from the electron traps and some of these reach luminescence centers (L); if so light (i.e. TL or OSL) is emitted as a result of the processes of recombining into these centres (Botter-Jensen, 2000).

2.2 Materials Studied By OSL

When dating with OSL quartz and feldspar in the sample are concerned as primary subjects since they are the light-sensitive minerals. Secondly, the effect of radiation from uranium, and other radioactive impurities in the sample itself and its surroundings is concerned.

2.3 Basic Principles of OSL Dating

The natural OSL is mainly the result of exposure to the nuclear radiations from naturally occuring radioisotopes, potassium, thorium and uranium; commonly these contribute in roughly enough equal portions but there can be strong variations from site to site.

The radiation types from the naturally occuring radioelements relevant to dating are alpha particles, beta particles and gamma rays including those from cosmic rays are involved. Alpha, beta particles and gamma rays produce secondary electrons. When these electrons have been slowed down enough the ionization occurs, with consequent trapping of ionized electrons at defects in the crystal lattice.

Radioactive isotope of potassium is the potassium-40 with an atomic abundance of close to 0.01 per cent in natural potassium. It emits both beta particles and gamma rays. In addition to these, thorium and uranium emit alpha particles.

The rate at which trapped electrons are accumulated is proportional to the rate at which energy is absorbed by a sample from flux of radiation. Thus it is possible to evaluate the annual dose by determining the concentration of radioelements in a sample.

In order to determine annual dose the alpha particles from uranium and thorium and potassium content of the sample must be known. In addition to these the cosmic ray contribution to annual dose must be determined. The annual dose given in equation 2.3,

$$\text{Annual Dose} = kD_{\alpha} + D_{\beta} + D_{\gamma} + D_{c} \qquad (2.3)$$

Where,

kD_{α} is effective alpha contribution,

D_{β} is beta dose-rate,

D_{γ} is gamma dose-rate,

D_{c} are cosmic dose-rate (Aitken, 1998).

In evaluating the equivalent dose, the natural OSL from sample is compared with OSL of same or similar sample that is irradiated with a calibrated radioisotope source. There are varios techniques to evaluate the equivalent dose. In this study Multiple Aliquots Additive Dose (MAAD) Technique is used. In this technique a number of equal portions (aliquots) of the sample are prepared and divided into groups. One group is reserved for measurement of the natural OSL and the other groups are given various doses of laboratory radiation before measurement, In this process members of each group exposed to the same dose. Before measurement, all groups, including those used for measurement of the natural radiation, are normally subjected to preheating (Aitken, 1998).

In Figure 2.3, a typical growth of OSL with dose has been shown as linear. A straight line is a good fit to the data points. The equivalent dose (Paleodose), P, is read off as the intercept on the dose axis.

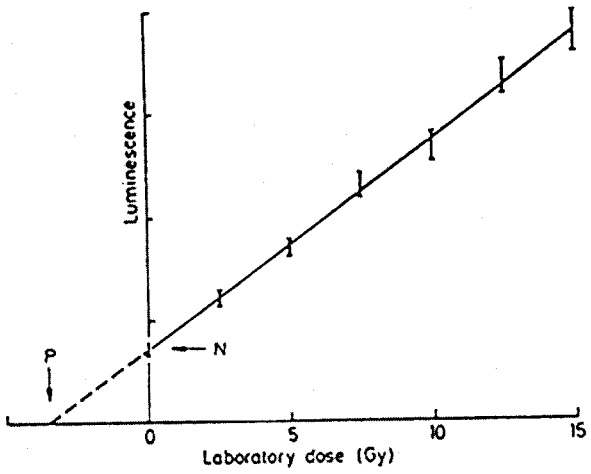


Figure 2.3 Each data point is the average OSL from a group of aliquots, all members having been given the same laboratoy dose (except for the lowest point, N, at which the laboratory dose is zero). In the case shown the growth is linear with dose, i.e. a straight line is a good fit to the data points; the paleodose (equivalent dose), P, is read off as the intercept on the dose axis (Aitken, 1998).

The water content of the sample plays an important role on the absorption of the radiation. If the moisture is high more radiation is absorbed and therefore, if it is ignored, appreciable underestimation of age occurs.

There are two main stimulation methods currently being used for routine OSL measurements. They are Infra-Red Stimulated Luminescence (IRSL) which is useful only for feldspars and visible light stimulated luminescence which works for both with quartz and feldspars. Since this was first observed using green light it is referred as Green Light Stimulated Luminescence (GLSL), Figure 2.4. In both methods it is vital to avoid the excitation light source affecting the PM tube. This is achieved by using suitable filters.

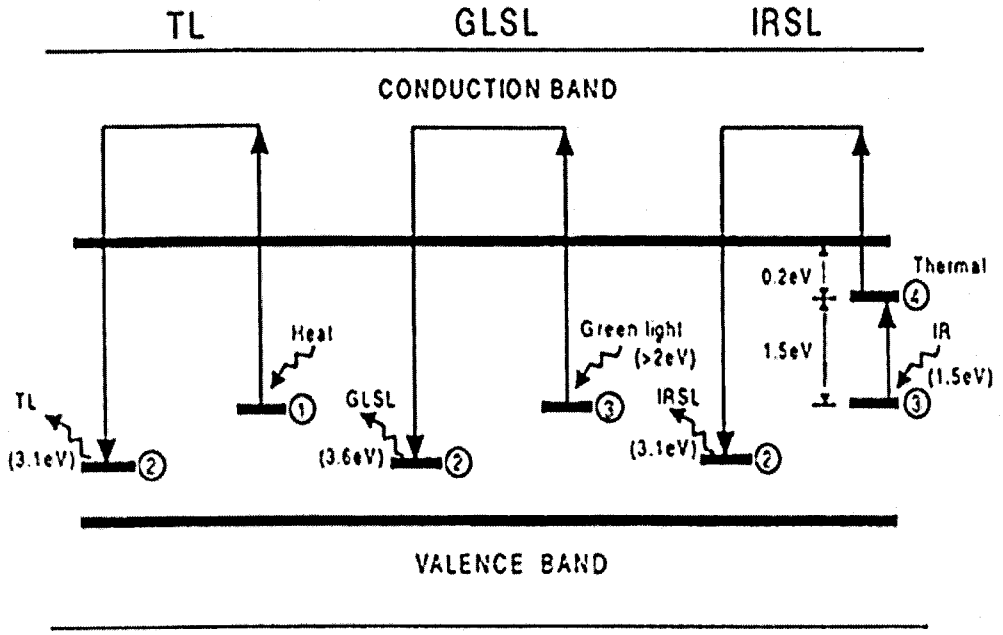


Figure 2.4 *1. TL Trap, 2. Radiative recombination center, 3. GLSL/IRSL trap, 4. Excited state.* Schematic energy diagrams for TL, green light stimulated luminescence and infrared stimulated luminescence. The latter process is based on the Hütt *et al.* (1988) model with the assumption that infrared photons raise electrons from the ground state into an intermediate excited state from where they are raised further by thermal effect into the conduction band i.e. by lattice vibrations (Botter-Jensen, 2000)

The primary source of electrons for the OSL signal has been reported to be from the trap that corresponds to 325^{O}C TL peak (Smith *et al.*, 1986 and Spooner *et al.*, 1988).

The depth of the trap responsible for the 325^{O}C TL peak, as measured by Wintle (1975), is 1.7 ± 0.1eV and by Rhodes (1990) and Smith *et al.* (1990) is 1.84 ± 0.07eV using isothermal decay and 1.69 ± 0.02eV using the peak shift with temperature method; the estimated lifetime at 20^{O}C is ~30 My, suggesting that the OSL signal associated with this peak should have adequate stability for Quaternary (which also includes archaeological times) dating.

The above discussion gives the considerable optimism for the success of dating; it also indicates that the eviction by photons is more complex than a simple excitation into the conduction band. This was pointed out by Goddfrey-Smith *et al* (1988) in commenting on their observation that OSL could be

stimulated from 10 ka undosed quartz sample by combined (799 + 753) nm krypton laser light. Taking the ratio of *E* (optical) to *E* (thermal) to be ~1.8 (the ratio of static and optical dielectric constants), it is to be expected that the eviction from a trap having *E* (thermal) = 1.7eV will require *E* (optical) = 3.06eV, i.e. a wavelength not longer than 405 nm; on this basis eviction from the 325^{O}C TL trap cannot be expected using 514 nm light let alone 753 nm light. There are two further indications that the mechanism is complex: first the selective bleaching of the 325^{O}C TL peak already noted, and secondly the strong temperature dependence, of ~1% / ^{O}C, observed for the rate of eviction. Godfrey-Smith *et al* (1988) suggest a complex, multi-step, multi-photon process, and note as an alternative the thermal assistance model suggested by Hütt *et al.* (1988) for feldspar.

For a given number of electrons evicted from the trap probability of photon emission is about 100 times greater for OSL (at 20^{O}C) than for TL. This means that it may be possible to obtain bright OSL from a sample despite its 325^{O}C TL peak being barely discernible. TL glow-curves of feldspars do not indicate selective bleaching in any particular region, which is unlikely in quartz (Smith *et al.*, 1986; Singhvi and Wagner, 1986; Li and Aitken, 1989). Some types of K-feldspars are even more remarkable that quartz in respect of the low value of photon energy necessary to stimulate luminescence. Thus at the 1987 Cambridge seminar, Hutt *et al.* (1988) reported a stable dating signal using 870 nm infrared, at the same time proposing the model just mentioned; the stimulation spectra showed a broad peak centred on that wavelength plus another centred on 550 nm. As with quartz the thermal assistance involved in the eviction process gives rise to a strong dependence of eviction rate on sample temperature.

2.3.1 Historical Development of OSL as A Method of Dating

The initial work of Huntley *et al.* (1985) showed the potential of OSL in dating applications. In this work Huntley *et al.* used the green light (514 nm) from an argon laser to stimulate luminescence from quartz for dating sediments. The

OSL properties of quartz have been used in dating applications and dosimetry studies intensively afterwards (Aitken and Smith, 1988; Aitken, 1990; Godfrey-Smith *et.al.*, 1988; Rhodes, 1988).

In 1988, Hütt *et.al.* discovered that infrared (IR) light could also be used for stimulation of luminescence in feldspars and subsequently Poolton and Bailiff (1989), Spooner et.al. (1990), and Botter-Jensen et.al. (1991) constructed units for stimulation based on systems of small IR light emitting diodes (LEDs). Broad-band emitters such as incandescent or arc lamps, in conjunction with selected filters, have also been used to produce both infrared and visible light stimulated luminescence from feldspars and quartz samples (Botter-Jensen, 2000).

A simple diagram of OSL system is given in Figure 2.5, with the main components only.

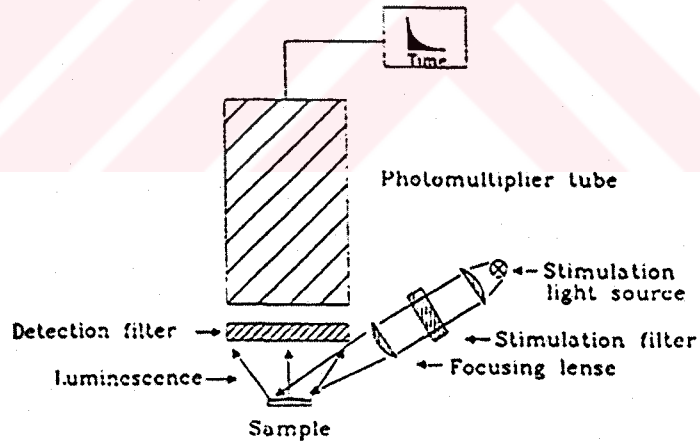


Figure 2.5 Simple diagram of an OSL system with stimulation light source, photomultiplier detector and readout electronics.

CHAPTER 3

EXPERIMENTAL METHODS

This chapter outlines the OSL dating and Alpha counting systems, their calibrations and sample preparations and measurements for age calculations.

3.1 OSL Dating System

In this work ELSEC 9010 Optical Dating System which is shown in, Figure 3.1, was used. The OSL measurements were conducted in the Archaeometry Research laboratories of METU Physics Department.

Figure 3.1 ELSEC 9010 Optical Dating System.

The parts of 9010 Optical dating system are:

1. **Control Computer:** An IBM-PC compatible with 80386 processor, 40 MB hard disc and a monitor (this is the very minimum configuration).

2. **Computer Interface Cards:** 2cards are required that plug into the computer ISA (standard IBM PC compatible) bus: Stepper driver and Counter card.

3. **Interface module:** This contains high and low voltage power supplies, control circuits for the IR LEDs, displays for LED current, sample temperature, IR intensity.
4. **Sample tray:** A black anodised aluminium tray (Figure 3.2) with 64 machined positions for standard (9.7 mm diameter) sample discs. A light-tight, screw-down lid fitted with a viton rubber o-ring provided for safe sample transport.

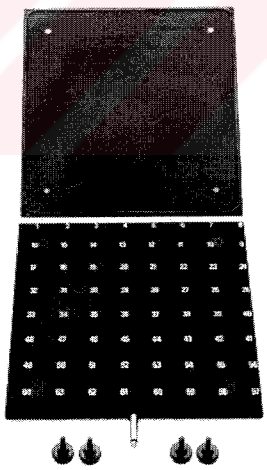


Figure 3.2 Sample Tray.

5. **Sample handling module:** An X-Y stepper-motor positioning unit takes the sample tray and positions samples under the photomultiplier on command from the control computer. It is mounted in a heavy-duty light

tight box. The sample is held on a heavy copper plate that is temperature controlled by Peltier devices at 22 ± 0.5 ^{O}C. This generates waste heat that needs to be removed with water-cooling. The water is connected to the back of the sample-positioning unit.

6. **Sample Illumination Assembly:**

   Sample illumination assembly contains two modules.

   A. IR LED Module:

   24 IR LEDs are mounted in machined aluminium block for accurate alignment and temperature stability. A photodiode is fitted for closed loop IR intensity control or LEDs can be operated in a constant current mode. Even illumination of the sample is ensured with a plastic diffuser. A fused silica light guide transmits the emitted luminescence to the photomultiplier.

   B. Filtered Lamp Module:

   The light from a 150 W tungsten-halogen lamp is optically filtered to provide light at any chosen wavelength. A high quality lens system with forced air cooling focuses light evenly onto the sample. This module has been designed particularly to provide light at 514 nm for use with quartz. An easily replaced filter cartridge uses 44 mm diameter glass filters and 25 mm interface filters. A high-speed mechanical shutter is used to accurately control the stimulation time (Figure 3.3).

7. **Photomultiplier assembly:** A low-noise photomultiplier in a light tight housing. Built in preamplifier with user adjustable discriminator. Provision for the use of optical filters (essential to reject stimulating light).

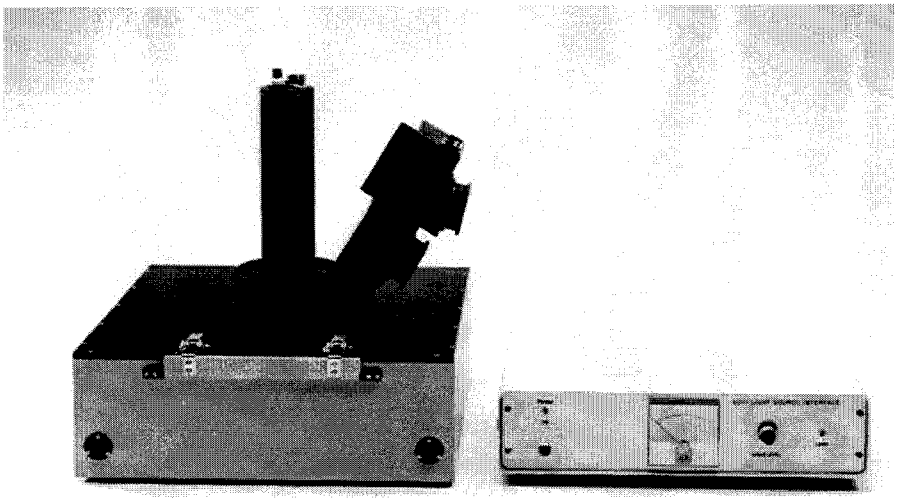


Figure 3.3 ELSEC 9010 Optical Dating System Filtered Lamp Module and Interface Unit.

8. **Beta Irradiator unit:** Sr-90 Beta source, shown in Figure 3.4, installed the system.

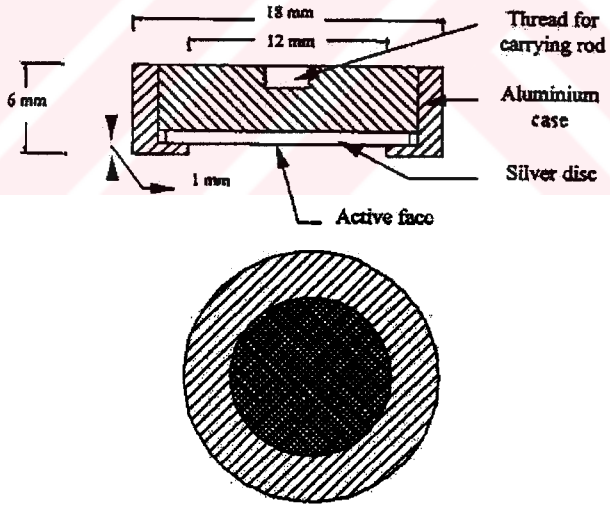


Figure 3.4 Strontium-90 beta source. Upper part shows section and lower part shows the front face. The radioisotope compound is incorporated into the front surface of the silver disc in a very thin layer (about 2mm for a 1.5 GBq source) there is then a 0.1 mm screen of silver on which is a protective coating of gold or palladium.

3.2 Alpha Counting System

The 7286 Low Level Alpha Counting System has been developed for studies where the counting rate is small which is also applicable to luminescence dating where the counting rate is also small. Up to four scintillation-photomultiplier assemblies can be controlled from the one printout control unit.

There is an adjustable high voltage supply and discriminator unit for each photomultiplier tube. The output pulses from the discriminator circuit are fed into an internal microprocessor, which keeps track of the total number of fast pairs, which occur within 4 msec, and slow pairs, which occur between 20 and 400 msec apart. If the user specifies it, the uranium and thorium contents can be estimated by using fast and slow pairs counting. ELSEC 7286 Low Level Alpha Counter is shown in Figure 3.5.

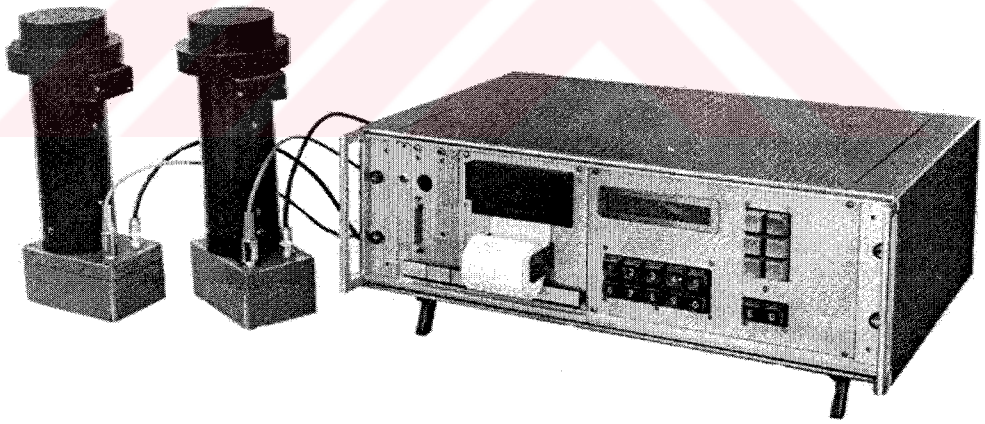


Figure 3.5 ELSEC 7286 Low Level Alpha Counter system.

In this technique a powdered layer of sample, thick compared with the 50 μm range of the most energetic alpha particles from thorium and uranium, is placed on top of a scintillation screen produces a scintillation, and this produces photoelectrons from the photocathode, after amplification, become an electrical

pulse at the anode of the photomultiplier. Then this pulse is registered on a counting device.

The scintillation screen is made by sprinkling zinc sulphide onto cello tape and the powdered sample is placed in direct contact with a suitable detergent or methanol to avoid errors coming from the previous usage. The advantage of zinc sulphide is that the pulses corresponding to the scintillation produced by alpha particles are much larger than those produced by beta particles and gamma radiation. This enables easy rejection of the latter in the electronics.

The sample plus photomultiplier must be enclosed in a dark box because of the photomultiplier's sensitivity to light.

For a screen diameter of 42 mm counts are received from the lower 0.2 g of the sample, about a quarter of the alpha particles originating in this amount actually reaching the screen. However, because of the difficulty of spreading evenly it is not practical to use less than about one gram of sample for this size of screen. The electronic layout of the alpha counting system is shown in Figure 3.6.

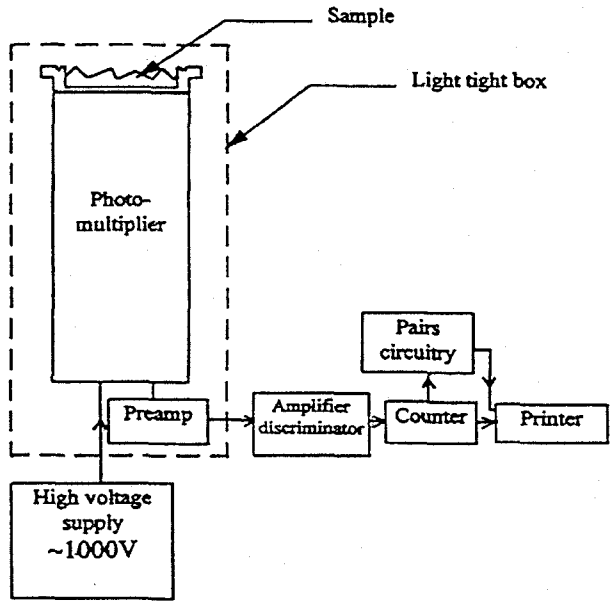


Figure 3.6 Alpha counting: electronic layout.

3.3 Çatalhöyük Site and Samples

Çatalhöyük is a tell settlement in the "Konya Plain" in Central Anatolia, Turkey. Although local people recognized it for centuries, it was "discovered" as an archaeological site in 1958 by a British team headed by James Mellaart and David French. The general view of the site is given in Figure 3.7.

There are in fact two mounds of accumulated debris from the Neolithic and Chalcolithic periods, the East and West Mounds. The "East Mound" - 16 hectares in area and rising to a maximum height of ca. 14 meters above the present level of the plain - was settled in the Early Neolithic.

The West Mound belongs to the Early Chalcolithic period, during which metal was known but stone was still the main material used for tools as it was in the neolithic settlement at Çatalhöyük East Mound. It was probably inhabited

about 7,500-8,000 years ago. Very little is known about this period in the Konya Plain, and therefore excavation is now being carried out at the West Mound in order to look at continuity and change between the two settlements.

Figure 3.7 View of Çatalhöyük from east-west direction from balloon.

In 1993 the Turkish Ministry of Culture granted permission for Ian Hodder, then of Cambridge University, UK, and now of Stanford University, USA to renew research at the site. Again with the sponsorship of the BIAA (British Institute of Archaeology at Ankara) he directs the Çatalhöyük Research Project, which is administered through the MacDonald Institute of Archaeology, University of Cambridge, UK. They maintain the facilities at the site where the combined teams live and work.

During 1993-94, the "Cambridge team" made an accurate detailed topographic map of the East mound. They also scraped the surface of a 40 x 40 meter area in the North part of the East mound, where they were able to draw plans of Neolithic architectural features at the surface of the mound. From 1995 until the present the "Cambridge team" has been excavating in both this area (referred to as North) and the area originally opened by Mellaart (referred to now as South).

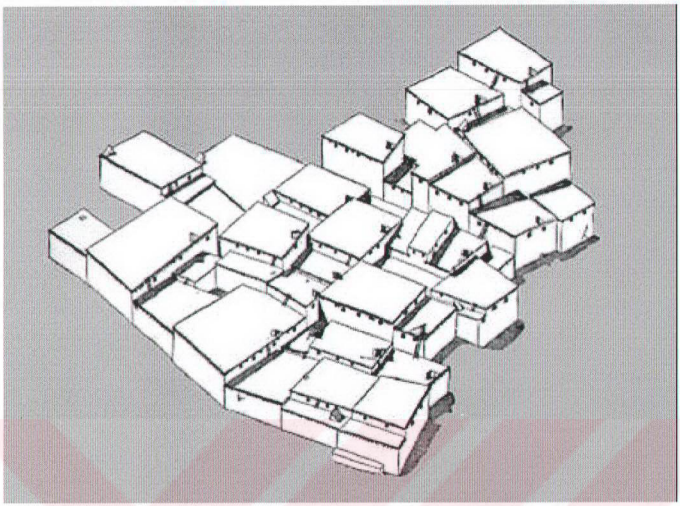


Figure 3.8 Schematic reconstruction of a section of Level VI with houses and shrines rising in terraces one above each other. (Melaart, 1967).

Çatalhöyük was phased by levels of buildings by Melaart(1967). He also discovered 15 Neolithic levels numbered 0 (latest) to XIII (earliest), with two sub levels at VI level as VIA and VIB.

The buildings in the settlement were built of sun-dried mud brick, plaster, wood beams, and woven mats. Excavations today reveal a plan of rectangular houses built in a labyrinth-like arrangement. Each house has its own separate walls but is built next to each other, Figure 3.8.

Five mud brick samples from different levels were collected and sent to the METU (Middle East Technical University), the Archaeometry Program of the Graduate school of Natural and Applied Sciences in 1998 and 1999. The collection of suitable samples must be done under very strictly controlled conditions during which the sample is exposed to no light, since the OSL signal is extremely sensitive to light.

Table 3.1 Description of samples. For detailed information please see the web page at http: catal.arch.cam.ac.uk/catal/database/excavation.html

SAMPLE S4240		**SAMPLE S5206**	
Mound: East		Mound: East	
Year: 1999		Year: 1999	
Area: South	Level:VII	Area: South	Level:IX
Feature: Wall 74		Feature: 554	
Space: 159		Space: 182	
Interpretive Category: Brick		Interpretive Category: Brick	
SAMPLE N3010		**SAMPLE S3707**	
Mound: East		Mound: East	
Year:1998		Year: 1998	
Area: North	Level: VI-V	Area: South	Level: VII
Feature: 4		Feature: 253	
Space:71/159/187		Space:	
Interpretive Category: Brick		Interpretive Category: Brick	
SAMPLE S2817			
Mound: East			
Year: 1997			
Area: South	Level: VII Phase: 1		
Feature: 52			
Space: 107/108			
Interpretive Category: Bricks in wall			

3.4 Sample Preparations

To conduct the measurements, samples were prepared by the methods explained in below sections. Mineralogical compositions of samples have been found before sample preparations.

3.4.1 X- Ray Diffraction (XRD) Analysis

In order to determine the mineral compositions of mud brick samples XRD analysis has been used. The analysis has been conducted on powdered samples

which had been dried for one night in oven at 105 ^{O}C for 16 hours. The Philips type PW 1353/20 X-Ray Diffractometer adjusted to 35 kV and 14 mA was used to determine the mineral compositions. The analysis was performed by using CoK_{α} with a Ni filter. In the analysis 2θ values were changed from 6^{O} to 75^{O}. The main minerals identified are quartz, feldspars and calcite dominantly. In Figure 3.9 XRD trace of S4240 is given as an example. The differences between mud bricks regarding mineral composition were negligible.

3.4.2 Sample Preparations for Equivalent Dose Determinations

In the laboratory, under the red light, the plastic covers around the samples were removed. The surfaces of mud bricks, which may have been exposed to light during their collection, were carefully scraped off. This scraping off the outer level had helped to eliminate the soil contamination that may lead to the additional beta dosage and the reduced level of luminescence in the outer surface because of the effect of sunlight. Samples are grounded in a mortar and sieved to a grain size <90 μm.

The Equivalent Dose (ED) was determined by the MAAD technique explained in Chapter 2, where the laboratory doses added to portions of the natural sample and the growth is extrapolated to zero luminescence signal to give the ED, Figure 2.3 (Rees-Jones and Tite, 1997). The amount of luminescence emitted by each portion is then measured using the 9010 reader, and the luminescence signal plotted against added dose.

When a curve is fitted to these data, the intercept with the dose = 0 axis gives the equivalent dose. For each dose value three discs as minimum are prepared and at least 7 dose points have been administered to sample including zero added dose. This implies that a sample to be dated needed to be divided into at least 21 aliquots.

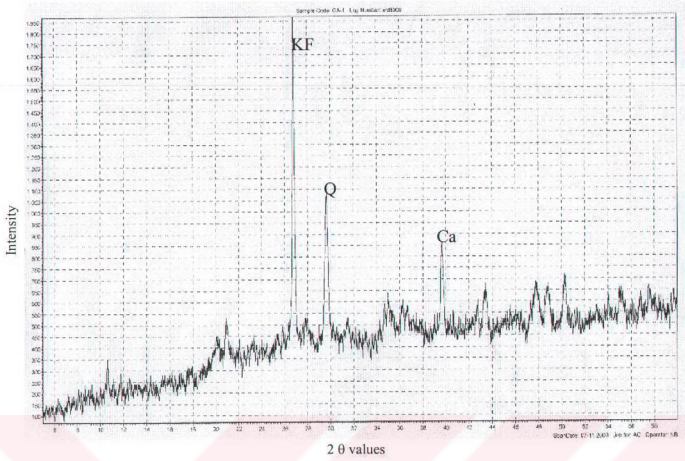


Figure 3.9 XRD trace of sample S4240. Where, KF is K-Feldspar, Q is Quartz, Ca is Calcite.

Sample Preparation for Feldspars

Samples for feldspar dating were prepared by following standard thermoluminescence dating procedures as described by Aitken (1985). Samples were treated with hydrochloric acid, HCl, 10%, v/v and hydrogen peroxide, H_2O_2, 38%, w/w to remove any organic matter and calcium carbonate then thoroughly rinsed with distilled water and acetone at least five times in order to remove all traces of chemicals from the previous treatments. Samples were re-sieved to <90 μm and then placed on aluminium discs that already have acetone film, the reason for adding acetone on discs is to make homogeneous distribution of powdered samples on the discs.

Sample Preparation for Quartz

Samples for quartz dating were prepared by following the procedure sample preparation procedures as described in Aitken (1985) with a few modifications. The procedure begins as the same steps with feldspar preparation. After the HCl and H_2O_2 treatment, samples were treated with hydrofluoric acid, HF,40%, w/w for 1 hr, then samples were left in fluoslicic acid (H_2SiF_6) for 12 hrs (Stokes, 1992), to remove chemicals from the previous treatments, samples were thoroughly washed with distilled water and acetone at least five times. Then sodium polytungstate solution with 2.78 g/cm^3 was prepared which is found to be quite enough to float quartz particles. Then heavy liquid separation with sodium polytungstate had been done and quartz particles collected with syringe and final mixture was washed through Whatman 40 filter paper then samples were dried. Finally they were re-sieved to <90 μm again and then placed on aluminium discs that already have acetone film, the reason for adding acetone on discs is to make homogeneous distribution of powdered samples on the discs.

3.4.3 Sample Preparations for Annual Dose Determinations

In order to determine the annual dose, alpha counting to determine the alpha particles resulting from the decay of U and Th, analysis by AES (Atomic Emission Spectrometry), to determine the potassium amount in samples and TLD discs placement on site were preferred. To find out the water effect on the dose-rate water take up evaluations were also conducted.

3.4.3.1 Sample Preparation for Potassium Analysis

To determine the potassium content of the samples AES (Atomic Emission Spectrometry) technique has been used. The full procedure of the sample

A 0.1 g sample of finely grounded soil was placed in a 30 mL. platinum crucible. The soil was wetted with a few drops of water, and 5 mL. of HF,48 %, W/W and 0.5 mL of $HClO_4$ to70 %,W/W were added then soil acid mixture was heated on a hot plate until fumes of $HClO_4$ appear, the crucible was cooled, then 5 mL of HF was added. Tthe crucible was placed in a sand bath, and covered about the nine-tenths of the crucible topped with a platinum lid. Heat the crucible at 220 - 225 °C, evaporate contents to dryness. The crucible was cooled, and 2 mL of water and a few drops of $HClO_4$ were added. The crucible was replaced in the sand bath, and the contents were evaporated to dryness. If organic matter stains are still present on the sides or lid of the crucible, the flame of a Meker burner directed onto the sides and lid until the organic matter is oxidized. A faint red heat is sufficient. The crucible was removed; and, when it is cool, 5mL of 6 M HCl and about 5mL of H_2O were added. The crucible was heated on a hot plate or over a burner until the solution boils gently. If the sample does not dissolve completely, the solution was evaporated to dryness, and the procedure was repeated, starting with 5mL of HF and 0.5 mL of $HClO_4$. When the residue completely dissolves in HCl, the sample was transfered to a 50 mL volumetric flask. The filter paper washed with water, and the solution was diluted to volume. Filtration is desirable to keep the solution free of solid particles which cause clogging of burner capillaries of flame photometers or Atomic Emission Spectrometers (Black, 1965).

Then the sample solutions in volumetric flasks were used in the determination of pottasium by the Atomic Emission Spectrometry with the device of Perkin-Elmer 305B Atomic Emission Spectrometer in Chemistry Department, METU.

3.4.3.2 Preparation of TLD Discs

To determine the gamma and cosmic ray component of the annual dose TLD (Thermolumuminescence Dosimeter) discs were used. Six Al_2O_3:C (carbon doped aluminum oxide) discs were placed at 30 cm depth and kept for 8 months at the site where the samples were taken. Before placements of discs at the site each had been left in Carbolite 1100 furnace for 1 hour at 400^0C. Then each had been covered with tin aluminium foil and plastic covers then put into black plastic box separately. After this preparation, the boxes had been placed at the site as mentioned above, within 24 hours at 24^{th} October 2001. 243 days later at 4^{th} July 2001 the discs had been taken out to the Ankara University Physics Engineering laboratories and measured in Harshaw 3500 TL device.

For each TLD discs the dose responses have been found by using the calibration graphs given in Figure 3.9. The average of the results, which is found as 0.155 mGy/y, is taken as the annual dose for the region where the dosimeters have been placed. When the effect of water to this amount is calculated according to formula below and found to be 0.146 mGy/y.

$$D_\gamma = D_{\gamma dry} / (1 + 1.14\ WF) \qquad (3.1)$$

Where the WF is the water correction factor for γ rays for Al_2O_3: C and has a value 0.54, $D\gamma$ is the dose-rate for gamma rays for Al_2O_3: C discs, $D\gamma_{dry}$ is the dose-rate for dry Al_2O_3:C discs.

3.4.3.3 Experimental Procedure For Water Uptake Measurements

Since the water content of the sample plays an important role om the absorption of the radiation, the water uptake measurements were conducted. First, five porcelain crucibles had been left for 30 minutes at 200°C then waited for several minutes for cooling and weighted; this was repeated to attain constant weight. Samples were put in the crucibles and weighed. The crucibles kept in desiccator. As the fourth step the samples were wetted with distilled water and heated at low temperature until they are truly wetted. The crucibles

with wet samples were weighed again. Lastly the samples were heated at 200^0 C to dryness and weighed again and the formula below has been used to calculate the water uptake during burial.

$$W = (\text{Saturation Weight} - \text{Dry Weight}) / \text{Dry Weight} \quad (3.2)$$

Where W, is the saturation water content.

Water Uptake During Burial is considered to be as the two thirds of the saturation water content (Aitken , 1985).

3.5 Calibrations And Measurements

Calibrations involve optical dating and alpha counter calibrations mainly.

3.5.1 Calibration of Optical Dating System

Calibration of the system consists of setting High Tension and Threshold voltage of PM tube and Beta Source calibration.

3.5.1.1 Setting High Tension (HT)

The sensitivity of the photomultiplier depends on the voltage applied to it, in general, larger the voltage the greater the sensitivity is. If the voltage is too high the background noise level is increased. As with threshold setting, the calibration light source (C-14 doped phosphorus) provided should be positioned under the PM tube to provide a stable light source. The threshold control potentiometer at the top of the PM housing should be set to a value about 3 or 4 during this measurement.

A series of measurements of the apparent intensity of the light source should be taken for various HT voltages (from 700V to 1400V). The correct HT voltage is found by using the above procedure (1260 ± 25) V, should be set on the front control panel.

3.5.1.2 Setting Threshold

The threshold value should be set to a level, which eliminates as much noise as possible, without also loosing the smaller true pulses. The calibration light source should be inserted in the same way as described above for setting HT voltage. The correct HT voltage should be set on the front panel control.

A series of readings should be taken for various threshold settings between 2.5 and 10.0 using the same shine as described above for setting HT voltage. When all readings have been taken, a graph is drawn of count rate against threshold setting. Then threshold setting is calculated, according to 85% rule. The value obtained is (2.75 ± 0.25) V.

3.5.1.3 Calibration of Beta Source

Since the source was out of usage for 4 years there was a problem occurred in the computerized control unit because of the nature of liquid strontium, also the test date was not known when the experiment started hence, the activity of the source had been found also experimentally.

In order to find the activity of the source experimentally Al_2O_3: C discs were used each of which have been dosed to 14.2 mGy and submitted by GSF (Forshüngenszentrum) Institute in Germany. For dose determination Harshaw 3500 Thermoluminescence equipment of Ankara University Physics Engineering Department was used. TL signals were read for the discs dosed to 14.2 mGy. Then the discs were dosed with the Sr-90 Beta source to 56 mGy. TL signals were taken again and found as 35.71 μC which is the charge amount when the discs are dosed to 39.375 mGy. Then the reduction in the dose amount delivered by Sr-90 beta source has been found and substracted from the first dose amount (0.055 Gy/sec) and found as 0.0386 Gy/sec. TL signals for different dose amount are tabulated in Table 3.2.

Table 3.2 Dose response of Al_2O_3: C discs which had been irradiated with a known activity source.

TLD Discs	Dose Response (μC) for 14.2mGy	Dose Response (μC) for 56mGy
1	12.78	35.04
2	13.35	35.12
3	10.63	33.50
4	14.33	37.99
AVERAGE	12.77	35.41

3.5.2 Calibration of Alpha Counter

Since every photomultiplier tube has different characteristics, the 7286 Low Level Alpha Counter has to be calibrated for a given tube. This involves adjusting the HT voltage and threshold on the back panel.

First threshold control on the back panel was set to 0.25 V. Then plot of counts versus voltage was obtained by using a source strong enough to provide about 10 counts per second (e.g. Monazite sand). It is recommended that this is done by starting at about 1250V and then lower voltages down top 800V in steps of 25 V and with a period length of 10 seconds. After changing the HT voltage counting should not be done for at least 5 minutes to allow the tube recovery time to settle.

Alternatively a good voltage to start with can be obtained by looking at the data supplied with the photomultiplier tube. HT voltage on the flat portion of the counts versus voltage curve obtained above was setted which is 1150 volts. This voltage was setted and a plot using the same source as above was obtained. At low threshold values below (0.25 volts)and above about 1 volt the curve will not be linear but between these values a reasonably straight line

should be obtained. When the line was produced to the Y axis; the value represents the count rate that should be obtained at zero threshold if there were no electrical noise or other imperfections in the system.The count rate at the line was multiplied by the required threshold fraction. This is 0.85 for the thorium series and 0.82 for the uranium series (usually 0.85 is used). The result gives the required threshold setting. With the settings obtained above count the sands provided to check the calibration.

HT voltage for alpha counting system has been found (1150 ± 25) V and threshold has been found as (1.75 ± 0.25) V

3.5.3 Measurements for Dose Determinations

Dose determinations mainly grouped in two categories, i.e, equivalent dose determinations and annual dose determinations.

3.5.3.1 Equivalent Dose Determinations

In this study the dating experiments were carried out using both IRLED module and Filtered Light Lamp Module of the ELSEC 9010 Optical Dating system.

The aim was by comparing the results of these two techniques; to obtain a confident determination of the ages of samples. In the initial studies of quartz, the use of green light (514.5 nm) from an argon laser operated at continuous wave mode demonstrated that the energy of visible light is sufficient to empty traps in this materials (Botter-Jensen, 2000). However, luminescence can be excited in feldspars with wavelengths near infrared (Hütt *et al*, 1988).

3.5.3.1.1 Beta Irradiation and Natural Normalizations

Equivalent dose had been found by using the multiple aliquots additive dose (MAAD) technique. In this method the sample is split into several portions, each of which is given a different radiation dose in addition to that naturally received. The amount of luminescence emitted by each portion is then measured using the 9010 reader, and the luminescence signal is plotted against added dose. When a curve is fitted to these data, the intercept with the dose=0 axis gives the estimated dose. For each dose value minimum of three discs are prepared and at least 7 dose points must be administered to sample including zero added dose. This implies that sample to be dated needs to be divided into at least 21 aliquots.

When aliquots are prepared, amount of samples on the discs may not have been equal. Therefore, 'Natural Normalization' technique was applied. This technique uses a normalization run in which a short shine is given to each aliquot before any are given an additional dose. The values from this run are used to normalise the data subsequently obtained after irradiation. For natural normalization short shines of 0.1 and 0.5 seconds are applied.

3.5.3.1.3 Preheating

For unburnt sediment there is strong advantage that only light sensitive traps are sampled and the unbleachable residual is very much smaller than that of TL. A disadvantage is that there is no built-in indication of stability as provided in the case of TL by the glow-curve; it is necessary to remove unstable components by a thermal pre-treatment before measurements (Aitken, 1998).

Although there may be some sample types for which preheating is unnecessary this is exceptional and, after irradiation all aliquots are subjected to this treatment including non-irradiated group (Aitken, 1998). All quartz samples are preheated at $220^{O}C$ for five minutes and all feldspar samples were preheated at $160^{O}C$ for two hours (Aitken, 1998).

3.5.3.2 Annual Dose Determinations

The contributions of alpha, beta particles and gamma rays to the annual dose are determined by alpha counting, Atomic Emission Spectrometry and by placing TLD discs in the site, which are explained in Chapter 2.

3.5.3.2.1 Alpha Counting

In order to determine the alpha contribution from U and Th to annual dose, alpha counting has been carried out in ELSEC 7286 Low Level Alpha Counter. Sealed and unsealed alpha counting for three days for each sample with a period of 1000 seconds has been conducted, results are tabulated in Table 3.3.

Table 3.3 Unsealed and sealed alpha counts for each sample.

Unsealed α Counts (S5206)	**Counts/ksec**	**Sealed α Counts (S3707)**	**Counts/ksec**
Mean counts	12.37 ± 3.76	Mean counts	13.93 ± 3.86
Mean thorium counts	5.72 ± 0.64	Mean thorium counts	9.10 ± 0.65
Mean uranium counts	6.64 ± 0.67	Mean uranium counts	4.83 ± 0.67
Sealed α Counts (S5206)		**Unsealed α Counts (N3010)**	
Mean counts	12.13 ± 3.76	Mean counts	11.14 ± 3.82
Mean thorium counts	6.63 ± 0.60	Mean thorium counts	7.67 ±1.46
Mean uranium counts	5.50 ± 0.62	Mean uranium counts	3.47 ±1.50
Unsealed α Counts (S4240)		**Sealed α Counts (N3010)**	
Mean counts	9.73 ± 3.37	Mean counts	10.95 ± 3.94
Mean thorium counts	5.74 ± 0.52	Mean thorium counts	5.73 ± 1.40
Mean uranium counts	3.98 ± 0.55	Mean uranium counts	5.22 ± 1.45
Sealed α Counts (S4240)	**Counts/sec**	**Unsealed α Counts (S2817)**	**Counts/ksec**
Mean counts	9.60 ± 3.32	Mean counts	11.71 ± 3.60
Mean thorium counts	3.69 ± 0.40	Mean thorium counts	4.56 ± 1.32
Mean uranium counts	5.71 ± 0.42	Mean uranium counts	7.15 ± 1.37
Unsealed α Counts(S3707)		**Sealed α Counts (S2817)**	
Mean counts	13.19 ± 3.82	Mean counts	12.68 ±3.56
Mean thorium counts	4.95 ± 0.64	Mean thorium counts	6.59 ± 1.46
Mean uranium counts	8.23 ± 0.67	Mean uranium counts	6.09 ± 1.51

3.5.3.2.2 Potassium Analysis

Results of Atomic Emission Spectrometry give us the potasium content of a sample examined in miligrams per liter, three measurements for each was done. Results are given in Table 3.4.

Table 3.4 Atomic Emission Spectrometry results for potassium content.

Sample	Intensity	K%	K2O%	Average	St.Dev.
S4240	61.8	1.95	2.35	2.34	0.04
	62.8	1.98	2.39		
	60.5	1.91	2.30		
S5206	97	3.08	3.72	3.69	0.02
	96.1	3.06	3.68		
	96.1	3.06	3.68		
S2817	89.8	2.32	2.79	2.80	0.01
	90.6	2.34	2.81		
	90.2	2.33	2.80		
N3010	74.9	1.91	2.29	2.30	0.01
	74.6	1.91	2.30		
	75.7	1.93	2.32		
S3707	62.8	3.16	3.79	3.77	0.02
	62.4	3.13	3.76		
	62.3	3.13	3.75		

3.5.3.2.3 Determination of Cosmic Ray Component of Annual Dose

In order to find the cosmic ray component of the annual dose TLD (Al_2O_3: C) dosimeters have been used. Primarily the calibration curves for TLD dosimeters have been assessed. To do that first discs are irradiated by doses given in Table 3.5, then TL counts for each dose have been taken 24 hours after irradiation. The dose response curves have been plotted as TL counts (μC) versus dose (mGy). The graph of luminescence from dosimeters versus dose

amount when they are irradiated is called as calibration curve. For a large dose range this graph must be linear for a good dosimeter.

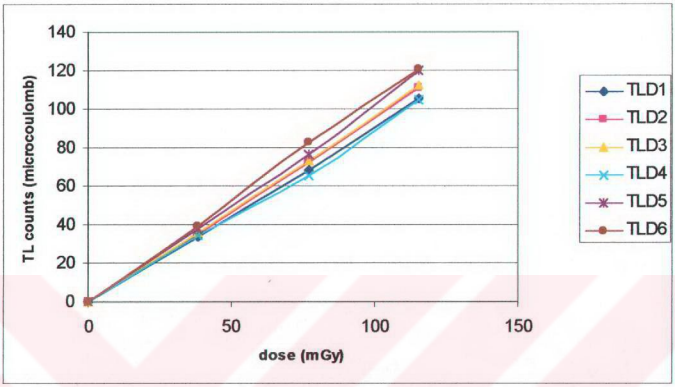


Figure 3.9 TLD discs calibration curves.

Table 3.5 Dose response (μC) for dose amounts introduced to TLD discs.

DOSE(mGy)	TLD1 dose response (μC)	TLD2 dose response (μC)	TLD3 dose response (μC)	TLD4 dose response (μC)	TLD5 dose response (μC)	TLD6 dose response (μC)
38.5	33.72	35.2	36.16	34.82	38.2	39.51
77	67.94	72.44	73.07	65.77	76.58	82.87
115.5	105.6	110.9	112.2	104.7	120.3	120.5

3.5.3.2.4 Water Content

Saturation water content and water uptake during burial for each sample are tabulated in Table 3.6.

Table 3.6 Water Uptake measurements.

Sample	Crucible Weight (gr)	Crucible and Dry Sample Weight (gr)	Crucible and Wet Sample Weight (gr)	Saturation Water Content as Percentage	Water Uptake During Burial
S4240	24.03	27.00	28.38	46.46	30.97
S5206	12.17	15.13	16.53	47.29	31.52
S2817	17.03	20.49	22.30	52.31	34.87
N3010	13.41	19.63	21.51	30.22	20.15
S3707	19.13	26.36	28.62	31.25	20.83

3.5.4 Values Needed for Age Evaluations

To determine the age of the bricks we now have the values needed to calculate the age for both IRSL and GLSL. These values are given for each sample in the following tables.

Table 3.7 Values needed to calculate age for fine grains of S4240 for IRSL.

SAMPLE: S4240 IRSL	
Unsealed α count rate:	9.79 counts / ksec
Unsealed α count rate due to Thorium:	5.74 counts / ksec
Unsealed α count rate due to Uranium:	3.98 counts / ksec
Sealed α count rate:	9.60 counts / ksec
K_2O %:	2.35
Saturation Water Content %:	0.46
Water Uptake During Burial%:	0.31
a value:	0.015
Cosmic ray dose rate:	0.146 mGy/y
Equivalent Dose:	11.928 Gy

Table 3.8 Values needed to evaluate age for fine grains of S5206 of IRSL

SAMPLE: S5206 IRSL	
Unsealed α count rate:	12.37 counts / ksec
Unsealed α count rate due to Thorium:	5.72 counts / ksec
Unsealed α count rate due to Uranium:	6.64 counts / ksec
Sealed α count rate:	12.13 counts / ksec
K_2O %:	3.69
Saturation Water Content %:	0.47
Water Uptake During Burial%:	0.31
a value:	0.15
Cosmic ray dose rate:	0.146 mGy / y
Equivalent Dose:	43.258 Gy

Table 3.9 Values needed to calculate age for fine grains of S2817 for IRSL.

SAMPLE: S2817 IRSL	
Unsealed α count rate:	11.71 counts / ksec
Unsealed α count rate due to Thorium:	4.56 counts / ksec
Unsealed α count rate due to Uranium:	7.15 counts / ksec
Sealed α count rate:	12.68 counts / ksec
K_2O %:	2.80
Saturation Water Content %:	0.52
Water Uptake During Burial %:	0.34
a value:	0.15
Cosmic ray dose rate:	0.146
Equivalent Dose:	46 Gy

Table 3.10 Values needed to calculate age for fine grains of N3010 for IRSL

SAMPLE: N3010 IRSL	
Unsealed α count rate:	11.14 counts / ksec
Unsealed α count rate due to Thorium:	7.67 counts / ksec
Unsealed α count rate due to Uranium:	3.47 counts / ksec
Sealed α count rate:	10.95 counts / ksec
K_2O %:	2.31
Saturation Water Content %:	0.30
Water Uptake During Burial %:	0.20
a value:	0.15
Cosmic ray dose rate:	0.146 mGy / y
Equivalent Dose:	15.8 Gy

Table 3.11 Values needed to calculate age for fine grains of S3707 for IRSL

SAMPLE: S3707 IRSL	
Unsealed α count rate:	13.19 counts / ksec
Unsealed α count rate due to Thorium:	4.95 counts / ksec
Unsealed α count rate due to Uranium:	8.23 counts / ksec
Sealed α count rate:	13.93 counts / ksec
K_2O %:	3.77
Saturation Water Content %:	0.31
Water Uptake During Burial%:	0.20
a value:	0.15
Cosmic ray dose rate:	0.146 mGy/y
Equivalent Dose:	18 Gy

Table 3.12 Values needed to calculate age for fine grains of S4240 for GLSL.

SAMPLE: S4240 GLSL	
Unsealed α count rate:	9.79 counts / ksec
Unsealed α count rate due to Thorium:	5.74 counts / ksec
Unsealed α count rate due to Uranium:	3.98 counts / ksec
Sealed α count rate:	9.60 counts / ksec
K_2O %:	2.35
Saturation Water Content %:	0.46
Water Uptake During Burial %:	0.31
a value:	0.07
Cosmic ray dose rate:	0.146 mGy/y
Equivalent Dose:	22.568 Gy

Table 3.13 Values needed to calculate age for fine grains of S5206 for GLSL.

SAMPLE: S5206 GLSL	
Unsealed α count rate:	12.37 counts / ksec
Unsealed α count rate due to Thorium:	5.72 counts / ksec
Unsealed α count rate due to Uranium:	6.64 counts / ksec
Sealed α count rate:	12.13 counts / ksec
K_2O %:	3.69
Saturation Water Content %:	0.47
Water Uptake During Burial %:	0.31
a value:	0.07
Cosmic ray dose rate:	0.146 mGy/y
Equivalent Dose:	32.4 Gy

Table 3.14 Values needed to calculate age for fine grains of S2817 for GLSL.

SAMPLE: S2817 GLSL	
Unsealed α count rate:	11.71 counts / ksec
Unsealed α count rate due to Thorium:	4.56 counts / ksec
Unsealed α count rate due to Uranium:	7.15 counts / ksec
Sealed α count rate:	12.68 counts / ksec
K_2O %:	2.80
Saturation Water Content %:	0.52
Water Uptake During Burial %:	0.34
a value:	0.07
Cosmic ray dose rate:	0.146
Equivalent Dose:	36.873 Gy

Table 3.15 Values needed to calculate age for fine grains N3010 for GLSL.

SAMPLE: N3010 GLSL	
Unsealed α count rate:	11.14 counts / ksec
Unsealed α count rate due to Thorium:	7.67 counts / ksec
Unsealed α count rate due to Uranium:	3.47 counts / ksec
Sealed α count rate:	10.95 counts / ksec
K_2O %:	2.31
Saturation Water Content %:	0.30
Water Uptake During Burial %:	0.20
a value:	0.07
Cosmic ray dose rate:	0.146 mGy/y
Equivalent Dose:	29.3 Gy

Table 3.16 Values needed to calculate age for fine grains of S3707 for GLSL.

SAMPLE: S3707 GLSL	
Unsealed α count rate:	13.19 counts / ksec
Unsealed α count rate due to Thorium:	4.95 counts / ksec
Unsealed α count rate due to Uranium:	8.23 counts / ksec
Sealed α count rate:	13.93 counts / ksec
K_2O %:	3.77
Saturation Water Content %:	0.31
Water Uptake During Burial %:	0.20
a value:	0.07
Cosmic ray dose rate:	0.146
Equivalent Dose:	17.8 Gy

These tabulated values, which were found by the experiments, are entered to the ELSEC 9010 OSL Dating System Software's age.exe computer program to calculate the dose-rates contributions of alpha-, beta- particles and gamma-rays. The equivalent dose and the cosmic ray component of the annual dose were entered the program manually. As a result of these values, age was calculated by the same program of the 9010 software.

CHAPTER 4

CALCULATED AGES AND DISCUSSION

In age calculations, Equation 2.3 was used. Equivalent doses of the samples are given in Tables 3.7- 3.16., for IRSL and GLSL respectively. For each sample GLSL and IRSL dating have been done. MAAD technique was used for the ED determination. The components of annual dose; $D\gamma$, D_β, D_C, $D\alpha$ dose rates are listed in the following tables (Table 4.1-4.10) By using the data given in Tables 3.7-3.16, the ages are calculated and listed below together with annual dose rate of alpha, beta particles, gamma and cosmic rays.

Table 4.1 Dose rates for fine grains of S4240 for IRSL.

SAMPLE: S4240 IRSL		
	DOSE RATES (mGy/y)	% OF TOTAL
α (D_α):	1.616	33.27
β from U:	0.419	8.62
β from Th:	0.192	3.96
β from K:	1.390	28.62
Total β (D_β):	2.001	41.02
γ from U:	0.330	6.80
γ from Th:	0.355	7.32
γ from K:	0.408	8.40
Total γ (D_γ):	1.093	22.52
Cosmic Ray:	0.146	3.02
Total (Annual Dose):	4.857	100
Age:	3.07±0.23 ky	

Table 4.2 Dose rates for fine grains of S5206 of IRSL

SAMPLE: S5206 IRSL		
	DOSE RATES (mGy/y)	% OF TOTAL
α (D_α):	1.941	34.85
β from U:	0.417	7.49
β from Th:	0.321	5.76
β from K:	1.409	25.30
Total β (D_β):	2.147	38.55
γ from U:	0.328	5.89
γ from Th:	0.593	10.65
γ from K:	0.413	7.42
Total γ (D_γ):	1.334	23.96
Cosmic Ray:	0.146	2.64
Total (Annual Dose):	5.77	100
Age:	7.77±0.62 ky	

Table 4.3 Dose rates for fine grains of S2817 for IRSL.

SAMPLE: S2817 IRSL		
	DOSE RATES (mGy/y)	% OF TOTAL
α (D_α):	1.867	34.66
β from U:	0.329	6.11
β from Th:	0.252	4.69
β from K:	1.598	29.67
Total β (D_β):	2.180	40.46
γ from U:	0.254	4.72
γ from Th:	0.469	8.70
γ from K:	0.470	8.72
Total γ (D_γ):	1.193	22.14
Cosmic Ray:	0.146	2.73
Total (Annual Dose):	5.387	100
Age:	8.54±0.69 ky	

Table 4.4 Dose rates for fine grains of N3010 for IRSL.

SAMPLE: N3010 IRSL		
	DOSE RATES (mGy/y)	% OF TOTAL
α (D_α):	1.964	34.68
β from U:	0.616	10.87
β from Th:	0.184	3.26
β from K:	1.498	26.44
Total β (D_β):	2.298	40.56
γ from U:	0.481	8.48
γ from Th:	0.339	5.98
γ from K:	0.437	7.71
Total γ (D_γ):	1.257	22.17
Cosmic Ray:	0.146	2.59
Total (Annual Dose):	5.665	100
Age:	2.79±0.27 ky	

Table 4.5 Dose rates for fine grains of S3707 for IRSL.

SAMPLE: S3707 IRSL		
	DOSE RATES (mGy/y)	% OF TOTAL
α (D_α):	2.345	30.81
β from U:	0.403	5.29
β from Th:	0.442	5.81
β from K:	2.439	32.05
Total β (D_β):	3.284	43.15
γ from U:	0.310	4.07
γ from Th:	0.813	10.69
γ from K:	0.711	9.34
Total γ (D_γ):	1.834	24.10
Cosmic Ray:	0.146	1.93
Total (Annual Dose):	7.609	100
Age:	2.37±0.23 ky	

Table 4.6 Dose rates for fine grains of S4240 for GLSL.

SAMPLE: S4240 GLSL		
	DOSE RATES (mGy/y)	% OF TOTAL
α (D_α):	0.730	18.38
β from U:	0.419	10.54
β from Th:	0.192	4.84
β from K:	1.390	35.01
Total β (D_β):	2.001	50.39
γ from U:	0.330	8.31
γ from Th:	0.355	8.95
γ from K:	0.408	10.27
Total γ (D_γ):	1.093	27.53
Cosmic Ray:	0.146	3.70
Total (Annual Dose):	3.972	100
Age:	5.68±0.92 ky	

Table 4.7 Dose rates for fine grains of S5206 for GLSL.

SAMPLE: S5206 GLSL		
	DOSE RATES (mGy/y)	% OF TOTAL
α (D_α):	0.906	19.98
β from U:	0.417	9.20
β from Th:	0.321	7.07
β from K:	1.409	31.08
Total β (D_β):	2.147	47.35
γ from U:	0.328	7.23
γ from Th:	0.593	13.08
γ from K:	0.413	9.12
Total γ (D_γ):	1.334	29.43
Cosmic Ray:	0.146	3.24
Total (Annual Dose):	4.535	100
Age:	7.14±0.57 ky	

Table 4.8 Dose rates for fine grains of S2817 for GLSL.

SAMPLE: S2817 GLSL		
	DOSE RATES (mGy/y)	% OF TOTAL
α (D_α):	0.844	19.34
β from U:	0.329	7.54
β from Th:	0.252	5.78
β from K:	1.598	36.63
Total β (D_β):	2.180	49.95
γ from U:	0.254	5.83
γ from Th:	0.469	10.74
γ from K:	0.470	10.77
Total γ (D_γ):	1.193	27.34
Cosmic Ray:	0.146	3.37
Total (Annual Dose):	4.364	100
Age:	8.45±0.73 ky	

Table 4.9 Dose rates for fine grains of N3010 for GLSL.

SAMPLE: N3010 GLSL		
	DOSE RATES (mGy/y)	% OF TOTAL
α (D_α):	0.922	19.94
β from U:	0.616	13.32
β from Th:	0.184	3.99
B from K:	1.498	32.40
Total β (D_β):	2.298	49.71
γ from U:	0.481	10.40
γ from Th:	0.339	7.33
γ from K:	0.437	9.44
Total γ (D_γ):	1.257	27.17
Cosmic Ray:	0.146	3.18
Total (Annual Dose):	4.622	100
Age:	6.34±0.57 ky	

Table 4.10 Dose rates for fine grains of S3707 for GLSL.

SAMPLE: S3707 GLSL		
	DOSE RATES (mGy/y)	% OF TOTAL
α (D_α):	1.094	17.21
β from U:	0.403	6.33
β from Th:	0.442	6.95
β from K:	2.439	38.35
Total β (D_β):	3.283	51.64
γ from U:	0.310	4.88
γ from Th:	0.813	12.79
γ from K:	0.711	11.18
Total γ (D_γ):	1.834	28.85
Cosmic Ray:	0.146	2.31
Total (Annual Dose):	6.359	100
Age:	2.80±0.28 ky	

The ages of five samples with their uncertainties, studied in this work are summarized in Table 4.11. Here age refers to the time past since the samples' last exposure to light.

Table 4.11 Green Light Stimulated Luminescence (GLSL) and Infra Red Stimulated Luminescence (IRSL) ages for each sample.

SAMPLE	LEVEL	IRSL AGE (ky)	GLSL AGE (ky)
S4240	VII	3.07 ± 0.23	5.68 ± 0.92
S5206	IX	7.77 ±0.62	7.14 ± 0.57
S2817	VII	8.54 ± 0.69	8.45 ± 0.73
N3010	VI-V	2.79 ±0.27	6.34 ± 0.57
S3707	VII	2.37 ± 0.23	2.80 ± 0.28

For each sample two comparisons have been done i.e.

1. Comparison between IRSL and GLSL ages,
2. Comparison between OSL ages and the ages determined by other methods.

As can be seen from Table 4.11, for the samples S5206 and S2817 the IRSL and GLSL ages are in agreement, whereas for the samples S4240 and N3010 these two ages are not in agreement. This disagreement may be explained by the different rates of eviction of electrons in quartz and feldspar minerals when they are exposed to light (Godfrey-Smith *et al.*1988).

Although for sample S3707 the IRSL and GLSL ages are in agreement they are much smaller than those given in Figure 4.1 for level VII (Newton and Kuniholm, 1999). In fact, this sample has the highest dose-rate among the others. Thus it can be thought that the saturation may have been encountered for this sample.

When the results for the sample S5206 and S2817 are compared with the overall dating sequence derived from radiocarbon and dendrochronology (Newton and Kuniholm, 1999), S2817 has a promising result, although S5206 stays younger in the overall dating sequence, which is given as 6650-6250 BC carbon dates by Newton and Kuniholm (1999).

IRSL and GLSL ages for samples S4240 and N3010 are not in agreement and both are from level VII. The disagreement for this level needs to be investigated with more samples from same level. In addition, archaeological interpretation is needed for this level.

In order to be certain, the OSL dating studies for the same and different levels of the same site must be repeated for a large number of samples. Also, the problems such as, insufficient bleaching, fading and saturation of the signal, can be minimized by repeating the dating measurements for more samples.

CHAPTER 5

CONCLUSIONS

The optically stimulated luminescence from samples was measured in ELSEC 9010 Optical Dating System. The 514 nm green light source was used as a stimulation source for quartz samples. An array of infrared diodes was used for the polymineral fine grains resulting in a signal only from feldspars. The "Natural Normalization" technique is used to normalize the data obtained after irradiation.

The Equivalent Dose (ED) was determined by the additive dose technique, where the laboratory doses added to aliquots of the natural sample and the growth is extrapolated to zero luminescence signal to give the ED. Samples were irradiated by a Strontium-90 source which is delivering 0.0386Gy/sec.

In order to remove unwanted charge traps, which are filled during the laboratory irradiation, preheating was used. All quartz samples were preheated at 220°C for 5min. while the polymineral samples stimulated by infrared were preheated at 160 °C for 2h. Water content was measured as a percentage of dry weight.

The uranium and thorium contribution to annual dose rate was determined by low level alpha counting (ELSEC Low level alpha counter 7286) system, Potassium contribution by finding the K_2O% content with Atomic Emission Spectrometry (AES) and the cosmic ray component of annual dose rate by placing TLD discs

(Al_2O_3:C) and kept for eight months in the site where samples were taken.

When the results for the sample S5206 and 2817 are compared with the overall dating sequence derived from radiocarbon and dendrochronology (Cessford, 2001; Newton and Kuniholm, 1999; Göktürk *et.al*, 2002), the OSL ages are promising.

The results given in Chapter 4 are promising and need further study. To establish a chronology for the site based on direct dating of building, OSL technique may have a considerable importance.

In order to be certain, the OSL dating studies for the same and different levels of the same site must be repeated for a large number of samples. Also, the problems such as, insufficient bleaching, fading and saturation of the signal, can be minimized by repeating the dating measurements for a large numbers of samples.

REFERENCES

Aitken, M.J., 1985, Thermoluminescence Dating, Academic Press Inc. (London).

Aitken, M.J., and Smith B.W., 1988, Optical Dating: recuperation after heating, *Quaternary Science Rev.*, 7, 387-393,

Aitken, M.J., 1989, Luminescence Dating: A Guide for Non-Specialists, *Archaeometry*, 31, 147-159.

Aitken, M.J., 1990, Optical Dating of Sediment: Initial Results from Oxford, *Archaeometry,* 32, 19-31

Aitken, M.J., 1992, Optical Dating, *Quaternary Science Rev.*, 11, 127-131

Aitken, M.J., 1998, An Introduction to Optical Dating, Oxford University Press.

Black, C.A., 1965, Methods for Soil Analysis, American Society of Argonomy, Inc., Publisher Madison, Wisconsin, USA.

Botter-Jensen, L., Ditlevseni C. And Mejdahl, V., 1991, Combined OSL (infrared) and TL studies of Feldspars, *Nucl. Tracks Radiat. Meas.*, 18, 257-263.

Botter-Jensen, L., 2000, Development of Optically stimulated Luminescence Techniques Using Natural Minerals and Ceramics and their application to Retrospective Dosimetry, Doctorate Thesis, Riso Natural Lab., Roskilde.

Cessford, C., 2001, A new dating sequence for Çatalhöyük, *Antiquity*, 75, 717-725

Göktürk, E.H., Hillegonds, D.J., Lipschutz, M.E., Hodder, I.,(2002), *Radiochim. Acta*, 90, 407-410.

Godfrey-Smith, D.I., Huntley, D.J. and Chen, W.-H, 1988, Optical dating studies of quartz and feldspar sediment extracts, *Quaternary Science Rev.*, 7, 373-380.

Harvey, E.N., 1957, A History of Luminescence, *Memoirs of the American Philosophical Society*, 44, 1-351.

Huntley, D.J., Godfrey-Smith, D.I. and Thewalt, M.L.W., 1985, Optically Dating of Sediments, *Nature*, 313, 105-107.

Hütt, G., Jack, I. and Tchonka, J., 1988, Optical Dating: K-Feldspars Optical Response Stimulation Spectra, *Ouaternary Science Rev.*, 7, 381-386.

Lang, A. and Wagner, G.A., 1996, Infrared Stimulated Luminescence dating of archaeosediments, *Archaeometry,* 38,129-141.

Li, S. and Aitken, M.J., 1989, How far back can we go with optical dating of K-Feldspars. In: Long & Short Range Limits in Luminescence dating, vol.9, 41-46. Occasional Publication (RLAHA).

Mellaart, J., 1967, Çatalhüyük: a Neolithic Town in Anatolia. London: Thames & Hudson.

Newton, W. M and Kuniholm, P.I., 1999, Wiggles Worth Matching-Making Radiocarbom Work. The Case of Çatalhöyük, Aegaeum 20, Annales d'archaeolgie egeenne de l'Universite de Liege et UT-PASP, 527-536.

Parish, R., 1996, On the Surface: Çatalhöyük 1993-95, ed. Ian Hodder , Cambridge MacDonald Inst. For Archaeological Research and British Institute of Archaeology at Ankara.

Poolton, N.R.J. and Bailiff, I.K., 1989, The use of LEDs as an excitation source for photoluminescence, *Ancient TL,* 7, 18-20.

Rees-Jones, J and Tite, M.S., 1997, Optical Dating Result of British Archaeological Sediments, *Archaeometry,* 39, 177-188.

Rhodes, E.J., 1988, Methodological considerations in the optical dating of quartz, *Ouaternary Science Rev.*, 7, 359-400.

Rhodes, E.J., 1990, Optical Dating of Quartz from Sediments, D.Phil Thesis, Oxford University.

Shingvi, A.K. and Wagner, G.A., 1986, TL dating and its applications to young sedimentary deposits. In: Hurford, A.J., Jager, E. and Ten Cate, J.A.M.(eds.) Dating Young Sediments, 159-197, CCop. Techn. Sec., Bangkok.

Smith, B.W., Aitken. M.J., Rhodes, E.J., Robinson, P.D. and Gelard, D.M., 1986, Optical dating: Methodological Aspects, *Radiation Protection Dosimetry,* 17, 229-233.

Smith, B. W, Rhodes, E.J, Stokes, S., and Spooner, N.A, 1990, The Optical Dating Of Sediments Using Quartz, *Radiation Protection Dosimetry,* 34, 75-78.

Spooner, N.A., Prescott, J.R. and Hutton, J.J.,1988, The effect of illumination wavelength on the bleaching of TL of Quartz, *Quaternary Science Rev.,7,* 325-330.

Spooner, N.A., Aitken, M.J., Smith, B.W., Franks, M. and Mcelroy, C., 1990, Archaeological Dating by infrared stimulated Luminescence using a diode array, *Radiation Prot. Dosimetry,* 34, 83-86.

Stokes, S., 1992, Optical dating of Young (Modern) Sediments Using Quartz: Results from a Selection of Depositional Environments, *Quaternary Science Rev.*, 11,153-159.

Wintle, A.G., 1975, Thermal Quenching of Thermoluminescence in Quartz, *Geophysical Journal of the Royal Astronomical Society*, 41, 107-113.