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Recent Developments in the Techniques of Luminescence Dating

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Abstract

Dating is critical for the development of theories relating to many aspects of geology over Quaternary timescales (0-2 millions). Among different dating methods luminescence has the potential for covering a significant range of Quaternary time. In the last 3 decays, from 1967 to 2003, the use of luminescence signals from naturally occurring minerals has gone through a major transformation, from thermoluminescence (TL) dating of pottery to optically stimulated luminescence (OSL) dating of sediments. The present work reports aspects of recent advances of luminescence technique from naturally occurring minerals for dating applications. It has provided information about basic principles of luminescence dating and shows very briefly its progress from blue TL to OSL and red luminescence.

Keywords: *Luminescence, Dating, Quartz, Feldspar, TL, OSL.*

1. Introduction

Luminescence dating methods are based on a radiation-produced charge population trapped within crystalline dosimeters (e.g., quartz and feldspar). These dosimeters are capable of determining the period of time, which has elapsed since the last time the trapped charge population was reset. For archeological and volcanic materials, the resetting events are heating of the material. The resetting event for sedimentary materials is the last exposure to daylight prior to deposition.

Following the successful application of thermoluminescence (TL) dating to pottery and other fired archeological materials in the 1960s

and 1970s (e.g., Aitken, 1985; Aitken, 1990), similar techniques were applied to geologically-fired volcanic deposits (Wintle, 1973; 1974) and sedimentary materials (Wintle and Huntley, 1980).

The possibility of obtaining an optically stimulated luminescence (OSL) signal from feldspar and quartz using light (an argon ion laser) was demonstrated by Huntley *et al*. (1985), and the use of OSL in dating applications is now well established especially in dating sedimentary deposits. Hütt *et al*. (1988) published the first stimulation spectrum for feldspar showing a large stimulation peak in the near IR (825-1030 nm). This unexpected stimulation peak in the near infrared for feldspars was confirmed in later studies and the results of such stimulation spectra and their applications are summarised by a number of authors (see Table 4 and Figure 9 of Duller, 1997).

Research in to TL and especially OSL methods have increased dramatically during the last 15 years. The advances include technology, methodology, physics and application of luminescence dating. More than 50 luminescence-dating laboratories now exist world-wide, particularly within Europe (14 in UK, alone), Australia and China. Most of these laboratories now exist within university establishment because the method has remained in a developing state, frequently distributed, and not yet widely applied as a routine dating method.

This paper seeks to report very briefly the recent advances in techniques of luminescence dating methods. It describes the age equation, the determination of the equivalent absorbed dose accumulated, the dose-rate determination common for both TL and OSL dating of the geological or archaeological event that took place in the past, and the probable problems. Further details could be found elsewhere (e.g., Aitken, 1985; 1990; 1998; Chen and McKeever, 1997; Berger, 1994; 1995; Wintle, 1997; Duller, 1996; 1997; Wagner, 1998; Stokes, 1999; Hütt, and Jaek, I 2001; Fattahi and Stokes, 2003a) and references there in.

2. Basis and methodology of luminescence dating

2.1. Basic principles

Radioactive elements (e.g., Uranium, Thorium, Potassium and Rubidium) are present in the Earth's crust and undergo radioactive

decay over long (>10^6 a) timescales. When minerals such as quartz, and feldspar (used as luminescence dosimeters) are exposed to ionising radiation, electrons may become detached from their parent nuclei in the crystal lattice and diffuse in the vicinity of defects in the lattice where they may become trapped. The number of trapped electrons increases proportionally with the duration and intensity of radiation exposure. Subsequent heating or illumination of a crystal can free trapped electrons, and they may return to a vacant position (a 'hole') left by the absence of a previously displaced electron where they will either emit part of the energy absorbed during irradiation as a photon, or they may become trapped. A convenient way of describing the luminescence mechanism is to display it as an 'energy level diagram' (Figure 1) in which a 'hole' is shown either as a 'recombination centre' for electrons, from which light (a photon) is emitted or, alternatively if heat is produced, as a 'killer centre' (Aitken, 1985, 1998).

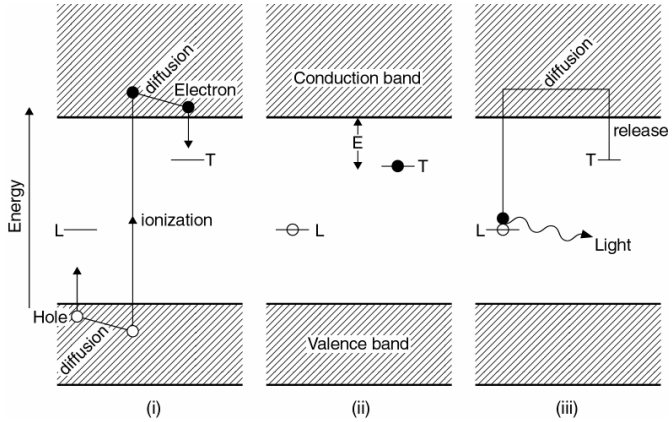

Figure 1 - An energy-level diagram of the luminescence process (after Aitken, 1998). (i) After the ionisation of atoms following exposure to radiation, an electron is ejected from the valence band to the conduction band and a hole is created in the valence band. Then electrons and holes are respectively trapped in T and L defects. (ii) Storage of electrons in electron traps and holes in the recombination centre, with life times ranging from seconds to millions of years. (iii) Electrons are elevated from the traps to the conduction band either by thermal or optical stimulation. Some of these electrons will recombine with the trapped hole, and will produce light (a photon of a specific wavelength) - and some will produce heat (a phonon).

Luminescence dating is based on the premise that luminescence dosimeters (e.g., quartz and feldspars) record the amount of radiation (?q ?q ?qnd cosmic) to which they have been exposed and this can be related to time. The ?, ? and ? radiation arise mainly from the ambient U and Th isotopes and their decay products, as well as from ^{40}K.

The age of a sample is calculated using the formula:

Age (ka) =Equivalent Dose (Gy) / Dose Rate (Gy/ka)

The Equivalent Dose (D_e) is the radiation dose, measured in the laboratory, received by a sample since some resetting event (such as a volcanic eruption). The dose is equivalent to a natural radiation dose. The Dose Rate is the amount of dose to which a sample has been exposed per year, and Gray (Gy) is the SI unit of absorbed radiation.

2.1. Equivalent dose determination

In the past 30 years much effort has been expanded to determine the optimal means of measuring the equivalent dose (D_e). Estimates of De are achieved by comparing the laboratory irradiation. A common way of making this comparison is by the use of dose response curves, often known as growth curves.

There are two main approaches for measuring D_e: (i) the additive dose method which involves extrapolation of the growth curve of the luminescence signal as a function of radiation added in the laboratory to the aliquots containing natural dose; and (ii) the regeneration method that involves interpolation and measuring the dose of ionising radiation that produces the same luminescence signal as was measured from the sample in its 'natural' state (Stokes et al., 2000). The essential basis of additive dose procedure is shown in Figure 2a. A number of equal aliquots (identical sub-samples) are divided into groups. One group is used to measure natural luminescence and the other groups are given various laboratory radiation doses in addition to natural before measurement. A luminescence growth curve is constructed by adding doses to the natural dose, except that instead of reading off the equivalent dose (ED) from the intercept on the dose axis, it is read off

from the intercept on a horizontal line through the latter being the level of TL remaining after a long laboratory bleaching. In the regeneration technique the sample gets a prolonged exposure to light until the TL level is reduced to the unbleachable residual signal (total bleached). The luminescence growth curve is then regenerated by adding doses to the residual dose and the palaeodose ED is taken as that required to reproduce the natural level (Figure 2b).

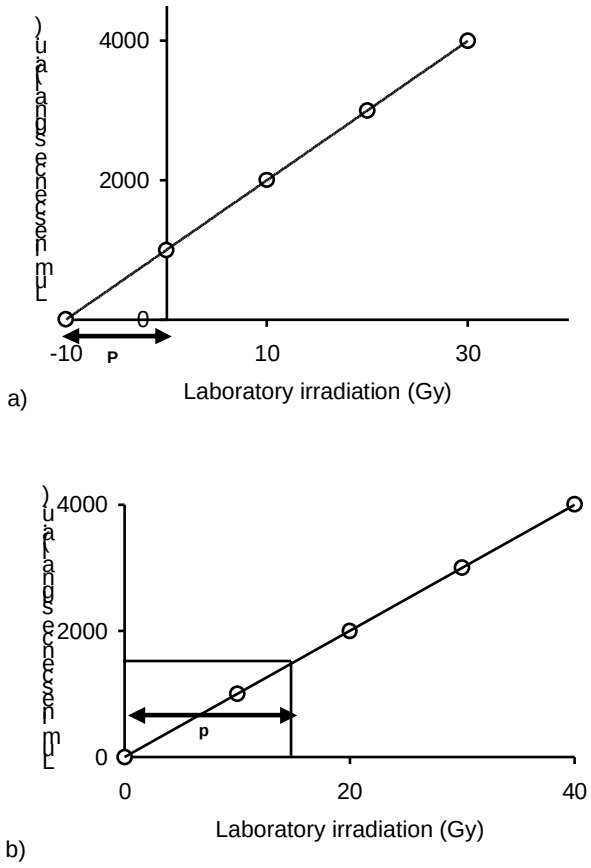

Figure 2 - Schematic representation of the two main methods used for equivalent dose determination (expressed here as the palaeodose, P); (a) additive dose, (b) regeneration.

The essential basis of regeneration dose procedure is shown in Figure 2b.

Conventional methods of D_e determination are divided to two groups: Multiple aliquot (identical sub-samples) in which several tens of separate aliquots of each sample is used and single aliquot that make all the measurements necessary for D_e measurement only on a single 5-10 mg sample aliquot (Duller, 1995).

Single aliquot methods of D_e determination are preferred over multiple-aliquot methods, as the former have been shown to provide better precision in equivalent dose determination (e.g., Murray and Wintle, 2000). The precision can be provided either by analysis of the fitting parameters for data from an individual aliquot, or by looking at the spread in equivalent dose from a set of aliquots (Duller 1995; Duller et al., 2000). The latter assume that the uncertainty in the estimate of equivalent dose from each aliquot is similar. This assumption is not valid when a single grain is used (Duller et al., 2000). It therefore becomes essential to assess correctly the uncertainty associated with each equivalent dose determination. Single aliquot protocols, especially the single aliquot regenerative-dose (SAR) protocol first suggested by Murray and Roberts (1998) are now widely used to measure D_e in feldspar and quartz OSL and TL dating of both heated and sedimentary materials (Murray and Wintle, 2000; Fattahi, 2001; Fattahi and Stokes 2003d and references there in).

2.3. Dose rate

The analytical techniques for determining ?‡?‡?‡dose-rate components are as follow:

a) neutron activation analysis,

b) alpha particle counting "pairs" technique (Huntley and Wintle, 1981; Liritzis and Galloway, 1982b), and potassium determination by XRF, atomic absorption or other nuclear or chemical techniques,

c) High purity Ge gamma and alpha spectrometry (Murray and Aitken, 1982; Liritzis and Danali- Kotsakis, 1985; Saro and Pikna, 1987),

d) portable ?s-scintillometer (Liritzis and Galloway, 1981),

e) plastic scintillator (Galloway and Liritzis, 1991),

f) Induced couple plasma mass spectrometry,

g) Geiger - Muller (GM) proportional counter (Botter-Jensen and Mejdahl, 1985),
h) TL dosimetry, employing phosphors e.g $CaSO_4$:Dy, $CaSO_4$:Mn, CaF_2, LiF, SiO_2 (Mejdahl, 1972; Liritzis and Galloway, 1980, 1982a, b; Liritzis, 1985).
The in situ dosimetry of (h) is suitable for heated materials not sediments. The final error in dose-rate is between 5-7%.
Further detail about the principles and practices of dose-rate evaluation could be found elsewhere (e.g., Aitken, 1985).

3. Probable problems

It seems appropriate to set out in summary form some of the issues that appeal to us as relevant to the actual practice of luminescence dating. Major categories of causal factors for the unreliability of luminescence dating of sediments include:

3.1. Type of fitting function to growth curves

The exact form of a linear or saturating exponential fit to the dose growth curves from "additive dose" or "regeneration" techniques, produce variable ED values, if the fitting parameters (exponents, least square test summed deviations) are not considered with care, and the experimental data points are a few.

3.2. Zeroing

Luminescence dating method measure the age of the sample since its latest signal resetting to zero. While after heating there is rarely any doubt about whether the luminescence signal was set to zero, resetting to zero is by no means certain when the mechanism is bleaching by exposure to light. TL at all glow curve temperatures for both quartz and feldspar (and mixtures of them) is reduced to a stable, low, but not zero level in times of the order of 24 h of full sun. It is necessary to determine.

In some cases there is clear evidence that zeroing has been incomplete. In such cases the partial bleach method (Wintle, 1997) and its variations, which restrict the measurement of TL light to that

fraction which is most easily bleached, are appropriate. It can be applied to any of the materials currently extracted for dating.

Sediments deposited by or under water are subject to uncertainty in the degree of resetting. Apart from uncertainty in the environment before the sediment grains entered the water, water itself attenuates the most effective wavelengths and this increases with increasing depth of the water column. OSL partial bleach protocol, together with thoughtful attention to the procedures to use in laboratory bleaching with the aim of emulating natural conditions may overcome this uncertainty.

3.3. Anomalous fading

The potential advantages of using feldspathic minerals as integrating dosimeters for utilization in thermoluminescence and optically-stimulated luminescence research have been complicated by the widespread observation of anomalous fading. A variety of mechanisms have been proposed to account for the presence of anomalous fading and while it is possible to detect fading via laboratory experimentation, a routinely applicable means by which the phenomenon may be overcome has not been forthcoming for the commonly used UV-blue emission. Studies by Visosekas and collaborators (e.g., Visosekas and Zink, 1999; Zink and Visosekas, 1997) proposed an alternative approach for use in TL dating applications involving the detection of the far red TL (RTL) emission in alkali feldspars. Their results suggest that the red (?j~ 600-750 nm) TL emission peaks (up to 300ºC) does not suffer from anomalous fading. Fattahi (2001) explored the stability of RTL (up to 600ºC); red and far red IRSL emission signals from potassium feldspars for dating applications. Fattahi and Stokes (2003, b-c) explored the suitability of infrared stimulated red luminescence from feldspar for dating applications. Short term fading experiments on samples which exhibit significant anomalous fading in the UV-blue portion of the emission spectrum suggest that anomalous fading is absent or significantly reduced. Furthermore, these emissions appear to be highly suited to long range dating applications (e.g., Fattahi and Stokes 2003d; Arnold *et al.*, 2003).

Further physics-based research into the causes of fading and methods of overcoming is required.

Since there is no confirmed evidence that quartz suffers from anomalous fading, it would appear to be a preferred dating material, although a preheat is needed to eliminate short-lived components of the glow curve after irradiation. The disadvantage of quartz for determination of large ages was that it saturates earlier than the best feldspars. This limitation could be overcome using red luminescence (Fattahi 2000a, b).

3.4. Dose-rate accuracies

The possible factors that could make a significant error in the corrected dose-rate value include:

1- The water content may not have been constant and the sediment may have become compacted (Prescott and Robertson, 1997).

2- Decay series U-disequilibrium (Radium-226 deficiency or excess, radon emanation, Th-230/U-234, U/Th), and mobility of such isotopes, water contents throughout the past (Prescott and Robertson, 1997).

In summary, equilibrium should never be assumed in luminescence dating and disequilibrium is almost guaranteed in wet environments, including those that were wet in the past.

4. Technology

Detection of the luminescence signal (very week light) requires the use of a photomultiplier tube (PMT) that is installed on a computer-controlled automated system. Both TL and OSL are normally detected using a PMT which, after 40 years, still constitutes the vital component in a luminescence measurement system**.**

The conventional PMTs are biased towards the UV-blue emission. The need for equipment, which is capable of measuring OSL and TL, has been influenced by studies, which have improved the physics, methodology and application of luminescence dating. Over the last few years, several new luminescence measurement systems and techniques have been developed. These include three new optical stimulation systems based on blue (470 nm) emitting diodes, an infrared (830 nm) laser diode and a solid state green (532 nm) laser for

rapid OSL measurements of individual sand-sized grains of a sample. Recently, three important enhancement in luminescence instrumentation have emerged: the linear modulation optically stimulated luminescence (LM-OSL) technique described by Bulur (1996); the single grain apparatus described by Duller et al., (1999) and red thermoluminescence and infrared stimulated red luminescence described by Fattahi and Stokes (2000a; 2003b, c respectively). The developed LM-OSL; single grain and red detection attachment for the Riso TL-OSL reader has been modified to achieve single grain LM-OSL and red emission LM-OSL (Bulur et al., 2002; Singarayer and Fattahi, 2003).

5. Age and environment ranges of luminescence dating

Previous studies show that luminescence dating methods have been applied to a very wide cross-section of depositional environment and their time range depend to the specified properties of dosimeter material in that environment (Stokes, 1999). These properties include the capacity of the dosimeter to accept and hold charge and the dose rate. Aeolian sediments, especially from warm desert environments have been the most extensively dated, due to their suitability (complete bleaching) for luminescence dating and the absence of other methods capable of providing age control (Stokes, 1997). The less ideal sedimentary environments (e.g., alluvial fans, rock shelter and fluvial) where bleaching by light could be limited have also been dated by luminescence (Stokes, 1999). In sand dominated sediments (dose rare # 1-2 Gy ka^{-1}) quartz can be used as a dosimeter for dating samples up to 150 ka (Stokes, 1999). In environments that ionizing radiation level are less, quartz has generated ages up to 800 ka (Huntley et al., 1993). Feldspathic minerals have provided ages of the order of hundred of thousands of years (e.g., Berger et al., 1992). In volcanic, quartz has extended the time range of luminescence to 1.2 Ma (Fattahi and Stokes, 2000b).

6. Conclusions

Luminescence dating methods are very powerful tools for providing an accurate index of the period of time since the deposition in many

sedimentary environment. Despite of these ability, the means by which the luminescence within minerals is produced, is not yet fully understood and luminescence dating methods are not used as a routine method for dating all geological environments and events and ongoing interactions between physicists and geoscientists (end-user) is a critical need. Luminescence is relatively a cheap dating method that can provide enormous advantageous for scientists and practitioners in Iran. It has vast geological applications. It can be used for dating past earthquakes and Volcanic activities up to 1.2 Ma, provide age control for sediments spanning the last interglacial cycle (0-150 ka), provides absolute age assessment over a broad cross-section of deposited sedimentary landscape components and to date very young (<1000 years old) sediments. Geoscientists with physics background have had a critical role in the ongoing development of luminescence dating and geologist and geomorphologists are employing these methods in their relevant activities.

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