Ancient TL

www.ancienttl.org · ISSN: 2693-0935

Li, S.-H., 1991. *Removal of the thermally unstable signal in optical dating of K-feldspar.* Ancient TL 9(2): 26-29. https://doi.org/10.26034/la.atl.1991.178

Removal of the thermally unstable signal in optical dating of K-feldspar

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Introduction

Optical dating has been developed over the past five years since it was first described by Huntley et al. (1985). However, one important aspect of optical dating has not received much discussion - the removal of the thermally unstable component of the signal induced by laboratory irradiation. This component is not stable at ambient temperature over the relevant geological time period. If this component is not removed by appropriate thermal pre-treatment, the equivalent dose, and hence the age of the sample, will be underestimated.

Preheating, or thermal washing, is often used to remove an unstable component in thermoluminescence (TL) dating (Mejdahl and Winter-Nielsen, 1982) and has been used in the optical dating of quartz (Huntley et al., 1985; Rhodes, 1988). Because the majority of the optically stimulated luminescence (OSL) signal from quartz is correlated to the 325 °C TL peak (Smith et al., 1986), a preheating procedure was developed which was based on the TL behaviour of this peak (Rhodes, 1988). However, this procedure would not be expected to be suitable for other minerals. For K-feldspar, the OSL is from at least two kind of traps, as demonstrated by the loss of signal from two TL peaks (Li and Aitken, 1989).

Experimental design

The design of a preheat procedure involves the choice of temperature and time. A very high preheating temperature can result in an extremely short preheating time with risk of failure to reach complete thermal equilibrium and of effects due to sample oxidization. On the other hand, if the preheating temperature is very low (say 50 °C), it may take months or years, making such a procedure impossible in practice.

For a particular preheating treatment to be appropriate, the thermally unstable signal should have been removed and enough stable signal left for measurement. Three possible tests are suggested.

a. Plateau method.

This is similar to the plateau method in TL dating (Aitken, 1985). Once the stable signal has been removed, a plateau of the OSL ratio for natural and irradiated samples is to be expected as the preheating time is increased.

b. Thermal decay rate.

The same thermal decay rate for the OSL signal is to be expected once sufficient preheating is achieved, because the remaining natural and irradiated signals will have the same thermal stability.

c. Similar shape of the TL glow curves.

After a suitable preheating, the TL glow curves should be similar for the natural and irradiated samples. Furthermore, the low temperature peak, which is thermally unstable, should be erased and only the high temperature peaks should be left.

Experimental details

Single wavelength (514.5 nm) light from an argon laser was used for stimulation (Smith et al. 1986). The power of the expanded laser beam incident on the sample was about 5 mW/cm^2. Detection of the stimulated luminescence was by means of a photomultiplier (EMI 9635QA) with four Corning 7-59 filters and one Schott BG39 filter in front. During measurement, the sample disc was placed on a heating plate which could be held at a precise temperature during illumination and all measurements are made at a sample temperature of 17 °C.

TL measurement was performed on a manual TL set with heating rate 5 °C/s. TL was detected with one Corning 7-59 filter and one HA3 filter in front of an EMI 9635QA photomultiplier.

K-feldspar was mounted on 10 mm diameter discs with a monolayer of 100-300 µm grains affixed by means of a silicone oil aerosol spray. Three K-feldspar samples separated from sediments from Scandinavia were used in this study. The Oxford Laboratory numbers are Z10, Z29 and Z30, and the TL equivalent doses are 70, 480 and 1120 Gy respectively (Li and Aitken, 1989). The Nordic TL Laboratory (Risø) reference numbers are 862403, 859008 and 869004.

A short shine (0.1 second) of laser light was used for the OSL measurements. This caused less than 0.4 % loss of the OSL signal and negligible loss of the TL signal (Li and Aitken, 1989). Hence, the discs could be used for more than one measurement.

A purpose-built thermostat-controlled oven was used in the 160 °C preheating studies.

Results and discussion

In practice, two different kinds of preheating are required. One involves a long time (a few hours) at a lower temperature, the other a short time (a few minutes) at a higher temperature. The former can be performed in an oven. The latter can be performed on a TL heating plate and is particularly useful if the same equipment is to be used for both TL and OSL measurements since measurements can be made without moving the discs.

1. Prolonged preheating

From other studies, it is known that there is a TL peak around 160 °C in the TL glow curve after laboratory irradiation. This TL maximum is not found in natural samples and is therefore considered to be an unstable signal.

For sample Z29, two discs, one containing the natural signal and one with an additional irradiation (200 Gy) were placed in a 160 °C oven for a total period of more

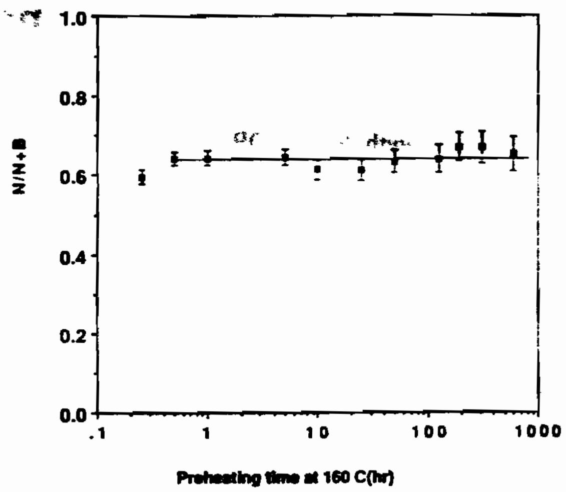

Figure 1.
Sample Z29 - OSL ratio versus preheating time at 160 °C. The ratio is between natural and natural + β dose (200 Gy) discs. (natural TL ≡ 480Gy)

than 200 hours. The discs were periodically taken out for OSL measurement. Figure 1 is the plot of the OSL ratio for these two discs and it shows that a plateau was reached after a half hour at 160 °C. Use of a 0.1 second laser exposure allows the discs to be used many times; hence no normalization is required to improve the precision. This is analogous to obtaining a plot of ED versus preheating time for a group of discs but this requires normalization and curve fitting procedures which reduce the precision.

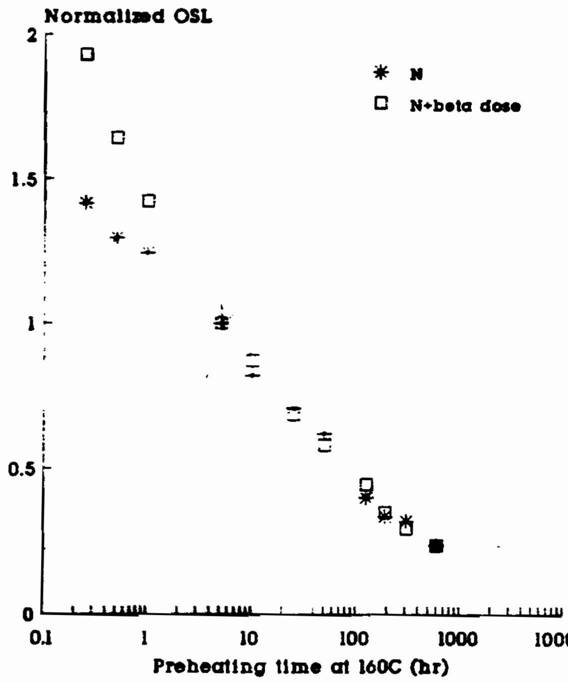

Figure 2.
Sample Z10-OSL signal isothermal decay curves at 160 °C; the added dose is 70 Gy. OSL was normalized to the OSL counts of after 5 hours preheating. (Natural TL ≡ 70 Gy)

Using a similar set of measurement, the thermal decay rates of the natural and irradiated signals can be compared.

Figure 2 shows the thermal decay of the OSL that occurs during the preheating for sample Z10. This shows that the thermal decay rates are the same once the two aliquots have been heated five hours or more and there is a thermally unstable signal in the dosed samples which can be removed after 5 hours preheating at 160 °C.

To demonstrate further the similar thermal response of natural and irradiated samples after preheating, a "quick heating" experiment was performed (Li and Wintle, 1991). The disc is heated to a given temperature, cooled to 17 °C and the OSL measured at 17 °C with the 0.1 second laser stimulation light. This measurement is repeated after heating to successively higher temperatures (figure 3). The responses were similar after preheating for half an hour at 160 °C. This implies that the OSL signal is from a similar temperature range for both discs after this preheating treatment. This was not the case for the OSL from discs which were not preheated.

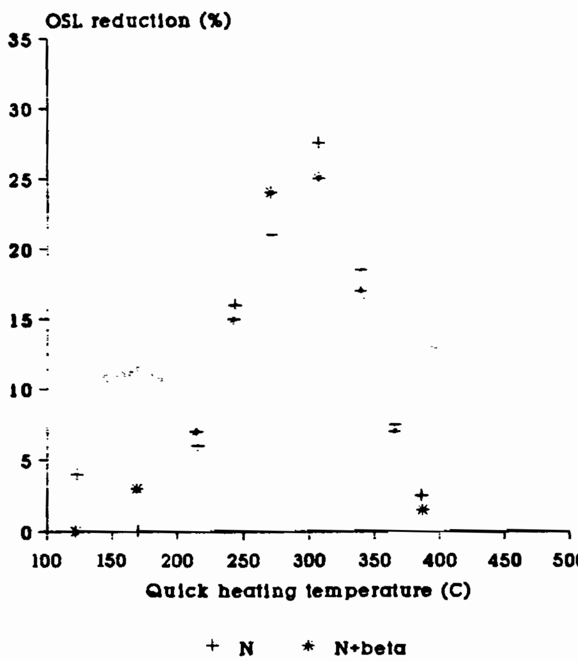

Figure 3.
Sample Z29-OSL reduction (% of whole OSL signal) resulting from each quick heating step, after preheating for half of an hour at 160 °C for natural and N+β samples. (100% is the whole OSL signal before quick heating)

2. Short preheating.

Other dating procedures require the removal of the thermally unstable signal in a short time. Ten minutes was chosen as a convenient preheat time, and the effects of different preheating temperatures were studied.

As already stated the OSL signal results from at least two populations of charge and it is impossible to distinguish their thermal stabilities using the OSL itself (Li and Wintle, 1991). To isolate the OSL signal with

the highest thermal stability, a preheat procedure was developed to leave only a high temperature TL peak.
Preheating experiments were carried out on the TL heating plate immediately before TL measurement. In an attempt to isolate a peak in the TL curve after preheating, the Width at Half the Height (WHH) of the maximum of the TL was measured for each preheating temperature. Because the WHH is independent of the mass of grains on the disc, normalization is not necessary. Figure 4 shows the values of the full WHH for the natural TL and for the second glow response for the same disc sample. The dose used was less than the ED and this caused the deviation of the two data sets. The left half of the WHH (LWHH) gave more consistent results. After ten minutes at 210 °C, the lower temperature peaks were removed, including the 280 °C peak which was found in the natural sample (figure 5). This experiment was repeated for all three samples and similar results were obtained.

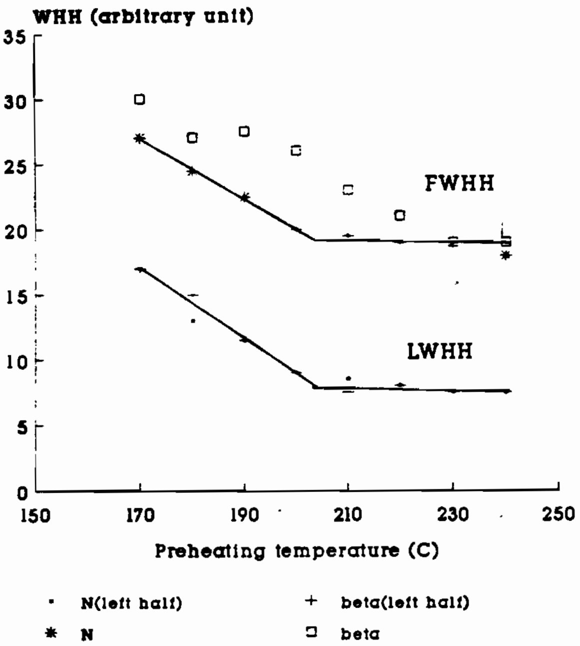

Figure 4.
Sample Z29, plot of WHH (see text) of TL glow curve versus preheating temperature for ten minutes. (*) (•) natural; (□) (+) dosed. The first symbol of each pair refers to FWHH and the second one to LWHH.

Figure 4 indicates that a stable TL signal can be isolated after ten minutes at 210 °C. This was further demonstrated by "quick heat" experiments (figure 6); those gave almost identical results.
In a final experiment, the equivalent dose was obtained for sample Z29 using both preheat procedures, 5 hours at 160 °C and 10 minutes at 220 °C. The values were 272^{+22}_{-32} Gy and 232^{+21}_{-17} Gy respectively. While these two values are mutually consistent, they are substantially less than the value of 480 Gy obtained by TL. This suggests either that a substantial relict TL signal remained at deposition in antiquity, or that there was substantial bleaching of the OSL signal during sample preparation.

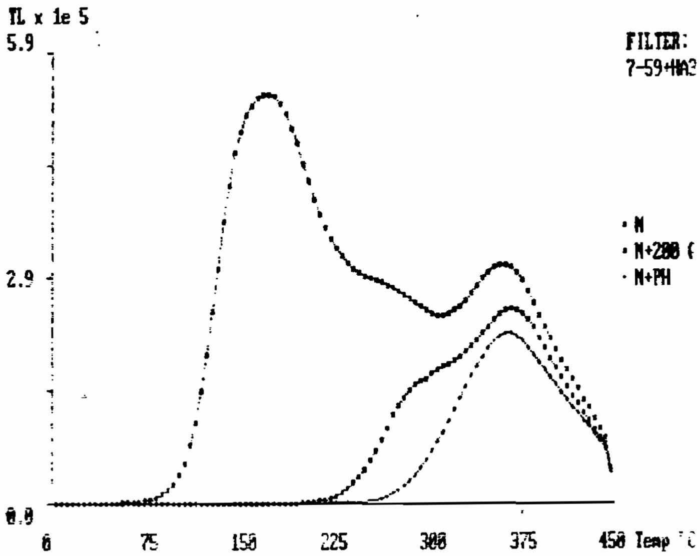

Figure 5.
Typical TL glow curves of samples Z29. (a) Natural+200 Gy dose; (b) Natural and (c) natural + preheating ten minutes at 220 °C.

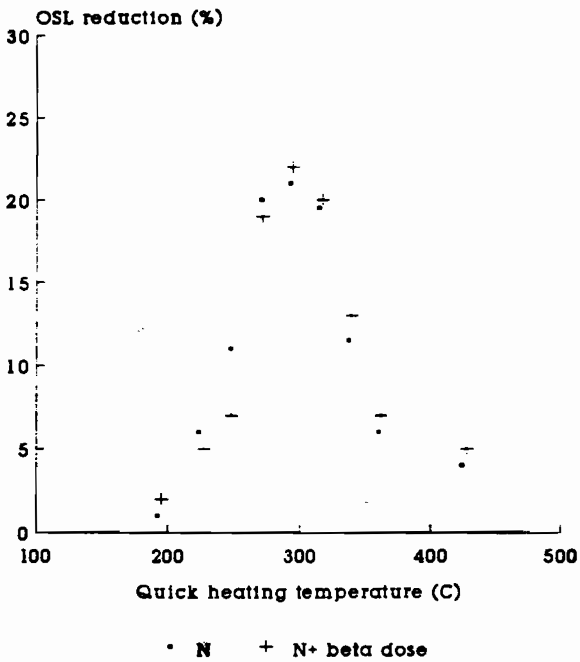

Figure 6.
Sample Z29, OSL reduction (% of whole OSL signal) by each quick heating step, after preheating 10 minutes at 210 °C for natural and N+β dose samples. (100% is the whole OSL signal before quick heating). Beta dose was 200 Gy.

Conclusion

Preheating is needed to remove the thermally unstable signal in optical dating. Combined OSL and TL studies provide an experimental approach to the choice of suitable preheating conditions. Based on results for three K-feldspar separate, preheating for ten minutes at 220 °C or five hours at 160 °C was recommended.

Acknowledgements

I wish to thank Professor M.J.Aitken for his encouragement and supervision and Dr. V. Mejdahl for provision of the samples and EDs measured by TL. Also, thanks to Dr. E. J. Rhodes for useful discussions during this study and Dr.A.G. Wintle for correcting the English and useful comments. Experiments were done at the Research Laboratory for Archaeology, Oxford University while the author was supported for part of the time by a Royal Society Fellowship.

References

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

Huntley, D.J., Godfrey-Smith, D.I. and Thewalt, M.L.W. (1985). Optical dating of sediments. *Nature* **313**, 105-107.

Li, S-H. and Aitken, M.J. (1989). How far can we go with the optical dating of K-feldspar. In synopsis from a workshop on *Long & Short Range Limits in Luminescence Dating*, Oxford, April 1989, Research Laboratory for Archaeology and the History of Art, Oxford University, Occasional Publication No. 9.

Li, S-H. and Wintle, A.G. (1991). A global view of the stability of the luminescence signal from loess. *Quaternary Science Reviews*, (in press).

Mejdahl, V. and Winter-Nielsen, M. (1982). TL dating based on feldspar inclusions. *PACT J*, **6**, 426-437.

Rhodes, E.J. (1988). Methodological considerations in the optical dating of quartz. *Quaternary Science Reviews* **7**, 395-400.

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

PI Reviewers comments (Martin Aitken)

Removal of the unstable component is essential if reliable results are to be obtained by optical dating - as indeed pointed out by Huntley et al (1985). Two points additional to those mentioned by the author are in respect of (i) use of ED in fig. 1, and (ii) the possiblity of non-plateau in fig. 1 because of dose-dependent sensitivity change being progressively stimulated by the preheating. As the author points out in respect of (i) use of (N/N+ß) avoids the need for normalization; but on the other hand if there is progressive change in the degree of non-linearity (of growth of OSL with dose) there may be a good plateau if ED is used, but not with (N/N+ß) - the latter giving an erroneously pessimistic assessment of the efficacy of the pre-heating. Evidently neither of these interfering affects are present with the K-feldspar samples under study.