Ancient TL

www.ancienttl.org · ISSN: 2693-0935

Wright, D., 1978. *Thermoluminescence dating: Apparatus and proceudre for measurment of the thermoluminescence.* Ancient TL 2(1): 2-5. https://doi.org/10.26034/la.atl.1978.004

THERMOLUMINESCENT DATING:
APPARATUS AND PROCEDURE FOR MEASUREMENT OF THERMOLUMINESCENCE

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Introduction

A study of thermoluminescent (TL) dating has recently commenced at Durham. The method of heating the sample of ceramic differs from the direct heating of a nichrome strip commonly employed in other laboratories, and the main purpose of this note is to outline the method and the results it is giving. Another point is that TL results are being obtained while heating the sample in vacuum rather than in a flow of nitrogen, which is the practice in most laboratories.

We have used indirect heating of the hot plate, requiring a current of little more than 3 amp. This results in an inexpensive control unit, as it avoids a heavy current transformer and heavy current control devices. Another advantage is that the thermocouple welded to the hot plate is isolated from the heating circuit. The use of vacuum rather than nitrogen started in the first place because access to this laboratory is rather difficult, and transport of nitrogen cylinders was to be avoided if possible.

Experimental Arrangement

A nickel hot plate is used as in Figure 1, 0.1 mm sheet, 45 mm diameter, with a shallow central recess to locate the 10 mm nickel disc which contains the sample. Another advantage of this system is that the presence of such a recess does not lead to any non-uniformity of temperature, as may occur in a directly heated strip. An 8 ohm helical winding of 30 gauge Kanthal A wire (0.0124", 17 ohm per 36") provides the indirect heating, and is insulated from the hot plate by the pyrophyllite base disc below and a thin mica sheet above. The thermocouple wires are welded separately to the top of the hot plate, one each side of the recess. Three brackets of 0.1 mm nickel sheet support the plate from the central spindle, giving very low heat loss by conduction.

The heating circuit involves a variable ramp generator and comparator, based on a circuit designed by W. L. Paterson, (Rev. Sci. Instr. 46 No. 2, pp. 196-197). An integrator drives a high-gain control element consisting of a darlington pair, which controls the heater input power, derived from a 30 volt D. C. supply. The ramp setting 4 was normally used, giving a heating rate of 5° per second linear with time to about 400°C. Higher ramp settings gave non-linearity before 400° was reached. For example, setting 6 which was sometimes used gave a linear rise of 10°/sec. to 150°C, falling at higher temperatures. These were the highest rates obtainable with this heater winding and supply. Higher rates could no doubt be obtained by re-design, but there us no evidence so far that this is necessary. A temperature cut-off was provided in the circuit, which was set either at 450 or 500°C for pottery samples, or at 320°C when working with phosphors in teflon for TL dosimetry.

The hot plate was mounted in a brass cylinder which could be evacuated using a Speedivac single stage pump 1SC30A. This has produced pressures between 0.2 and 0.4 mm. The cylinder was enclosed in a lead castle so that irradiation of the sample could be carried out in situ when required. The lid to the cylinder, mounted via an o-ring seal, carried the photomultiplier (PM) type 9635 Q/B. The light from

the sample passed through a spectrosil window, which also gave a vacuum seal via an O-ring; then through an OB14 blue filter, an HA3 infra-red absorbing filter, and finally a silica light-collecting cylinder 16 mm long and 35 mm diameter. The multiplier was normally operated at 1200V. A microswitch on the brass lid protected the PM in case it was lifted and exposed to light with the volts applied.

The signal from the PM passed through a pluse amplifier/photon ratemeter (Littlemore Sci. Eng.) to the Y-axis of a Bryans X-Y recorder 25000 A4. The thermocouple wires were connected to the control circuit which gave an output to the X-axis of the recorder.

The disadvantage of the above scheme is the slow cooling rate, particularly in vacuum. The time from 500° to 125°C is 5 minutes, and the total time to 30° is about 15 minutes. This can be accelerated a little by opening to air on falling to 80°, which seems to do no harm, or passing a flow of N_2 at any stage (4 litres/min., O_2 free). As explained, this is avoided if possible. It might be feasible to provide water-cooling for the hot-plate, but the structure is rather fragile, and the whole point of the design is to minimize heat loss, so that this possibility has not so far been seriously considered.

An artificial dose of β-radiation could be provided using a Sr90 1mCi foil source SIC12, originally from Harwell. This was mounted with Prespex, lead and brass protection on a circular brass plate the same size as the lid carrying the PM. This could be removed and replaced by the Sr90 source, which was then 0.6" above the Ni disc when it was in position in the recess in the hot plate. The source was calibrated by exposing phosphor test specimens to the source for various times, the specimens being in position on one of the Ni discs. The phosphors were provided and the calibration carried out by Dr. A. McKinlay of the National Radiological Protection Board at Glasglow. It has so far been assumed that the dose received by the ceramic samples is the same as that received by the phosphor when in the same position on the hot plate.

Comparison of vacuum and nitrogen atmospheres

(a) Wear-side sand. Samples of sand from the river-side at Willington gave a large and complex TL output above 250°C, either as received or after subsequent β-doses. Clear grains were prepared by washing in dilute HCl for 15 min., and etching in HF for 30 min. The geological TL was cleared by heating to 450°C, and standard artificial doses of 10 rad were given. Figure 2 shows the resultant TL in vacuum compared with that in O_2-free nitrogen, flowing at 4 litres per min. The quartz peak is seen at 105°C, and peaks attributable to CaF_2 at 205 and 305°C.

It will be recalled that the thermocouple is welded to the hot plate, so that the sample will be at a lower temperature than that recorded. Figure 2 shows that the temperature lag in vacuum is greater than that in nitrogen, by about 20° at 100° and about 50° at 300°C. This would lead to some difficulty in interpreting conventional TL plots obtained in vacuum, though this could be overcome. There would, however, be no difficulty in interpreting predose results using quartz inclusions, and the height of the 105° quartz peak is the same in vacuum as in N_2. Thus this result indicates that there is no objection to the use of vacuum. Of course, it is appreciated that the lag between vacuum and N_2 would be greater with better vacuum or higher heating rates.

Incidentially, the presence of CaF_2 in the sand had not been expected when the work started, but the conclusion from TL has been confirmed by X-ray analysis.

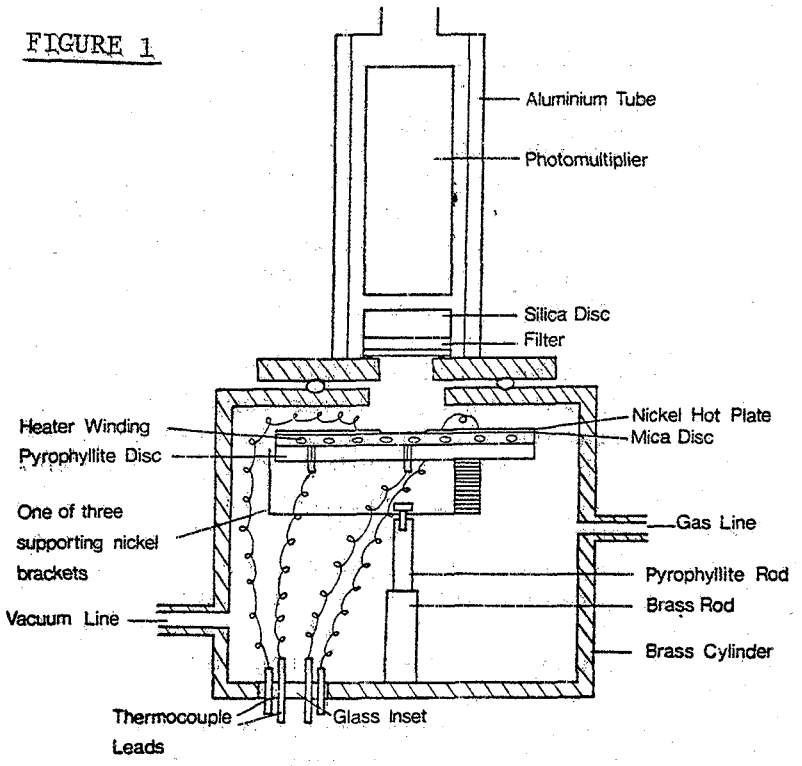
(b) North-east ceramics. Measurements are proceeding on pottery and tiles from two sites in Co. Durham, and on hearth tiles from the York Minster undercroft project. (D. Phillips, 1975, Friends of York Minster 46th Annual Report, pp. 19-27.) Figure 3 shows some typical plots of TL against thermocouple temperature in vacuum obtained from quartz grains from a York Minster sample of Roman tile. Our predose procedure follows Aitken and Murray (1976 Edinburgh Symposium Report), and it is hoped to publish the results of both predose and conventional TL when the self and soil dose-rates have been determined. In the meantime, Figure 3 indicates that the rather slow heating rate nevertheless leads to satisfactory TL plots. The poor vacuum may not be adequate for conventional TL, but is expected to be satisfactory for predose measurements. The archaeological doses so far determined for samples of medieval pottery from the two sites in Durham are in fact of the magnitude expected.

Acknowledgement

The ramp generator was designed and built by P. Bedall, final year undergraduate project in Applied Physics, 1976, aided by P. Friend, Electronics Technician in Applied Physics Department, University of Durham.

[Editor's note: As alluded to in the text, the dose rate from a beta source to a calibration phosphor such as CaF_2 or LiF is not identical to that to a sample of quartz but differs by the ratio of their specific electron stopping powers. The specific electron stopping power is a function of sample composition and beta particle energy and is most conveniently obtained from tables (e. g. M. J. Berger and S. M. Seltzer, Tables of Energy Loses and Ranges of Electrons and Positrons, NASA Report SP-3012, 1964). The variation between most common phosphors is generally less than 10%.]

FIGURE 1



Hot Plate Assembly

Willington Sand:

A Comparison of heating in N_2 and vacuum

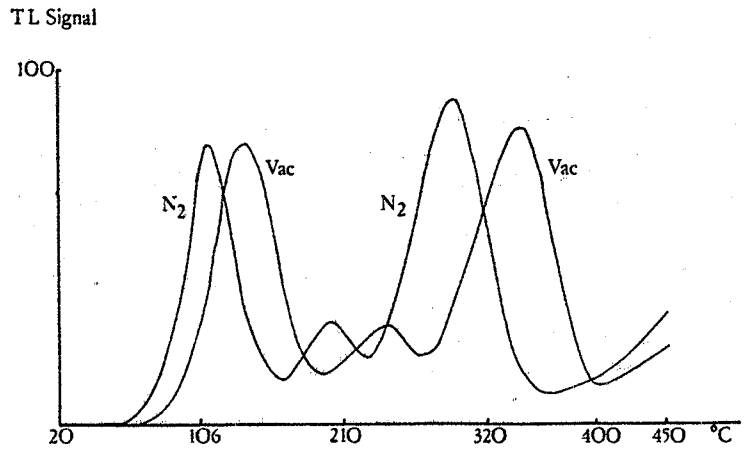


FIGURE 2

TL plots obtained in vacuum using 10 grains of quartz from "York Roman Tile"

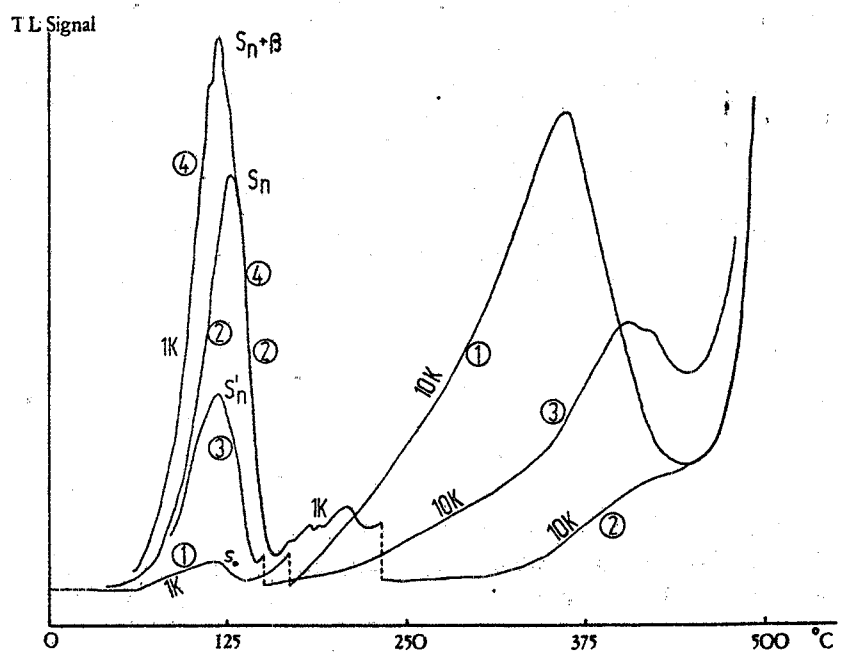


FIGURE 3