

THE APPLICATION OF THERMOLUMINESCENCE DATING TO THE PALAEOLITHIC

by

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ABSTRACT

The aim of this paper is to provide a brief introduction to the thermoluminescence (TL) mechanism and its application to dating of the Palaeolithic. The relevance of the TL signal measured in the laboratory to the archaeological event to be dated is discussed, together in general terms with the types of material that are datable. The stability and saturation of the signal and the bearing of these factors on the upper age limit of the method are also considered. The necessity for *in situ* measurement of radiation levels on any site to be dated is emphasised.

The development, limitations and some examples of the application of TL dating to three materials is then discussed in more detail. These materials are burnt flint, stalagmitic calcite and sediment and are those most commonly used for TL dating the Palaeolithic.

THE MECHANISM

Thermoluminescence (TL) is the light emitted by a crystalline material when it is heated after previously being exposed to radiation. This light is additional to the incandescence produced by heat alone.

In nature, the radiation is alpha, beta and gamma from the decay of uranium, thorium and potassium (^{40}K isotope) both in the sample itself and in the burial environment. In addition there is a small contribution from cosmic rays. When these ionizing radiations pass through matter, some of the free electrons they produce will be trapped at lattice defects if the material is crystalline. Depending on the nature of the defect (trap) the electrons can remain trapped at ambient temperature for long periods of time (millions of years in some cases). When heat is applied the electrons can be given sufficient energy to be released and, if recombination at a luminescence centre occurs, the light known as thermoluminescence is emitted.

"ZEROING THE TIME CLOCK"

The light emitted on first heating an archaeological sample in the laboratory is known as the natural TL. Taking for example pottery, this natural TL in simple terms is proportional

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to three factors: the annual amount of radiation received by the pottery, the effective TL sensitivity of the mineral inclusions to radiation and the time that has elapsed since the pottery was last heated i.e. was fired. In principle therefore the age of the pottery can be determined if the other two factors and the natural TL can be determined. This is equivalent to the age equation:

$$\text{age} = \frac{\text{natural TL}}{(\text{TL per unit of radiation}) \times (\text{annual number of radiation units})}$$

or

$$\text{age} = \frac{\text{archaeological (or palaeo) dose}}{\text{annual radiation dose}}$$

where the archaeological dose (or palaeodose) is a measure of the total radiation received in antiquity, and is derived from the ratio of the natural TL to the effective TL sensitivity to radiation.

For pottery, firing acts as a "zeroing mechanism" that can be thought of as removing the geological TL so that the natural TL measured in the laboratory relates to a specific archaeological event. Temperatures in excess of about 400°C are required and such temperatures are readily reached in a fire. Hence hearth material is datable as demonstrated by HUXTABLE and AITKEN (1977) for baked clay from a fireplace at Lake Mungo. Similarly burnt flint or stone are potential candidates for TL dating. Lava flows should also be datable, but as discussed below stability of the TL signal can be a problem.

Apart from heat, there are two other main zeroing mechanisms: crystal formation and exposure to sunlight. Without a crystal structure, there can be no TL; hence, the formation of a crystal such as calcite acts as a "zeroing mechanism" for TL dating. It is important that stalagmites or flow stones rather than stalactites are dated since the formation of the latter is not readily relatable to any archaeological event. With *in situ* stalagmitic calcite, the relative stratigraphic position of the calcite to the archaeological levels will provide at least a *terminus* result, and in some cases the archaeology may fortuitously be sandwiched between two datable calcite layers.

In some of the early work on TL dating of ocean sediments it was thought that the TL observed was from foraminifera or radiolaria and that the zeroing mechanism was the formation of the shells of these organisms (BOTHNER and JOHNSON, 1969; HUNTLEY and JOHNSON, 1976). In fact sediment particles attached to the shells were producing the TL and these were most likely to have been "zeroed" by the action of sunlight prior to deposition: the extent of zeroing is discussed further below. In recent years the application of TL to the dating of terrestrial sediments has greatly expanded, especially in geology. Loess, in particular, is an obvious sediment for TL dating since it is fine-grained and through aeolian transport will have been exposed to sunlight for a considerable time prior to deposition.

SIGNAL STABILITY AND SATURATION

A zeroing mechanism and association of the TL material with the event to be dated are essential prerequisites of the technique. In addition it is necessary that the TL signals measured in the laboratory have certain characteristics: stability at ambient temperature for time periods well in excess of that to be determined and continuing growth with addition of further radiation dose.

Stability of both the trapped electrons and the activated luminescence centres is required, and two forms of instability exist. One is a function of the properties of the defects themselves and of the storage temperature of the sample, whereas the other, referred to as anomalous fading, is a less predictable phenomenon.

In the laboratory it is possible to determine the characteristics of a trap and predict the mean lifetime of an electron at ambient temperature in that trap. For example, the trap giving rise to the TL signal at about 370°C in figure 1 has a lifetime of approximately 600 Ma (1 Ma = one million years) at ambient temperature (BOWMAN, 1982). Hence the flint, which gave rise to this signal and which was burnt in the Palaeolithic, should be datable. In general terms traps giving rise to TL signals that require laboratory temperatures in excess of about 250°C for their measurement have lifetimes more than adequate for dating the Palaeolithic (see AITKEN, 1985, Table E.1, p. 272). It is less easy to determine the stability of the luminescence centres. If one such centre is dominant in a particular type of material, its lifetime can only be assessed by TL dating known age examples of that material as discussed below for sediments.

More problematic is the phenomenon of anomalous fading, so called because, despite the defect characteristics predicting long-term stability of the TL signal at ambient temperature, extremely rapid loss is observed. WINTLE (1973) found this to be a particular feature of volcanic feldspars and obtained TL ages an order of magnitude lower than expected. Quartz, however, does not seem to be subject to anomalous fading and lava flows have been successfully dated using quartz pebbles found within the lava (GILLOT *et al.*, 1978) or quartz extracted from sediment underlying a lava flow, the heat from which "zeroed" the sediment (HUXTABLE *et al.*, 1978). Similarly BERGER and HUNTLEY (1983) have dated Holocene volcanic ash falls using the fine-grained glass fraction.

Recent work (TEMPLER, 1985) has indicated that it might be possible to circumvent the effects of anomalous fading by storing samples at elevated temperature (*c* 120°C) prior to measurement of the TL signal. This method, however, is yet to be applied to TL dating of volcanic feldspars.

Saturation refers to the stage at which the TL signal no longer increases significantly with the addition of further radiation. This is due to the filling of the traps with time; when most or all of the traps are filled, little or no further growth of the signal can occur. The total radiation dose corresponding to this is a function of the concentration of defects in the crystal. However, the time at which this level of radiation dose is reached depends on the rate at which radiation dose is received; if this is low, saturation will be reached after a longer period of time.

The upper age limit of TL dating is therefore dependent on the stability of the signal, on the characteristics of the TL material and on the annual radiation dose. In consequence it is not possible to attach a single number to this limit, rather it is specific to each application. Nevertheless a few generalisations can be made and these are discussed below in the sections specific to materials.

ANNUAL RADIATION DOSE

Alpha, beta and gamma radiations have very different ranges in matter: approximately 0.03, 3 and 300 mm respectively in typical TL materials. If a fragment of flint weighing a few tens of grammes is considered, these ranges mean that the vast majority of the gamma contribution comes from the burial environment, not from the flint itself. If the outer 2-3 mm of the flint is removed, the alpha and beta contributions will then only be from the flint. Assessment of these is referred to as the internal dosimetry and the gamma plus cosmic contribution is the external, or environmental dosimetry.

Two further considerations are the effect of water and of radon emanation. Water absorbs radiation more than air does, hence the wetter the environment and sample are, the lower is the annual radiation dose received by the TL producing minerals. Radon emanation refers to the escape of the radioactive gas, radon, which is one of the members of the uranium series. Its loss makes the evaluation of the annual radiation dose, in particular the environmental contribution, difficult (see e.g. BOWMAN, 1985).

These and other factors mean that laboratory measurements to assess environmental dosimetry on samples isolated from the bulk of the burial environment may not be representative. It is better to make *in situ* measurements using either a portable gamma ray spectrometer or capsules containing a sensitive TL phosphor (see e.g. AITKEN, 1985). In the latter case the capsules must remain in the levels to be dated for a period of about a year. Without the burial environment information a site cannot be dated by TL, and without *in situ* measurements, errors may be introduced.

Consultation between TL laboratories and archaeologists are therefore essential before samples are submitted for dating so that the best samples are collected, the correct collection methods are employed and *in situ* measurements are arranged.

BURNT FLINT

Dating of other heated stones has received rather less attention than flint, perhaps due to factors such as inhomogeneity and higher levels of internal radioactivity that would restrict the upper age achievable (see below). This section will concentrate on flint, however, examples of TL dating of other stones include sandstone (HUXTABLE *et al.*, 1976; VALLADAS, 1981) and a biotite microgranite (SCHVOERER *et al.*, 1977).

For flint, crazing of the surface is a clear sign of burning, however, the absence of crazing does not necessarily mean the flint has not been adequately heated, since laboratory experiments have shown that the temperatures needed to produce crazing and other very obvious visual changes are in excess of those required to zero the geological TL. Laboratory tests however can readily determine if adequate heating was achieved in antiquity.

The initial work on burnt flint was hampered by non-radiation induced signals (GÖKSU and FREMLIN, 1972) that resulted from crushing in the preparation of samples for TL. These effects were overcome using a slice technique (GÖKSU *et al.*, 1972). This technique has the disadvantage that the optical attenuation characteristics of the flint must be measured to evaluate the effective TL sensitivity to radiation (AITKEN and WINTLE, 1977). It was, however, used to obtain two dates averaging to 230 ± 40 ka for the Lower Palaeolithic site of Terra Amata (WINTLE and AITKEN, 1977; 1 ka = one thousand years). Further disadvantages are the sample size required to facilitate preparation of slices and that the TL properties of a series of slices can be variable (BOWMAN and SEELEY, 1978).

VALLADAS (1978) showed that not all crushed flint exhibited the interfering signals observed in the early work, particularly if acid treatment was used in the sample preparation. This presumably removes unconverted calcium carbonate that can comprise up to 3% of the sample (COWELL and BOWMAN, 1985). Valladas used 100 μm grains for her TL measurements, and other workers (e.g. DANON *et al.*, 1982) have also had success with similar grain sizes. Crushing and deposition of the 1-8 μm grains as frequently used for pottery dating (ZIMMERMAN, 1971) greatly simplifies evaluation of the radiation contribution from alpha particles, and dating programmes employing this sample preparation technique are now being reported (e.g. HUXTABLE, 1982).

The stability of the signal in flint appears in general to provide no problems in dating

back as far as the Lower Palaeolithic at least. However substantial underestimates have been reported (BOWMAN *et al.*, 1982) and investigated (BOWMAN, 1982). Though no clear indication of instability of the signal could be found, equally no other unequivocal cause was identified.

It is interesting to note that where flint and other burnt stones are available on a site, the latter are more likely to be in saturation (e.g. the site of Sclayn reported by AITKEN *et al.*, 1986). Although the typical radiation dose needed to saturate the traps in flint is not high, the internal radioactivity is very low and hence the age limit determined by saturation depends on the level of external (gamma) radiation. For many environments, such as limestone caves, this will also be low, and the samples that are datable will be correspondingly older.

Other difficulties do, however, exist. When a flint is heated it tends to shatter into pieces that are often too small to be dated since the outer 2 mm is removed to simplify the evaluation of the annual dose (see above). Equally, insufficient heating will be a problem. Also low TL sensitivity to radiation can render the natural signal too low to be measurable. Large numbers of datable flints from a single site are consequently rare; the exception being the thirty four dates reported by VALLADAS *et al.* (1986) for Le Moustier. In contrast, only six of the fifty samples investigated from Combe Grenal were datable (BOWMAN and SIEVEKING, 1983). Despite being small in number these remain the only sequence of dates for a site covering a long time span: the interpretation of these dates in relation to the regional chronology is not straightforward as discussed by the authors (see also ASHTON and COOK, 1986, and MELLARS 1986 a,b). Such discussions emphasise that currently there are too few absolute dates for much of the Palaeolithic.

STALAGMITIC CALCITE

The first attempts at TL dating of stalagmitic calcite revealed the problem of interference from spurious luminescences, which were chemically rather than radiation induced. To overcome this problem, samples must be heated in an atmosphere of low oxygen and water vapour concentrations. WINTLE (1975) also showed the importance of careful preparation of samples, recommending a slight acid etch of the calcite grains following crushing of the stalagmite. In further studies (WINTLE, 1977) she examined the stability of the dominant TL glow curve peak, at a temperature of 275°C, and deduced that this signal was adequately stable for dating stalagmites throughout the Quaternary. Stalagmitic calcite suffers, however, from having a low TL intensity relative to other minerals, and BANGERT and HENNIG (1978) emphasised the difficulty of measuring samples with a high level of detrital contamination.

In a study of six stalagmites, WINTLE (1978) compared TL measurements of the archaeological doses with those calculated from uranium series disequilibrium data. Agreement was not encouraging, and several possible explanations were put forward to account for the discrepancies. One suggestion was that TL sensitivity variations within the stalagmites may be spatially correlated with concentrations of the radionuclides emitting short-ranged alpha particles. This possibility was investigated by WALTON and DEBENHAM (1982) who found evidence for anti-correlation in a small number of samples, but the effect was not thought to have major consequences. Meanwhile, spectral studies of calcite TL (DEBENHAM *et al.*, 1982) suggested an alternative explanation for the discrepancies by showing that rejection of an unwanted TL signal, present in newly formed samples, could be achieved by selecting emissions at blue wavelengths. The unwanted signal was thought to originate from limestone detritus embedded in the stalagmites.

This work was followed by a study of twenty seven stalagmites from Pontnewydd Cave, Wales, and Caune de l'Arago, France (DEBENHAM and AITKEN, 1984), in which TL age measurements were compared with uranium disequilibrium dates. Generally good

agreement was found between the two methods, suggesting that TL does provide a useful basis for dating stalagmites. However, the limitations of the method are at present not well defined. In particular, it is not yet clear how serious a limitation is imposed by the requirement for samples to have low levels of clay and limestone detritus. There may also be difficulties at some sites caused by geochemical action on the stalagmites during their burial. Only the application of the method to a wider range of sites can answer these questions.

Additionally, doubt still surrounds the time range over which TL dating can yield reliable dates for stalagmites. Because saturation of the TL does not occur until very high radiation doses, and because annual radiation doses to stalagmites are generally low, the upper age limit is almost certainly set by the lifetime of the signal. The oldest samples dated by DEBENHAM and AITKEN (1984) were 350 ka old, and independent testing of these pieces (DEBENHAM, 1983) has suggested that, at most, 15% of the TL signal may have been lost due to instability over this time. Further testing of older stalagmites is required before the upper age limit of the method can be established. The lower limit is set by the reliability of the assumption that the measured TL signal was at zero in the newly formed stalagmite. This in turn will depend on the level of detrital contamination. At present, dates younger than 30 ka should perhaps not be regarded as accurate to better than about $\pm 50\%$.

Despite these uncertainties, it would appear that TL has the potential for usefully supplementing the uranium series disequilibrium dating technique in the age range 50-300 ka, and possibly for extending the range considerably further.

SEDIMENT

The first attempts to date sediments by TL were carried out in the Soviet Union and in East Europe, and this early work has been reviewed by HÜTT and SMIRNOV (1982) and by WINTLE and HUNTLEY (1982). Interest in the technique in the West was aroused by studies on ocean sediments by WINTLE and HUNTLEY (1980), who found that TL measured palaeodoses increased with depth and suggested that sunlight exposure before deposition had caused a significant reduction (bleaching) of the TL signal. An example of the effect of sunlight on a TL signal from sediment is shown in figure 2. The method was next applied to Devensian loess (WINTLE, 1981), to sand dunes (SINGHVI *et al.*, 1982) and to glacial deposits (HÜTT and SMIRNOV, 1982a). WINTLE (1982) distinguished between a quartz TL peak at 320°C and a feldspar component at lower temperatures. While the first was found to saturate at too low a radiation dose to allow dating of sediments older than 30 ka, the feldspar signal was suspected to be unstable. DEBENHAM and WALTON (1983) studied the spectra of these two components, and showed that a much purer feldspar signal could be obtained by recording the TL at ultra-violet wavelengths. This signal, peaking at 260°C, was found to be removed much more rapidly by sunlight than was the quartz signal. Later investigation (DEBENHAM, 1985) showed that the 260°C signal did not suffer from saturation, but that a long-term instability apparently limited its application to sediments younger than 100 ka. This was revealed by TL dating a series of deposits whose ages, estimated on other evidence, ranged over the past 700 ka. Figure 3, in which the measured TL ages are plotted against their stratigraphic dates, shows severe underestimation of the TL results for older sediments. It was thought that this effect was due to instability of the luminescence centres since no loss of trapped electrons was apparent.

On the basis of these results, therefore, it seemed that neither the quartz nor the feldspar TL components could be used for dating sediments over 100 ka old; the former because of saturation of the TL, and the latter due to inadequate stability. If no other signals are present in TL from sediments, this implied that no dating of the older deposits was possible by TL. Many workers, however, disagree with this conclusion, believing either that the two signals do not invariably suffer these limitations, or that additional signals are available which are free of such problems. Thus, for instance, BRIDGLAND *et al.* (1985) and HUNTLEY *et al.*

(1985) both observe non-saturation of the quartz signal, with the former using it to date loams at Swanscombe to around 220 ka. Alternative signals are proposed by BERGER (1984) and MEJDAHL (1985 a,b) who employ different sample preparation techniques to separate mineral components, and Mejdahl obtains a date of 1.07 Ma for a sample of marine sediment. Similarly, WINTLE (1985) suggests a heat pretreatment in order to separate a more stable feldspar signal; however, the validity of this approach has been questioned (DEBENHAM, 1987).

Thus, there exists at present no consensus of opinion on the age range of sediments that are datable by TL, although it appears that valid ages back to 100 ka are obtainable. There is also some uncertainty about the range of sediment types that may be expected to have received sufficient sunlight exposure at deposition to adequately bleach the TL signal, but indications gained from many investigations (e.g. HUNTLEY, 1985; KRONBERG, 1983; GEMMEL, 1985) are that aeolian and slowly deposited fluvial or lacustrine sediments at least should be datable. For other types of sediment, which may have undergone only partial bleaching at deposition, techniques have been described (MEJDAHL, 1985c) to overcome this problem. Active research continues in all areas of uncertainty.

SUMMARY

The non-pottery materials datable by TL and the limitations of the method have been briefly discussed. Stability and saturation problems should present no problems at least back to, and including, the Middle Palaeolithic. However, consultation between TL laboratories and archaeologists are essential before samples are submitted for dating so that the best samples are collected, the correct collection methods are employed and *in situ* measurements are arranged.

A better absolute chronology is required for the Palaeolithic and by collaboration between archaeologists actively involved in excavations and TL laboratories, this should be achievable.

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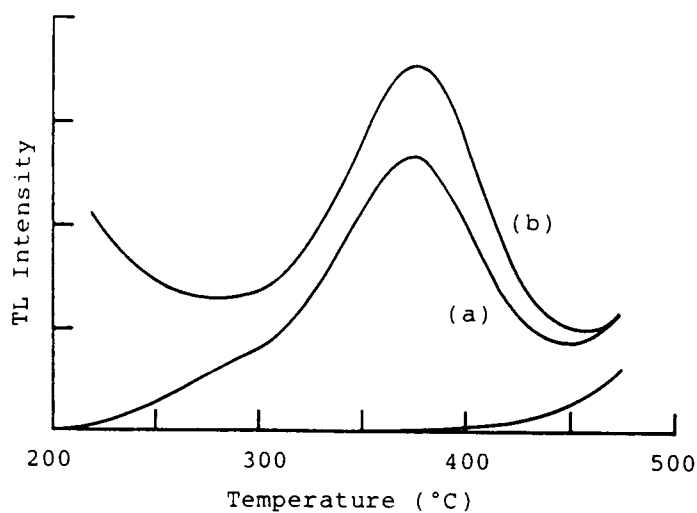


FIGURE 1

Typical TL signals from burnt flint, (a) is the natural TL and (b) is the signal in response to a known laboratory radiation dose (The TL in the blue part of the spectrum has been recorded).

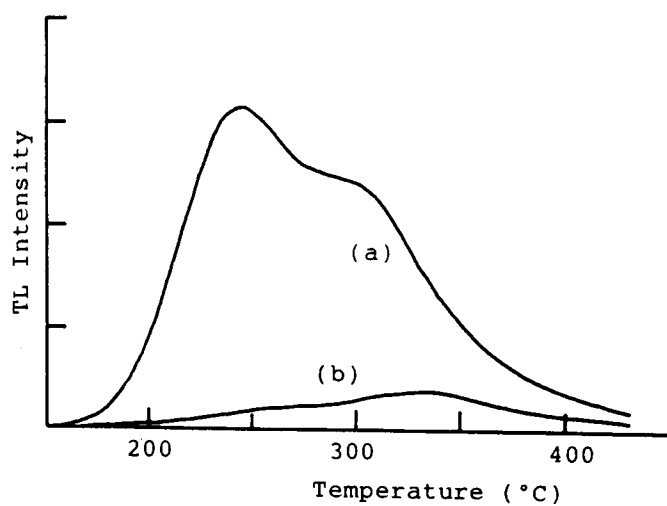


FIGURE 2

TL signals from fine-grains of a sediment from Susterseel, W. Germany, (a) is the natural TL of grains not exposed to light since the time of their deposition and (b) is the signal from grains exposed to sunlight for 16 hours (Only the TL emitted in the near ultra-violet part of the spectrum has been recorded).

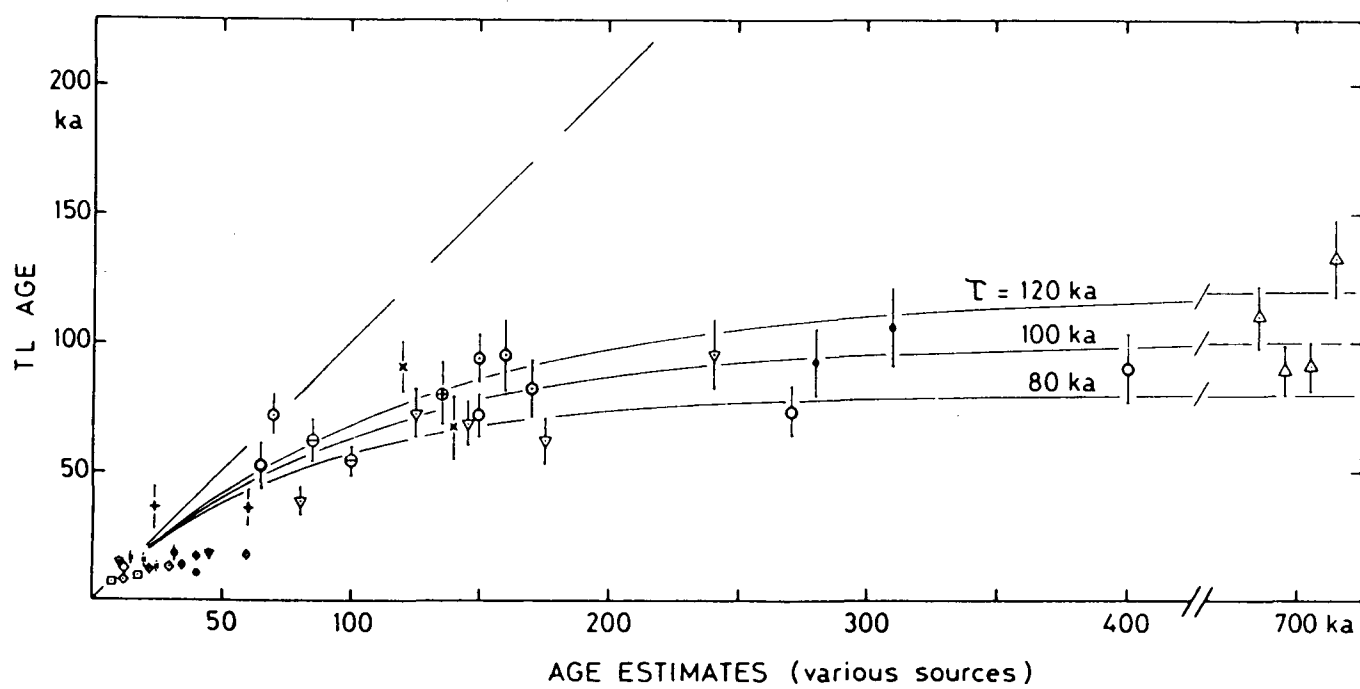


FIGURE 3

TL ages of a series of sediments from N.W. Europe plotted against their estimated ages based on geological evidence. The TL ages of the older sediments are seriously underestimated and appear to approach a limit of around 100 ka.