Thermoluminescence method: outline of history, foundations, principle, possibilities, perspectives and limitations

Stanisław Fedorowicz

Fedorowicz St. Thermoluminescence method: outline of history, foundations, principle, possibilities, perspectives and limitations. *Geologija*. Vilnius. 2003. No. 41. P. 28–35. ISSN 1392–110X.

Thermoluminescence dating (TL) has been applied in natural sciences, particularly in geology and geography, for more than 40 years.

The paper presents an outline of its history and the physical foundations of TL dating. TL research methods have been changing over the years. The additive method was the fist to be used, followed by the total bleach method, regeneration method and optically stimulated luminescence (OSL). These research methods are discussed and schematically presented in diagrams. The paper ends with the presentation of the Gdańsk University TL Laboratory founded and managed by the author since 1981.

Keywords: thermoluminescence (TL), luminescence, optically stimulated luminescence (OSL), equivalent dose (ED), dose rate (Dr)

Received 20 October 2002, accepted 26 November 2002

Stanislaw Fedorowicz, Department of Geomorphology and Quaternary Geology, University of Gdańsk, Dmowskiego 16 A, Poland. E-mail: geosf@univ.gda.pl

INTRODUCTION

The luminescence method is applied in natural sciences, including geology and geography. It enables researchers to determine the age of Quaternary deposits from several thousand to several hundreds of thousand years BP. This time span covers a large part of the Quaternary. The method has the above time limit due to the fact that when laboratories examine deposits younger than thousand years and older than 400,000–500,000 years BP, they find the nonlinear characteristics of the amount of the energy stored. This fact may be a reason for any age determination errors larger than the estimated 15%.

The development of luminescence methods (TL and OSL), as well as their joint application provide additional information on deposition conditions. In 2001, the TL Laboratory at the University of Gdańsk started to examine samples from Lithuania. The first results of a joint research on the Vilkiškės profile have already been published (Gaigalas, Fedorowicz, 2002). At present, further works are conducted on the above-mentioned profile and on other bench mark profiles from Lithuania. After they are completed, their results will be published in the journal “Geologija”. They will certainly give an impetus to the development of chronostratigraphic studies in Lithuania.

HISTORY OF THE METHOD

Attempts to apply thermoluminescence dating (TL) were undertaken in the fifties of the 20th century. The first publication on this method was written by Daniels (Daniels et al., 1953). In 1956, E. Zeller (Zeller, Ronca, 1963) was the first to use the TL method to determine the age of limestone rocks. At the beginning of the sixties, N. Glogler, F. G. Hautermans and H. Stauffer (Glogler et al., 1960) used the TL method to date ceramic products, trying to determine when the product was burnt for the last time. The above experiments were extensively developed in the works of M. J. Aitken and M. Tite (Aitken, Tite, 1964). Scientists who were involved in methodological works at the end of the 60-s aimed at determining the age of ceramic products include J. Bongfinioli (Bongfinioli, 1968), S. J. Fremlin (Fremlin, 1968), J. Kaufold and W. Herr (Kaufold, Herr, 1971), F.R. Siegel, J. E. Vaz, L. Ronca, P. Juciano (Siegel et al., 1968). They conducted extensive research to measure TL in clay materials. They found that the burning process reduces the energy stored in the sample to the background level. Observing the light of samples due to thermoluminescence, E. Zeller and L. Ronca (Zeller, Ronca, 1964) noticed that maximum light was obtained at a temperature of 230 °C. They found that, both in archaeological and geological samples, the temperature peak changes together with the energy stored in the sample. When J. K. Kaul, A. K. Bhattacharya, and P. Tolpadi (Kaul et al., 1970) were studying thermoluminescence in quartz, they came to the conclusion that quartz is the most promising mineral for specifying the absolute age of ceramic products. M. H. Bothner and N. M. Johnson (Bothner, Johnson, 1969) conducted research and dating of deep-sea cores.

The Ukrainian scientist V. Shelkoplyas is regarded as the pioneer of TL applications for geological purposes (Shelkoplyas, Morozov, 1965).

The TL method has been used in Poland since the eighties of the 20th century. At that time four TL laboratories were opened successively at the University of Warsaw, University of Lublin (Maria Curie-Skłodowska University), University of Gdańsk and the Silesian Technical University in Gliwice.

The TL method developed further in the eighties and in the nineties, when new measurement methods were created.

A related method was introduced in 1985. It was called OSL (Optically Stimulated Luminescence) and was authored by Huntley, Godfrey–Smith and Thewalt (Aitken, 1998).

Luminescence methods are based on fundamentals of physics and physical phenomena.

Luminescence is a physical phenomenon consisting in the emission of light by bodies. It results from the return to the ground state of molecules or atoms activated to higher electron states. A number of luminescence types are known. One of them is thermoluminescence (TL). Thermoluminescence is induced by heating a substance to a temperature of *ca.* 500 °C.

BASICS OF LUMINESCENCE DATING

U-238, U-235 and Th-232 are the main radiation components in the crust of the earth and at the same time they form the beginning of the radio-

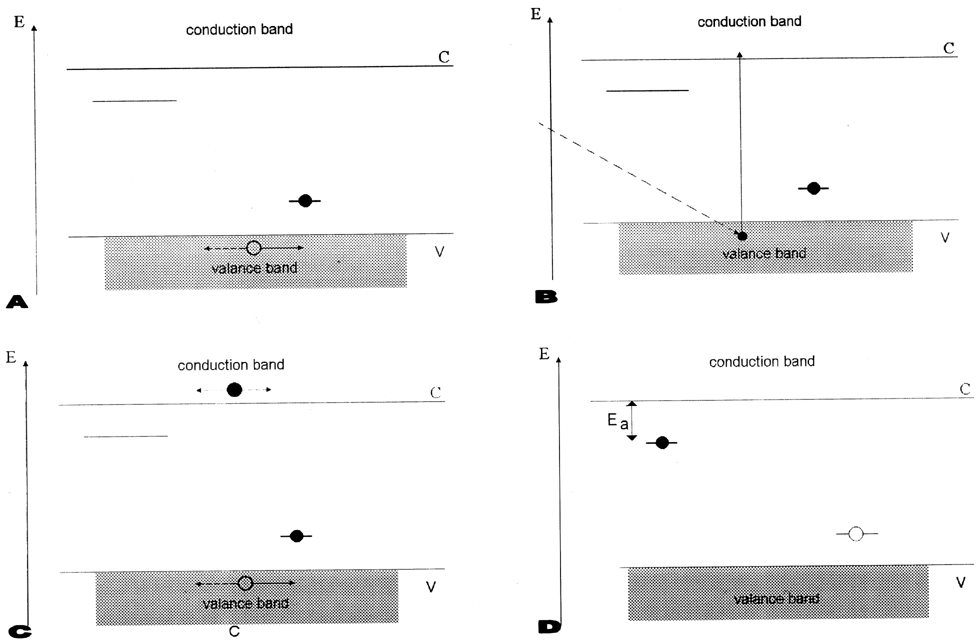

Fig. 1. Simplified structure of energy levels in a crystal with ionising radiation energy absorption and storage processes shown. *A* – initial state (equilibrium state). *B* – ionising radiation delivers energy to an electron in the valence band and drives it to the conduction band. *C* – a hole in the valence band and an electron in the conduction band move freely through the crystal lattice. *D* – final state: the electron has been trapped in the trap center and the hole in the recombination center. Legend: *E* – electron energy, *C* – conduction band, *V* – valence band, *Ea* – electron activation energy (trap depth with relation to the bottom of conduction band) (Bluszcz, 2000)

1 pav. Energijos lygių kristale supaprastinta struktūra su jonizuojančios radiacijos energijos absorbcijos ir kaupimo procesų išraiška. *A* – pradinė (pusiausvyros) būklė, *B* – jonizuojančios radiacijos energijos virtimas elektronų valentinėje juostelėje ir perėjimas į laidų sluoksnį, *C* – skylė valentiniame sluoksnyje ir elektronas laidžiame sluoksnyje juda laisvai per kristalo gardelę, *D* – galutinė būklė: elektronas patenka į gaudyklės centrą ir skylė užsitraukia. Sutartiniai ženklai: *E* – elektrono energija, *C* – laidus sluoksnis, *V* – valentinis sluoksnis, *Ea* – elektrono aktyvacijos centras (gaudyklės gylis, priklausantis nuo laidaus sluoksnio dugno padėties) (Bluszcz, 2000)

active series. As a result of natural radioactive decay, tens of radioisotopes and isotope K-40 come into being in the crust of the earth. This radiation produces energy that is absorbed by all the substances on the earth. The energy of natural radiation is measurable in some substances by physical methods.

The measure of energy absorbed by a given substance is called an absorbed radiation dose. It is the ratio of radiation energy in a specific volume to its mass. It is expressed in grays (1 Gy = 1J/ 1 kg).

The rate of radiation energy absorption is called a dose rate and is expressed in 1 Gy/ 1 s. As regards TL measurements, they are calculated in Gy/ thousand years (Gy/ka).

According to the method assumptions, ionising radiation intensity and dose rate are constant values due to a huge, billion-year period of partial isotope decay which gives origin to natural radioactive series.

The absorbed radiation dose (D) is directly proportional to the dose rate (Dr) and time (t):

$$D = Dr\ t.$$

The absorbed energy is almost completely dissipated in the form of heat. A small part of it can be stored in the crystal lattice.

In real crystal, in the forbidden band between the valence band and the conduction band, there are local energy states connected with various defects of the crystal lattice (Bluszcz, 2000) (Fig. 1).

Some of them may have a form of traps catching electrons from the conduction band and keeping them for a long time until they receive activation energy for the conduction band. The state of a crystal in which part of or all the traps are filled with caught electrons is marked by an energy surplus compared to the ground state (Bluszcz, 2000).

Such a surplus may be released by heating or lighting. This way of energy liberation forms a basis for the two luminescence methods: thermoluminescence (TL) and optically stimulated luminescence (OSL).

The both methods are applied for dating geological deposits and archaeological structures.

AGE OF QUATERNARY DEPOSITS

The age of Quaternary deposits may be determined both via TL and OSL.

Prerequisites for determining the age of deposits include exposure to solar radiation in the past and heating of mineral grains.

Those two factors induce emptying of traps and removal of any luminescence stored earlier. This process is called optical bleaching or thermal bleaching.

Thermal bleaching up to the temperature of *ca*. 500 °C results in a total emptying of traps. Optical bleaching does not cause such disappearance of electrons from traps. Long optical bleaching (exposure to solar radiation or ultraviolet lamp UV radiation) does not reduce the energy stored in grains to zero. Grains retain some energy which may not be get rid of. This residue is called residual thermoluminescence.

As regards luminescence methods, the age (T) is determined according to the following formula:

$$T = \frac{ED}{Dr(ef)},$$

where ED is the equivalent dose and Dr (ef) is the effective annual dose.

Analogous formulae are applied in TL and OSL methods.

TL METHOD

Thermoluminescence is light emitted by crystalline, natural and artificial substances when they are heated to a temperature of *ca*. 500 °C. When a substance is heated to this temperature, electrons leave traps and return to the valence band. This phase is accompanied by emission of light. As a result, the glow curve is measured (Fig. 2). It is a ratio of TL intensity to temperature. The curve differs depending on materials.

The energy released as glow (thermoluminescence) is proportional to the energy stored in the crystal. The amount of thermoluminescence is measurable. It is possible to specify both the absorbed radiation dose and the time when it was absorbed.

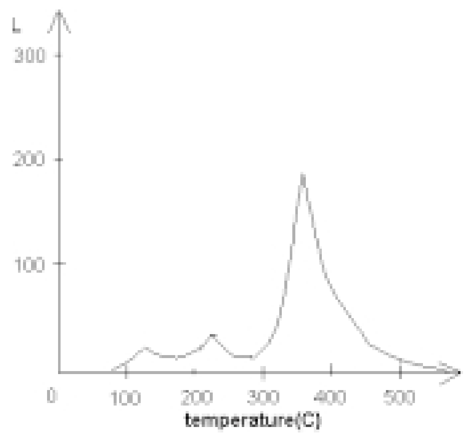

Fig. 2. Example of TL glow curve measured for quartz gains
2 pav. Kvarco grūdelių įkaitinimo TL kreivės pavyzdys

OSL METHOD

OSL (Optically Stimulated Luminescence) is luminescence that takes place as a result of material lighting with the light of appropriately long waves.

Like the TL spectrum, the OSL spectrum is unique for a given material. A scheme of optical activation process is presented in Fig. 3.

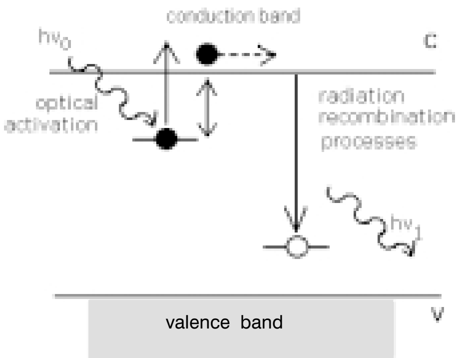

Fig. 3. Diagram showing optical activation and radiation recombination processes leading to OSL emission (Bluszcz, 2000)
3 pav. OSL emisiją lydinčios optinės aktyvacijos ir radiacijos sukeltų procesų diagrama (Bluszcz, 2000)

The intensity of luminescence decreases exponentially to the time of stimulation. This process presents shine-down curves (Fig. 4).

The amount of luminescence emitted as a result of optical activation is proportional to the number of carriers stored in traps. Thus, it is possible to determine the ionising radiation dose absorbed in the material and hence the time when it was absorbed.

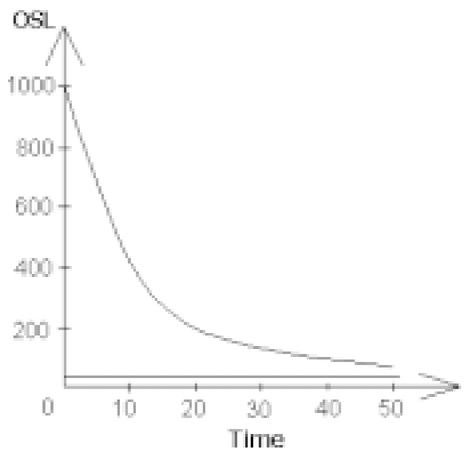

Fig. 4. Example of OSL, shine-down curve from quartz grains excited with green light of 514 nm wavelength (Bluszcz 2000)
4 pav. Kvarco grūdelių švytėjimo, sukelto žalios šviesos 514 nm bangų ilgio, žemėjančios kreivės OSL pavyzdys (Bluszcz, 2000)

The OSL method significantly changed the measurement of a dose absorbed in the deposit. Luminescence accompanying light-stimulated bleaching is recorded directly. The OSL luminescence spectrum is moved towards shorter waves compared to stimulating radiation. Different stimulated lengths of waves are applied for respective minerals.

ED determination is simpler in the OSL method, and the time required to determine ED is much shorter than in the TL method.

DETERMINATION OF EQUIVALENT DOSE (ED)

A dose absorbed by mineral grains is determined using laboratory means by comparing the natural luminescence of grains extracted from the deposit to the luminescence induced by absorption of a known beta or gamma radiation dose in the laboratory conditions.

There are several methods of equivalent dose calculation.

A. Additive method

This method allows for the determination of ED by way of extrapolation. Grains extracted from the deposit are irradiated with specific doses. The amount of ED is extrapolated on the extension of a function made up of measurement points. The resulting curve may be a linear or exponential function (Fig. 5). This method is limited, because it may be applied only if initial thermoluminescence is zero; only burnt materials may have this value.

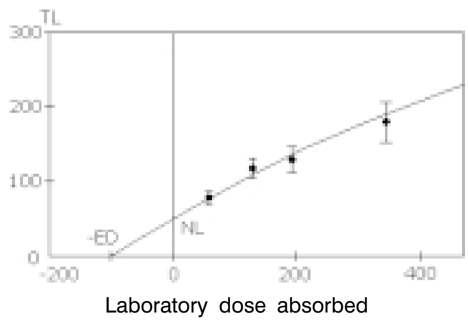

Fig. 5. Illustration of the additive method of equivalent dose determination
5 pav. Ekvivalentinės dozės nustatymo įterpimo metodu pavyzdys

B. Total bleach method

Separated grains are subjected to a long exposure to light (bleaching). It causes a reduction of natural luminescence to the level of residual thermoluminescence (TLo). ED is reduced (Fig. 6). This method may be applied for deposits formed under long-

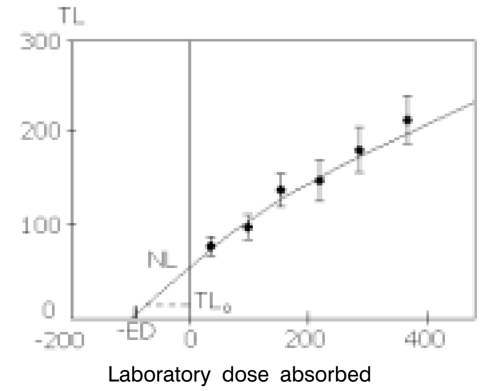

Fig. 6. Illustration of the total bleach method of equivalent dose determination
6 pav. Ekvivalentinės dozės nustatymo visiško išblukinimo metodu pavyzdys

-term exposure to solar radiation (*e.g.*, dune sands or Aeolian sands).

C. Regeneration method

Separated quartz grains are first bleached and then irradiated with specific doses of gamma or beta radiation. And this is how the increase of signal may be regenerated with the absorbed dose (Fig. 7). A certain modification of the regeneration method is achieved by the simultaneous application of the regeneration method and the additive method, which jointly also check the sensitivity of grains. Analysis correctness is verified by the plateau test.

The plateau test is an ED diagram from the glow temperature (Fig. 8). It allows for a correct determination of the initial TL value for a deposit. The diagram is made on the basis of the glow curve measured. ED values are minor at first; then they grow and stabilise above 300 °C to finally reach a constant value (plateau). Further calculations should be based on the ED as read out from the diagram. Another method to verify the correctness of analysis is to examine quartz and feldspar at the same

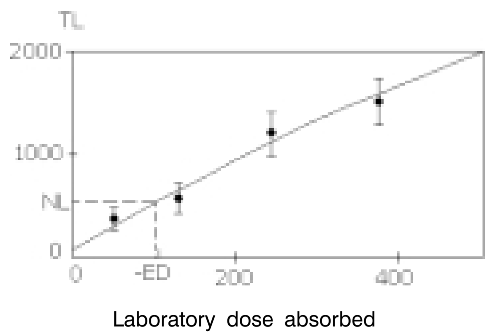

Fig. 7. Illustration of the regeneration method
7 pav. Atstatomojo metodo pavyzdys

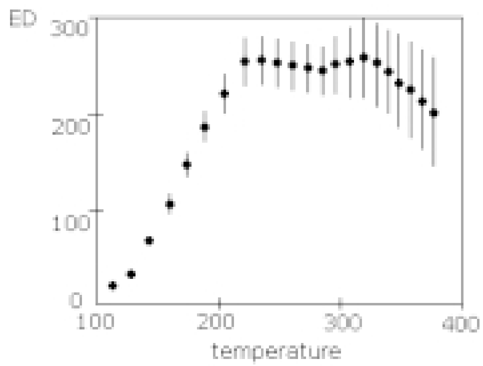

Fig. 8. Example of ED dependence on glow curve temperature exhibiting a characteristic plateau
8 pav. ED priklausomybės nuo įkaitinimo temperatūros plokščiaviršūnės kreivės pavyzdys

time. The deposit may often contain quartz and feldspar minerals. Both minerals show different susceptibility to bleaching (feldspar grains lose thermoluminescence at a faster rate). Both minerals should be bleached in the same conditions, and the time of bleaching should be selected so as to ensure that its effect is the same for the both minerals.

DOSE RATE MEASUREMENTS (DR)

There are several methods to calculate the dose rate. Most popular are TL dosimetry and gamma radiation spectrometry.

In respect of TL dosimetry, special dosimeters made of LiF, CaF2, Al2O3 are placed directly in the deposit to be examined for a precisely defined period of time (*e.g.*, a year).

Gamma radiation spectrometry is based on the scintillating spectrometer. It calculates the dose rate of alpha, beta and gamma radiation from radioactive isotopes U, Th, K. A number of calculation formulae are used to determine dose rates for respective radiation types.

The total effective dose rate is a sum of all types of doses and cosmic radiation dose.

Spectrometric measurement of dose rate is conducted on a dried sample. Calculations are based on formulae that take account of sample humidity (Aitken, Xie, 1985).

Important information for TL and OSL users

The following deposits may be used for TL and OSL dating:
- Aeolian deposits (loess and dune sands)
- sand deposits (beach sands and fluvial sands)
- calcite dripstone.

The following materials are not useful for dating:
- fluvioglacial sands

– glacial till and boulder clay
– landslide deposits.

The latter's lack of application results from the fact that during their deposition mineral grains were not exposed to solar radiation. Some research was conducted to date those deposits but its results are questionable.

In the case of such deposits, dating results should be treated as an indicator, not as an absolute date. Some methodological research was carried out to determine how much of grain energy decreases during transport and how the freezing and defreezing processes impact energy decrease (Fedorowicz, 1998).

The way how the method's usefulness is evaluated by its users was analysed among those who received TL dating results from the Gdańsk University laboratory in 1998.

The above finding was confirmed by the users: the best results were obtained for Aeolian deposits, while the worst for boulder clays (Fedorowicz, 1998).

Furthermore, the following information concerning the collection of samples for luminescence dating may be vital for method users:

– samples should be collected from geologically well-identified profiles
– the researcher should avoid collecting single samples (it is recommended to collect a larger number of samples in the vertical or horizontal profile)
– better samples are received from an open pit than from drilling (possibility of mixing with foreign material)
– when samples are to be collected from an exposure, it is necessary to remove the external layer of the deposit to a depth of a dozen or so centimetres
– the deposit collected should be well protected against exposure to solar radiation (black packaging).

TL Laboratory at the University of Gdańsk

The Gdańsk Laboratory has been operating since 1981. It was created from scratch by the author of this article. Since that time we have already replaced the equipment that was installed 20 years ago. That equipment was completely used up. At present the Laboratory has the following equipment at its disposal:

– two spectrometers (MAZAR and TUKAN)
– reader-analyser (RA 770A).

TL MEASUREMENT METHODOLOGY

As regards a deposit obtained for examination, first its humidity is measured and next it is dried at room temperature.

Dose rate measurement (Dr)

A dried deposit undergoes spectrometric measurement of U, Th, K. The deposit is put into a plastic Marinelli-type container with a volume of 0.5 cu. dm or 1.5 cu. dm (depending on the volume of the sample to be examined) and placed in the protective chamber of one of the two spectrometers. Twenty measurements of 2,000 s each are conducted. The values obtained with U-238, Th-232 and K-40 are calculated into rate doses for alpha, beta and gamma radiation and adjusted by deposit humidity as measured earlier. A cosmic radiation dose (dc) is added to such dose rates of alpha, beta and gamma radiation. This dose decreases with the depth from 0.15 Gy/ka on the land surface to 0 at a depth of ca. 8 m. The Dr measurement error range is *ca.* 3%.

Equivalent dose measurement (ED)

A fraction of 80–100-micrometer grains is separated from the entire sample. Grains separated with the sieve method undergo initial extraction. The aim of initial extraction is to remove the external layer from quartz grains and to clean the external surface of grains. Grains are treated with 10% HCl and HF acids for 1 hour. Samples are next washed with distilled water and dried at room temperature. The dried sample weighs *ca.* 2 g. Then it is divided into a number of portions. All the portions of extracted grains will be used to measure ED by the regeneration method.

The first portion is used to measure natural thermoluminescence (NTL), while the remaining part of extracted grains is exposed to ultraviolet lamp (UV). During one day the UV lamp reduces the stored energy of grains to the lowest level, *i.e.* the so-called residual thermoluminescence (TLo). Thus zeroed material is divided into a number (at least 5) of portions. The first portion is used for the measurement of the above-mentioned residual thermoluminescence, while the remaining portions are exposed to gamma rays from a cobalt bomb of the parameters ensuring that they regenerate the energy stored when the sample was in the bed.

Ten to 10–20 diagrams of the glow curve are made for each portion of extracted grains. The plateau test is also conducted for verification purposes and is also checked if the sample is not in saturated condition. The error range of ED measurements is estimated at 10%.

ED measurements are made with the use of 770 A a reader-analyser. Samples are heated up to a temperature of 500 °C at a heatup rate of 8 degrees/s.

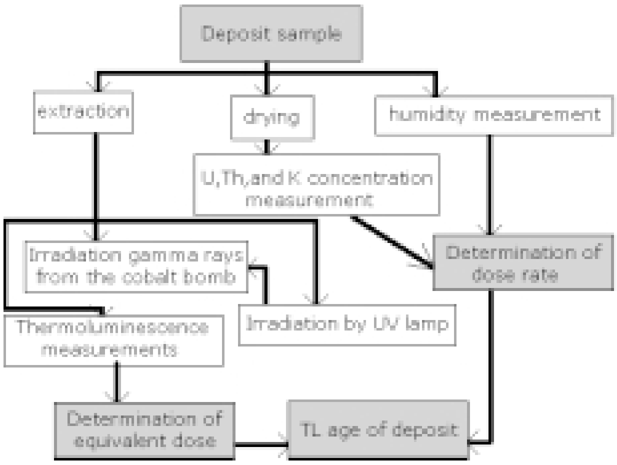

Fig. 9. A diagram showing the dating by thermoluminescence measurement
9 pav. Termoliuminescenciniu matavimu paremto datavimo diagrama

A scheme of dating is presented in Fig. 9.

The TL age is a quotient of ED and Dr. The error range of the method is estimated at 15%.

PERSPECTIVES

The Gdańsk Laboratory has conducted 5,500 datings during its existence. The rest of Polish laboratories carried out a similar number of analyses. Therefore, nearly 20,000 datings were made in Poland; this number is still much higher in the world. As far as Poland is concerned, some of these analyses were made for geological purposes. The largest numbers of dates were applied for compiling detailed geological maps of Poland. Dates were also used in numerous geological and geographical studies.

All the Polish laboratories use different preparations for dating, research methods, applications of measuring devices. This fact often explains the discrepancy of the results. Only a few interlaboratory comparative works have been undertaken. This fact does not facilitate comparison and use of results in joint studies. It would be perfect if such interlaboratory collaboration were established.

Luminescence dating is also influenced by the development of new research methods. It is important to correlate radiometric methods which are often applied in profiles (TL, OSL, ESR, C-14).

Each result of dating provides additional information; conclusions should be drawn from numerous datings, however, with regard to assumptions and possibilities of the methods applied. It should be borne in mind that before collecting the samples, results of earlier research and geological and botanical surveys should be first studied, and not *vice versa*.

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Stanisławas Fedorowiczius

TERMOLIUMINESCENCIJOS METODAS: ISTORINĖ APŽVALGA, PAGRINDAI IR PRINCIPAI, GALIMYBĖS, PERSPEKTYVOS IR RIBOTUMAS

S a n t r a u k a

Termoliuminescencinis datavimas (TL) naudojamas gamtos moksluose – visų pirma geologijoje ir geografijoje jau daugiau negu keturiasdešimt metų. Straipsnyje apžvelgiama metodo atsiradimo istorija ir TL datavimo fizikiniai pagrindai. Termoliuminescencijos matavimo metodai laikui bėgant keitėsi. Pirmiausia naudotas adityvinis metodas, vėliau – visiško išbalinimo, atkūrimo ir optiškai stimuliuotos liuminescencijos (OSL). Šiuos metodus autorius nagrinėja ir schematizuoja diagramose. Autorius atstovauja Gdansko universiteto laboratorijai, kurią pats sukūrė ir joje dirba nuo 1981 metų.

Станислав Федорович

ТЕРМОЛЮМИНЕСЦЕНТНЫЙ МЕТОД: ИСТОРИЧЕСКИЙ ОЧЕРК, ОСНОВЫ И ПРИНЦИПЫ, ВОЗМОЖНОСТИ, ПЕРСПЕКТИВЫ И ОГРАНИЧЕНИЯ

Р е з ю м е

Термолюминесцентные датирования (TL) используются в естественных науках, прежде всего в геологии и географии, уже сорок лет. В статье представлены исторический очерк метода, а также физические основы датирования TL.

Методы измерения люминесценции менялись с течением времени. Первый – метод аддитивный, следующие методы – совершенного отбеливания, воспроизведения и метод оптически стимулированной люминесценции (OSL). Автором настоящей статьи эти методы и обсуждаются, а также схематизированы на диаграммах.

Автор представляет лабораторию Гданьского университета, которую основал и с 1981 г. проводит в ней исследовательские работы.