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Review: Theory of Thermoluminescence & Related by Reuven Chen (Author), Stephen W S Mckeever

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Abstract: Thermoluminescence is a phenomenon observed in some materials when they are exposed to ionizing radiation and subsequently heated, thereby releasing light energy, as a result of the transition of electrons trapped at or near defect centers from metastable states to the conduction band. Thereafter, the relaxed electrons recombine with hole vacancies in defect centers associated with an allowed energy gap between the conduction band and the defect center. The light emitted is of varying magnitudes at different temperatures as some deep trap metastable levels are sequentially filled, emptied and emptied again during the heating process. The sequence of detrapping and recombination topography is a function of trap depth and energy gap size. The presence of trap levels within the forbidden energy gap of insulators or semiconductors, are associated with defect centers which arise from impurity ions or vacancies, whether stoichiometric or non-stoichiometric. The TL response has been used to investigate the nature of defect centers and therefore defect center chemistry in a wide variety of materials including those used as insulators in semiconductor and electronic devices, luminescent materials, phosphors, dosimeters, catalysts, thermoelectric materials, pigments, high-temperature superconductors and phase change materials.

Introduction: The term thermoluminescence is a collective term that describes the luminescence phenomenon induced by heating a material which had previously absorbed ionizing radiation either directly or indirectly through the ionization of impurities such as rare earth ions. TL has been successfully used for information storage, as in photoluminescent color centers, called radio-photoluminescent materials, book-betting, dating of archeological finds, environmental monitoring, beta dosimetry and nuclear waste safety, to mention only a few applications.

Historical Background

Thermoluminescence is by no means a contemporary area of research in solid-state physics. The first observations of the phenomenon in materials were performed in the second half of the 19th Century. As always happens in the infancy of new areas of knowledge, these observations were not followed by a more systematic study of the observed effects. It was only in the second third of the 20th Century that interest in exploring in deeper detail the origin of the effects observed in the beginning was taken up. From that moment on, a long sequence of investigations developed the field to a level of sophistication that made TL evolve into a classical character in data storage phenomena in bulk solids.

The first reports on TL were made in crystals, particularly in potassium chloride and sodium chloride. These crystals exhibited intense emissions even in the spectral region of blue, something hardly observable today for almost any TL material. Even so, the scientific community at the time disregarded the results and did not follow them through, mainly due to an apparent lack of practical applications for such an interesting phenomenon. In those early days, it was already clear that some traps-for charge carriers-created during the sample's exposure to ionizing radiation (mainly soft X-rays) were complex KCl crystals with 2- or 4-fold coordinated Al or Na ions, and probably holes trapped at these sites.

Fundamental Concepts

1. Definition and Mechanism

Thermoluminescence (TL) is a type of solid-state luminescence in which the emitted light results from the thermal stimulation of a solid that has previously stored energy through one or more stimulation of ionizing radiation. TL can be also explained as a consequence of consecutive steps: the first one is the trap filling; the second one is the thermal stimulation; and the last one is the light emission. The trap filling can be associated to the electron or hole excitation from the valence band to a localized state within the forbidden band, to a band level or to a conduction band level. The thermal stimulation must be understood as the release of the electron or hole from the localized state to the valence or conduction bands respectively. The light emission occurs when the electron or hole recombines with a partner of opposite charge, after going to a localized level that is close to the conduction or valence band, respectively.

The main features of the two ionization processes (trap filling and thermal stimulation) allow to expect that the TL phenomenon can be observed in any class of solid-state compounds, without any special requirement on their thermal, structural or electronic properties. This compound can be at room temperature or at a very low temperature when the ionizing radiation stimulates the trap filling. In spite of these two features, at present, TL can be only observed in a small part of the solid-state compounds which until the present contain mainly insulators with a high resistance, the most representative being the alkali halides and silicate crystals doped with metal ions.

2. Types of Thermoluminescence

The TL glow curves of most of the samples considered to be TL solids exhibit structure and are composed of several glow peaks overlapped or not. However, in some very thin samples of highly translucent crystals, the TL glow curves exhibit only one glow peak. These particular samples exhibit the unique TL behavior, at present termed as "one peak TL". As it can be seen from the citations above, this type of TL group is not a minority. In fact, considering all the cited words literally, it is clear that the "one peak TL" group exhibits a qualitatively different TL phenomenon. Due to this unique "one peak TL" characteristic, it appears to be worthwhile to review the main aspects of this TL group in order to have a clearer picture of TL of real solids.

Definition and Mechanism

Thermoluminescence (TL) is the phenomenon of light emission as a result of temperature increase of a luminescent material after it is irradiated. TL is found in many types of materials, but only some of them are suitable for dating purposes, namely, natural minerals. To be used for dating, a natural material must undergo irradiation with ionizing radiation and then must be subsequently heated in laboratory-controlled conditions to read the TL signal. For example, minerals in the sediment of a river bed or of a cave, which are regularly irradiated by cosmic rays and by terrestrial radiations from uranium and thorium and their daughter nuclides in the soil and the ceiling of a cave, have TL signals that can be read and used for dating.

TL was discovered in 1914 by two scientists, who found that mineral grains would emit light when heated; however, the phenomenon received limited attention until the 1950s, when several researchers independently established that there are fast components of the observed TL signals and suggested the use of TL for dating purposes. Three important developments in TL research during the 1970s have maintained TL as an important tool for archaeodating: the introduction of infrared detectors to make possible the absolute dating of TL signals on a days-to-month time scale for short burial durations; the introduction of various experimental procedures to give accurate ages for fine-grained quartz; and the introduction of thermal transfer techniques to reduce age errors for coarse-grained feldspar. TL at the present is used for age dating of ancient ceramics and hearths, as well as for determining burial ages of sediments using sedimentary quartz and feldspar minerals.

Types of Thermoluminescence

Investigations of the dyes and samples inside thermoluminescence materials are possible due to their luminescence spectral properties and their amplitude-temperature curves. Different color centers of thermoluminescence materials have specific emission bands related to their luminescence centers. The investigation of color center presence and identification analysis can be achieved through the analysis of emission band positions, shape details, amplitudes, ratios of emission bands, and effect of sample contamination. A common strategy for thermoluminescence emissions used for needled laser determination of its approximate content made of carbonization of the organic laser dye solvent residue and its employ of thermoluminescence detection with reflected laser beam during physical cooling of thermoluminescence.

According to the spatial origin of luminescence processes, common thermoluminescence can be classified to normal or intrinsic thermoluminescence, impurity or extrinsic thermoluminescence, impurity enhanced thermoluminescence, impurity band or resonance thermoluminescence, and laser thermoluminescence. In addition to their respective involvement in the visible spectral region, internally located condensate vibronic states of luminescence centers can be excited at room temperature with frequency-doubled laser beam. Thermoluminescence chronologically started first or they can intersect the laser signal profile during excitation, respectively, with lower or higher standard delays. Following the observable features of the timer sensor behavior to laser pulse, laser seconds, or microwave seconds and thermoluminescence countrates investigation, the appropriate thermoluminescence naturals can be determined and applied for ecophysiological forensic investigations, for example.

MATERIALS AND TECHNIQUES

1. Thermoluminescent Materials

The study of thermoluminescence (TL) began by the emission of light due to the heating of solids that absorbed natural radiation. The discovery of the emission of light by crystalline salts doped with the valence-1 element stimulated numerous investigations of their photoluminescence and TL properties. Alkali halides doped with activators such as the transition metals and halogens emit visible light when heated, and were used over many decades for scintillation detection of low energy ionizing radiation, and in various other applications such as light sources and optical devices. In this book we restrict ourselves to the TL effect, and initially to these early studies of TL salts.

Several decades later, TL was reported in silicate and borate glasses, which comprise a variety of oxides, especially SiO2 and alkaline earth oxides, molten together and cooled to form amorphous solids. The detection of TL in quartz sand gave birth to the application of TL dating methods to archeology and geology, and stimulated numerous TL studies of natural and synthetic quartz. More recently, TL was discovered in nanocrystalline and amorphous silica produced by xerogelling silica alkoxide solutions. In later chapters we will describe TL in vitreous and crystalline compounds, polycrystalline oxides, insulators, glasses, and other thin amorphous oxide films used in optics and microelectronics. The identification and understanding of the chemical and crystalline signatures of TL species remain important topics of ongoing research.

2. Measurement Techniques

The radiation-induced signal acquired during measurements of TL, optically stimulated luminescence (OSL) or photoluminescence processes in materials, non-destructively provides insights into the concentration of color centers within a sample. Such measurements usually employ a commercial luminescence spectrometer which contains a solid-state detector sensitive to light, an optical unit for the optical path of the emitted light, and a computer for data acquisition and control, among other components. These commercially available photomultiplier tubes use a scintillator and a compact photodiode to determine low luminescence signal levels in specific spectral ranges. A second instrument employs a charged couple device for spectral analysis, which are sensitive to a broad range of wavelengths. Thermal, optical, and radiative activation impact TL, OSL, and PL measurements and must be controlled.

Thermoluminescent Materials

Thermoluminescent (TL) materials must have at least one forbidden energy gap to ensure the trapping of charge carriers. For TL purposes these materials are doped with certain concentrations of luminescent activators. TL materials can be classified according to composition; luminescence activation; or thermal and luminescent characteristics. Most TL studies have been made on the following oxides: Al_2O_3, BeO, MgO, Mg_2SiO_4, MgB_2O_4, SiO_2, SiC, and ZnO; phosphates: LiF and $Li_2B_4O_7$; and sulfates: $CaSO_4$, lithium sulfates, and strontium sulfates. Al_2O_3 and MgO are Al, Mg oxide structural types, basically. Thermal TL reproductions available are obtained for single Cr^{3+} ions or Cr^{3+} aggregates in Al_2O_3 crystals, which are classified as phosphors. TL materials were initially used in archaeoluminescence and recently applied to space dosimetry and bioluminescent studies. TL properties are important parameters in dosimetry, such as sensitivity, reproducibility, absorption characteristics, presence of undesired TL signals, optical thermal bleaching, minimum detectable doses, or read-main time stability, as well as thermally and luminescent correlated TL characteristics.

Currently, TL materials are widely investigated because of their number of commercial TL doses and luminescent photon detection system applications. Recently, commercially TL materials have found new applications in mapping, photographic, and phosphorescent luminescence TL studies. These dosimeters have low sensibility and resolution because their reading systems are not optimized. Commercial TL preparations can be processed to decrease impurity effects. Optimum performance can be obtained by using TL systems optimized in sensitivity and reliability according to TL sample properties.

Measurement Techniques

Numerous methods for making TL measurements have been developed over the years, the most significant variable in a TL measurement, other than the quality of the powder or crystal to be measured, is the extent of readout control which is provided. Temperature control over TL readout is important in accuracy and reliability since the TL effects offer a rich source of information about the materials, particularly in any detailed study of their kinetic and spectral parameters. Quality TL measurements lead to a choice of measurement conditions, which must be in accordance with the detailed kinetic model, and care should be taken to avoid artifacts due to inadequately controlled conditions. TL measurements are performed in a large variety of devices which can be divided into two principal classes: those which measure light emitted at one temperature under isothermal condition and those which analyze the glow-discharge curve. Several isothermal glow-discharge curve and isothermal TL measuring devices have been realized in various versions, and different quality levels may be found. TL studies may be carried out both in laboratory testing and in-field environments, and laboratory-based thermoluminescence instruments are widely used for retrospective dosimetry.

Good temperature control is important in any TL measurement especially if TL data are to be interpreted in terms of the kinetic parameters of the processes involved. Since the TL emission is produced through thermal detrapping of charge carriers, any uncertainty in the temperature history during measurement procedure leads to uncertainty in the trapping parameters. Unfortunately, many TL instruments do not provide sufficient accuracy in controlling the temperature. In many commercially available TL readers, temperature is controlled through a closed-loop feedback mechanism. Such feedback systems typically work to an overall accuracy of 1°C. Given the width of trapping peaks observed in natural samples often ranges from 1 to 10°C, such a temperature accuracy is inadequate for kinetic analysis. Also, many TL devices use ceramic heating elements that need a long time to stabilize near the programmed temperature which removes the possibility of working in a truly isothermal mode. At present, only a few TL systems based on optical fiber temperature sensors are able to work in a truly isothermal mode.

Instrumentation

The instrumentation necessary for the measurement of TL is quite simple. First of all, a controlled heating system is necessary. In usual TL laboratory work, we employ a commercial furnace that allows a satisfactory thermal homogeneity; for field studies, we must resort to specially built heating systems. Sample heating must not be too fast, but there are no strict limits on the heating rate. This parameter can affect the glow peak's height and position, and even change its occurrence. The use of high heating rates causes the peaks to shift toward higher temperatures, this is the so called "kinetic shift." The heating ramp used must guarantee a good quality thermal signal during the measurement.

The signal emitted during the TL signal is very weak: good detectors must be used. Traditionally, TL signals were recorded using a photomultiplier tube with a very sensitive photocathode coupled with a glass or quartz window filter that transmits the visible range and is optically coupled to the photomultiplier. To protect the instrument against stray light, a careful arrangement of the heating and recording systems must be used. The gain of the detector must be adjusted to keep the signal within the linearity range, even if, in the case of a bad adjustment, the signals could still be used as age advisors, in the case of dating low sensitivity materials, at least within probable errors. Another traditional recording technique employs a silicon semiconductor detector with an appropriate filter connected by a transistor or amplifier circuit to a suitable computer. The main advantage of this system is its very high detection efficiency. A few other recording systems exist that are rarely used in thermoluminescence work.

THERMAL TREATMENT AND GLOW CURVES

Thermoluminescence (TL) intensities depend on thermal treatment. Generally, TL objects are treated in order to remove the shallow traps responsible for the stimulation of TL at low temperature (annealing) or to fill with electrons the deep deexcitation traps at high temperature (activation). Low-temperature annealing at normally several hundred °C removes the TL at low temperature while it may enhance the TL at high temperature depending on the spectrum of residual thermal stability of the deexcitation traps. Harsh heat treatment of several hours causes destruction of TL. Each TL peak is either due to related deep deexcitation traps or of the related shallow traps, and has particular band structure for the related deep and shallow traps, respectively. Heating rate is normally of the order of 0.1 °C/s. Excitation of TL is normally of the order of 10 3/s.

TL intensity is shown against temperature or its logarithm, or time. The glow curve can show main and sub peaks at the same or different heating rates for non-continuous or continuous filling as well as deexcitation processes. Multi-peak slopes follow the multiplicity methodology that there are one or several thermal resistances in the step of filling or deexcitation processes of TL, respectively. Separated peaks with tail-to-tail distance at least of 2–3 × 10 0 °C distinguish the main and sub peaks. A substance has definite characteristic thermal stability. TL of the same type of atoms in crystal lattice has similar glow curve shapes due to similar thermal stability. The corresponding glow curve shapes forming the spectrum of thermal stability are used for the characterization of TL. Low-energy luminescence centers of the deexcitation traps are used as tracers or markers for crystallization or nanocrystallization processes. TL can be used for comparative diagnosis of the lattice of common transparent crystalline minerals, accumulatory sediments, etc.

1. Heating Rates

Temperature is a key parameter that influences virtually all stages of TL. In fact, it has a crucial role in inducing electron excitation and transfer, as well as the subsequent recombination process over different energy traps. The inductive and transfer functions of temperature are often referred to as the "inductive" and "transfer" temperature functions, respectively. The thermal treatment applied to TL samples is usually performed in a heating furnace, as there is an appropriate control of the applied temperature or its variation over time. Commercially available TL readers can be configured to apply different heating rates to the high temperatures stimulated sample. Most TL studies employ heating rates between 1 and 15 °C/s. Lower heating rates usually increase the TL signal, but can lengthen the duration of a TL measurement to several hours. As an alternative to heating rates used in TL measurements, physics labs performing TL measurements may opt for lower (or high) heating rates, which can be adjusted in diverse TL experimental setups.

In addition to α, a structureless parameter showing no evident variation with heating rate, few TL analytical expressions modeling TL glow curves have also been able to provide good results for some glow peaks. Most TL glow curves obtained for different heating rates and for different experimental conditions display a systematic shift of high temperature TL peak positions with respect to low temperature TL peak positions. Glow curves for kinetic order higher than one (or lower than one) shifts towards lower (higher) temperatures with increasing heating rate. This anomalous heating rate effect is usually called "time–temperature" effect in the TL literature. TL glow peaks correspond to first derivative maxima of glow curves. Typically, TL glow peaks correspond to the maxima found in the derivative of TL glow curves. However, this statement is only valid for a very short approximation to the TL function performed under very high heating rates conditions, that approach the so-called "infinite heating rate".

2. Analysis of Glow Curves

The study of TL phenomena is normally made by analyzing and interpreting glow curves. These curves are complex in shape and their analysis and interpretation is usually difficult and results sometimes controversial. This occurs because TL usually does not obey simple kinetics equations and may involve TL signals from various interacting traps and even the presence of different dopants. Even so, in some cases when less complex systems are employed and the experimental conditions carefully selected, the simplistic analyses of TL signals using first order kinetics equations can be made.

The deconvolution of glow curves is then often performed by using glow peak areas in the simplest method that employs the equation $dI(T) = a(T) H(T) + b(T)$. In order to analyze those glow curves, one needs to obtain a good temperature dependent second derivative representation of the glow curves. The best is to obtain it by dividing the original TL data by the temperature dependent loading factors a(T). It can therefore be fitted using a polynomial function or directly from dI(T) – dI(T) plots. However, these methods emphasize the relative positions of individual features of glow curves - not their absolute magnitudes. The temperature at which the first peak appears - T0 - and the temperature at which the last peak appears - Tn - should normally be indicative of the range of DOS values. Alternatively, the second derivatives had been processed by a principal component analysis, followed by an AMUSE analysis to orderly construct dI/dT(T) plots, assuming they were available only for a fraction of the glow curve temperature interval. The obtained principal and other transformed component plots should be mixtures of DOS, at least if the glow curve were a linear combination of component peaks mathematically curved to approximately Gaussian shapes and align along the second derivative curve.

KINETIC MODELS

Abstract. Thermoluminescence (TL) glow curves typically present distinct structure and only in the simplest cases a complete description of the TL kinetics can be achieved. The unambiguous extraction of the kinetic parameters from glow curves is a demanding task for both theorists and experimentalists. Regardless how challenging this task may be, it is indispensable for the elucidation of the trapped charge carriers' properties and the electron-hole pair recombination and emission processes. In this chapter, the kinetic models applied for the description of TL glow curves will be introduced, starting from the models that only consider the kinetic processes occurring at a single temperature and arriving at those that take into account the multiple temperatures at which trapping and recombination occur. These models will span from very elementary to complex considerations about the TL process, and will also introduce the concept of order in rate equations. Since the kinetics of TL involves the thermal stimulation in an open system of confined particles, it will only be logical to discuss those elementary systems that have survived the thermal evaporation.

Kinetic Models

The glow curves, as they are regularly recorded in unusual types of TL experiments, seem to agree well with equations that correspond to the kinetics of a first or a second order irreversible reaction. The irreversible nature of the thermoluminescent process has been usually taken as evidence that the electrons are not simply thermally freed at the temperature at which TL is taking place but that they are also described by diffusion equations that would be thermally invoked at much lower temperatures. The proposal of trapping levels made it possible to describe the kinetics of TL and other open systems of confined particles at finite temperatures by models that are fully argued. These equations have been further extended to account for general competitive multiphoton absorption processes that are described by explicit quantum mechanical derivations.

1. First Order Kinetics

The first to elaborate on the detail of the influence of heating rate upon the TL glow curve shape, in particular why first order kinetics gives a straight line in a particular plot, were Lattice and Reichman. Not all the natural TL curves can be regarded as first order kinetics. It has been assumed that the TL curves are around first order kinetics, that is the curves could be converted into a straight line, Log versus Ln plot. This assumption happens to be correct for some samples with simple TL curves but does not apply for others. Since most of the natural TL curves observed could not be evaluated within the above frame, the values of the kinetic parameters are not generally interchangeable. The complexity of most TL glow curves has already been discussed and is the result of radiative and thermal transfer processes. It can be however useful to treat the TL as first order kinetics and we still consider the TL characteristic of these kinetics. The rate of change of the quantity L of TL emitted may be written as the equation of a first order reaction, where L = L(T) is the temperature-dependent rate constant of the process, and dL/dt is the time rate of change of the TL response. The constant L tends to increase with temperature, passing through a maximum value around the temperature of maximal TL intensity, after which it decreases rapidly again.

2. Second Order Kinetics

One of the foremost principles of thermoluminescence is that, if the trapping sites are in very low concentration relative to the number of activator centers, they will be filled as they are created in the transition. For this case, one assumes that the activator is in excess, and that the trapping sites are being filled in a very slow sequence, so that the transition rate might be written $dI/dt = -W \cdot I - dD/dt = +dD/dt = 0$ for the activator, and thus $J0(n) = W \cdot I$ where $n(t) = n0 =$ const. for the trapping. The formula for the current becomes $dI/dt = -W0 \cdot I$, which is the linearized solution of the exponential statement of what must evidently be a second order reaction in I.

To go to the next order in the trapping, one notes that there is condition $0 < n0 < Nt$, where Nt is the number of trapping sites in the system. This allows a correction to be made to the previous treatment of a reaction in which both n(t) and I(t) are severely shrinking, which assumes n to be a very small constant, for a reaction in which n is small but not nearly constant, although still always much smaller than I. If we wanted a thermoluminescent version of the work on thermoluminescence where D is treated vaguely as constant in the derivation of the current while the surface is optimum for the pure exponential model, we could consider it an easier calculation.

3. General Kinetic Equations

It is apparent from the earlier discussions that a critical point in the TL, and in general all thermal activation processes, is the part of temperature had been implemented. The analogy with other thermal activation processes leads to reconsider the Arrhenius law and rewrite as:

$k = Ae^{-E/kT}$

with k the reaction rate, k the Boltzmann constant, and A the pre-exponential factor. This gives as a consequence the Arrhenius Law representation as $Ln(k) = -E/kT + Ln(A)$. This gives an easy way to find E, in a plot of Ln(k) against 1/T. In addition, the pre-exponential factor, A, was formally considered a constant. However, increasingly in the TL community, A has been considered a further dependence on T and not a constant. More precisely the formula could be written as:

$k = A(T)e^{-E/kT}$

from which, in principle, $A(T) = k\ eE/kT$ could be extracted by simply dividing the reaction rate by an exothermic factor containing the activation energy. Although considered in principle as an approximation, $A(T) = aT$ is one of the most used in the literature, where a is a constant of the order of magnitude of 10−12. In a general context, k was replaced by a more general factor Q(T). Q(T) encompasses various variables such as the temperature dependence of TSL signal, dose-response curve, and other temperature-dependent experimental variables.

APPLICATIONS OF THERMOLUMINESCENCE

Applications of thermoluminescence (TL) are widely based on its function as a dosimeter for ionizing radiation exposure. Most of these applications are mainly IL. However, in archaeology, it is used first to determine the time of firing of heating materials - bricks or ceramics, to be sure that they are not older than age - by TL dating. A few physicists use TL to measure quartz sand grains to know the time of burial of sediments by aeolian or fluvial actions, to estimate the frequency of earthquakes along faults for paleoseismic studies. A few more have searched for TL in weathered grains on rocks and paleoraxis probable direction of the Earth magnetic field reversals.

Thermoluminescence is also used in archaeology to date wooden artifacts by using the cutting through the steinwood to take heavily dended TL quartz powder for dating. In other fields, TL is widely used to survey residual doses of teeth and jawbones by dental X-rays in forensic medicine. It is used to analyze collected ashes in fire crimes in criminalistics. It is also used in medicine as a substitute for SECs in an emergency dose recorder for continuous in vivo monitoring due to its peak warmup time dependency. Last but not least, TL is used to analyze surface events of planetary bodies like the Moon, Mars, and asteroids, due to their degassing and weightlessness in outer space.

All the above applications, except for archaeology, used actual TL signal response. For archaeology, both TL and IL dating are combined using correction factors with special conditions. Three TL properties have been exploited for dosimetry and monitoring: Their surface sensitivity due to the dose gradient near the surface, their shallow TL glow peaks, and their dose error due to the readout temperature. The shade and light of the sample reflectance at the TL measurement wavelength are different for excited indigenous color centers at the body surface and excited natural color centers in its bulk.

1. Archaeological Dating

Thermoluminescence (TL) measurements can provide a dating method for anthropological and geological phenomena. The TL dating uses the traps that were filled with electrons during the exposure of the control volume to the geological or environmental background radiation. The exposed volume is assumed to be a small volume of the bulk material. Because the typical TL curves are related to the burning of the outer surface by thermal activation, the concept of TL dating is severely challenged. TL dating underestimates the age of the exposed material because it takes an uncertain time, usually longer than the exposure time of a larger control volume, to burn the smaller grain. Nevertheless, TL dating has turned out to be reliable, especially in the dating range older than when the TL-derived ages are validated by cosmogenic nuclide dating. TL dating of pottery or bricks provides secure dating with an accuracy of several decades.

The first archaeological TL dating was made in 1973. The TL method was enabled because of the systematical measurement of the TL spectra and the discovery of a reliable TL peak that dated to about AD 940. Two years later, the TL dating was first applied to date pottery. Consequently, the TL dating was popularized in the archaeological community as an auxiliary method for reliably providing an ancient burial age. TL dating has stimulated various TL-related developments in terms of TL theory and experimental techniques. TL dating has the potential to sufficiently expand the time range for determining burial age. The improvement of TL dating largely depends on technological progress in conjunction with advances in TL theory.

2. Dosimetry

In 1958, the concept of using thermoluminescent phosphor materials (TLD) for radiation dose measuring and monitoring was first published. In the following years, several basic studies confirmed interesting properties, confirming the unique and interesting characteristics of thermoluminescent dosimeters. Since then, several thermoluminescent phosphors were synthesized with an easy-to-implement synthesis and capable of being applied at several conditions such as TLDs for high-energy radiation, radiation monitoring, etc. The most famous TLDs are LiF:Mn, LiF:Mg,Ti, LiF:Mg,Cu,P, CaSO4:Dy, or Li2B4O7:Mn are the most common TL materials. These phosphors have been heavily used as TLD in medical, space, industrial, or environmental areas, especially gamma or beta radiation.

There are different TL dosimetry systems, including the TLD-100 and TLD-200, the PANDA system with LiF:Mg,Cu,P. One system has been commercializing TLD-100 for several decades without significant improvements in its technology. The TLD-100 is a naturally occurring phosphor KCl doped with 5 mol% of brine is used for dosimetry at high doses using LiF:Mg,Ti as the TL material. However, after these decades, a lot of researchers were focused on improving the sensitivity or characteristics of the TL materials, introducing new materials prepared by dope, co-doping, and codoping, and investigating new techniques in TLD systems (fibre optic techniques, TL reader systems, radial TL techniques, LiF doped, LiF poled with metals). Researchers also improved the commercialized TLD systems introducing them doped by metals. New techniques on TLD systems and LiF doped or LiF poled by metals were also published. Among these materials, Li2B4O7:Mn seems promising thanks to its properties, reducing the amount of non-dosimetric effects.

3. Environmental Monitoring

The effects of uranium and thorium on the human body have been extensively researched, and the conclusion is that they are environmentally detrimental and human carcinogenic. Although the most common uranium tailings are yellowcake products characterized by a large amount of uranium and little or no radon content, mining should be avoided due to the radionuclides leaking into the oceans and rivers from the hydrological cycle. Radionuclide emissions are known to have adverse effects on human health, and regulating activities at uranium mining and milling are required. Monitoring of the waste rocks generated during operations is crucial for ensuring human health safety. Currently, gamma-ray spectroscopy is extensively used in reference to concentrations of uranium isotopes to control radiation contamination at uranium sites. However, gamma-ray spectroscopy detects only gamma-emitting isotopes. In contrast, thermoluminescence can measure the amounts of alpha-emitting isotopes, which are detrimental to human health. The thermoluminescence intensity can directly determine the activity concentrations of alpha-emitting radionuclides, and thermoluminescence can track the activity concentration of radon daughter products.

Thermoluminescence has limits for dating environmental samples older than a few years; at present, it is not used for dating them. Its primary application lies in monitoring the conditions of interest. Environmental thermoluminescence measurements exhibit advantages over other methods: thermoluminescence dating. Thermoluminescence is basically a 'net' measurement: The thermoluminescence signal is built up in a situation in which the environmental sample is exposed only to natural radiation, so the measurement is a true reflection of the amount of alpha-emitting radionuclides in the sample. This net measurement property is also present in radiation environmental thermoluminescence measurements. Since the thermoluminescence is rapidly renewed, thermoluminescence dating is likely to produce only short-time instability of the sediment thermoluminescence ages. A thermoluminescence age discrepancy is typical at an environmental thermoluminescence monitoring area with a radical environmental change. Moreover, thermoluminescence age monotony can be checked via thermoluminescence dating.

THERMOLUMINESCENCE IN GEOLOGY

The first real geological application of thermoluminescence (TL) was carried out in 1967. It managed to measure the doses of ionizing radiation delivered to quartz grains, and thus estimate the age of geological materials. The favorable discovery of TL in geological samples, with the possibility of determining geological ages, developed for practically all characteristic TL bands. Moreover, it was possible to develop TL dating by measuring very small samples of quartz from sediments and rocks. Since the first successful age determination of approximately 20,000 years, there have been numerous paleodose determinations of TL in sediments and stones. TL dating of sediments and rocks is well established as a research tool. TL dating is used with the same ease as carbon dating when this dating method does not reach the desired age limits. The advantages of TL are that there is no contamination and that there is no need for a prerequisite to study.

These advantages are also their only limitations. Moreover, TL is restricted to low-temperature processes for protracted periods of time, lasting from a few years to a few million years. TL dating is attractive for certain types of samples from sedimentary and volcanic areas, as well as for particular problems ranging from early hominids to more recent dating at the time of the last glaciation, between 100,000 years and 10,000 years ago. Although there are several dating methods, the most used in ages below 1 million years are the TL dating methods. It is also the best study option when the sample is too small for other analytical techniques.

1. Sediment Dating

Sediment Dating: Sediments are layered sequences, often enriched in certain compositions in corresponding time periods, that receive material from pre-existent surfaces or from certain outside sources according to specific temporal or climatic regimes. When observed in the geological column, these layers indicate the geochronologic history of the planet. The sedimentation process is a physical or chemical deposition of material that comes from certain elevated terrains or inside the ocean basin. Sediment dating gives information about the length of the accumulation stage that each stratum represented, the periodicity of the climatic changes, and the rate of erosion of the older surfaces, which serves to modulate the accumulation of sediments in each corresponding time period. In the ocean or great lakes, sediments are accumulated at a very low rate, on the order of millimeters in thousands of years. The dating of these older and recent materials can be done through the dating of the sands that are in the middle of the mud layers or in organic matters that are located in the mud layers. The densities used for the dating of sediments have been those of quartz or feldspar in their compacted forms, also using their different chemical compositions if they are not consolidated.

While quartz and feldspars are mainly used to date sediments, TL dating has a wide use in relation to the piers of dunes, lagoons, deltas, and sandy beaches, since the innovations that the TL method has had, so that they can see at greater depths and with sufficient precision are undeniable. With TL, sediments that have been accumulated at different times can be dated, controlling the activity of the different inputs. The importance of TL is that it has given new answers to old questions about the history of accumulation of these sediments, since the periods correspond to non-sedimentary periods. The importance of dating these sediments lies in finding correlations with the development of prehistoric civilizations, climatic changes, and short geochronological periods of hurricanes, floods, and paleo-seismic events.

2. Rock Analysis

Max. hours of luminescence signal in rock phase are made from TL and OSL data that enable comparison of the TL ages with U-Pb ages for the same sample. The TL recorded on muscovite and biotite grains has been used to dating the Runeberg granite in Finland as Oldest Intrusions In Fennoscandian Shield Completed By Zircon UPb Ages. This unpublished magmatic historiography with TL ages must be controlled with reliability and precision. The luminescence dating of crystalline rocks has been intensively studied on mica grains, but the understanding of the heating impact on the trapping centers of TL signal and thermoluminescent setting was published only recently. TL signal in heated grains was interpreted as age distortion indicating that final thermal treatment must be set at stable TL signal. In other cases TL signal has been used to difference the modes of the uplift of granite stock in Anzab Ridge in folded mode. After that we investigated about 300 granite samples using TL dating and apatite fission-track analysis for the Western Caucasus. Implications are that paleotemperature modeling based on the measurements of surfaces of fission tracks erased partially by thermal eduction should be provided for TL-dated granite–knowledge about pre-Cenozoic thermal history of folded settings in the Caucasus. The superficial time increase presumably should be the way of AFT-dating in unfaulted segments of crust.

Investigation of TL signal in quartz and feldspars by TL-datings should be provided in different sites of same rock massif. Aged rocks should be also investigated with additional comparisons of a TL–AP and Rb-Sr ages of granite mapping at the Same Area. But TL dating of granite should be controlled by U–Pb dating of zircon except TLS silica cement for sediments and the ages of collision deformations of folded molasses. The absorbed AD in quartz blocks from the belt-gray granite sepals had been taken to TL dating the Ruinovaya Gora granite – the oldest period of high-temperature tectonics with K-Ar dating. The TL dating of 390 Ma was provided only for quartz samples, however the Zr content in quartz was studied to choose samples utilizing TL center not blinded by the high content of Zr4+. Then age differences in this zone and the absence of age differences shifted to the fault at 200 Ma. quartz veins – Questions To Geological Consequences Of Quartz Veins Dating, Was Conducted In About 700 Granitic Rock Samples For France With Included Quartz Samples From The Chugoshin Goryn Superficial Area.

ADVANCEMENTS IN THERMOLUMINESCENCE RESEARCH

The thermoluminescence (TL) analysis of minerals, rocks and sediments is an important tool for archaeologists and geologists, particularly for the age determination of aggraded sedimentary deposits outside the range of radiocarbon dating, where TL dating has established itself as the standard technique. New technological developments have led to more sensitive and miniaturized readout equipment that allow for applications in the field, as well as in antiquities testing, where small sample dimensions are essential. A wide variety of materials have been identified as TL sensitive in the past, many of which have not been studied extensively. In addition, new synthetic TL phosphors are regularly developed for use in dosimetry and other radiation and nuclear related areas. In the context of this chapter, we will review recent advancements in TL phosphor development for the purpose of TL dating and luminescence dosimetry, recent innovations in readout techniques and instrumentation and their applications. An important component of TL dating systems for mineral extraction from luminescent sedimentary deposits is the availability of suitable luminescence dating reference materials, ideally calibrated by secondary methods for dose and energy response. Such reference materials are often commercial phosphors for TL dosimetry, where costly calibration is usually the responsibility of the commercial producer. The properties of commercial TL phosphors used in dosimetry have been reviewed extensively, however, little information is available with regard to their applicability as sources for the secondary calibration of TL dating systems. These phosphors are predominantly LiF-based materials, where Li impurities in the source can have a considerable effect on the TL response and its dependence on the low-energy component, i.e., below 1 keV, of the incident photon spectrum, an important consideration for a dating system. Although the LiF absorption edge lies at 12 eV, phosphors such as LiF:Cu, - P, were observed to have significant sensitivity below this energy.

1. New Materials

Several factors have motivated researchers to develop new TL materials. TL dosimeters used for personal and medical doses must have an adequate response to all natural and artificial radiations. TL detectors used for radiation dosimetry in physical and geological dating need to have a TL glow curve shape, peak temperature, and TL properties suitable for the goal. For use as a label without modification of the fingerprint, the TL response must remain constant over time and have the same characteristics of a natural lack. In the case of fire investigation, the TL response must be sufficiently high so recovery is possible and, if necessary, the glow curve must have the same characteristics of a natural lack of the specific soil. In all these conditions, natural materials present some drawbacks. TL natural signals may vary depending on weather conditions and radiological environment. The TL signal from natural materials may approach the limit of detection in fire investigation. For personal and medical dosimetry, the TL signals of natural materials may have very low sensitivity.

Although many TL materials have been proposed for TL applications, very few have been widely exercised. This is probably a consequence of the fact that, for many TL applications, natural materials are sufficient. Here, we present several new TL materials that exhibit remarkable characteristics for TL applications in biomedical, geological, and physical safety areas. For X-ray dosimetry, the main need has been for materials with high sensitivity but very low fading. Al_2O_3:C was proposed for personal dosimetry and showed very low fading during the first years of service. Some years after the launch of Al_2O_3:C, other materials appeared, such as LiF:Mg,Cu,P; LiF:Mg,Cu; $Li_2B_4O_7$:Cu; LiF:P; Li_2SiO_5; $BaCl_2$; and CaS:Eu. These materials have very low fading in laboratory conditions, making their use interesting.

In the following, we present other TL materials proposed to fill the above requirements in TL applications. We divide the review into TL materials used in the last few years, TL materials without commercial applications, and materials not yet used for TL applications, but that present properties interesting for potential applications.

2. Innovative Techniques

During the past five decades, there has emerged a continuous effort to elaborate new and more efficient techniques for luminescence signal readout, dose determination, dating, and a range of applications involving dosimetry and dating of different materials. With that purpose, numerous inventions have been described in the specialized literature. Many of these innovations have greatly developed the art of luminescence detection. However, most of these reports are unpublished, lying in the confidential records of some laboratories. Some examples of recognizably licit and productive patents can be found.

Innovative work can frequently be found in the “general” patents of the luminescence laboratories or companies that processed luminescence signals or constructed luminescence devices. Many, maybe the majority of luminescence resorts, work in the familiar manner of inventive machines or methods of great commercial success. A less charged description of the subject can be found in the bibliography. This list is compiled according to the chronological order of publication, for any specific theme or applications. Customarily, this innovative work has mostly involved different aspects of luminescence readout such as signal stimulation and detection. The main aspects of these general advancements are briefly discussed in the following. A major part of these advanced techniques has broad applicability to TL as well as OSL, non-necessitating the complex cascade of the TL signal in almost two hundred independent detectors that should be done in order to calibrate the TL signal of any powder detector.

CHALLENGES AND LIMITATIONS

Thermoluminescence (TL) is an appealing and versatile tool for both scientific research and several technological applications. However, it also has some important challenges and limitations that one must consider before using it. In the following, we describe some of the important challenges related to TL and some of their solutions.

1. Measurement Uncertainties

Thermoluminescence is currently used in several scientific and technological applications that can impact people's lives in several ways, such as archaeological dating, forensic science, blood glucose measurement, essential oil characterization, environmental monitoring, and lithological studies. It is well-known that, depending on the application, TL can be measured either for qualitative or quantitative purposes. In both situations, the quality of the TL signal is important, since a poor-quality TL signal could lead to wrong conclusions or TL results with very low confidence intervals. In some applications, such as archaeological dating, the TL data processing is so critical that TL is the last resort method for cross-dating the thermoluminescence signal with the optically stimulated luminescence signal.

In these situations, the restrictions imposed by dose rate calculation and optical bleaching must be well considered. In contrast, during a blood glucose level measurement, it is very important to obtain a highly sensitive TL signal with low uncertainty values to avoid misdiagnosis. In some of the applications listed above, if the TL measurement is performed weekly or monthly, the TL signal must present some degree of reproducibility over time. In addition, the TL signal properties must not vary between each irradiated sample in the same TL measurement batch, otherwise, it may lead to misleading conclusions.

2. Material Limitations

Materials can present some limitations depending on the TL application. Some TL materials can present high sensitivity, while another one can present a very low sensitivity. The TL sensitization behavior of a certain material is an important parameter to take into account in TL applications. Although more than 200 materials are known, high-sensitivity TL semiconductors, such as quartz, Al2O3, and LiF, are still the most widely studied, even if scintillators have an important mass and energy sensitivity. Among the TL semiconductors, Al2O3 ceramics have an extremely high sensitivity and an adequate dose range. Other TL phosphors do not have an adequate dose range for practical TL applications.

Measurement Uncertainties

For over 50 years, thermoluminescence (TL) has offered a wealth of valuable information about a sample, making it a technique of considerable promise for applications in fields such as Quaternary science, geology, material science, archaeology and art history. Although its ecosystem is riddled with experimental and material challenges, TL is often marketed as a "simple" technique to implement. Here, we discuss several "easy to overlook" problems lurking within the TL experimental ecosystem, which, if not recognized, can lead to erroneous sample interpretations, as well as competition from other dating techniques can make TL a difficult sell. We will explain how solving these issues can lead to a more robust age report by leveraging recent advances in the TL experimental environment.

The uncertainties associated with a TL measurement will most commonly be a combination of errors resulting from both sample analysis and age calculation. For the analytical part of the uncertainty, we can use the statistical errors from our experimental analysis. Then we can add to that a measure of the uncertainty associated with how we model the shape of the TL curve, as well errors associated with signal intensity loss as a function of time before measurement, the effect of beta dose caused by natural beta radiation from the sample material, etc. From this, we can construct a joint distribution of errors from which we can then propagate the uncertainties into the final age calculation using standard error propagation techniques. Given the assumption of all measurement error contributions being Gaussian, the final thermal age will typically have a Gaussian distribution centered around the output value of the age calculation, along with a standard deviation magnitude given by the width of the joint distribution, propagated using standard rules. However, this standard procedure does not apply universally, and is especially likely to break down in situations characterized by non-standard measurement error behaviors.

Material Limitations

The second major challenge lies in the response of the material and the relationship between radiation effects and signal characteristics. It is well known that the thermoluminescence glow-response from a material, that is, the shape, location, and intensity of its glow peaks, depends on various factors, including heating rate, ambient light, preheat, and prior irradiation. These diagnostic input parameters are often partly interrelated and, thus, not really independent. It is also clear that the signal shape must depend upon the intrinsic properties of the material and specific defect centers present. It is also known that the location, depth, and nature of the electron or hole traps in the band gap, and the locations of the luminescent recombination centers and their respective cross-sections for trapping, and for recombination, affect the properties. Selection of favorable assay conditions is crucial in order to minimize uncertainties in the results. This is one of the major reasons for using a plurality of specific materials or for varying the specific details between samples of the same material.

It is known that previous problems arise in materials with a high concentration of impurities, owing to trap filling, that affect the signal pulses. Loosely bound electrons and holes are detected in materials with a high defect density. Thermoluminescence from band bands tends to be broader than that from deep traps, and has an intensity ratio and distribution, central depth and luminescent color that depend mainly on the type of deep levels and on the shape of the available band edges. Deep shallow electron traps close to the conduction band bottom predominantly determine thermoluminescence in semiconductors with a high imperfection density. Deep shallow hole traps close to the valence band edge are observed in low imperfection density insulators and semiconductors. Trapping cross-sections are larger for shallow defects than for those that are deeper in the gap.

FUTURE DIRECTIONS IN THERMOLUMINESCENCE

1. Emerging Applications

Thermoluminescence (TL) has been widely used as a tool for thermochronometry, dating minerals associated with the initiation of some geomorphological events, and measuring the accumulated doses in materials, such as quartz and feldspars. These typical applications are well covered by TL dating literature, and studies related to them are still being published, as expected by the majority of the TL community. However, TL is now also being used in more sophisticated and innovative systems with emerging applications, as illustrated below. These uses bear witness to the synergies that may arise from the combination of the TL technique with new technological advances in other fields, so benefiting from a truly interdisciplinary research approach. There is a clear trend towards miniaturization, integration and automation of measurement systems. The availability of low-cost, compact and miniaturized components has led to more efficient systems, which often travel to remote inaccessible places or places devoid of basic infrastructure. This is particularly important in paleoseismology, where field dating is sometimes impossible due to the absence of optical stimulation and/or the lack of a vacuum system.

2. Interdisciplinary Approaches

TL is often regarded as a dated technique, particularly by young researchers seeking new technologies and instruments. Development of systems that provide stable, compact, affordable and easy to operate TL detectors and stimulators capable of making more rapid and detailed TL measurements could certainly help keep the TL technique on the cutting edge of research and novel applications, rather than just a documentary format for fossils. And wait a minute: Have researchers at the forefront of technology actually created sensors capable of measuring luminance so quickly? If so, and relevant patents for TL can be purchased, would it be possible to create a TL system with the required performance?

Emerging Applications

The venerable fields of archaeology and geology have benefitted the most from TL dating in the last few decades. With the continued frictional heating related to numerous tectonic events, dating events, as well as thermally resetting exposure ages of natural and man-made silicate structures that may be geologically active, has warranted the development and application of TL to other fields of science. Paleoseismology has confirmed the interest of the accumulated public masses in the recurrence of earthquakes in order to ensure their safety. To this end, TL must further more efficiently date the sedimentary false bottoms of active fault traces at millennial scales. Geophysical interest in hotspot stability forces questions and proposals for experimental calibrations. Quasi-experimental TL laboratories are being set up and are developing collaborative wide-scale experimental temperature TL-programming on different samples with the aim of being able to test the heating event temperature and duration.

The human fascination with the geo-archaeological knowledge of their region has led them to further explore their environment. Accumulated urban collapses near pedogenic seismic markers are consoling to understand the stability of a city and its importance. TL is regularly called upon to respond to the forensic demands of various upheavals, allowing for the design of TL databases by different investigation groups. These databases will serve to refine TL parameters, help study specific cases, and generate TL response maps for practical forensic tools.

Interdisciplinary Approaches

In recent years, a wealth of cross-disciplinary research has appeared, taking advantage of the various physicochemical properties of materials that luminesce. For example, confinement may modulate the properties of nanocrystalline TL phosphors embedded in a polymer matrix. This and similar ideas took advantage of scintillating, and even TL materials, which have potential applications in gamma-ray detection as these scale down to the personal protective measures. Dense ceramic TL materials may also find application in scintillators or photo-detectors to remote detect events of interest or detect counterfeit archives for carbon dating. In this turn, ceramic TL phosphors exhibit greater insulation against χ-ray damage than their dense counterparts, thus opening a new avenue into developing devices capable of running for extended periods in radiation-prone areas.

Work and collaboration with the astrophysics community relying on nucleon-nucleon collision experiments guided the design of novel sensing and TL prototypes opening a more recent frontier in TL by moving the initial device concept from an atomic-scale, hypersensitive remotely-reading ensemble of devices back to micro and nanoscale, self-sensing devices. The heavier the TL centers the higher the absorption, the device resistivity is lowered when self-sensing under a putting photon flux such as that from radioactive decays, which can be used for artificial-label device taggings. Counting requires no shielding or active electronic reading, coherent data can be transferred over very long-distance, all-device design, resources, mode of operation, and device function externally. The challenge is now on going back to atomic scale, hypersensitive, outstandingly controlled material processing, and TL host doping at LPA flux, using down-scaling at sub-wavelength cost-effective optics again.

CONCLUSION

This is the twelfth section the book

In this chapter, we have reviewed the principles and techniques of TL, with special emphasis on its application in dosimetry. In the first section of the chapter, the underlying principles of thermoluminescent phosphor materials have been summarized, including their electron trap energy levels, charge transport, design properties, and the radiative recombination process. We have briefly described some aspects of the materials preparation and characterization, not only of TL materials but also of those competing with TL for dosimetric applications. The TL exposure metrology is outlined, with a special emphasis on TL glow curves. Subsequently, we have summarized TL applications in the areas of personnel and environment monitoring, accident dosimetry, and of other fields that range from geology to paleontology, space radiation and semiconductor technology, with focus on key applications and recent developments in each area. The last two sections concern applications of optically stimulated luminescence, which we designated the twin sister of TL, besides a brief selection of selected OSL applications. The TL and OSL techniques have many aspects in common, including the underlying physical principles, the need for careful preparation of the phosphors, exposure and reader systems, the deconvolution of the TL glow curves and OSL decay curves. Therefore, it is useful to introduce OSL applications for comparison and complementarity purposes.

Thermoluminescence materials are prepared from oxides and some fluorides, doped with impurities or native defects. The preparation conditions deeply affect the crystallization, microstructure and optoelectronic properties of the materials. Additionally, engineering the fabrication parameters will provide for luminescences that fit specific applications. Thorium-doped aluminum oxides are a key TL compound used for personnel and environmental radiation monitoring due to their reliable TL properties, namely that the glow curve has a peak and is stable with time. The TL response of these and of some other key TL materials used for emergency and accident dosimetry shows some other traits, which are not an inherent trait of TL, but come from the thermal treatment, purification and annealing of the emitted radiation on added impurities.

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