Thermoluminescence as a Research Tool

Farrington Daniels, Charles A. Boyd, and Donald F. Saunders[1]

Department of Chemistry, University of Wisconsin, Madison

THERMOLUMINESCENCE IS THE EMISSION OF LIGHT produced by heating a solid to a temperature below that of incandescence. It is exhibited by crystals, such as alkali halides, that have been exposed to x-rays or radioactivity and then heated rapidly. The high-energy radiation dislodges electrons, some of which become trapped in lattice imperfections, and are later driven out with the accompanying emission of light when the temperature is raised to supply the necessary amount of kinetic energy.

Thermoluminescence is a property that is extremely sensitive to changes in the structure of crystals. Changes in minor impurity concentrations, crystallization techniques, and physical treatments all produce profound effects. This structure sensitivity is found in other solid state properties such as catalytic activity, dielectric constant, magnetic susceptibility, light absorption, fluorescence, and phosphorescence. Because of the similarities between thermoluminescence and these other properties it is thought that thermoluminescence measurements may be of value in their interpretation.

The application of thermoluminescence in the analysis of minerals and control of feldspars in the ceramics industry has been described by Deribere (*1*, *2*). It has been used in the interpretation of phosphorescence mechanisms by Garlick and others (*3*). Further interesting uses of this phenomenon were suggested in preliminary reports (*4–6*), and it is the purpose of the present communication to outline progress in the application of thermoluminescence to a variety of research problems.

Apparatus

The technique of measuring thermoluminescence by the so-called glow curves, developed initially by Urbach (*7*) and modified by Randall (*8*) and Boyd (*9*), has been simplified so that many thermoluminescent substances can be studied rapidly and conveniently.

The intensity of light as measured with a multiplier phototube, and the temperature as determined by a thermocouple, are recorded simultaneously, using a recording potentiometer. The specimen is heated at a uniform rate of 1° C per second, producing records such as that shown in Fig. 1. The straight line is a record of the temperature, and the light intensity passes through a series of maxima. The crystalline material is prepared either as a powder or as a plate about 1 cm square and 1.5 mm thick, obtained by cleaving crystals or cutting rocks with a diamond saw. The powder usually gives a lower light intensity on account of the scattering of the light within the sample, but it is more reproducible because it averages out the heterogeneity of the specimen. It is ground to 100–200 mesh size, and a weighed quantity of about 20 mg is placed on a thin glass plate and moistened with a drop of water containing a trace of detergent. After drying, the powder sticks to the glass well enough to be handled. The electrically heated silver hot plate is adjusted by means of a variac to give a uniform temperature rise as recorded on the graph, more current being allowed to pass as the furnace heats up and the heat losses become greater. The apparatus is enclosed in a light-tight box and, after heating the sample to dull red heat, where thermoluminescence can no longer be detected, the hot plate is removed and cooled quickly with dry ice for the next determination.

Most of the irradiation of crystals has been carried out in two special γ-ray irradiators, each consisting of two concentric aluminum tubes, with cobalt powder filling the annular space between them (*10*). We are indebted to the Argonne National Laboratory and the Oak Ridge National Laboratory for placing these irradiators in nuclear reactors to produce radioactive cobalt (Co^{60}). In this way a space 2.2 cm in diameter by 8 cm long is available for uniform irradiation with 1.1 and 1.3 mev γ-rays at intensities of 6000 or 1400 roentgens per hour for 4 or 1.3 curies of Co^{60}, respectively.

Mechanisms

To exhibit thermoluminescence, a substance must have an ordered structure such as is found in crystals, or a semiordered structure as in glasses. In addition, it must be electrically an insulator or semiconductor. The crystal must be exposed to ionizing radiation that will produce a cloud of electrons within the lattice when the radiation is absorbed.

The ability to exhibit thermoluminescence in certain crystals can be produced by a variety of high-energy radiations—hard or soft x-rays, γ-rays, α-particles, and β-rays. An energy of about 10 ev is sufficient to remove electrons from ions in most crystal lattices and to provide them with energy to move around in the crystal, but unless there are traps into which the released electrons can go, there is no mechanism to provide for thermoluminescence. There are several different kinds of possible electron traps: (*a*) imperfections and vacancies in the crystal lattice produced at the time the crystal is formed, or created later by

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[1] The authors are pleased to acknowledge the support of this project by grants from the Atomic Energy Commission.

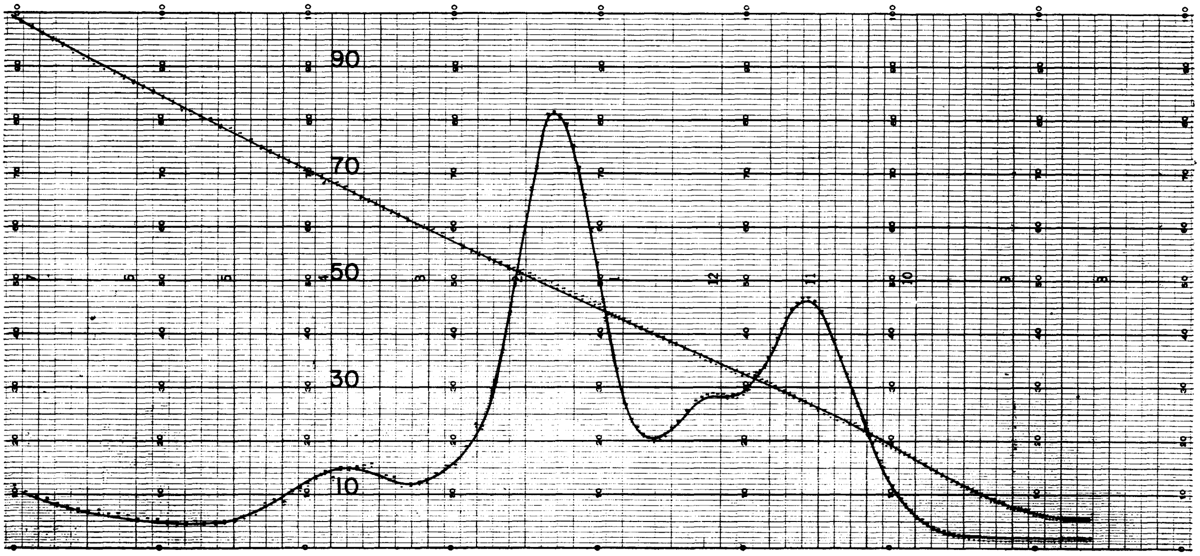

FIG. 1. Typical glow curve recording; γ-activated thermoluminescence of a limestone. 0 = 0°C; 100 = 500°C; 0 = 0 microamp; 100 = 1 microamp. Heating rate = 0.8°C/sec. 115 hr-exposure in γ-source.

mechanical pressure or thermal treatment; (*b*) statistical imperfections that are due to kinetic motions and that increase in number at higher temperatures; (*c*) distortions produced by impurity ions of larger or smaller size than those comprising the crystal lattice; and (*d*) ion dislocations, or "holes," produced by radioactive bombardment.

One of the common types of traps is a negative ion vacancy—for example, a chloride ion missing from a sodium chloride lattice. When such a vacancy is filled with a dislodged electron, it is called an F-center, and it acts as a light-absorbing unit. The color produced by x-radiation in crystals and that found in some fluorites and other minerals containing radioactive impurities are thought to be due to the creation of these F-center and other arrangements of trapped electrons (*11, 12*). It has long been known that this coloration can be bleached out by heating (*13*); it is not quite so well known that thermoluminescence usually accompanies the removal of the color.

A crystal may possess many trapped electrons within its lattice and may not produce thermoluminescence when heated unless there is some mechanism for releasing the extra energy of the electrons as visible light. This mechanism is thought to take place in certain emission centers in the lattice, associated with impurities, as is the case in many artificial phosphors and natural fluorites (*14–16*), or perhaps with displaced lattice ions or other irregularities in the lattice.

The role of impurities in determining the character of thermoluminescence has been discussed by Alt and Steinmetz (*17*). Our investigation has shown that individual peaks in fluorite glow curves emit different colors of light. This would seem to indicate that these electron traps are intimately associated with particular emission centers, which have been demonstrated to be rare earth impurities (*16*). In this case it is thought that the electrons are trapped in distortions produced by the foreign ions.

The production of ion dislocations in various materials by α-particles has been studied by Seitz (*18*) and Slater (*19*). Halos of color around radioactive inclusions in minerals are thought to be due to electrons trapped in dislocations produced by α-particles. Certain feldspars exhibit very intense thermoluminescence from such halos, and it is concluded that the electron traps responsible were produced by the α-bombardment. Estermann (*20*) detected changes in density in KCl crystals exposed to x-rays. These were ascribed to the production of vacancies in the lattice by the radiation. Lithium fluoride has been found to produce increased thermoluminescence on each successive equal exposure to γ-rays from Co^{60}. The increased sensitivity is thought to be the result of the production of additional electron traps by γ-radiation.

Each peak in a glow curve corresponds to a definite energy level of trapped electrons, and low-temperature peaks can be drained out without affecting the high-temperature peaks if the heating is stopped before the high temperatures are reached. The amount of thermoluminescence of a given peak is a result of a balance between the rate at which electrons have been driven into the traps by radiation and their rate of thermal escape at the temperature of irradiation. When the temperature of the irradiated crystal is raised quickly and then maintained constant, the intensity of luminescence decays, with a definite mathematical relation from which the energies of activation (*9*) of a specified type of trapped electron can be calculated.

The thermoluminescence behavior on continued irradiation has been studied in considerable detail and will be reported elsewhere. In general, saturation occurs at an energy input that varies with the crystal and the type of radiation. Low-temperature peaks are apt to become less prominent with continued irradiation, and new high-temperature peaks are created. The alkali halides and limestones are apt to reach saturation after about 100,000 roentgens of Co^{60} γ-radiation. Continued irradiation may simply give a constant thermoluminescence intensity or it may give a decreasing intensity.

TYPES OF THERMOLUMINESCENT MATERIALS

The alkali halides are all thermoluminescent. In general they have two prominent peaks which shift with the size of the ions, the smaller atoms giving thermoluminescence which comes in at higher temperatures and coloration from F-centers with absorption maxima at shorter wavelengths (*21*). In lithium fluoride, where both ions are small, the coloration produced by radiation comes in the ultraviolet, and the maxima in the thermoluminescence curves occur at about 220° C and at 320° C.

In addition to the alkali halides, the following give thermoluminescence after irradiation with γ-radiation —calcite, dolomite, fluorite, aluminum oxide, magnesium oxide, gypsum, quartz, glass, certain catalysts, feldspars, feldspathoids, certain dried clays, and ceramics. A large variety of inorganic crystals is being investigated for thermoluminescence, and more sensitive apparatus for testing thermoluminescence is being developed. In general, hard, transparent or transluscent crystals of simple crystal structure are most apt to exhibit thermoluminescence after exposure to γ-rays.

Sometimes, of course, the thermoluminescent light emitted probably escapes detection with the present apparatus because it is in the ultraviolet or the infrared. Organic material cannot be studied in air because combustion emits obscuring light, but there is reason to believe that, if certain organic substances are irradiated at liquid air temperatures and then heated to room temperature and above, they, too, will exhibit thermoluminescence. Prominent additional, low-temperature peaks were found in the glow curves when the alkali halides were irradiated at liquid air temperatures (*21*). These peaks are, of course, registered as fluorescence when irradiated at room temperature, the light being emitted at the time of irradiation.

The presence of impurities is an important factor in thermoluminescence glow curves, and, until theories can be developed to account for the different activation energies of the various energy traps, it is difficult to know whether a given peak is characteristic of a given crystal lattice or of a given impurity, perhaps present in traces.

Preliminary work has been done on the effect of added impurities and mixtures of salts crystallized out together from a given melt (*22*). When 1 mole per cent of silver chloride is added to fused sodium chloride, for example, the crystallized salt gives 100 times as much intensity of thermoluminescence as the pure sodium chloride alone. Traces of cupric chloride and manganese chloride, on the other hand, quench the thermoluminescence. When potassium bromide is added to sodium chloride and the two are fused and crystallized, a curve of thermoluminescence intensity plotted against mole fraction gives a maximum at which the intensity of the mixed crystals is considerably greater than that of either sodium chloride or potassium bromide alone. The distortion of the lattice by the introduction of ions of different sizes seems to create holes in which electrons, released by γ-rays, can be trapped.

The color of the thermoluminescence varies at least over the whole visible spectrum with different crystals, and it is quite sensitive to impurities.

DOSIMETRY

Since in many crystals the intensity of thermoluminescence is nearly proportional to the amount of γ-radiation received, a considerable effort has been devoted to developing a practical means of measuring the exposure to γ-radiation (*23*). Lithium fluoride has been found to be the best crystal for this purpose, and it has been possible to measure exposures of 10–1000 roentgens and more with considerable ease and accuracy (*21*) by means of a portable photomultiplier tube and microammeter. Measurements of less than 1 roentgen have been made with a nonportable instrument. Lithium fluoride is stable, insoluble, and nondeliquescent, and ⅓ gram of artificial crystal grown by the Harshaw Chemical Company is sufficient for a measurement. The crystals used originally are about 1 cm^2, but more recently powdered lithium fluoride has been pressed into pellets, in a thin metal casing. Experiments are still under way to obtain greater uniformity, but successful tests with different laboratory and atomic radiations have been made. The dosimeter crystals, or pellets, are very small and can be placed in experimental apparatus, which is inaccessible to ordinary radioactivity meters. They have been used successfully by Marshall Brucer, of the Hospital of the Oak Ridge Institute of Nuclear Studies, to obtain measurements of internal radiation intensity in cancerous patients injected with radioactive isotopes. The crystals were swallowed by the patients, recovered one or two days later, and the accumulated dosage in roentgens was measured by matching the thermoluminescence intensity with that produced in the crystals by a known roentgen dosage.

IDENTIFICATION

The glow curves are characteristic of specific substances, with definite peaks at definite temperatures, similar in a sense to spectrograms with their lines of light at definite wavelengths. They are typical of crystalline materials that contain specific impurities and that have had definite heat treatments and physical histories. Thermoluminescence measurements are thus not suitable for analysis of chemical compounds, but they may find use in identification and control. For example, different types of aluminum oxide will give different glow curves depending on impurities and previous heat treatment, but all samples of the same type will give the same glow curve. If one type is desired perhaps it could be selected on the basis of the glow curve. Again, the thermoluminescence glow curve of window glass will vary greatly with the impurities present, chiefly ferric oxide, but if a particular quality of glass is required the thermoluminescence glow curve may become a convenient control test to assure that each batch has the same quality. Limestones, ceramics, and optical crystals are among the materials that may be classified, identified, and controlled.

A collection of about 30 standard clays has been the object of an intensive cooperative research program involving many different measurements, such as viscosity, infrared absorption, and chemical tests. Some, but not all, of these standard clays exhibit characteristic thermoluminescence glow curves which merit further investigation. The nonorganic parts of certain soils should show characteristic glow curves that may be useful in identification and classification of soils.

The color of the light emitted on heating a previously irradiated material is often characteristic. Limestones give a yellow light, and dolomites give an orange light. The orange color may be helpful as a field test in identifying the presence of magnesium carbonate, although it may well be that the color is not related to the magnesium carbonate, but to an impurity associated with it.

Catalysis

It was suggested by Boyd and Hirschfelder (*22*) that thermoluminescence curves might be useful in evaluating the efficiency of surface catalysts. The same strains and lattice imperfections that permit adsorbed molecules to come within optimum distances from each other and bring about chemical reactions might also provide traps into which electrons could be driven by γ-radiation, and later released by heating. The intensity and nature of the thermoluminescence might be a measure of the catalytic efficiency. At least the thermoluminescence glow curve could be used as a criterion for controlling the preparation of a desired catalyst. A standard, successful catalyst should have a specific type of glow curve.

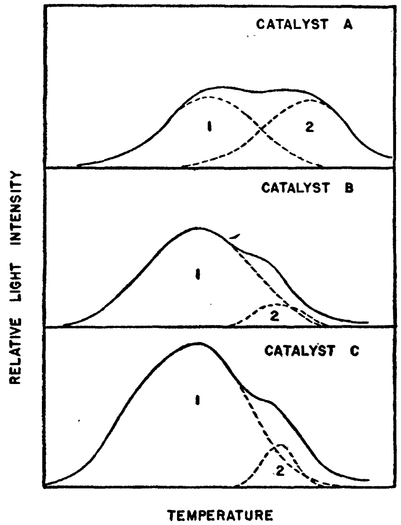

FIG. 2. Glow curves of catalysts.

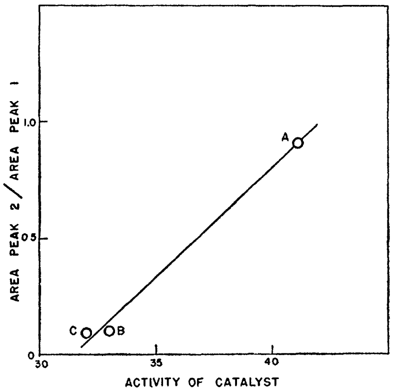

FIG. 3. Thermoluminescence and catalytic activity.

In the first test a good aluminum oxide catalyst prepared by Homer Adkins was found to give considerably more thermoluminescence than a poor catalyst of the same material. Several commercial catalysts have been examined, three of which are shown in Fig. 2. In this case the areas under the No. 2 peak are correlated with the catalytic efficiency, as shown in Fig. 3. Many catalysts, particularly of the gelatinous type, do not give any thermoluminescence, and in many others there is no apparent correlation, but enough catalysts have been examined to justify the conclusion that thermoluminescence is a new tool in catalyst evaluation and that it may become useful in researches to find new catalysts.

Radiation Damage

Most high-energy radiations are effective in producing damage to crystals. The investigation of this phenomenon is of importance in the design of nuclear reactors and in interpreting changes in crystal structure occurring in radioactive minerals over millions of years.

The fraction of γ-radiation energy that can be stored as trapped electrons in crystals is very small. With a photomultiplier tube calibrated with a thermocouple and standard lamp it was found that less than 1/10,000 of the energy of the absorbed γ-radiation was given out as thermoluminescence in lithium fluoride (*24*).

Thermoluminescence is one means of studying the storage of energy from high-energy bombardment and the crystal damage which can be produced by it. Atom displacements, if they occur, might lead to the storage of greater amounts of energy than that observed in thermoluminescence, but the means for detecting heat are much less sensitive than the means for detecting light. Thermal analysis has failed thus far to reveal the storage of heat energy in γ-irradiated crystals, but more sensitive methods and greater exposures to radioactivity are now under investigation. In the case of certain metamict crystals containing considerable quantities of uranium the α-ray bombardment for millions of years has caused serious dislocations of the crystal lattice; so much so that x-ray diffraction patterns characteristic of crystals have been obliterated. When these minerals with accumulated radiation damage are raised in temperature, the stored energy is released as heat, and the x-ray diffraction pattern characteristic of a normal lattice is restored. In one case the energy released amounted to as much as 25 calories per gram (*24*).

THERMOLUMINESCENCE OF ROCKS

Many natural rocks and minerals are thermoluminescent without exposure to x- or γ-rays in the laboratory. Most limestones crushed to millimeter size and dropped onto a faintly red-hot frying pan in a darkroom will emit a bright white or orange light for several seconds. After they are once heated and cooled, no light is emitted if they are heated a second time. However, brief exposure to x- or γ-rays will bring back the property of thermoluminescence, and by proper adjustment of the exposure the same intensity of thermoluminescence can be reproduced. Natural thermoluminescence has been frequently recorded in the geological literature (*13, 25, 26*) but it has generally been regarded as a curiosity. Some attributed the light to the burning of organic material. It has been repeatedly proved in the course of this investigation that the cause of the thermoluminescence is the presence of traces of uranium, thorium, and other radioactive elements contained in the rocks as impurities (*6, 27*). These radioactive impurities may be present only to the extent of one part per million or so, but they have been giving off α-, β-, and γ-rays for millions of years, and part of the thermoluminescence effect is cumulative. In fact, natural thermoluminescence of a rock is often a more sensitive means of detecting traces of radioactivity than is a Geiger counter or scintillometer. The relation between radioactivity and thermoluminescence in certain minerals had been earlier pointed out by Ellsworth (*25*), Kohler (*28*), and Alt and Steinmetz (*17*).

Surprisingly many rocks and minerals are naturally thermoluminescent. Over 3000 samples have been studied, mostly limestones and granites, of which about 75 per cent showed visible thermoluminescence, and still more would undoubtedly show thermoluminescence if measured with the most sensitive photomultiplier apparatus. Nearly all limestones and acid igneous rocks are naturally thermoluminescent. Calcium and magnesium carbonates show light-yellow to orange thermoluminescence, and potassium and sodium feldspars show white to blue-violet. These minerals are responsible for much of the thermoluminescence in the rocks examined, as, for example, in calcareous fossils or inclusions in shales, and in the cementing material between the grains of sandstone. Some fluorites containing uranium give a particularly brilliant thermoluminescence. Quartz, nepheline, topaz, halite, and spodumene react when they contain impurities of uranium, or thorium.

In a Boy Scout collection of 36 minerals over one third gave visible thermoluminescence. Out of 65 rock-forming minerals selected from the mineralogy laboratory's collection at the University of Wisconsin, 34 showed natural thermoluminescence. The intensity of light ranged all the way from that sufficient for reading a newspaper to "barely detectable."

All the minerals that displayed natural thermoluminescence could be made much brighter by additional exposure to γ- or x-rays, and many that did not give any detectable thermoluminescence were made thermoluminescent by a brief exposure to these rays. Of the 65 minerals just mentioned, 47 were thermoluminescent following exposure to γ-rays.

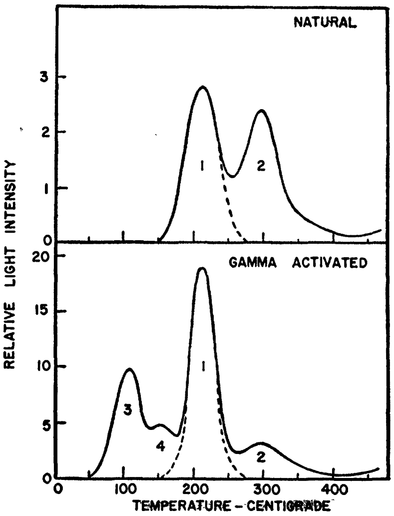

FIG. 4. Glow curves for Escabrosa limestone.

The increase in thermoluminescence resulting from a γ-radiation exposure of 140,000 roentgens is shown in the glow curves of Fig. 4 for the Escabrosa limestone. The No. 2 peak coming in at 300°C is about the same, but the No. 1 peak at 215° is seven times as intense. The No. 3 and No. 4 peaks at lower temperatures are fairly intense in irradiated thermoluminescence, but are completely missing in natural thermoluminescence, because the earth temperature has been high enough to drive all the displaced electrons out of their traps. The time elapsing between irradiation in the laboratory and testing is too short to permit any measurable loss. The high-temperature peak at 300° involves so great a kinetic energy that most of the trapped electrons have remained trapped over the geological ages, and a still longer exposure to natural radioactivity would of course increase the intensity at peak No. 2 still higher—until saturation is reached.

The thermoluminescence of rocks is apt to be quite heterogeneous—some of the mineral constituents give off light when heated, and others do not. Thin specimens of limestones and granites have been polished and placed on a hot plate, with a camera focused on the surface. The light emitted by the heated specimen (below incandescent red heat) is sufficient to produce a photograph. An example is given in Fig. 5, where the calcareous fossils are clearly shown at the left. The light is due to the release of electrons that were originally dislodged by traces of uranium present and trapped in the calcium carbonate lattice of the fossils. The brighter picture at the right was produced by the same sample after an exposure to 140,000 roentgens of γ-rays. The thermoluminescence patterns displayed by granite surfaces commonly reveal spots of brighter light adjacent to inclusions of material of high radioactivity.

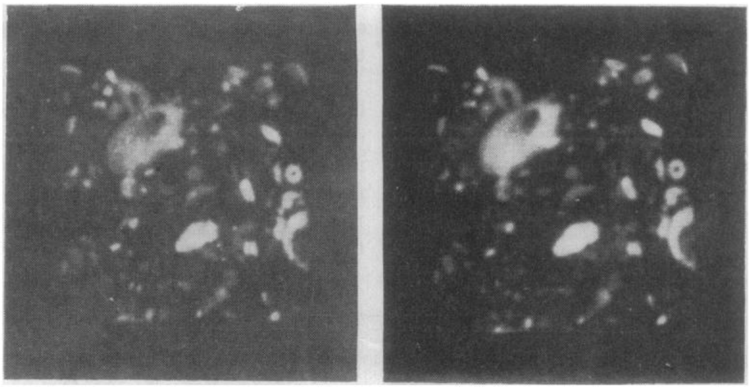

FIG. 5. Photographs of thermoluminescence of a typical limestone. Floyd's Knob formation. Left: natural; right, γ-activated.

Whereas at high altitudes cosmic radiation may be a factor in some of the activation for thermoluminescence, it becomes practically negligible in comparison with the activation caused by radioactivity in rocks below ground level or in rocks that contain one or more parts per million of uranium or thorium.

The thermoluminescence glow curves of rocks vary greatly—i.e., the heights of the peaks of the glow curves and the temperatures of their maxima change from rock to rock. The greater the concentration of uranium or thorium and the older the rock, the greater is the thermoluminescence, because the rock has been subjected to greater effects from radioactivity. The presence or absence of chemical impurities and the physical imperfections are, however, factors that may be just as important. The chemical environment at the time of crystallization and the physical effects of temperature and pressure on the rocks during their geological history have a great deal to do with the nature and intensity of the thermoluminescence.

STRATIGRAPHY

Because the glow curves are so responsive to chemical and physical conditions at the time a rock is formed, they can be used to determine whether two samples of sedimentary rocks were laid down at the same time in the same environment, even if the samples are widely separated geographically. Fossils and other criteria are now used to determine whether two rock samples belong to the same stratum, and these tests have practical value in petroleum exploration. If a certain sequence of strata is found as one drills down from the surface and eventually strikes oil, then a repetition of the sequence in another location may indicate that the geological features are the same as found before. Thermoluminescence glow curves add one more test to those already available for correlating sedimentary formations. In some rock types—e.g., volcanic ash and Pre-Cambrian limestones—no fossils are present to aid in the identification of the strata.

One of the first tests of the reliability of the application of thermoluminescence was carried out in a large limestone quarry, where the various strata can be differentiated visually for nearly half a mile. Thermoluminescence curves of ten samples taken from the same stratum across this half-mile face were identical in shape, but samples from other strata a few feet above or below gave entirely different glow curves (*29*).

Extensive studies have been made of thermoluminescence as a means of correlating limestones. For this purpose the glow curves obtained after excitation with γ-rays are more useful than the natural thermoluminescence curves, because many more peaks are available for identification in the low-temperature region—peaks that have been annealed out of the natural thermoluminescence at earth temperatures. In general, there are four prominent peaks in the glow curves of most limestones. They occur at 120°–140°, 150°–190°, 210°–250°, and 290°–310°C. Saunders (*27, 29*) and Bergstrom (*30*) have made a special study of the correlation of limestones as revealed by their thermoluminescence glow curves. Particular attention has been given to the Pennsylvanian limestone outcrops in Iowa. Parks (*31*) has made a special study of subsurface stratigraphy as revealed by glow curves, using core samples from areas around southern Indiana and southern Illinois.

AGE DETERMINATION

If suitable corrections can be made for chemical impurities and physical imperfections, and for the light absorption in the rock itself, one should be able to estimate the age of the rock from the thermoluminescence intensity and from the uranium or thorium content as determined from the α-ray activity of the sample. Each α-particle produces a given number of trapped electrons that will emit thermoluminescence. The light emitted should be proportional to the number of α-particles per year multiplied by the number of years since the crystal was formed. This method is much less reliable than the method of radioactive carbon for dating material, but the carbon method is satisfactory only for carbonaceous materials less than 25,000 years old. It is still less reliable than the lead-uranium, helium-uranium, potassium-argon ratios, but these radioactive methods also have their limitations. Any new method for dating rocks and minerals is well worth exploring, and progress is being made in the thermoluminescence method, which can give only the time since the mineral was last crystallized. It has been developed for limestones by Zeller (*32*) and for fluorites by Saunders (*33*). The α-activities are obtained with a scintillometer, applied by Ockerman (*34*) to a large area of the powdered rock. If the uranium content is only 1 ppm, the accuracy in determining the α-activities becomes the limiting factor. The area under a selected high-temperature peak in the natural glow curve is used for comparison—or, in another method, the electron traps thought to be produced by α-particles are filled by saturating with γ-rays. The light absorption of each sample is determined experimentally so that corrections can be made for light losses in samples that are not completely transparent. Limestones of known geological ages are used for a calibration scale.

The greatest uncertainties lie in the presence of chemical impurities and imperfections in the crystal lattice, which will increase or decrease the thermoluminescence. It is hoped that better results will be obtained through the use of a 1-curie polonium source, lent by the Atomic Energy Commission, with which an α-ray bombardment can be given to a thin surface layer in a second, which is equivalent to a million-year bombardment in the limestone with its mere trace of uranium. With this technique it should be possible to cancel out the effects of impurities; and, except for uncertainties in the radiation damage, the time required for the polonium source to match the natural thermoluminescence of the rock would give a direct measure of the α-particles emitted since the rock was formed. Knowing the rate of α-particle emission per hour and per year in the rock, the time required to produce the observed thermoluminescence could be easily calculated.

The possibility of estimating the dates at which limestones and ancient pottery were heated to high temperatures is now being explored. Many other applications of thermoluminescence to various laboratory techniques and applied problems are under investigation.

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