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DETECTION OF STRAIN IN ROCKS USING AN INTRINSIC "SEMI-INSULATOR" CHARACTERISTIC OF SOME MINERALS

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Abstract—Strain induced by impact or static loading will cause changes in some of the solid-state characteristics of minerals. Although exact relationships have not yet been demonstrated, thermoluminescence provides one means of obtaining a *relative* indication of these changes.

Data from investigations of suites of rocks from the vicinity of the Gaspe Copper "A" fault; the Charlevoix, Holleford and Brent "meteorite" craters; and the Sedan nuclear event, are used to illustrate some of the variations of strain-induced thermoluminescence. The results of artificial static loading of high-purity limestone from Bedford, Quebec, and of crushing and grinding of several rock types are also described.

An idealized thermoluminescence vs load relationship is suggested as a partial explanation of the changes in "semi-insulator" characteristics due to deformation.

Strain-induced lattice defects may produce marked changes in the solid state characteristics of some minerals. One method of investigating strain effects has been the observation of x-ray defraction line broadening or the blurring of Laue patterns (Johnson and Daniels, 1961; Roach, 1961, 1968; Aitken, 1965; Myner, 1967). A second line of investigation has been based on the observation of variations of a solid state characteristic such as: the formation of *F*-centers due to translational gliding in calcite and halite (Handin *et al.*, 1957; Agullo-Lopez and Levy, 1964); the increase in the number of etch pits in Iceland spar (Keith and Gilman, 1960; Kolantsova *et al.*, 1962; Thomas and Renshaw, 1967; d'Albissin and Fornaca-Rinaldi, 1968); measurement of the electronic charge on lattice dislocations in NaCl (Turner and Whitworth, 1968); and the strain-induced thermoluminescence of carbonate minerals, feldspars, and quartz. Exact relationships have not yet been demonstrated, but since many of the principles involved are similar to those for silicon whisker semi-conductor strain gauges, these "semi-insulator" phenomena in minerals may ultimately provide a means of measuring the amount of strain to which a rock has been subjected. For the present, where permament deformation has occured, a comparison of strained and unstrained samples may provide a relative indication of the amount of deformation.

Strain-induced thermoluminescence can be defined as thermoluminescence due solely to physical deformation and not to ionizing radiation. A number of investigators have previously reported studies of strain-induced thermoluminescence by: (i) uniaxial and triaxial loading (Zeller *et al.*, 1955; Lewis *et al.*, 1956; Handin *et al.*, 1957; d'Albissin, 1963; Ovchinnikov and Maxsenkov, 1963; Angino, 1964; Morency and McDougall, 1964; Morency, 1968; d'Albissin and Fornaca-Rinaldi, 1968); (ii) projectile impact (Roach *et al.*, 1961); and (iii) grinding (Debenidetti, 1958; Johnson and Daniels, 1961; Fornaca-Rinaldi, and Tongiorgi, 1961; Lewis, 1968). Field studies of natural strain-induced thermoluminescence have included investigations in the vicinity of alpine nappes (d'Albissin *et al.*, 1962, d'Albissin, 1963); meteorite craters (Roach *et al.*, 1962; Fuex, 1967); underground nuclear events (Dickey, 1960; Fuex, 1967; Roach, 1968); and faults (McDougall, 1968a, 1968b).

There appears to be some disagreement in the literature, but the *initial* effect of increasing strain is usually reported as a change in amplitude of one or more glow curve peaks and an increase in the total emission. Still greater strain usually results in a decrease in thermoluminescence. In some cases, strain may cause either the appearance or disappearance of certain glow curve peaks. The causes of changes in thermoluminescence due to strain are not well understood, but, in part at least, appear to be due to variations in the free energy of the crystal (McDougall, 1968b) which in turn is related to the formation and annihilation of lattice dislocations. Over some ranges of increasing deformation, thermoluminescence varies uniformly with strain, but over greater ranges, this simple relationship is not always apparent.

The studies reported below include; (a) investigations of rocks in which varying amounts of strain are either obvious or can be inferred; and (b) material which has been artificially strained in the laboratory. In them, the total emission (kE), natural glow curve peak heights (Pk_1, Pk_2) and peak height ratios (Pk_2/Pk_1) (all in arbitrary units) have been used to try to relate thermoluminescence to strain. In addition, in order to combine information on the amplitude of the peaks and total emission, the following relationship has been devised by one of the authors (G. D.)

$$\frac{kE}{(Pk_2/Pk_1) + 1}$$

FAULTS

One of the authors has previously reported examples of increased thermoluminescence in the vicinity of faults (McDougall, 1968a, 1968b). In the earlier discussions it was uncertain as to what extent the rocks might have been affected by post-faulting metamorphism or metasomatism. However, the Gaspe Copper "A" fault appears to be a case where the faulting has not been affected by more recent events. This fault is a well defined normal fault on which the north side has moved up. At the outcrop surface the fault intersects Grande Greve (Devonian) calcareous siltstones, including a portion which is within the metamorphic aureole surrounding the ore zones of the Gaspe Copper mine. During detailed geological mapping of the region surrounding the mine, the presence of a nearby body of the younger York Lake sandstone was thought to be due to graben-like faulting, and two additional faults ("C" and "D") have been postulated (Brummer, 1966). However, other than the presence of sandstone, there does not appear to be any real evidence for these two faults.

Figure 1 illustrates several thermoluminescence parameters of Grande Greve outcrop samples along a section approximately at right angles to the strike of the "A" fault. The two samples approximately 500 feet north and 800 feet south of the fault have been projected into the section from points about 1000 feet to the east.

The samples within and north of the fault are inside the alteration aureole, and the most northerly sample is at the southern edge of the Needle Mountain ore zone. In the direction of the fault, the thermoluminescence parameters kE (total emission), $kE/[(Pk_2/Pk_1 + 1)]$, Pk_1 (amplitude of first peak), Pk_2 (amplitude of second peak), all show a distinct increase, and the ratio Pk_2/Pk_1, shows a distinct decrease. All these changes are believed to have resulted from increased strain in the direction of the fault. The lack of similar effects near the postulated "C" and "D" faults suggest that they probably do not exist. In this general connection it may be noted that, the thermoluminescence of the alteration aureole is always distinctly higher than that of the unaltered rocks, and that samples taken elsewhere along the fault may have peak height ratios as low as zero.

METEORITE CRATERS

Shock effects have been shown to modify the thermoluminescence of rocks in the vicinity of meteorite craters (Roach *et al.*, 1962). Some aspects of the thermoluminescence response from samples taken from the vicinity of three Canadian "craters" are described below.

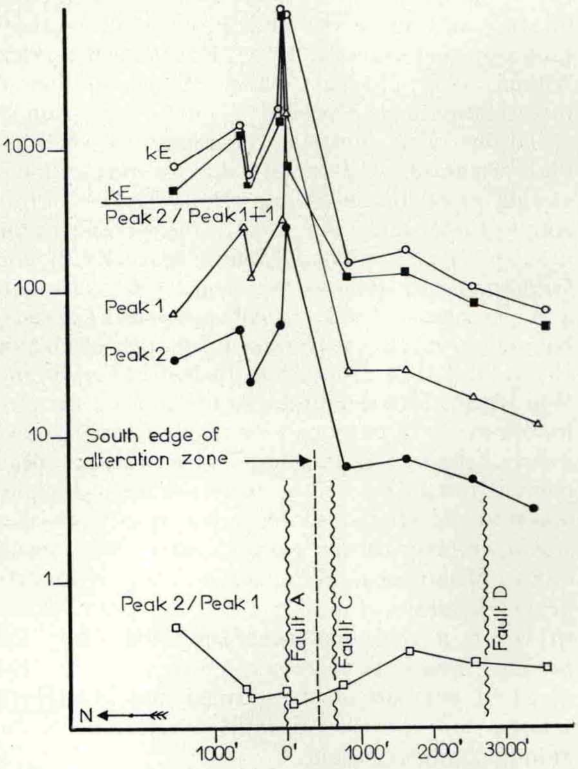

FIG. 1. Variations in thermoluminescence parameters in the vicinity of faults and zone of recrystallization (alteration zone).

(i) The recently described semi-circular Charlevoix structure (Rondot, 1968), lies along the north shore of the St. Lawrence river between Baie St. Paul and La Malbaie, Quebec. The rocks within the structure include Precambrian metasedimentary gneisses, anorthosite, charnockitic rocks, granodiorite, granite, and Ordovician sedimentary rocks including sandstones, limestones, and shales. Six samples, taken in pairs, from the Ordovician (Trenton?) limestone within the structure and a pair of samples of Trenton limestone from outside the structure were collected by E. H. Chown. Data was obtained for the total natural emission (kE), the peak amplitude (Pk_2), and the total emission from x-ray excited low temperature peak. One of the peaks (Pk_1) could not always be satisfactorily resolved, so that the values for the peak ratio (Pk_2/Pk_1) are only approximations. Figure 2 shows the spread of values from each collection site and the general tendency for a decrease towards the center of the structure. For the radiation-induced peak in particular, this decrease suggests that there has been a progressive distruction of a solid-state characteristic with increasing levels of shock.

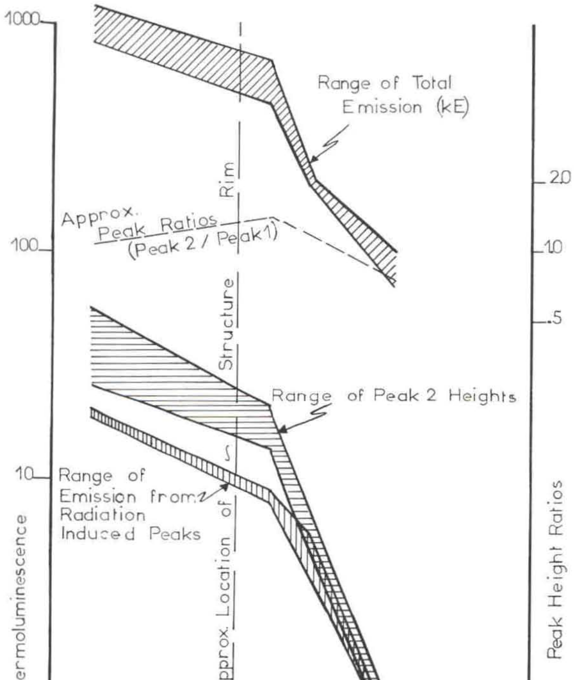

FIG. 2. Variation in thermoluminescence parameters in vicinity of meteorite "crater" (0 miles at crater center).

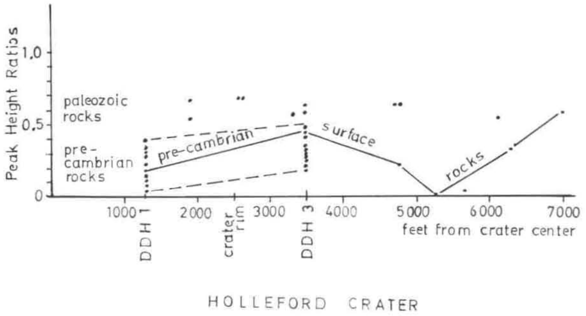

FIG. 3. Thermoluminescence peak height ratio in the vicinity of meteorite "crater" (0 feet at crater center).

(ii) The Holleford structure is located about 17 miles northwest of Kingston, Ontario. The present topographic surface is a basin about 5000 feet in diameter in which there are saucer-shaped beds of limestones which are not found outside the basin. Underlying these there is a sequence of buff and grey limestones with some shale; and basal beds of red and green shales, green calcareous arkose, greywackes, and conglomerates. These beds are middle Ordovician and rest unconformably on upper Cambrian (Potsdam) sandstones, or Precambrian marbles, quartzites, and gneisses. Drilling has shown that within the basin, the undeformed sediments overlie a polymict breccia of fragments of Precambrian rocks in a fine grained matrix. Similar breccias are not exposed in the Precambrian rocks outside the structure (Gold, 1964).

Sixteen samples of Precambrian marbles and Paleozoic limestones and calcareous sandstones were collected by the senior author from surface outcrops in and near the basin. Twenty-one samples of Paleozoic limestones and Precambrian marbles from a diamond drill hole (DDH 3) outside the basin rim and thirty-one samples of Paleozoic limestones, Precambrian gneisses and polymict breccia from a diamond drill hole (DDH 1) were supplied by D. P. Gold. Figure 3 illustrates the variations of the Pk_2/Pk_1 ratio of samples of the Paleozoic and Precambrian rocks at increasing distances away from the basin center. The ratios for most of the Paleozoic samples are rather uniformly between 0.7 and 0.5, while most of the Precambrian samples are less than 0.5. The low ratios for most of the Precambrian samples suggest high levels of strain. A possible explanation for the low ratios in some of the Paleozoic sedimentary rocks (DDH 1) is that they are derived in part from the strained Precambrian rocks.

(iii) The Brent structure in the upper Ottawa valley, Ontario, is a "super-imposed circular structure, some 10,000 feet across, in alamandine–amphibolite facies quartzo–feldspathic gneiss of Grenville (Precambrian) age. A bowl-shaped structure underlain by a pod of breccia and shattered country rock has been delinated by drilling'' (Gold, 1966). Forty-two outcrop samples from an area south and east of the structure were provided by D. P. Gold. The amplitudes of the single peak glow curves from these samples were generally low with some higher values. There is an apparent tendency for the higher values to be arranged in two zones, roughly concentric to the structure rim. The first high zone is about 500 feet from the rim, while the second varies in distance from the rim from about 2000 feet on the east to about 3500 feet on the south. The more common low values appear to be in accord with the usual effects of high levels of shock, but the higher values cannot be explained on this basis. An alternative explanation, which is not related to shock, can be found in the observation that thermoluminescence should be high in the greenschist and granulite facies and low in the almandine–amphibolite facies. (McDougall, 1970)

NUCLEAR EXPLOSIONS

Glow curves from twelve samples of quartzite and granodiorite from the vicinity of the Sedan nuclear crater, Nevada Test Site, which were supplied by N. M. Short, are illustrated in Figure 4. The glow curves are numbered on an arbitrary scale of increasing shock based on increasing amounts of isotropized quartz and glassy material (zero indicates unshocked samples). In general, the shocked samples are less thermoluminescent than the unshocked samples. Examination of the glow curves from the quartzites shows that with increasing shock: (a) the level of thermoluminescence does not decrease uniformly; (b) the shape of the curves changes non-uniformly; (c) there is no systematic increase in individual peak amplitude which might be ascribed to radioactivity from the nuclear explosion; (d) there is apparently no systematic change in the peak ratio which can be ascribed to either low level strain or heating; and (e) the decrease in thermoluminescence is not the same as the decrease due to crushing. A very recent study of the quartzites has shown that with increasing shock, peaks due to artificial irradiation decrease uniformly and the thermal activation energy of these peaks increases fairly uniformly (Manconi and McDougall, 1970).

CRUSHING EFFECT

The decrease in thermoluminescence due to decreasing grain size is principally due to increased light scattering and absorption by fine particles (Fornaca-Rinaldi and Tongiorgi, 1961). This effect is illustrated in Figure 4 for unshocked quartzite from the vicinity of the Sedan nuclear crater; syenite from Brome Mountain, Quebec; and high purity limestone from Bedford, Quebec. In each case the effect of decreased grain size has been to reduce the amplitude of the glow curves with virtually no change in the peak ratios or the position of the peaks. Further reduction in size by a few minutes of hand grinding of the finest fraction caused a further reduction in the glow curves. Both Fornaca-Rinaldi and Tongiorgi, (1961) and Lewis (1968) have observed increases in emission from specific peaks due to fine grinding, but this effect was not detectable in any of this group of samples. Except as noted above, the reduction in size of the particles was accomplished with a laboratory jaw crusher and it is believed that this treatment inhibits the formation of lattice dislocations during communution. The justification for this belief lies in the observation that very few dislocations are formed in Iceland spar if it is cleaved quickly (Gilman and Johnston, 1957).

ARTIFICIAL STATIC LOADING

Artificially deformed carbonate rocks usually show an initial increase in thermoluminescence after they have been subjected to low strain followed by a reduction in thermoluminescence at higher strain (Angino, 1964; Morency and McDougall, 1964; d'Albissin and Fornaca-Rinaldi, 1968). However, triaxial loading of machined samples of limestone from Bedford, Quebec, and syenite from Brome Mountain, Quebec, at axial load increments of 1000 psi, and several comparatively low confining pressures, resulted in a cyclical variation in thermoluminescence, with apparently no real change in thermoluminescence over the ranges tested. At the same time, direct comparisons of the variations of thermoluminescence with stress-strain curves, suggested but did not prove that the

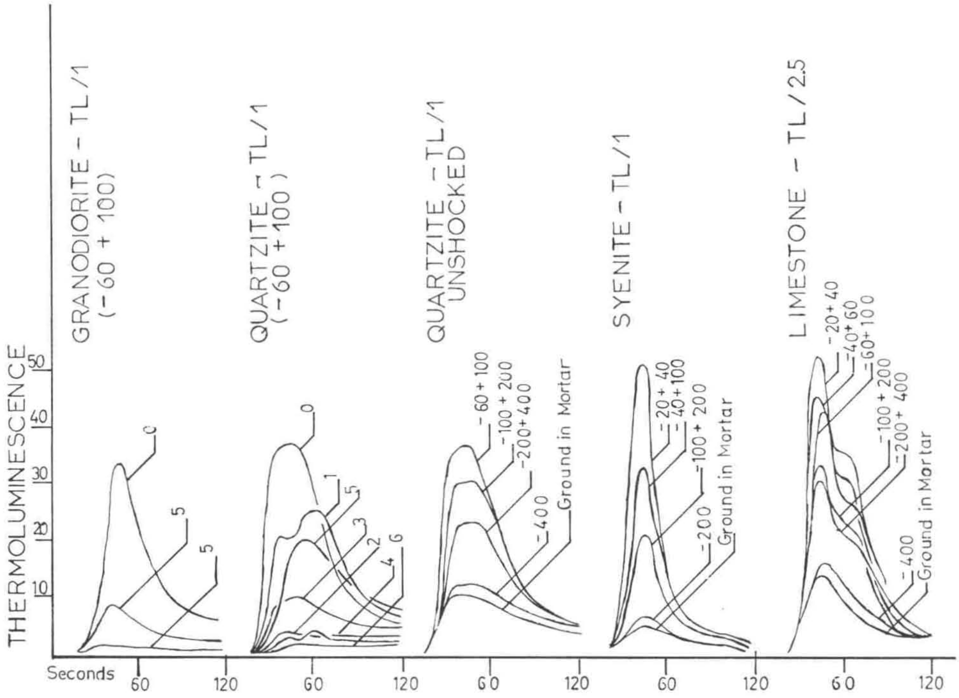

FIG. 4. Thermoluminescence glow curves comparing: (a) unshocked (0) and shocked (5) granodiorite; (b) unshocked (0) and shocked (1 to 6) quartzite; (c, d, e) effect of particle size reduction in unshocked quartizite, syenite, and limestone. The peak heights of the limestone samples should be multiplied by a factor of 2.5 for direct comparison with the other cases.

increases in thermoluminescence may be related to stages of virtually no deformation, whereas decreases in thermoluminescence may be related to stages of deformation (Morency, 1968). Figure 5 illustrates various thermoluminescence parameters of the Bedford limestone at a confining pressure of 2000 psi. There is questionable increase in thermoluminescence at about the middle of the axial load range and a very definite decrease in thermoluminescence at failure (29,000 psi). The peak height ratio curve appears to reflect comparatively low strain conditions up to about 8000 psi (ratio about 0.7), followed by higher strain conditions up to about 28,000 psi (ratio about 0.4), and finally a release of strain at 29,000 psi (ratio about 0.9).

Over a period of a few months there did not appear to be any appreciable change in the strain-induced thermoluminescence due to aging.

DISCUSSION

Stress-induced changes in the intrinsic semi-insulator characteristics of some minerals, as reflected in their thermoluminescence, can apparently be considered in three stages:

(1) When strain has caused an initial increase in thermoluminescence, it appears probable that at least part of the photon energy has been stored in lattice dislocations. In some, but not all cases, it is possible to assume that the release of photons on heating may have been accomplished by the annealing of some part of the dislocation network.

(2) Not uncommonly, further strain results in a decreased level of thermoluminescence. A possible explanation lies in the annihilation of previously formed dislocations. The cyclical increase and decrease of thermoluminescence which has sometimes been observed with increasing strain, would thus be due to a cyclical process of formation and repair of dislocations. This cyclical process is readily explained in polycrystalline materials by assuming different levels of strain in crystals of different orientations.

(3) In some cases, very high levels of strain (failure and impact) will cause a marked decrease in thermoluminescence. In part this effect may be

due to the formation of numerous fracture surfaces in the crystals and a resultant increase in light scattering and absorption. However, the effect often appears to be more complex than what might be anticipated from a simple reduction in particle size and may be the result of a change from long-range to short-range order in the material. Some lines of evidence indicate that the change involves approaching the condition of being an insulator instead of a semi-insulator.

A rather unsatisfactory hypothesis to explain the initial increase in thermoluminescence (stage 1 above) is that it may correspond to an anelastic damping effect. Ideally, such a lag of strain behind stress should decrease exponentially with time at constant temperature. However, the continued presence of strain-induced thermoluminescence in artificially deformed materials for several months, as well as its presence in the vicinity of geologically-old faults and meteorite craters would suggest that if there is any time-dependant decrease, it must proceed at an extremely slow rate.

On the other hand, there appears to be some grounds for comparing the first two stages noted above with stress-strain relationships in crystalline materials. The initial increase in thermoluminescence may correspond to strain-hardening due to the development of dislocation nets in the crystals. If this relationship is valid, then strain-induced thermoluminescence should be initiated at about the elastic limit and reach a maximum during the early stages of permanent deformation. The subsequent decrease in thermoluminescence would then correspond to the later stage of plastic deformation and result from the annihilation of dislocations during the recovery from strain-hardening. This suggested relationship is illustrated in Figure 6. The first portion of this curve resembles a Gaussian distribution. If it is assumed that the thermoluminescence is approximately proportional to the number of dislocations and that the number of dislocations are approximately inversely proportional to the amount of deformation, then a suitable cumulative plot of the thermoluminescence should resemble a stress-strain diagram. Some of the cyclical strain-induced thermoluminescence data obtained by one of the authors (M. M.) has recently been treated in this fashion by the senior author, and curves obtained which approximate the somewhat irregular stress-strain curves from the same material. These results, although encouraging, have not yet been able to provide a satisfactory correlation between the amount of deformation and thermoluminescence.

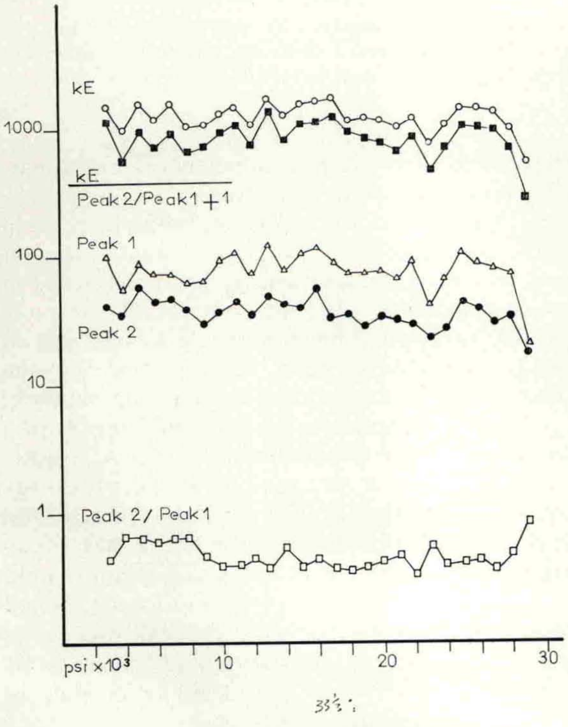

FIG. 5. Variations in thermoluminescence parameters in artificially loaded limestone samples at constant confining pressures.

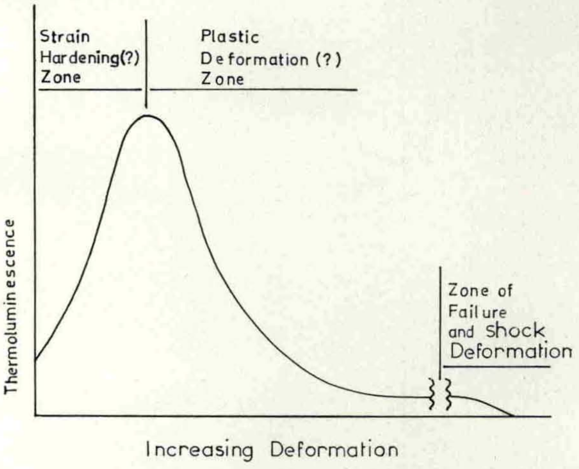

FIG. 6. Tentative proposal of relationships of thermoluminescence to deformation (strain).

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