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ANOMALOUS FADING OF THERMOLUMINESCENCE IN METEORITES

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Anomalous fading in meteorites of diverse origin and history has been studied. The meteorites included the four known members of the shergottite class, Shergotty, Allan Hills A77005, Zagami and Elephant Moraine A79001, the type 3 ordinary chondrite Dhajala and the howardite Kapoeta. After irradiation and 15 days storage at room temperature, considerable fading of the TL of the Kapoeta meteorite and two of the shergottites (Shergotty and 77005) was observed, while the TL of the other shergottites and Dhajala remained constant. Mineral separation experiments and energy-dispersive X-ray analyses indicate that the phosphor responsible for the TL is located in the low-density feldspar rich fraction in the Dhajala meteorite and the shergottites and this is probably also true of Kapoeta. Despite the diverse history of the samples and major differences in composition, it is only the samples in which feldspar is thought to be present in the low-temperature form which display anomalous fading. It is argued that the present data are most consistent with the mechanism for anomalous fading involving overlap between the wavefunctions for the excited and ground state electrons. Fortunately, most meteorites contain feldspar in the high-temperature form and large scale studies of their natural TL should not be hampered by anomalous fading.

1. Introduction

Thermoluminescence (TL) measurements on meteorites have been used for investigating various aspects of their history, including shock and metamorphism [1–3], anomalous orbits [4,5], and terrestrial age [6,7]. The last two of these applications involve measurement of the natural TL level and implicitly involve assumptions about the thermal stability and decay rate of the TL. At room temperature, typical lifetimes for a TL peak can be as short as a few seconds for the shallower traps or as long as many millions of years for the deepest traps. However, a property of many terrestrial and lunar samples is that they suffer the phenomenon of "anomalous fading".

Anomalous fading is the name given to a loss of TL at high glow curve temperatures that cannot be explained by thermal decay as predicted by kinetic models. Although the effect could constitute a serious problem for natural TL studies, little progress has been made in understanding the phenomenon. Probably the most systematic recent study is that of Wintle [8] who observed anomalous fading while examining the TL properties of volcanic lavas of known age. A loss of 10–40% of the TL in the 350–500°C region of the glow curve occurred after periods of a few hours to a few days in samples of sanidine, labradorite, andesine, and bytownite and also in fluorapatite and zircon from non-volcanic sources. In a separate study, Wintle [9] showed that the activation energy responsible for this kind of fading is considerably less than the trap depth (~ 0.2 eV compared with 1.6 eV). Moreover, the fading can be either temperature-dependent or temperature-independent; anomalous fading in sanidine, for instance, was temperature-dependent above $\sim -18°$C, but temperature-independent at lower temperatures.

Several mechanisms have been proposed to account for anomalous fading. Garlick et al. [10] suggested that quantum-mechanical tunneling occurred as the wavefunction of the trapped charge overlapped that of the ground state electron. Townsend [11] suggested that defects could diffuse through the lattice and release trapped electrons non-radiatively when they encountered a trapping center. Wintle [9] suggested that anomalous fading

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could be caused by a reduction in the probability of luminescence being produced by an electron when released from its trap during the glow curve measurement, but does not provide a detailed model. At the present time, the tunneling theory is probably the strongest since the anomalous fading of labradorite and calcite shows a logarithmic time-dependence [33–35].

The present paper reports a study of the anomalous fading of the TL of a suite of meteorites of diverse origins and histories. The meteorites studied are the shergottites, Shergotty, Allan Hills A77005, Zagami and Elephant Moraine A79001, the howardite, Kapoeta, and the Dhajala ordinary chondrite. These rocks all have feldspar as their dominant TL phosphor, and their TL properties are similar in many respect, yet differ in that some show anomalous fading and others do not.

The shergottite meteorites are the products of igneous processes, probably on the planet Mars [12–16]. They are coarse-grained assemblages of pyroxene, olivine, whitlockite and a shock-produced glass with plagioclase composition called maskelynite. Hasan and Sears [17] have discussed the TL sensitivity values of the shergottites and concluded that the individual members of this meteorite class suffered post-shock recrystallization at a variety of cooling rates. The howardite Kapoeta, is an assemblage of plagioclase and pyroxene with textures and mineralogy similar to terrestrial dolerites and basalts. The textures reflect crystallization from a melt. The ordinary chondrites are aggregates of material present in the early solar system. Most of them suffered metamorphism, solid state recrystallization at elevated temperatures, during or after accretion, and this metamorphism caused a 10^5-fold increase in TL sensitivity. It seems that this increase reflects the formation of the TL phosphor, feldspar, by devitrification of a primary igneous glass.

Table 1
Samples studied, their sources and masses

Meteorite	Source [a]	Mass (mg)	Fading at 300°C [b] (%)
Shergotty, 20	GSI	172	35 ± 10
ALHA 77005, 68	MWG	57	65 ± 8
Zagami	BM	70	≤ 6
EETA79001 [c],170	MWG	30	≤ 5
Kapoeta	BM	(20) [d]	50 ± 3
Dhajala	PRL	(20) [d]	≤ 7

[a] MWG: Meteorite Working Group of NASA/NSF.
GSI: Geological Survey of India, Calcutta (Curatorial fragment number indicated).
BM: British Museum (R. Hutchison, Catalog Number BM1966,54).
PRL: Physical Research Laboratory, Ahmedabad (D. Lal).

[b] Errors quoted are 1 sigma.

[c] Lithology A [32].

[d] 20 mg aliquot taken from a large (~ 3 g) homogenized mass.

2. Experimental

Two fragments from each meteorite, whose sources and masses are listed in table 1, were ground and, where necessary, the magnetic component removed with a magnet. 5 mg aliquots were placed in a Cu dish for TL measurement using TL apparatus manufactured by Daybreak Nuclear and Medical, Salem, CT, with the solenoid valves replaced with metering valves and 0.5 mm aperture placed over the sample to shield against thermal emission from the heating strip [18]. The temperature of the nichrome heating strip was monitored with a chromel–alumel thermocouple, and the samples heated at a rate of 15°C/s. Corning 7-59 and 4-69 filters were placed in front of the sample assemblage to cut down black-body radiation. The TL signal was detected with an EM 9635B photomultiplier tube, which has a wavelength range of 320–600 nm and peak sensitivity around 400 nm. Photon counting electronics were used. The glow curves were obtained by removing the natural TL by heating the samples to 500°C in the TL apparatus and irradiating to 1 Mrad with 250 mCi Sr-90 beta source. Such a high dose was necessary to ensure reasonable filling of the deepest traps and accounts for the differences in peak temperature and width between the present data and those in ref. [17]; the latter dealt with the low-temperature regions of the glow curve and used a test dose of 25–50 krad. After measuring the induced TL curve, the samples were irradiated a second time with the same dose and then stored in darkness at room temperature for 15 days and their TL measured again. The samples were then irradiated a third time and the TL measured to check for changes in TL sensitivity.

To identify the mineral(s) responsible for the TL, a mineral separation for Allan Hills A77005 was performed using a density gradient column with Clerici solution as the heavy liquid and water as a light liquid [17,19]. Six density separates were obtained. Samples from each fraction were deposited on 10 × 0.45 mm copper disks for TL measurements and mineral identification. No attempt was made to calibrate the density of the column, instead the minerals on each disk were identified by a Tracor Northern NS-880 X-ray analysis system attached to a Cambridge S600 Scanning Electron Microscope.

3. Results

Our data for samples of bulk powder are shown in figs. 1–3. Figure 1(a) shows the TL glow curves for Allan Hills A77005 obtained immediately after irradiation (curve A) and after storage for 15 days at room temperature (curve B). The ratio of the two glow curves for each shergottite is shown in fig. 1(b). In the 300°C region of the glow curve, there is almost 65 and 35% loss of TL for A77005 and Shergotty, respectively after 15 days, while Zagami and A79001 show no loss above 300°C. Similar glow curves, and a plot of the ratio of the

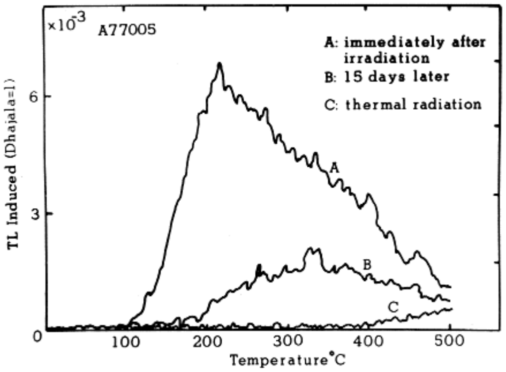

Fig. 1a. TL glow curves for the Allan Hills A77005 obtained immediately after beta irradiations (A) and after storing for 15 days at room temperature (B).

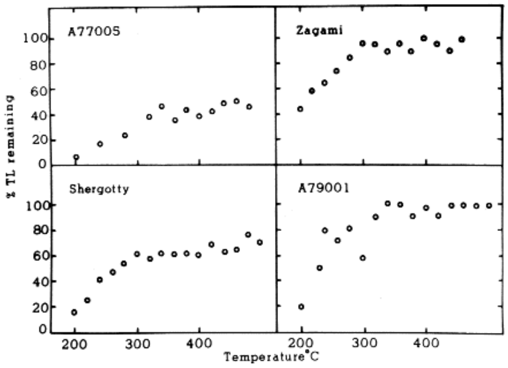

Fig. 1b. Ratio of the TL measured 15 days after irradiation to the TL measured immediately after irradiation as a function of glow curve temperature for the four shergottite meteorites.

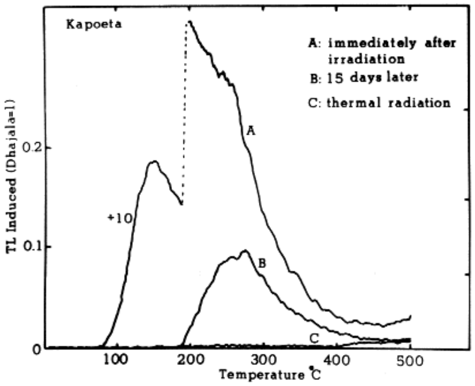

Fig. 2a. TL glow curves for the Kapoeta howardite obtained immediately after beta irradiation (A) and after storing for 15 days at room temperature (B).

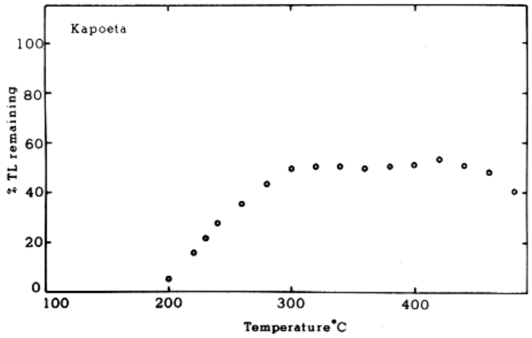

Fig. 2b. Ratio of the TL measured 15 days after irradiation to the TL measured immediately after irradiation as a function of glow curve temperature for Kapoeta meteorite.

TL before and after storage against glow curve temperature, are shown in fig. 2 for the Kapoeta meteorite. The 15 day storage at room temperature caused almost 50% loss of TL in the 300°C region of the glow curve. The Dhajala meteorite, whose data appear in fig. 3, showed no loss of TL above ~ 300°C in the glow curve.

We found that the anomalous fading in shergottites is temperature-dependent by placing one sample in a refrigerator (−5°C) for one week, one in an oven at 65°C for one week, and leaving another at room temperature (fig. 4). The refrigerated sample showed no loss of TL above 300°C in the glow curve, while for the others there was decay throughout the glow curve; at 400°C, the room temperature sample had lost 20% of its TL and the sample kept at 65°C had lost 40% of its TL.

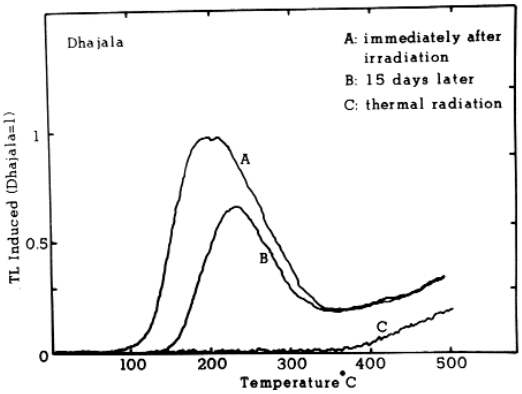

Fig. 3a. TL glow curves for the type 3 ordinary chondrite Dhajala obtained immediately after beta irradiation (A) and after storing for 15 days at room temperature (B).

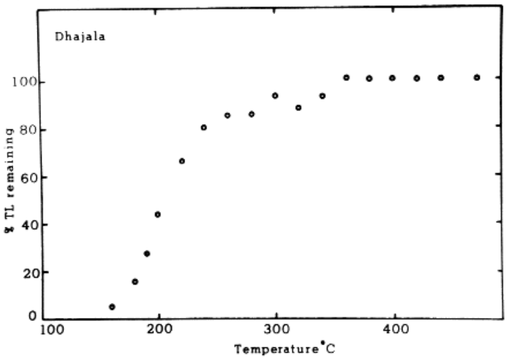

Fig. 3b. Ratio of the TL measured 15 days after irradiation to the TL measured immediately after irradiation as a function of glow curve temperature for the type 3 ordinary chondrite Dhajala.

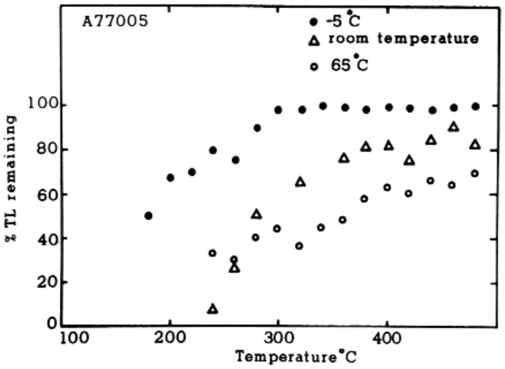

Fig. 4. Percent TL remaining after 7 days for samples of the Allan Hills A77005 shergottite meteorite stored at three different temperatures. The decay is clearly temperature-dependent.

Mineral separation experiments have shown that the TL phosphor in ordinary chondrites is feldspar [20], and our data for shergottites indicates that feldspar is also the TL phosphor in this class. The results of our mineral separation experiments on the shergottite A77005 appear in table 2. The bulk of the TL sensitivity lies in the light fractions in which maskelynite is located. The TL per unit mass for the light fraction is 18, compared with 0.079 for the most dense fractions, indicating that the TL is associated with the maskelynite. Annealing treatments under relatively mild conditions are known to cause crystallization of the maskelynite [21] and also a large increase in the TL sensitivity [17], so it seems very clear that it is crystalline feldspar in the maskelynite which produces the TL in shergottites. Although a mineral separation was performed only on A77005, because of the similar mineralogy of the four shergottites, it seems very likely that they would yield similar results. There are no data on the carrier phase for the TL in howardites, however since feldspar is abundant in this class, and there are no obvious alternatives, it seems most likely that the TL carrier in this class is also feldspar.

Table 2
Thermoluminescence sensitivity of 6 density separates from Allan Hills A77005

Fraction	Density	TL sens. (arb)	Mass (%)	Mineral composition[a]				
				Pyrox.	Oliv.	Feld.	Phos.	Metal
A	heavy	4.9	62	4	7	1	–	–
B	↓	6.6	13	10	3	5	–	–
C		8.3	10	6	4	3	–	–
D		5.1	8	8	7	4	–	–
E		12.5	5	4	10	4	1	–
F	light	36.0	2	2	2	13	–	1

[a] Number of grains identified by an energy-dispersive X-ray spectrometer on a Scanning Electron microscope.

4. Discussion

(a) The anomalous fading mechanism

We think it highly significant that some of our samples display anomalous fading, and others do not and there is no association between the occurrence of the phenomenon and the type of meteorite; two of the shergottites and the howardite display anomalous fading, while the other two shergottites and the ordinary chondrite apparently do not. These samples are highly diverse in their origin and history, and the feldspar they contain is very different in its petrologic setting and composition. In the shergottites, the feldspar is ~ 50 mole percent An (An_{50}) and is present in nanogram/gram quantities in an unusual shock-produced glass; in the Dhajala meteorite it is An_{10} in milligram/gram quantities in an igneous glass; in the howardite it is millimeter-sized crystals of An_{50} and constitutes about 40% of the meteorite. Despite these considerable differences, there is no obvious relationship between the composition of the feldspar or its mode of occurrence in the meteorite and anomalous fading. However, there does appear to be a relationship between anomalous fading and the degree of ordering in the feldspar.

It was discovered by Cole et al. in 1949 that the distribution of Al and Si atoms throughout the feldspar framework was regular ("ordered") at low temperature but became random ("disordered") at high temperatures [36]. Most silicate minerals show similar high-temperature disordering. The disordering occurs between 600 and 700°C for the sodium-rich feldspar, albite, and causes changes in the optical properties (e.g. refractive index) and cell parameters (e.g. the difference in the 2θ angle between the reflections from the 131 and $\bar{1}31$ planes). It also causes the TL peak temperature to increase from 120 to 210°C, even when the disordering is very slight [22,37,39].

The metamorphism experienced by the ordinary chondrite class of meteorites is described in terms of petrologic "types" 3 to 6, with the type 3 chondrites further subdivided into types 3.0–3.9 [3]. Type 3.3–3.5 chondrites have peak temperatures similar to feldspar in the low-temperature form, while type 3.6 and greater have peak temperatures similar to feldspar which has been partially converted to the high-temperature form by annealing treatments [2]. Thus greater degrees of metamorphism experienced are associated with the high-temperature form. Furthermore, annealing a type 3.4 meteorite at a variety of temperatures for 10–100 h causes TL peak temperature to increase from 120 to 220°C when the annealing temperature exceeds the low–high transformation temperature (fig. 5) [23]. It seems clear that the peak temperature is reflecting the state of disorder in the feldspar. There is also an increase in the width of the TL peak associated with the disordering, presumably because of the appearance of extra peaks in the glow curve [23].

We have used the same concepts to interpret trends in TL sensitivity, peak temperature and peak widths for the shergottites. Shergotty and Allan Hills A77005 apparently contain feldspar which is predominantly in the low form, while Elephant Moraine A79001 and Zagami contain

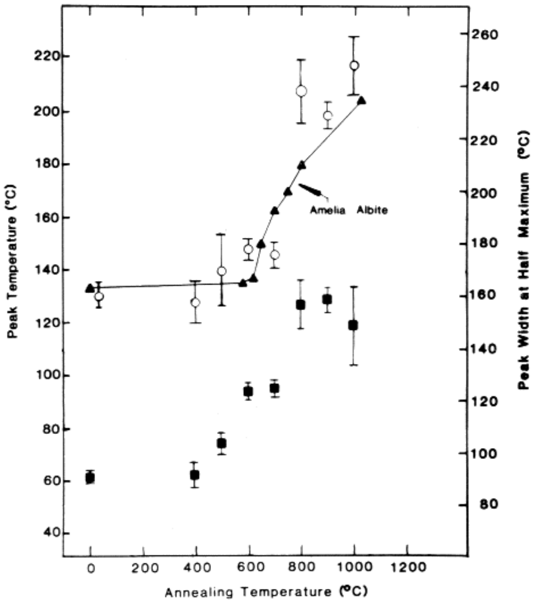

Fig. 5. Plot of TL peak temperature (circles and triangles, left axis) and peak width at half maximum intensity (squares, right axis) as a function of the temperature to which they had been annealed for 100 h. The circles and squares refer to a meteorite sample (Allan Hills A77011, which is petrologic type 3.4 [23]) and the triangle refer to a terrestrial feldspar (albite from Amelia, Virginia [23]). X-ray diffraction data showed that the annealing treatment caused the terrestrial feldspar to partially transform from the low-temperature form (ordered Al, Si structure) to the high-temperature form (disordered Al, Si structure) after annealing > 700°C. The feldspar in the meteorite is well below the detection limit for XRD, but a variety of evidence indicates that the source of TL in the meteorite is feldspar [21].

feldspar which is predominantly in the high form [17]. Kapoeta has a fairly narrow peak at 140°C inferring that its feldspar is predominantly in the low form.

Akber and Prescott [44] have suggested that the calcium-rich feldspars show anomalous fading while the Na-poor feldspars do not. Unfortunately, Akber and Prescott did not give structural details for their samples, and it is impossible from the data presented to make reasonable deductions based on source regions. However, it seems that their data too might be indicating that it is the state of disorder which is determining the extent of anomalous fading, not the Ca/Na. Figure 6 shows Akber and Prescott's data for an oligoclase sample of unknown provenance and a sample of oligoclase from Muskwa Lake (Canada) provided to us by Dr. B. Robertson of the Canadian Department of Energy, Mines and Resources, Ottowa. Our sample is known from X-ray diffraction studies to be in the low-temperature form [42,43] and we found that it showed fading of its TL up to at least 420°C in the glow curve when treated in the way of our meteorite samples. The prominent peak in our sample of oligoclase is at 120°C, while Akber and Prescott's sample has the dominant peak at almost 300°C, with a lesser peak at 220°C and a slightly smaller peak at 150°C; we surmise that their sample was in the high-temperature form. In any event, we do not believe that it is the Ca/Na which determines the extent of fading since the feldspars in our four shergottite samples are compositionally almost

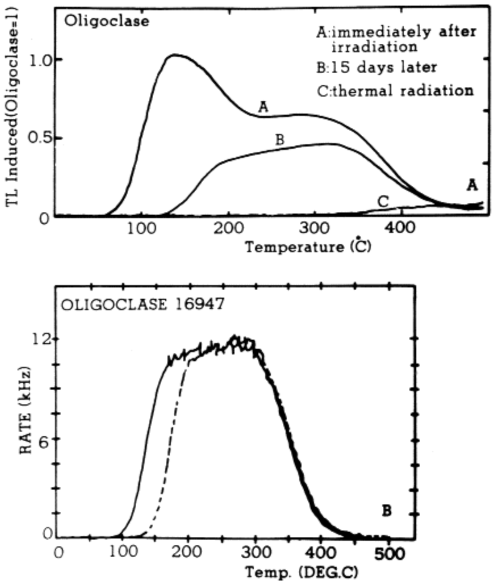

Fig. 6. Glow curves for two samples of the terrestrial feldspar oligoclase before and after storing for 15 days at room temperature. The upper curves are from the present work and are for a sample in the low-temperature form. The lower curves are from Akber and Prescott [44] and, on the basis of glow curve shape, probably refer to a sample in the high-temperature form.

Table 3
Thermoluminescence sensitivity values for plagioclase feldspars normalized to oligoclase

Feldspar	Source [a]	Present work [b]	Akber and Prescott [c]
Albite	Amelia, Virginia	$\left.\begin{array}{l}6.2\ \pm 0.4\\ 10.1\ \pm 0.8\end{array}\right\} 7.5 \pm 1$	3.8
Oligoclase	Muskwa Lake, Canada	$\left.\begin{array}{l}0.85\ \pm 0.03\\ 1.53\ \pm 0.08\\ 0.52\ \pm 0.03\\ 1.14\ \pm 0.11\end{array}\right\} 1.0 \pm 0.4$	1.0
Bytownite	Crystal Bay, Minnesota	0.028 ± 0.003	0.028

[a] Samples for the present work were provided by R. Ostertag (Univ. Munster, oligoclase and bytownite), B. Robertson (Canadian Dept. of Energy, Mines and Resources, Ottawa, oligoclase) and R.S. Clarke (Smithsonian Institution, Washington, DC, albite). The samples are described in refs. [22], [42] and [43].

[b] Errors are ±1 sigma based on 3–5 replicate measurements. Each value quoted refers to a separate chip; the range of values displayed by the oligoclase samples is associated, at least in part, to varying the grain size used.

[c] Read from fig. 1 of Akber and Prescott (44).

identical, yet two show anomalous fading and two do not. Akber and Prescott [44] also suggest that the TL sensitivity of feldspars depends on Ca/Na, with calcic plagioclases having lower TL sensitivity; our data confirm this suggestion (table 3).

Visocekas and co-workers have provided strong evidence that the mechanism for anomalous fading in labradorite (and calcite) involves quantum mechanical tunneling, since the level of TL remaining after fading varies with the logarithm of the fading time [33,34,35]. The data are consistent with the Medlin's model for the TL mechanism for calcite, which attributes both the trapping centre and the luminescent centre to the presence of Mn in the Ca sites [40]. Doping experiments have shown that the TL of feldspars is also associated with Mn substitution in Ca sites [26]. In $CaCO_3:Mn$ the TL emission is red and is caused by the 4g–4s transition in the d orbitals [40], but Geake et al. [26] have argued that the higher ligand field strength experienced by the Mn in feldspar would move the emission into the blue/green. Pasternak found that the disordering caused a slight change in the TL spectra, the wavelength of maximum emission increasing from 5580 Å to 5669 Å as the TL peak temperature changed from 120 to 210°C. Following Geake et al. [26] we may argue that this change in emission wavelength is associated with changes in the M–O bond lengths which accompany the disordering (where M = Na or Ca). There are 7 oxygen atoms surrounding the M atom in albite, and the distance of the two closest oxygen atoms increases almost 25% with disordering [21].

These changes in M–O bond length also provide a plausible explanation for the presence of anomalous fading in low-temperature feldspar and its absence in the high-temperature form. A reasonable model for the luminescence in feldspar involves electron transfer between Mn and O; such would be the case if the trapping involved the formation of Mn^+ and O^- and detrapping involved reforming Mn^{2+} and O^{2-}. The activation barrier which would have to be penetrated would then be thicker in the disordered phase than that ordered phase, and various studies have shown that the mean-life for quantum-mechanical tunnelling is very strongly dependent on the thickness of the barrier (e.g. [41]).

On the basis of the present data it appears that it is the meteorites that contain feldspar in the low form which display anomalous fading. The shergottites are particularly significant in this context, since those which display anomalous fading are similar in most respects to those which do not, the major difference being their TL peak temperatures and widths. We also note that the feldspar-bearing or feldspar samples which Wintle [9] and Akber and Prescott [44] showed were undergoing anomalous fading, had a TL peak temperature we believe to be associated with the low-temperature form (120–140°C).

Attempts have been made to separate the TL into a component which shows anomalous fading, and one which does not, in the hope that the "stable" component could be used for dating samples which show anomalous fading. Wintle suggested that 36% of the TL of a labradorite did not show anomalous fading, while the remainder did [9]. This would seem to indicate that 64% of Wintle's feldspar samples were in the low-temperature form.

The concept of defect diffusion as a mechanism for anomalous fading, as proposed by Townsend [11], faces the major difficulty that diffusion is very sluggish at low temperatures in a framework silicate structure. Diffusion of small cations appears to require temperatures above 500°C for ~ 1 week [24] and rearranging the Al and Si order requires temperatures of $\geqslant 800$°C for ~ 1000 h. Defect diffusion probably has lower activation energy than these two processes, but it is highly doubtful that it could occur at liquid hydrogen temperatures [9]. Conversely, if defect diffusion were the major mechanism for anomalous fading in feldspar, then it is hard to understand why the fading is even temperature-independent.

(b) Implications for TL studies of meteorites and dating

Ultimately, an understanding of the mechanism for anomalous fading could help in circumventing the difficulties it poses for dating and similar applications where thermal equilibrium or secular build-up of the TL are assumed. At present, it is important that the phenomenon be borne in mind when interpreting natural TL levels in any sample. Tests of a few weeks duration can be readily made, but there is always the possibility that the decay is occurring at a rate too small to detect in laboratory experiments, but fast enough to affect the natural samples. The present data suggest that TL fading in feldspars may be determined by structural factors, in which case a knowledge of the structure would provide an indication of the problems due to anomalous fading which are likely to be encountered.

The problems caused by anomalous fading in pottery dating applications are well-known [27]. For meteorites, the situation is different, but the potential problems equally serious. The period over which meteorites have been exposed to internal and cosmic radiation without complete draining is usually $\sim 10^6$ y, so that throughout the glow curve the TL is either at thermal equilibrium or saturated. Applications of natural TL in meteorites have therefore focused on variations in the natural TL level caused by changes in dose rate, ambient temperature, or both. The period of time on earth (terrestrial age) [6,7] and unusual orbits [4,28] involve the meteorite experiencing a change in both temperature and dose rate, while TL variations within meteorites may be due either to purely thermal effects (draining of the TL near the surface of the meteorite during atmospheric passage [29]) or, if the meteorite is sufficiently large, purely dose-rate effects (changes in the particle flux throughout the meteorite [5]).

The first two of these applications could be seriously affected by anomalous fading. This is especially important since it has been suggested that, because of its relevance to orbits and terrestrial age [30,31], the TL of the many thousands of meteorites being recovered in the Antarctic should be measured as part of their routine preliminary examination. The present data are encouraging in that over 90% of Antarctic stony meteorites are ordinary chondrites whose feldspar is in the high-temperature form and not expected to show anomalous fading.

Acknowledgements

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