ANOMALOUS FADING AND CRYSTALLINE STRUCTURE: STUDIES ON INDIVIDUAL CHONDRULES FROM THE SAME PARENT BODY

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Abstract: Plagioclase feldspar is the major luminescent mineral in meteorites. Thermoluminescence (TL) characteristics, peak temperature (T_m), full width at half maximum (FWHM), ratio of high (HT) to low temperature (LT) peak, and TL sensitivity (TL/dose/mass) to an extent reflect degree of crystallinity of the mineral. The present study explores and establishes a correlation between quantum mechanical anomalous (athermal) fading and structural state by examining TL of individual chondrules. Chondrules were separated using freeze-thaw technique from a single fragment of Dhajala meteorite. The results show large variation in T_m (155-230°C), FWHM (80-210°C) and HT/LT (0.07-0.47) and seem to be positively correlated. TL sensitivity (ranging from 14 to 554 counts/s/Gy/mg) decreases with increasing T_m and FWHM. Large variations in TL parameters (Tm, FWHM, HT/LT, and Sensitivty) suggest that individual chondrules had different degree of crystallization. Thermal annealing experiments suggest that comparatively ordered form of feldspar can be converted to a disordered form by annealing the sample at high temperatures (1000°C) for long time (10 hr) in vacuum (1 mbar pressure) condition and rapidly cooling it. Measured anomalous fading suggest that fading rate increases as the crystal form changes from an ordered state to a disordered state. However, the fading rate becomes nearly negligible for the most disordered feldspars.

Keywords: TL of chondrules, anomalous fading, degree of crystallinity, thermal metamorphism, thermal annealing.

1. INTRODUCTION

The natural and laboratory induced thermoluminescence (TL) of meteorites have been extensively used to inform on the meteorite properties and several planetary parameters. For example, the natural TL (NTL) of meteorites provides information on the perihelion of the meteoroid orbit (Melcher, 1981); terrestrial age (Sears and Durrani, 1980) and possible estimation of cosmic ray exposure age (Biswas *et al.*, 2011). The laboratory induced TL, on the other hand has provided a way for detailed classification of meteorites (Sears *et al.*, 1980).

Applications of luminescence to the estimation of terrestrial ages have been somewhat limited due the anomalous (athermal) loss of luminescence signal. Anomalous fading was first observed by Garlick and Robinson (1972) in lunar samples and later by Wintle (1973) in volcanic feldspars. This was explained as non-radiative quantum mechanical tunnelling of charges (Visocekas, 1985). More recent studies suggest that the trapped charges quantum mechanically tunnel from the ground state of the trap to the recombination centre (Poolton *et al.*, 2002a; Poolton *et al.*, 2002b). Despite the recent advance in the understanding of anomalous fading, the

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relationships of fading rate with lattice structure, chemical composition and the activation energies are not clear, and thus the fact that different grains from feldspars of the same provenance display different luminescence behaviour, needed elucidation (Huntley and Lamothe, 2001; Huntley and Lian, 2006; Jaek *et al.*, 2007).

In Shergottites (of different origin and history), that comprise feldspar as the most common luminescent mineral, Hasan *et al.* (1986) observed that anomalous fading occurred in only those meteorites in which plagioclase was in a low-temperature ordered form. They generalized that feldspars with lower degree of thermal metamorphism (petrologic type 3.3-3.5) exhibited anomalous fading and feldspar with higher degree of metamorphism (type 3.6 and above) did not exhibit fading. Later, Tyler and McKeever (1988) reported that both the low- and the high-temperature form of oligoclase exhibited fading. Visocekas (2002) suggested that for terrestrial feldspar, only disordered one showed fading and ordered feldspar gave a stable luminescence signal. Thus, the basic question of elucidation of the state of lattice, when a feldspar crystal becomes prone to anomalous fading still remains unresolved. In the present study we explored a near ideal possibility to establish the correlation between anomalous fading and structural state by measuring fading rate of individual grains from same provenance and composition but with different degree of crystallinity.

Towards this a comparison of the luminescence properties of individual chondrules from the same meteorite fragment (Dhajala meteorite; type 3.8) was attempted. Chondrules are sub millimetre to a millimetre sized spherical objects comprising silicate such as olivine and pyroxenes and these are surrounded by mesostasis which in type 3 ordinary chondrites generally consist of feldspathic glass, either glassy or crystalline (Huss *et al.,* 2006; Sears *et al.*, 1984). Although the exact mechanism of their formation is not understood, it is suggested that chondrules are formed by flash heating and cooling (Zanda, 2004) and that they were heated to about 1500-1600°C and cooled at rate ranging from 10 to 1000°C/h (Yu and Hewins, 1998).

It has been suggested that TL parameters such as temperature at which glow peak is maximum (T_m), full width at half maximum (FWHM), and sensitivity (TL/unit dose/unit mass) are related to the degree of crystallinity of the mineral (Sears, 1988). Sears *et al.* (1984) separated 58 chondrules from Dhajala meteorite and found a large variation in T_m (~125-225°C), FWHM (~55-190°C) and sensitivity (0.002-0.12 of the bulk sensitivity = 1). In that study, the TL of chondrules was measured without any optical filter, allowing whole TL emission limited only by the photomultiplier response. The present work extends this study by examining 17 chondrules separated from Dhajala meteorite and measuring their TL characteristics (T_m, FWHM and sensitivity), constraining the TL emission in violet-blue (395±30 nm) region, the major TL emission band, and additionally conducting measurements on the anomalous fading for each of these chondrules.

2. EXPERIMENTAL DETAILS

The chondrules were separated from the matrix using freeze-thaw technique. The bulk sample was repeatedly recycled between liquid nitrogen temperature and room temperature in an 800 W ultrasonic bath. After around 50 cycles, about a gram of chondrule and matrix were recovered. The samples were then sieved to 300-1000 μm grain fractions and the chondrules were manually picked under an optical microscope. Chondrules which had adherent matrix were not used. The separated chondrules were again cleaned in ultrasonic bath to remove any residual grains from matrix adhering to the chondrule surface. Individual chondrules were then mounted on stainless steel aliquots using silkospray. The weight of each chondrule was computed by measuring its dimension using an optical microscope, and assuming it to be an ellipsoid with a density of 3.4 g/cm^3.

The TL measurements were made using a Risø TL/OSL reader (TL/OSL DA-20; Bøtter-Jensen *et al.*, 2010). The detection optics comprised a Thorn-EMI 9235 QA photomultiplier tube coupled to Corning 7-59 and BG-39 filter combination (transmission 395±30 nm). The system had on-plate $^{90}Sr/^{90}Y$ beta irradiator. A linear heating rate of 2°C/s under constant flow of ultrapure nitrogen was used.

3. RESULTS

TL characteristics

The chondrules were heated to 500°C to record the natural TL signal, and then a 100 Gy beta dose was given to each followed by 12 h storage at room temperature, and a TL glow up to 500°C was measured. The background was then recorded for each and subtracted. The laboratory dosed TL glow curves of chondrules had two glow peaks, i.e. low (LT) and high temperature (HT) peaks (**Fig. 1a**). The chondrules with low sensitivity had significant fluctuations due to dark noise being a significant component in the TL glow curves, and hence these were smoothed using 5 point running average to identify the exact peak position and to calculate FWHM accurately (inset of **Fig. 1a**). The peak temperature (T_m) of the LT varied from 155-230°C between chondrules and the T_m of HT occurred in the range ~400-450°C. The FWHM of LT ranged from 80-210°C and that of HT was difficult to estimate because of contribution of the high temperature region of LT to HT. The peak height ratio of HT to LT varied from 0.07-0.47. The TL sensitivity, calculated by taking peak height of LT, varied from 14-554 counts/s/Gy/mg (**Table 1**), and the maximum TL sensitivity was ~40 times that of the least sensitive chondrule. **Table 1** provides the experimental data.
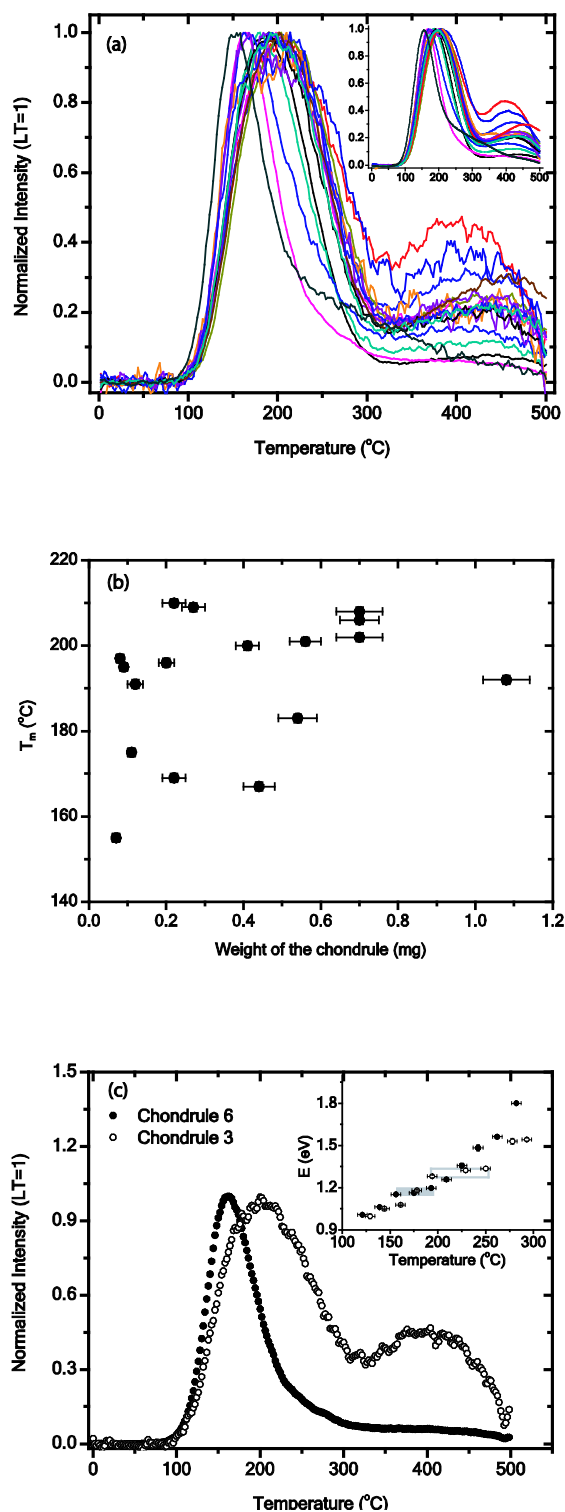
Fig. 1. *a) The normalized (LT = 1) TL glow curves of chondrules of 17 chondrules. The inset of the figure shows a smoothed curve obtained by taking running average, b) variation of peak temperature (T_m) with mass (size) of the chondrules, c) the normalized TL glow curves of two extreme chondrules, Chondrule 6 (maximum sensitivity) and Chondrule 3 (minimum sensitivity). The insets of the figure shows trap depth distributions of the two chondrules at different temperature of the glow peak and the bars indicate distinctive traps at ~1.17 and ~1.31 eV for chondrule 6 and 3 respectively.*

The TL glow curves were measured with a heating rate of 2°C/s under constant nitrogen flow and it was reasonably assumed that thermal lag even for larger grains was not an issue. To further examine this assumption, T_m of LT of all chondrules were plotted against their computed masses (**Fig. 1b**). The random distribution of the points suggested that size dependent thermal lag was not a matter of concern.

The normalized TL glow curves of two extreme chondrules, Chondrule 6 (maximum sensitivity) and Chondrule 3 (minimum sensitivity) were then compared (**Fig. 1c**). TL of both the chondrules had LT and HT but their TL parameters (T_m, FWHM, HT/LT and sensitivity) were different. To know further characteristics of traps, trap depth distribution of the two chondrules were calculated using the initial rise method (Randall and Wilkinson, 1945). The trap depth of the Chondrule 6 (higher sensitivity) vary from 1.0 to 1.8 eV and for Chondrule 3 (lower sensitivity) it vary from 1.0 to 1.6 eV (inset of **Fig. 1c**). Although they have nearly same range, the trap depth distribution patterns are different. A clear distinct trap, where the trap depth distribution is parallel to temperature axis, is observed for both the chondrules, but the values are somewhat different; for Chondrule 6 it is ~1.17 eV whereas for chondrule 3 it is 1.31 eV. These traps appear in the temperature ranges of ~156-193°C and ~194-250°C respectively and correspond to the two T_m (LT peak), 167°C and 202°C of the two respective chondrules. Considering the fact that TL of chondrule arises from feldspar mineral in an identical detection window, it can be plausibly suggested that the variation in TL parameters is due to different physical environment of the same defect.

Degree of crystallinity and TL parameters

Mutual correlation between TL parameters (T_m, FWHM, HT/LT, sensitivity) of LT peak was explored for clues to reason for differences between chondrules. A large variation in T_m (155-229°C), FWHM (80-210°C), HT/LT (0.07-0.47) and sensitivity (14-554 count/s/Gy/mg) is seen and these parameters are correlated. The FWHM vs. T_m (**Fig. 2a**), HT/LT vs. T_m (**Fig. 2b**) and HT/LT vs. FWHM (**Fig. 2c**) plots, depict some correlations. Although the data are scattered, an increasing correlation is discernible. **Figs. 2a** and **2c** show clear increasing trend, whereas the **Fig. 2b** shows larger scatter in the data and more like a cluster structure, still do discern an increasing trend.

The sensitivities of individual chondrules decrease with an increase of T_m and FWHM (**Figs. 3a** and **3b**). The sensitivities are presented in $\log_{10}$ scale. A similar trend between sensitivity, T_m and FWHM was observed by Sears *et al.* (1984).

Keck *et al.* (1986) used laboratory annealing experiment to suggest that T_m and FWHM increase if the crystallinity decreases. Since the parameter HT/LT follows similar trend as T_m and FWHM, it can be implicitly sug-

Table 1. *List of different measured quantities, like mass, peak temperature, FWHM, peak ratio HT/LT, sensitivity and fading, for 17 chondrules. The error in masses were calculated by propagating uncertainty in the measurement of dimension using optical microscope and for peak temperature ±1°C and for FWHM ±2°C error was considered. All TL parameters mentioned here are for LT peak.*

Chondrule	Mass (mg)	T_m (°C)	FWHM (°C)	(HT/LT) × 10	Sensitivity	Fading (%)
2	1.08±0.06	192	106	0.79±0.02	146±9	19.1±4.1
3	0.7±0.06	202	145	4.73±0.22	22±2	5±0.5
4	0.7±0.05	206	128	3.3±0.13	36±3	4.9±0.4
5	0.54±0.05	183	103	1.19±0.05	89±8	13.7±1.2
6	0.44±0.04	167	80	0.64±0.02	544±49	5.2±0.3
7	0.27±0.03	209	128	2.5±0.08	168±19	1±0.1
8	0.7±0.06	208	152	4.05±0.24	14±1	3.8±0.5
10	0.41±0.03	200	125	2.39±0.12	56±5	1.5±0.1
12	0.22±0.03	169	99	1.62±0.08	169±20	10.2±1.1
13	0.22±0.03	210	144	2.67±0.24	34±4	1.8±0.4
14	0.11±0.01	175	138	2.43±0.21	75±11	4.3±1
15	0.2±0.02	196	124	2.62±0.1	160±20	4.6±0.4
16	0.12±0.02	191	120	2.19±0.1	225±33	0±0
17	0.56±0.04	201	121	3.07±0.07	130±9	5.7±0.4
18	0.08±0.01	197	135	2.34±0.14	209±35	6.1±0.7
19	0.09±0.01	195	121	2.26±0.11	310±47	3.7±0.4
20	0.07±0.01	155	81	1.28±0.07	465±82	4.2±0.5

gested that the ratio HT/LT should also be correlated with crystallinity. Assuming this to be so, chondrules with lower T_m, FWHM and HT/LT, and higher sensitivity should have comparatively a more ordered form of feldspar glass, and chondrules with higher T_m, FWHM and HT/LT, and lower sensitivity should comprise more disordered form of feldspar glass. These results suggest that within a small piece of meteorite, individual chondrules have different degree of crystallinity.

Fading and crystallinity

Anomalous fading rate of individual chondrules was calculated by measuring instant TL signal (TL_1) immediately after artificial irradiation (100 Gy) and after storage for 43 days (TL_2) at room temperature (21°C). TL glow curves with and without fading are shown in **Fig. 4a**. Given that the sensitivity can change due to heating associated with a TL glow curve recording, sensitivity correction for the TL signal was made using a test dose (100 Gy) TL following each TL measurements. The test dose signal was used to normalize the corresponding TL signals. The ratio of sensitivity corrected TL_2 to TL_1 in the temperature region 300-400°C (inset of **Fig. 4a**), where the signals are thermally stable and plateaus of TL_2/TL_1 with temperature, was used for the estimation of fading for 43 days.

The fading rates of all the chondrules are plotted as a function of T_m (**Fig. 4b**) and FWHM (**Fig. 4c**). The results show two clusters, A and B. In region A (lower T_m and FWHM) fading rate increases with T_m and FWHM, whereas in region B (higher T_m and FWHM) fading rate is independent of T_m and FWHM and the values are near zero. In other word the fading rate initially increases with increasing the T_m and FWHM and then reduces to a lower value beyond $T_m = \sim190°C$ and FWHM = 110°C.

In region A, where the chondrules are in comparatively ordered state (lower T_m and FWHM, and higher sensitivity), the fading rate increases with decreasing the degree of crystallinity. And in region B, where the chondrules are in comparatively disordered state (higher T_m and FWHM, and lower sensitivity), the fading rate is negligible. Thus disordered form of feldspathic glass shows reduced fading. Short range order and low sensitivity in region B suggest that the volume density of traps is low and thereby the probability of overlap and tunnelling is also low, leading to low athermal fading.

Effect of thermal annealing

Two chondrules, Chondrule 6 (most ordered) and Chondrule 3 (most disordered), were annealed under identical conditions, at 1000°C under 1 mbar pressure for 10 h and then allowed to rapidly cool to room temperature by leaving the sealed capsules containing the samples in air. The TL parameters (T_m, FWHM, HT/LT, and sensitivity), before and after the thermal cycling, were then compared (**Table 2**). The TL glow curves, before and after annealing are shown in **Fig. 5**.

For Chondrule 6, due to thermal treatment, T_m marginally increased by 3°C, the FWHM increased by 86°C and the peak became skewed towards high temperature, the peak ratio HT/LT increased by factor of 4, and the sensitivity decreased by a factor of 6. For Chondrule 3, the change in TL parameters (particularly FWHM, HT/LT and sensitivity) was minimal, T_m increased by 5°C, the FWHM increased by 2°C, the peak ratio HT/LT increased by factor of 3, and the sensitivity decreased by a factor of 3 (**Table 2**). These results suggest that the annealing (temperature and cooling rate) change the crystal properties and the change is more for the chondrule with higher order.
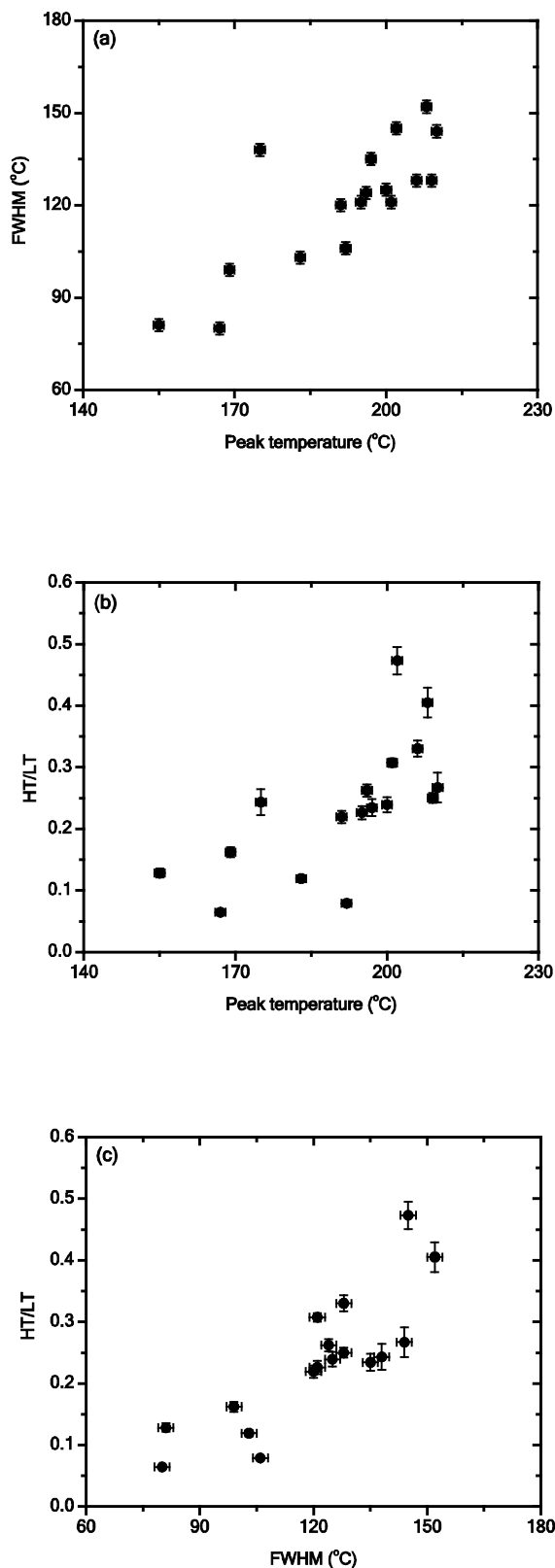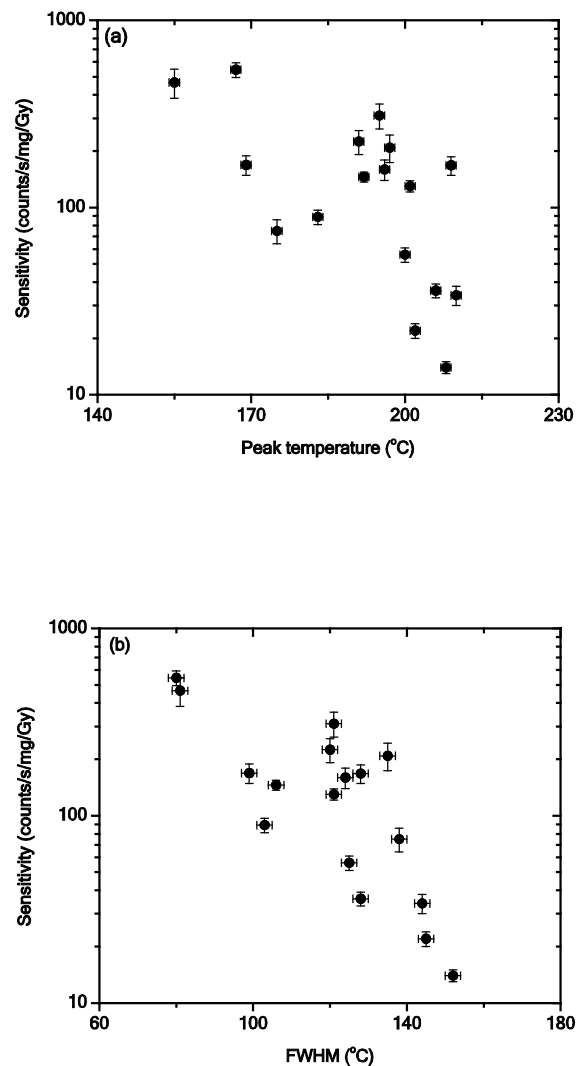
Fig. 3. *Variation of TL sensitivity with a) peak temperature and b) FWHM. The error in sensitivity is propagated error of peak height (counting error) and error in weight.*

Fig. 2. *Relationship between TL parameters (Tm, FWHM, and peak ratio HT/LT). a) FWHM vs. peak temperature, b) HT/LT vs. FWHM, c) HT/LT vs. Peak temperature. For peak temperature ±1°C and for FWHM ±2°C error was considered. The error for HT/LT is propagated counting statistics error of two peak height.*

A similar annealing exercise by Keck *et al.* (1986) for bulk Dhajala meteorite sample (at 800°C for 14 h), gave an increase of 36°C in the peak temperature, 40°C in FWHM and a decrease in sensitivity by a factor of 2.5. The results by Keck *et al.* (1986) cannot be compared directly with the results presented above, since their measurement was made for bulk sample and without any filter whereas the present measurements were made for chondrules and in blue emission window and also the measurement conditions were different. But these two results however do suggest that thermal treatment at high temperature followed by rapid cooling reduces the crystalline order of feldspathic glass. In the present case this change was discernible. However the activation energy distribution of Chondrule 6 (most ordered) did not change due to thermal annealing (**Fig. 6**).
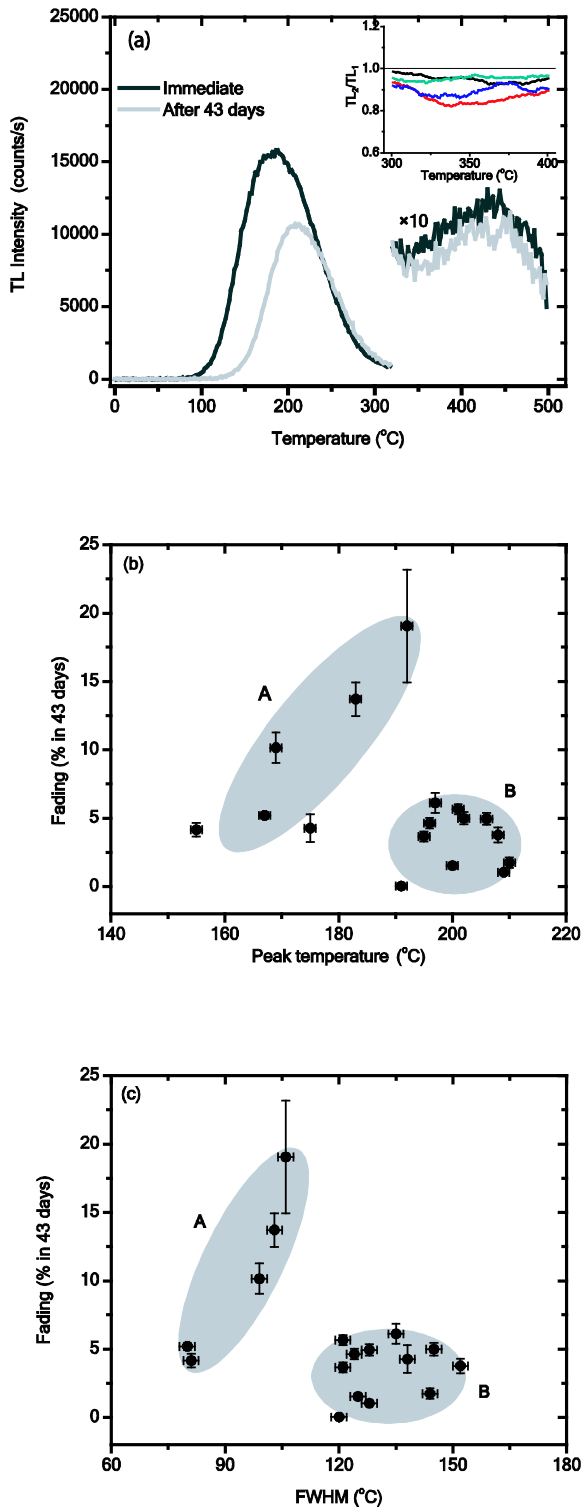
Fig. 5. *TL glow curves before and after annealing for a) Chondrule 6 (most ordered) and b) Chondrule 3 (most disordered). The insets of both the figures show normalized plot (LT=1).*

Fig. 4. *a) TL glow curves of the 100 Gy dosed chondrule (Chondrule 2) with (43 days storage after irradiation) and without (immediate after irradiation) fading. The high temperature parts of the TL glow curve (300-500°C) are blown up by multiplying a factor 10 to make it visible. The inset of the figure shows the ratio of TL₂ (with fading) to TL₁ (without fading) with glow curve temperature in the range 300-400°C; b, c) fading in % for 43 days vs. peak temperature and FWHM respectively. Two clear regions, A and B, are marked in both the figures.*
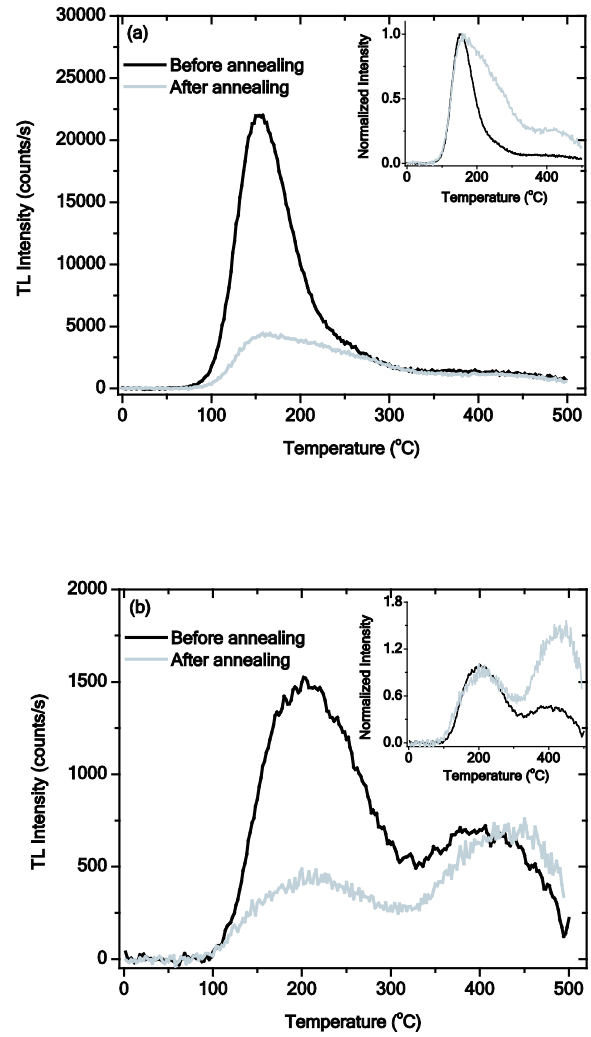
Table 2. *List of TL parameters of LT peak of Chondrule 6 and Chondrule 3 before and after annealing.*

		T_m (°C)	FWHM (°C)	HT/LT	Sensitivity
Chond 6	Before annealing	167±1	80±2	0.07±0.01	554±49
	After annealing	170±1	166±2	0.27±0.01	95±9
	Change	+3	+86	×4	/6
Chond 3	Before annealing	202±1	145±2	0.47±0.02	22±2
	After annealing	207±1*	147±2*	1.54±0.09	7±1
	Change	+5	+2	×3	/3

*Due to significant overlap between the LT and HT peak, the two peaks were de-convoluted using computerized curve de-convolution (CCD; Afouxenidis et al. 2012) and the parameters were calculated.
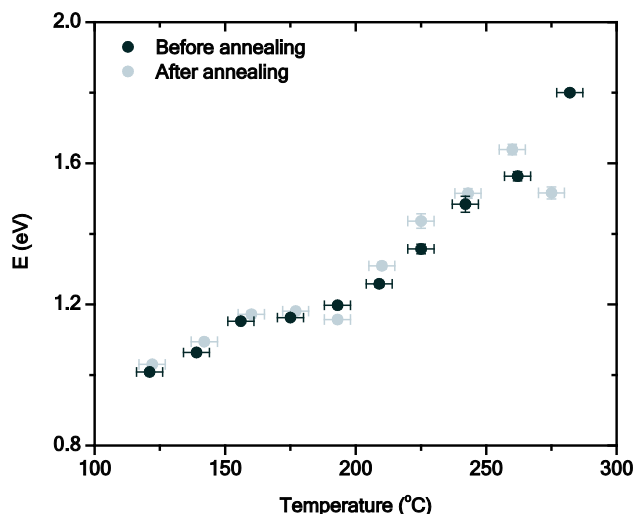
255

Fig. 6. *Plot of thermal activation energy at different temperature of the glow curves of Chondrule 6 before and after annealing.*

4. DISCUSSION

Variation of TL parameters from chondrule to chondrule

The present study indicates that within a single meteorite (Dhajala) body, individual chondrules have a range of TL parameters (T_m, FWHM, HT/LT and sensitivity) and as a first order approximation this can be attributed to variation in the crystallinity. Two possible reasons for such variations are: 1) the individual chondrules experienced different degrees of in situ thermal metamorphism after accretion in to parent body, and 2) chondrules formed under different crystallization temperature and/or experienced different cooling rate during crystallization, prior to their accretion in the parent body.

From the point of view of TL, the following is to be noted. The chondrules were collected from a meteorite piece of dimension ~1 cm^3 and within such a small volume element heterogeneity in thermal metamorphism is unlikely. Further, the variation of TL sensitivity with T_m and FWHM for chondrules is opposite to those present in such plot for bulk chondrites (Sears and Weeks, 1983; Sears *et al.*, 1984). Studies on different bulk chondrites by Sears and Weeks (1983) show that the TL sensitivity of chondrite increases with T_m and FWHM due to different degree of thermal metamorphism, occurs at temperatures ~400-600°C (Dodd, 1981). The present study and earlier studies by Sears *et al.* (1984) on individual chondrules show a decreasing trend of TL sensitivity with T_m and FWHM. These enable a logical inference that variation of TL parameters from chondrule to chondrule is not due to thermal metamorphism but due to variation in crystallization temperature or cooling rate during formation from a high temperature melt.

Variation of anomalous fading rate with degree of crystallinity

As the T_m and FWHM increase or the crystal becomes more amorphous, the anomalous fading rate first increase and then suddenly reduces to near zero value. This result accords with Hasan *et al.* (1986), where they concluded that ordered form of meteorite feldspar exhibited fading whereas disordered form of meteorite feldspar did not exhibit fading. A nearly similar study but on different type of sample by Berger (1985), showed that 100% clear volcanic glass, with a disordered lattice, did not show any anomalous fading.

Dependency of anomalous fading rate on crystallinity (section 3) suggests that the temperature during chondrule formation decides the crystallinity and anomalous fading rate. Higher cooling rate during crystallization leads to higher degree of amorphous phase. The plot of anomalous fading rate with T_m and FWHM (first increase and then reduction to near zero value) suggests that beyond certain annealing condition (temperature and cooling rate) a definite phase occurs and the anomalous fading rate of the mineral becomes nearly zero. And before that critical annealing condition, anomalous fading increases as the crystal moves towards amorphous side. A question that still persists is: why fading with crystallinity does not follow a monotonic trend, rather the fading parameter suddenly drops down to near zero value beyond certain degree of crystallinity? An incremental annealing condition (temperature and cooling rate) on easily available terrestrial feldspar coupled with X-ray diffraction (XRD) analysis on single grain may elucidate the processes better.

5. SUMMARY

1) In a single meteorite fragment (Dhajala) different chondrules have different TL parameters (T_m, FWHM, HT/LT and sensitivity),
2) T_m, FWHM and HT/LT are positively correlated, whereas sensitivity is negatively correlated with these parameters.
3) Chondrule with lower T_m, FWHM and HT/LT, and higher sensitivity have higher degree of crystallinity and vice versa.
4) As the crystallinity of the chondrules (feldspar) decreases, the anomalous fading rate first increases and then suddenly drops down to a near zero value.
5) The most disordered chondrules show negligible fading.

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