***IN-SITU* DATING ON MARS: PROCEDURES AND CHARACTERIZATION OF LUMINESCENCE FROM A MARTIAN SOIL SIMULANT AND MARTIAN METEORITES.**
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Introduction: The search for past and present environments on Mars that could have supported life will be assisted by *in-situ* measurements and future sample return missions which provide a chronology of water activity and aeolian processes, and establish the age of geomorphological features on Mars.

With the current focus on *in-situ* studies, Lepper and McKeever(1) proposed using luminescence dating techniques to help construct a geological history of Mars over the last one million years. Aeolian processes have long been suggested to shape the surface of Mars,(2) and studies are still underway to understand these processes.(3) Recent work has also shown that water could be present on the Martian surface for brief periods of time either currently or in the recent past(4) and could result in features such as young flow channels(5).

New luminescence dating procedures(6,7) could provide ages for several depositional environments, and analysis of the dose distributions among aliquots from a sample might help in inferring the nature of the depositional environments,(8) e. g., aeolian vs. waterlain deposits(9). The equipment required for *in-situ* luminescence measurements is straightforward, and the procedures used for mineral separates (i.e., quartz and feldspars) can be adapted for polymineral materials such as those expected to be encountered on Mars. The current work focuses on determining the luminescence properties of Martian soil simulants, analogs, and meteorites, and using this information to develop luminescence dating procedures for use on the Martian surface.

Luminescence Dating Principles: Luminescence dating is based on solid-state properties of mineral grains that allow them to record their exposure to radiation. Absorption of radiation causes ionization of electrons, which subsequently become trapped at electron trapping centers within the mineral. The concentration of such trapped electrons is a measure of the dose of radiation absorbed. When the irradiated sample is stimulated with light or heat, the electrons are released from their traps and recombine, releasing a portion of the stored energy as luminescence. If the luminescence is stimulated by light, it is called optically stimulated luminescence (OSL); if it is stimulated by heat it is called thermoluminescence (TL). The intensity of the luminescence emitted from the mineral during stimulation is a measure of the dose of radiation initially absorbed. The value of the absorbed dose can be determined using appropriate calibration techniques.

The sources of natural radiation are the decay of natural radioactive elements in the soil (U, Th and K), galactic cosmic rays (GCR), and solar particle events (SPE). By determining the *rate* of natural irradiation (either by direct measurement or calculation) of the mineral one can estimate the radiation exposure age according to:

$$\text{Age}, T\ (\text{a}) = \frac{\text{Equivalent natural dose}, D\ (\text{Gy})}{\text{Annual dose rate}, R\ (\text{Gy/a})}$$

where the SI unit for dose is the Gray (Gy, 1 J/kg).

The radiation environment on the surface of planet Mars is determined by four factors: the ambient radiation environment, the properties of the Martian atmosphere, the properties of the Martian surface, and the radiation interaction models. Using these data the average annual surface dose is calculated to be 51 mGy from GCR particles and 2.7 mGy from solar (SPE) particles (based on an 11-year solar cycle)(10). Estimates from Martian meteorites suggest an average background dose rate of just 0.4 mGy/year due to U, Th and K(11). Thus, the radiation environment is dominated by external radiation, even down to depths of >2m(9).

Determination of the annual dose rate is thus simplified for those deposits that are shallow (less than, say, 2 m) and are stratigraphically stable. For such deposits one can estimate a fixed annual average of approximately 54 mGy/year. These estimates are a global average and can be refined for the particular location of any future landing. On the other hand, estimates of ages for deeply buried deposits would require a measurement or estimate of the background dose rate due to the presence of natural radioisotopes.

The age being determined by this method is the time since the luminescence signal was last zeroed. For OSL dating of sediments the zeroing event is the last exposure of the sediment grains to sunlight during transport, prior to deposition. Deposition then results in shielding of the sediment from further sunlight exposure and therefore, the “age” determined by OSL is the time since the last exposure to sunlight – i.e. the depositional age of the sediment.

Procedures[6] have been developed that allow equivalent doses to be measured on one aliquot (sub-sample) thus greatly simplifying the process (as opposed to earlier multiple-aliquot methods). The single-aliquot regenerative-dose (SAR) procedure uses the sample's response to a fixed test dose to correct for any sensitivity changes (changes in OSL response/ unit radiation dose) that occur during the measurement procedure. The equivalent dose is determined by comparing the sensitivity-corrected OSL signals of the natural irradiation and known laboratory radiation doses. Table 1 outlines the SAR procedure.

Material Characterization: Any materials encountered on Mars will more than likely be different than materials normally used for terrestrial luminescence dating studies. As a result, it is necessary to characterize the luminescence properties of Martian soil simulants and analogs. In this paper, we describe the luminescence signals from polymineral fine-grains of a Martian soil simulant, JSC Mars-1, and the bulk fraction of an SNC Martian meteorite (ALH 77005,74).

TL studies. The TL glow curve of JSC Mars-1 shows a single broad peak between ~100°C and 370°C, with the maximum intensity at around 270°C[11]. The Martian meteorite ALH 77005,74 has a TL glow curve with two peaks (Fig. 1). The first peak can be observed between the temperatures ~80°C and 160°C with the maximum at ~140°C, while the second broad peak ranges from 180°C to at least 400°C (the maximum annealing temperature) with a peak near 340°C. Many Martian meteorites have peaks at ~120°C, suggesting that feldspar is in the low temperature ordered state[12]. The presence of broad peaks in such samples could be a consequence of a single broad peak (due to a distribution of charge trapping states) or the superposition of several smaller peaks.

OSL studies. To determine the maximum dose estimable using blue stimulated luminescence signal (Fig. 2), the SAR method[6,7] was used to generate blue stimulated luminescence growth curves for JSC Mars-1 (Fig. 3) and ALH 77005,74 (Fig 4). For JSC Mars-1, the maximum estimable dose for blue optical stimulation is ~7500 Gy, which corresponds to an age of 0.14-18.8 Ma using the previous estimates of the minimum and maximum dose rates for Mars. The maximum estimable dose for the ALH 77005,74 sample is ~2500 Gy, implying an upper age range of 0.05-6.3 Ma.

For any dose estimation procedure such as SAR, a fundamental test is to demonstrate that a known dose can be recovered in the laboratory. To this end, dose recovery experiments were performed for the blue stimulated luminescence signals from the JSC Mars-1 and the ALH 77005,74 sample. The equivalent dose ratios (measured/given) for JSC Mars-1 were 0.96 and 0.84 for two different aliquots, and the corresponding ratio for ALH 77005, 74 was 0.98. The results of these experiments show that a known dose can be estimated to within 5% for both these samples.

Procedure Development: The SAR procedure was developed specifically for coarse-grain quartz samples and was later extended to polymineral fine-grain (4-11 μm) samples.[7] As yet, however, no procedure for coarse-grain (or mixture of grain sizes) has been developed for polymineral samples. In fact, attempts to apply the SAR procedure to coarse-grain feldspars have been unsuccessful[13].

With the eventual goal of developing a true polymineral procedure, experiments were undertaken to find a single-aliquot procedure for coarse-grain feldspars. Five feldspar samples (microcline, oligoclase, anorthoclase, albite, and andesine) were examined to determine their general luminescence characteristics, characterize and understand any sensitivity changes experienced during repeated measurement cycles, develop sensitivity-correction procedures, and recover a known laboratory dose using the procedure.

Sensitivity changes do occur in these samples under repeated cycles of radiation dose, preheating, and OSL measurement. However, if TL (to 450°C) is used to measure the luminescence signal, no sensitivity changes are seen. Consequently, measuring TL to 450°C after each OSL measurement eliminates sensitivity changes from repeated cycles of measurement. This result is consistent with sensitivity changes caused by competition among trapping states during irradiation and measurement.

To correct for sensitivity changes, the SAR procedure (Table 1) was used as an outline. Previous work has shown that the entire TL glowcurve is affected by optical stimulation, and this result indicates that the regeneration dose and test dose irradiations need to be preheated in the same manner. The SAR procedure was then modified so that the heating in steps 2 and 5 were identical (in both temperature and duration), and the sensitivity correction was tested by giving a fixed regeneration dose for 7 cycles. If the procedure is effective, the same sensitivity-corrected ratio (L_i) should be obtained for all 7 cycles and the regeneration and test dose signals should correlate (i.e., show the same pattern of sensitivity change).

The results showed that not only is the same L_i obtained for the 7 cycles, but that the regeneration and test dose OSL signals correlate. This is not the case when the test dose is heated in a different manner than the regeneration dose. Further tests showed that a TL measurement could effectively be used each cycle (after the test dose OSL), although, as expected, very little sensitivity change is seen in this case.

For any luminescence dating procedure, the minimum requirement is that a laboratory radiation dose can be recovered using the procedure. We used our modified SAR procedure to recover a known dose (~9 Gy) for all the samples using the blue-stimulated OSL signal. The samples were given a large "geologic" dose and bleached with blue diodes for an extended period of time to simulate natural bleaching. The known dose was then given and recovered, both with and without a TL measurement after each test dose OSL. Table 2 summarizes the results of the dose recovery experiments. For either case (no TL, or TL each cycle), the dose can be recovered to within 5 % when one standard deviation is considered. However, a procedure that does not use TL is preferable. Not only is measurement time reduced, but using only OSL measurements (assuming the natural sample was bleached and not heated) allows measurement of the same trapped charge. Thus, different charge populations may be measured in the natural and regenerated OSL signals. For this reason, it is suggested that the modified SAR procedure be used without any TL measurements for coarse-grain feldspars.

The above outlined procedure is far from complete. Feldspathic materials have long been known to suffer from anomalous fading,(14) and either a correction(15) or elimination(16) of this problem must be found. The full procedure must then be tested using feldspar samples with independent age controls.

Eventually, a true polymineral procedure needs to be developed. The previous experiments (including an future anomalous fading corrections) will need to be repeated with admixtures of minerals (i.e., quartz and feldspars). Finally, polymineral (and possibly mixtures of grain sizes) samples will also need to be tested against independent age controls.

Conclusions: The thermoluminescence and blue stimulated luminescence signals from the JSC Mars-1 and the Martian meteorite ALH 77005,74 have been characterized in this study. Dose recovery experiments show that radiation doses given in the laboratory can be estimated to within 5% using single-aliquot procedures. The blue stimulated luminescence growth curves suggest that the maximum theoretical estimable dose is ~7500 Gy for the JSC Mars-1 sample, and ~2500 Gy for the ALH 77005,74 sample.

A single-aliquot procedure for coarse-grain feldspars (based on the SAR procedure) has been outlined. The procedure effectively corrects for sensitivity changes in the investigated samples by applying the same preheating regimen after all irradiation doses. Although natural samples with independent age controls have not been dated, known laboratory doses could be recovered to within 5%.

References: [1] Lepper, K. and McKeever, S. W. S. (2000) *Icarus*, 144, 295-301. [2] McLaughlin, D. (1954) *Bull. Soc. Amer.* 65, 715-717. [3] Kuzmin *et. al.* (2001) *Icarus,* 153, 61-70. [4] Hecht, M. H. (2002) *Icarus* 156, 373-386. [5] Malin, M. C. and Edgett, K. S. (2000) *Science* 288, 2330.[6] Murray, A. S., and Wintle, A. G. (2000) *Radiation Measurements*, 32, 57-73. [7] Banerjee, D., Bøtter-Jensen, L., Murray, A. S. (2000) *Applied Radiat. And Isotopes*, 52, 831-844. [8] Olley, J. M., Caitcheon, G. and Murray, A. S. (2000) *QSR*, 17, 1033-1040. [9] McKeever *et. al.* (2003) *Radiat. Meas.* In press. [10] Wilson *et. al.* (1995) *NASA* TP-3495. [11] Banerjee *et. al.* (2002) *Rad. Prot. Dosim.* 101, 321-326. [12] Hasan, F. A., Haq, M. and Sears, D. W. G. (1986) *Geochim. Cosmochim. Acta,* 50, 1031-1038. [13] Wallinga *et. al.* (2000) *Rad. Meas.* 32, 691-695. [14] Wintle, A. G. (1973) *Nature* 245, 143-144. [15] Huntley, D. J., and Lamothe, M. (2001) *Can. J. Earth Sci.* 38, 1093-1106. [16] Vicosekas, R. and Zink, A. (1999) *Quat. Geo.* 18, 271-278.

1. Regeneration radiation dose (D_i), 0 Gy if natural signal
2. Preheat at T_x°C for 10 s*
3. Measure OSL at 125°C
4. Fixed test radiation dose (TD_i)
5. Heat to T_y°C*
6. Measure OSL at 125°C
7. Repeat steps 1-6 for a range of regeneration doses, including a repeat point and 0 Gy dose
8. Find sensitivity-corrected OSL ($L_i=R_i/T_i$)

* T_x and T_y determined from experiment

Table 1: Outline of the SAR procedure.

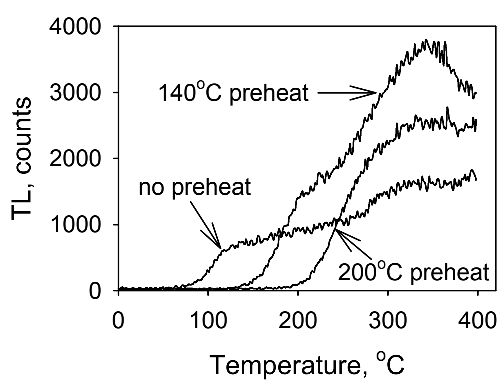

Figure 1: TL glowcurve of ALH 77005,74. The curves were produced after a 300 Gy dose. The three

graphs represent the TL immediately after irradiation, after a 140°C preheat, and after a 200°C preheat as indicated.

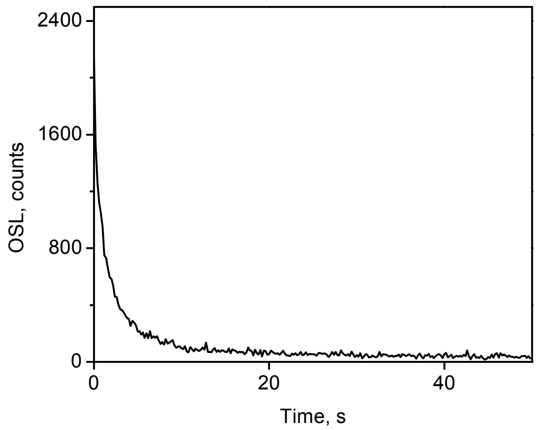

Figure 2: Blue-stimlated OSL decay curve for JSC Mars-1.

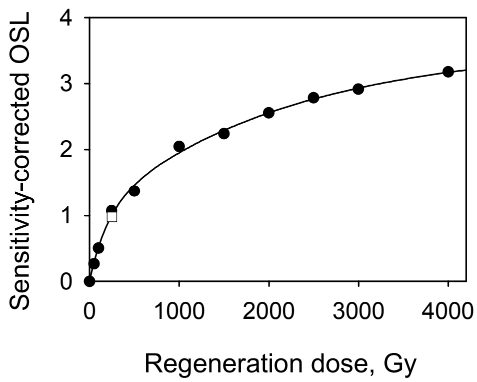

Figure 3: Sensitivity-corrected dose-response curve for the fine-grain fraction of JSC Mars-1. The signals have been integrated over the first 1 s of stimulation. The open square represents a repeat of the first regeneration dose.

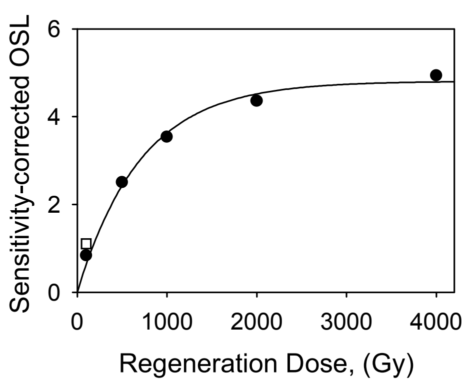

Figure 4: Sensitivity-corrected dose-response curve for ALH 77005,74.

Sample		% St. Err.	R4/R1	0 dose
Microcline				
	no TL	-0.29 +/- 0.59	1.00	0.01
	w/ TL*	4.52 +/- 0.37	0.99	0.00
Oligoclase				
	no TL	-1.25 +/- 0.25	1.00	0.01
	w/ TL*	0.79 +/- 0.31	1.00	0.01
Anorthoclase				
	no TL			
	w/ TL			
Albite				
	no TL	-1.59 +/- 0.12	1.00	0.02
	w/ TL	1.71 +/- 0.19	1.00	0.00
Andesine				
	no TL	0.40 +/- 0.68	1.00	0.00
	w/ TL	5.11 +/- 0.86	1.00	0.01

Table 2: Results of feldspar dose recovery experiments.
An OSL signal could not be detected for Anorthoclase.