A PROTOTYPE INSTRUMENT FOR *IN SITU* LUMINESCENCE DATING OF SEDIMENTS ON MARS.

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**Introduction:** Studies indicate that Mars might even today be a climatically active planet. Optically stimulated luminescence (OSL) dating has been suggested as a technique that can be adapted for robotic *in situ* dating of martian sediments that have been transported and deposited by wind or water over the last $10^5$-$10^6$ years [1-4]. In this presentation we describe efforts and challenges to adapt OSL dating for robotic *in situ* dating of martian sediments. We describe the prototype for a robotic luminescence reader, discuss the constraints on the design of a space-approved instrument and the handling of the sample material.

**General Principles of Luminescence Dating:**
OSL dating works on the principle that ionizing radiation – from U, Th, K, and cosmic rays – ionizes atoms within silicate mineral grains like quartz and feldspar. The freed electrons become trapped at light sensitive crystal defects within the mineral. The number of trapped electrons increases over geologic time and is a direct measure for the radiation dose absorbed by the mineral. Exposure to sunlight releases the electrons from the traps and resets or “zeroes” the luminescence clock. In the laboratory the sample is stimulated with light of one wavelength and the luminescence (OSL) emitted from the sediment is monitored at another wavelength. The OSL signal is proportional to the time elapsed since the mineral grains were last exposed to daylight (i.e., the time since burial). The amount of absorbed energy, the "equivalent dose" (unit: Gray; 1Gy = 1J/kg), is determined by comparing the natural luminescence signal with the signals obtained after known radiation exposures administered in the laboratory. If the rate of natural irradiation, the “dose rate” (in Gy/year), is known, the age of the sample is derived from: \( \text{Age} = \frac{\text{Equivalent Dose}}{\text{Dose Rate}}. \)

**Experimental Details:** OSL measurements at room temperature or above were made using a Riso TL/OSL-DA-15 reader with a built-in $^{85}$Sr,$^{88}$Y beta source. Optical stimulation was carried out with blue LEDs (470 nm), or an IR LED array at 875 nm. The low temperature TL and OSL measurements were made using the low temperature TL/OSL system described in [5]. Irradiation was carried out using a 40 kVp Moxtek miniature x-ray system. Optical stimulation was done using a 100 mW diode pump solid state green laser (532 nm). Hoya U-340 filters (290-370 nm) were used for detection.

**Equivalent Dose:** A robotic luminescence dating instrument for Mars requires the development of new measurement techniques in order to deal with the multiple challenges of the martian environment not usually found when using OSL to date terrestrial sediments. A prerequisite to this development is to identify the predominant minerals likely to be found in the martian regolith, and to examine the fundamental OSL properties of these materials (dose response, fading/stability, stimulation spectrum, etc.).

**Mineral identification.** Spectra from the thermal emission spectrometer (TES) distinguish two different types of regolith on Mars: Type I (basaltic) and Type II (andesitic). Both types of regolith are composed of plagioclase feldspars, pyroxenes and hematite, and the Type II material contains an abundance of obsidian or volcanic glass [6, 7]. Plagioclase feldspars are found to have a calcium content of 30-70% [8]. Based on these data we devised two mineral mixtures as surrogates for martian sediments [9]. The compositions of the mixtures OSU Mars-1 and OSU Mars-2 are as follows:

<table>
<thead>
<tr>
<th></th>
<th>Mars 1</th>
<th>Mars 2</th>
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<tbody>
<tr>
<td>Andesine</td>
<td>22 %</td>
<td>15 %</td>
</tr>
<tr>
<td>Labradorite</td>
<td>22 %</td>
<td>15 %</td>
</tr>
<tr>
<td>Bytownite</td>
<td>22 %</td>
<td>15 %</td>
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<tr>
<td>Augite</td>
<td>15 %</td>
<td>5 %</td>
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<tr>
<td>Diopside</td>
<td>15 %</td>
<td>5 %</td>
</tr>
<tr>
<td>Hematite</td>
<td>5 %</td>
<td>5 %</td>
</tr>
<tr>
<td>Obsidian</td>
<td>40 %</td>
<td></td>
</tr>
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**Figure 1**: TL emission spectrum of OSU Mars-2, after irradiation with 100 Gy. The numbers in the figure and the colour scale indicate the measured counts.

The TL-emission spectrum of OSU Mars-2 (Fig. 1) reveals a dominant red emission and weaker emissions in the blue and UV wavelength band.
Kalchgruber et al. [9] found that a major part of the signal emitted by the mineral mixtures originates from labradorite and andesine, with minor contributions from diopside. To separate the blue stimulation light from the luminescence signal we focused in our experiments on the UV-emission.

Measurement procedure. An in situ OSL dating instrument for martian surface sediments will likely not allow chemical treatment and mineral separation of the regolith samples. The measurement of the equivalent dose requires therefore a procedure that is suitable for polynomineral samples. Kalchgruber et al. [9] suggested a measurement procedure with combined IR and blue stimulation for the OSU martian regolith simulants. Although a post-IR blue procedure is more power-consuming than an IR-only or OSL-only procedure, it yielded better results for high doses. The accuracy for a 1600 Gy laboratory dose was 6.1% using the IR stimulated luminescence (IRSL) signal and 4.0% for the post-IR OSL. Additionally it has the advantage that two independent dose values are measured.

Low temperature measurements. It is necessary to consider that the ambient temperatures on the martian surface are significantly lower than on Earth, with large variations (from -140 °C to +20 °C). Thus, the influence of the temperature variation during the irradiation period on evaluated dose must be determined. OSU Mars-1 and OSU Mars-2 were irradiated and stimulated at varying temperatures between -125°C and +200°C. Simulations and experiments showed that for accurate dose measurements, the stimulation temperature must be significantly higher than the highest irradiation temperature in nature [5], i.e. in the case of Mars at least 80-100°C.

Solar resetting. The entire premise of OSL dating is that the sediments to be dated were exposed to sufficient amounts of light at the time of deposition to erase any previously accumulated signal. In terrestrial applications, this “zeroing” of the signal is accomplished within a few minutes of exposure to sunlight, but the solar spectrum on Mars is different from that on Earth and may lead to different bleaching efficiencies.

No measured data exist for the spectral irradiance at the surface of Mars. The wavelength range between 200 and 900 nm is particularly important for the solar resetting of sediments. We carried out radiative transfer calculations for the atmosphere of Mars using the libRadtran software package of Mayer and Kylling [10], (http://www.libradtran.org). The calculation is based on a vertical atmospheric profile and accounts for absorption by CO₂, O₂, O₃, H₂O, and Rayleigh scattering. Lambertian surface albedo and extinction by dust particles were also included. A more detailed description of the simulations is given by Deo et al. [11].

Fig. 2 shows a comparison of the total spectral irradiances for a clear day on Earth and typical summer and winter days on Mars. The integrated (200-900 nm) total irradiance at the surface of Mars is similar for both seasons. The irradiance in the northern summer amounts to 49% of the value for Earth. Due to the higher dust load and the increased solar zenith angle in winter, the irradiance is only 37% of the Earth-value. Whereas practically no radiation below 300 nm reaches the surface of Earth, sediments on Mars are exposed to UV radiation starting from 200 nm.

We have simulated the martian spectrum with a solar simulator and tested the bleaching characteristics of the martian regolith simulants. A detailed description of the experiments can be found in McKeever et al. [4]. After 10 min of exposure to the solar simulator only 5% of the original IRSL signal (10% for blue-stimulated OSL) was left and after 200 min the sample was fully bleached.

Fading. A remaining significant issue for the estimation of the equivalent dose is anomalous fading. Feldspars have been found to show a loss of signal during storage [12], known as anomalous fading. Experiments suggest that 53% of the IRSL and 58% of the OSL signal are stable and that the unstable signals have already completely decayed after two days. The most likely and least energy consuming means to correct for fading is to allow time for fading after each calibration irradiation.
Dose-rate: Unlike Earth, background radiation from U, Th and K, as derived from meteorites [13], is not the dominant radiation source in the upper 2-3 m of the martian surface. The main source for the dose rate on Mars is from galactic cosmic rays (GCR) and solar energetic particles (SEP), which consist predominantly of protons [14]. Dose rates expected on the martian surface (dependent upon altitude) are typically of the order of 200–300 mSv/year (~80-120 mGy/year; [15]). Furthermore, the composition of the cosmic radiation field will change with increasing depth in the regolith due to absorption and scattering. At 700 g/cm$^2$ the dose rate is expected to be reduced to approximately 0.5 mGy/year [3, 16]. The composition of the radiation varies with depth as well, and various computer models exist to determine the radiation environment on the martian surface and in the martian regolith.

For a constant administered dose, the intensity of the luminescence signal depends on the type and energy of ionizing radiation. Since the equivalent dose will be determined in relation to an X-ray or beta source in a robotic instrument, the relative efficiency of the GCR in producing OSL compared with the onboard radiation source needs to be determined.

![Figure 3: Normalized dose deposited at various depths in regolith simulant, after exposure to 1 GeV protons. Error bars result from the standard error of 4 aliquots.](image)

We carried out accelerator-based experiments with simulated cosmic rays at HIMAC in Chiba, Japan, and NSRL at Brookhaven National Laboratory. During the irradiations, small containers with OSU Mars-1, OSU Mars-2, and sedimentary quartz were sandwiched between columns of martian regolith simulant of various thickness, simulating a variety of burial depths in the martian regolith. The normalized dose for 1GeV protons (Fig. 3) shows a maximum at 10 cm depth, followed by a slow decrease. The maximum is caused by the build up of secondary particles and the concomitant loss of primary particles. The expected range of 1 GeV protons in regolith material is ~ 2m, however the range for the heavier particles is considerably less. The efficiency, i.e. the normalized dose at zero-depth, for the various ion beams and minerals is plotted against the LET in water in Fig. 4. The efficiency for beta and gamma radiation is assumed to be equal 1. The efficiency decreases with increasing LET.

![Figure 4: Efficiency in OSL production of various ion beams for OSU Mars-1, OSU Mars-2 and quartz.](image)

The results of our experiments can be combined with simulations to determine an “equivalent natural dose-rate” at a given depth for age calculation. GCR and SEP spectra are dominated by the lower LET parts of the spectrum, to the extent that an estimated 95% of the dose will be deposited by these particles. As a result, the drop in efficiency with LET may only induce a small error. A bigger source of error will be the fact that the dose rate will not be constant over the lifetime of the buried sediment due to the slow burial of the sediment.

Equipment Design: The basic requirements for all components of spacecraft instruments are low power, low weight, low volume and economical data acquisition. The prototype instrument was designed with these requirements in view. Kalchgruber et al. [9] found that a post-IR blue sequence yields better results in dose-recovery experiments than an IR-only or blue-only sequence. It has been shown that the stimulation temperature must be significantly higher than the highest irradiation temperature in nature. The instrument therefore requires facilities for IR and blue stimulation, as well as a heating plate, a luminescence detector, and an irradiation unit for on-board calibration.

A prototype luminescence / irradiation unit is shown in Fig. 5. The length of the base part is 15 cm. The chamber incorporates optical stimulation and irradiation at the sample position, thereby reducing the
complexity of the design and sample transport. A copper cover blocks light and X-rays from queued samples in the 20 aliquot rotating disk. One sample at a time is presented for irradiation or stimulation. An overhead PMT measures the OSL emission. A sample delivery system is under design. Samples can be stimulated with blue (470 nm, 50 mW/cm² at sample position) or IR LEDs (870 nm, 8.5 mW/cm²). Broadband interference filters are used to narrow the bandwidth of the stimulation light and to prevent an overlap with the detection band. A Hamamatsu H9319-11 photomultiplier with filters is used to detect the luminescence signal in the band 330 to 370nm. A heater allows the temperature to be ramped at computer-controlled rates. The upper temperature limit is 200 °C. A Moxtek "Bullet" miniature x-ray source (4W, 40 kV) is used as an x-ray system for dose calibration. The peak power of the system expected in operation is 5-6 W. A software program was developed to control the required sequence of heating, cooling, optical stimulation, OSL measurement and irradiation. The OSL system can be controlled from a notebook computer.

Conclusions: Assuming a lower measurable dose limit of 5 Gy [11] and a surface dose rate of 100 mGy/year, the lower age limit can be expected to be few decades. With an upper measurable dose limit of 1500 Gy the upper age limit would be only 10,000 years and thus considerably lower than on Earth. It has to be taken into account, however, that the dose rate decreases rapidly with increasing burial depth. Therefore the upper age limit will likely be several 10⁵ years. Burial over a depth of 1 m will result in a dose difference of about 50% and burial models might have to be developed. In the worst case the age can be bracketed by calculating an age based on the surface dose rate, or the dose rate at the depth from which the sample is recovered. These ages will then vary by approximately a factor of 2. The significance of such a result would be quite limited in terrestrial applications. But, given the age range covered with luminescence dating, and the fact that ages smaller than at least 1 Myr cannot be resolved by crater counting, even such results will be a major improvement to current Mars dating techniques.

An OSL prototype instrument is under development that can be controlled from a notebook computer and allows heating, cooling, irradiation, optical stimulation and OSL measurement. Initial dose-recovery tests resulted in 2.5% reproducibility in the laboratory. The goal is a dating technique that can elucidate a wealth of knowledge about the recent geological and climatic activity on the martian surface.

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