

**SINGLE GRAIN SPECTROSCOPIC ANALYSIS OF OLIVINE, PYROXENE, AND ILMENITE.** E. A. Stanish<sup>1</sup>, N. N. Turenne<sup>1</sup>, S. A. Connell<sup>1</sup>, E. A. Cloutis<sup>1</sup>, D. M. Applin<sup>1</sup>. <sup>1</sup>Department of Geography, University of Winnipeg, 515 Portage Avenue, Winnipeg, MB, Canada; [stanish-e@webmail.uwinnipeg.ca](mailto:stanish-e@webmail.uwinnipeg.ca)

**Introduction:** Spectroscopy is a useful tool for probing the composition of both planetary and terrestrial materials. Previous work has been done using Raman and reflectance spectroscopy to characterize fine grain bulk powder and separates while single grain analysis has yet to be routinely employed with reflectance spectroscopy [1].

This study is an offshoot of ongoing work to analyze single grain kimberlite indicator minerals and diamond inclusions. The goal is to create a non-destructive methodology to identify single mineral grains facilitating exploration techniques using reflectance and Raman spectroscopy. It will be particularly useful for analysis of planetary materials, which are either present as small, single grains (e.g., micrometeorites) or for comprehensive characterization of rare planetary materials (such as returned asteroidal samples) on a grain-by-grain basis. Here we report on the development of ongoing methods for spectroscopic analysis of single mineral grains in the sub-mm size range to derive spectra that can be compared to bulk powders and can be quantified for composition. Results for olivine, pyroxene, and ilmenite are reported.

**Methodology:** Samples of olivine, orthopyroxene (chrome diopside), and ilmenite were hand crushed using an alumina mortar and pestle, then dry sieved to 1000-500  $\mu\text{m}$  and 250-500  $\mu\text{m}$  particle sizes by hand shaking and brushing the samples through a stainless-steel sieve. The powders were characterized by X-ray diffractometry (XRD). Individual mineral grains were picked from these powders.

Raman spectra were collected in the 170-4000  $\text{cm}^{-1}$  range at a nominal resolution of  $\sim 4 \text{ cm}^{-1}$  with a B&W Tek iRaman instrument. A 532 nm excitation was produced at  $\sim 50 \text{ mW}$  and focused to a spot size of  $\sim 80$  microns in diameter.

For the Raman spectral measurements, the grains were placed onto a black 2% reflectance Spectralon puck. One grain of each sample was picked from the vial with tweezers and carefully set on the black Spectralon. The sample grains were not oriented and were placed on an adjustable stage and were brought into the focus of the laser.

When using the BAC151B microscope, objectives of 5x, 20x, and 40x magnification were employed, which enabled a laser beam spot size of 42  $\mu\text{m}$ , 105  $\mu\text{m}$ , 52  $\mu\text{m}$  respectively. The same sample placement

methods were used. Integration time was adjusted to approach 80% of saturation, giving an optimal integration time and signal-to-noise ratio. Instrument performance was measured before sample collection against either a polystyrene or gypsum standard. Measurements for each sample were made by first acquiring a dark current spectrum, followed by measurement of the sample. All measurements were made using an identical viewing geometry, integration time, and number of averaged spectra.

Reflectance spectra were obtained using an Analytical Spectral Device (ASD) LabSpec 4 Hi-Res (350-2500 nm). Spectra were acquired with a viewing geometry of  $i = 30^\circ$  and  $e = 0^\circ$ . Incident light was provided using an in-house tungsten light source with the beam focused into  $\text{SiO}_2$  fibers. The nominal diameter of the beam on the sample was  $\sim 1 \text{ mm}$ . Sample spectra were measured relative to a calibrated Spectralon 99% diffuse reflectance standard (7458) and corrected for dark current. For every acquisition, 500 spectra of dark current, standard, and sample were acquired and averaged providing sufficient signal-to-noise levels.

For spectral reflectance measurements, the mineral grains were placed on white Spectralon. The grains were rotated to find a non-angular surface area, to attempt capturing maximum light transmittance. Different grains of the same sample were selected and shot for comparison. This method attempts to collect repeat-pass transmittance while minimizing specular reflectance.

**Discussion:** Both methods of using the Raman with and without the microscope displayed the expected spectral signatures. The initial testing of the microscope objectives of 5x, 20x and 40x magnification collected the same data with all the expected peaks for ilmenite and olivine. The chrome diopside grains of 500-1000  $\mu\text{m}$  did not clearly identify Raman peaks from the 10x or 20x objectives, however the 40x lens produced expected results. For the duration of subsequent data collection, the 40x magnification lens was utilized.

Mineral grain size affected the Raman scattering in that smaller grains had stronger Raman peaks compared to larger grains.

Compared to spectra taken from previous studies, the single grain spectra are similar in quality and give

enough data to identify the minerals. Figure 1 shows the Raman spectra of a 250-500  $\mu\text{m}$  olivine grain and a 500-1000  $\mu\text{m}$  grain exhibiting the characteristic peaks around 800  $\Delta\text{cm}^{-1}$  [2]. The spectra obtained of ilmenite via Raman spectroscopy displayed in Figure 2 for a 250 – 500  $\mu\text{m}$  grain is usable to identify the grain as ilmenite.

Multiple-pass transmittance in a reflectance geometry of the mineral grains yielded similar results. Absorption band depth changed with grain size, and usable data was obtained for all minerals except ilmenite. Opaque minerals did not produce transmittance features, therefore this is not a viable method for analysis. Figure 3 illustrates the viability of reflectance spectroscopy for the identification of single grains of chrome diopside. The 500-1000  $\mu\text{m}$  grain correlates well with 45-90  $\mu\text{m}$  powdered pyroxenes previously analyzed by Cloutis (2002) [3].

**Conclusions:** The results obtained so far indicate that the methodology described above produce viable results. The single grain mineral species have comparable spectral properties to what has been gathered for bulk powders. This methodology is cost- and time-effective because it negates sample preparation while conserving the material by probing it nondestructively.

**Acknowledgments:** This research was supported by The Canadian Space Agency, NSERC, CFI, MRIF, and The University of Winnipeg.

**References:** [1] Cloutis E.A. and Norman L. (2009) *LRO Science Targeting Meeting* abstract. [2] Mouri T. and Enami M. (2008) *JMPS*, 103, 100-104. [3] Cloutis E.A. (2002) *J. Geophys*, 107, 6-12.

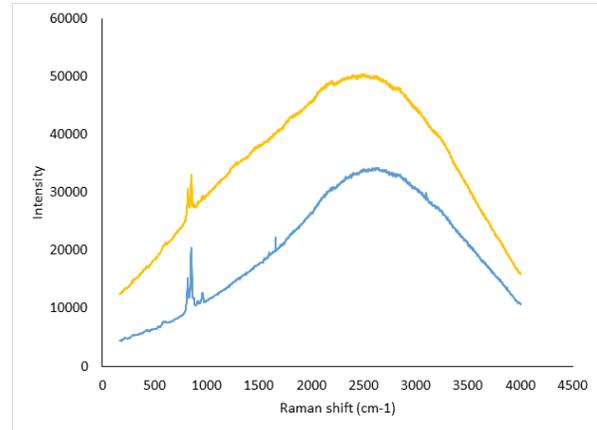


Figure 1: Raman spectra of different sized olivine mineral grains; 250 – 500  $\mu\text{m}$  (OLV007) on the bottom, and 500 - 1000  $\mu\text{m}$  (OLV007) on the top. Notice the stronger Raman peaks on the smaller grain and the characteristic peaks around 800  $\text{cm}^{-1}$ .

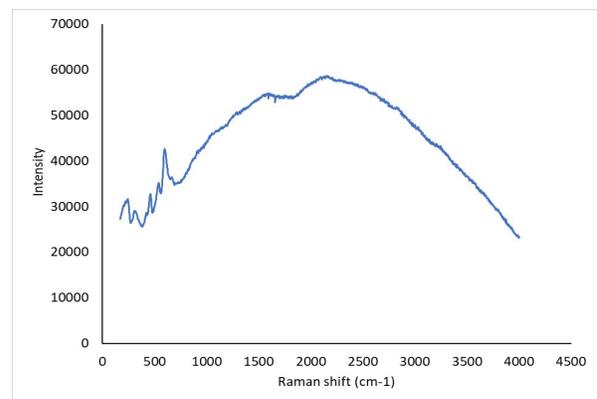


Figure 2: Raman spectra of 250 – 500  $\mu\text{m}$  grain size ilmenite (ILM117) exhibiting spectral data in anticipated range.

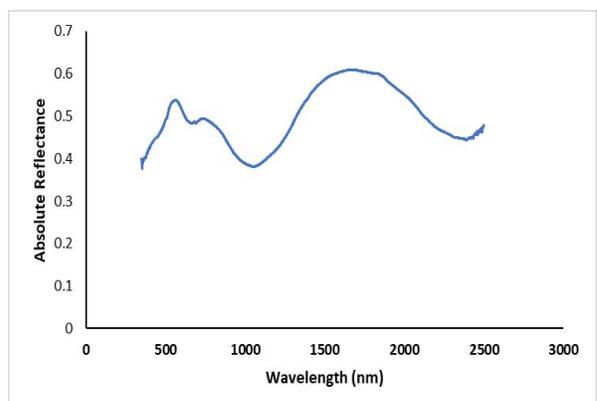


Figure 3: Reflectance spectra of 500 - 1000  $\mu\text{m}$  grain size chrome diopside (PYX191) showing predicted absorption bands.