SINGLE GRAIN ANALYSIS OF OLIVINE USING REFLECTANCE SPECTROSCOPY TO SUPPORT ASTEROID SAMPLE RETURN MISSIONS. S. A. Connell[1*], Z. U. Wolf[1], D. M. Applin[1], S. Sidhu[1], N. N. Turenne[1], E. A. Cloutis[1], [1]Centre for Terrestrial and Planetary Exploration, University of Winnipeg, 515 Portage Avenue, Winnipeg, Manitoba, R3B 2E9, Canada; [*]connell-s@webmail.uwinnipeg.ca.

Introduction: There are and have been multiple missions observing and collecting samples from near-Earth asteroids. To date, two have returned materials to Earth for analyses: (1) the JAXA Hayabusa1 mission to near-Earth asteroid (25143) Itokawa [1], and (2) the JAXA Hayabusa-2 mission to the near-Earth asteroid (162173) Ryugu [2]. The NASA OSIRIS-REx mission collected a sample from near-Earth asteroid (101955) Bennu and is scheduled to return to Earth in 2023 [3]. The China National Space Administration (CNSA) is planning a sample return mission (Zhenghe) to near-Earth asteroid 2016 HO3 [4].

An example of a returned sample from an olivine-bearing near-Earth asteroid is (25143) Itokawa - an LL ordinary chondrite [5]. The mineralogy of (25143) Itokawa is dominated by olivine, Ca-poor and Ca-rich pyroxene, plagioclase, and troilite, plus some accessory minerals [6]. Other olivine-bearing asteroids include Vesta and vestoids and some basaltic asteroids [7, 8, 9]. There are also numerous olivine-dominated asteroids in the main asteroid belt [e.g., 10, 11].

We are exploring how to acquire reflectance spectra of single grains of olivine that allow us to determine its composition. Olivine was selected due to its relative transparency and expectation that we can detect spectral features at different configurations, for single grains of various shapes and sizes that span the range of grain sizes for returned asteroid samples.

Methodology: Olivine (Sample ID: OLV003 (Fa9.6)) was analyzed using visible-near-infrared (VISNIR) reflectance as single grains and as bulk powders. Samples were powdered by hand and wet sieved to 850-1000, 425-500, 212-250, 106-125, 45-63, 25-38, and <38 µm particle size, and were measured as flat matte-surface bulk powders at a viewing geometry of *i=0°*, *e=0°*. Single grains from the 850-1000, 425-500, 212-250, 106-125 µm particle size fractions were measured using an aluminum sample holder with the grain placed in a well at a depth of 10 mm. The well had roughened walls and a tapered bottom, and a diameter of 6 mm to accommodate the bifurcated sample probe. This type of sample holder was expected to reflect the light off the walls of the well in multiple directions, to enable the grain to be illuminated from all directions and to pick up reflected/transmitted light from multiple directions. The bifurcated fiber optic probe was placed in the well at distances of 2 mm, 6 mm, and 12 mm between the end of the probe and the sample (Figure 1). The reference spectrum used was the empty aluminum well.

Reflectance spectra were collected with an ASD LabSpec4 Hi-Res spectrometer (350-2500 nm), at a viewing geometry of *i=0°*, *e=0°* using an 80-watt halogen operated at 150-watt light source directed into a bifurcated cable, with an off-axis parabolic mirror and an approximate measured area of 6.2 mm. The purity and composition of the olivine was determined for a bulk <45 µm particle size powder with X-ray diffraction (XRD) to verify the mineralogy. For XRD, a Bruker D8 Advance was used, with a DaVinci automated diffractometer, with an integration time of 1 second per 0.02º step.

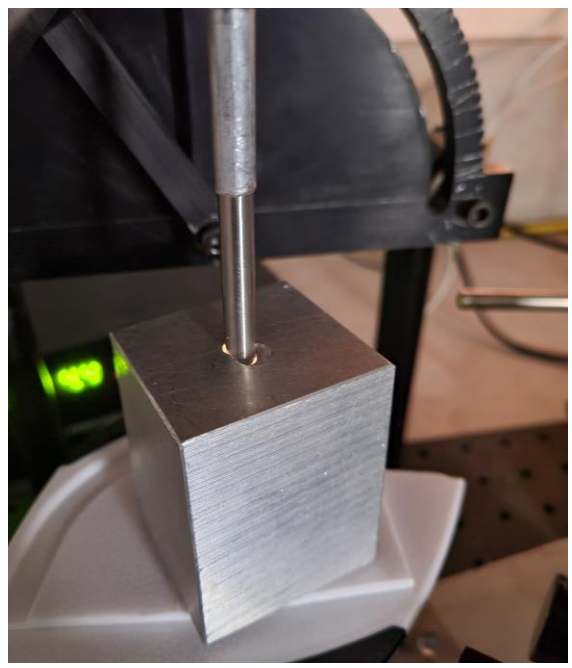

Figure 1. Image of the sample holder with the bifurcated fiber optic probe inserted in the well. The light source and probe (bifurcated cable) are at a viewing geometry of *i=0°*, *e=0°* at a distance of 2 mm from the sample.

Results: Each figure shows spectra of olivine at various grains sizes as single grains and as bulk powders that show characteristic absorption bands for olivine. The main spectral features of olivine that are of interest are the wide absorption band centered near 1050 nm (due to spin-allowed crystal field transitions of Fe^{2+}), local reflectance maximum near 550 nm, flat

spectral slope beyond ~1800 nm, and narrow absorption bands near 400-500 nm, and 620 nm [12].

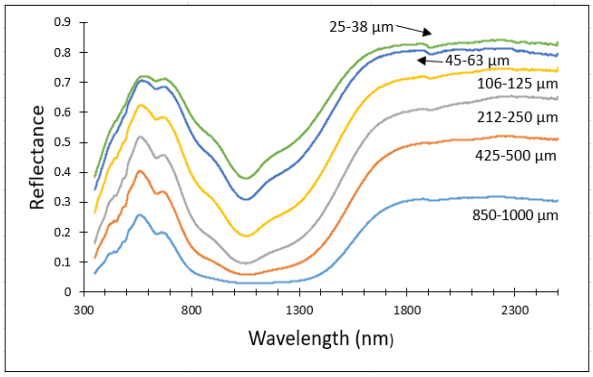

Figure 2. Bulk powder reflectance spectra of olivine at grain sizes of 850-1000, 425-500, 212-250, 106-125, 45-63, 25-38, and <38 µm particle size measured at a viewing geometry of *i=0°*, *e=0°*.

As expected, the reflectance spectra of the bulk powders show the expected absorption bands and spectral features. The spectra are dominated by a broad absorption band at 1050 nm. With increased grain size the 1050 nm absorption band becomes broader. Minor absorption bands are shown in the visible spectrum at ~400-500 nm and are indicative of spin forbidden transitions in Fe^{2+}, and are more apparent at larger grain sizes. As the grain size increases overall reflectance decreases.

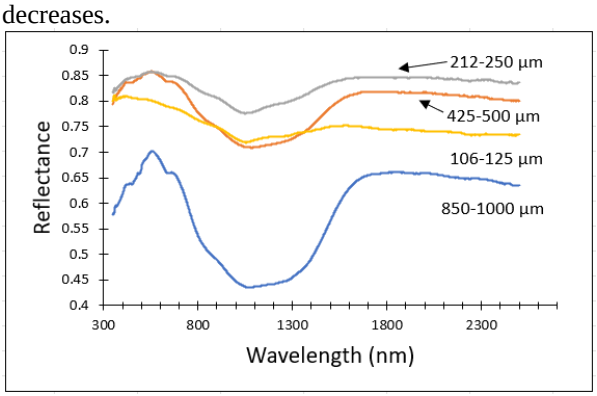

Figure 3. Reflectance spectra of olivine single grains at grain sizes of 850-1000, 425-500, 212-250, and 106-125 µm measured with the bifurcated fiber optic probe at a distance of 6 mm from the end of the probe to the sample.

The single grain reflectance spectra in Figure 3 of olivine exhibit definitive absorption bands for all grain sizes. The diagnostic 1050 nm absorption band is still evident in the single grains; however, it is shallower than the band in the corresponding bulk powder spectra. The Fe^{2+} absorption feature in the 1050 nm region is evident but weaker, and this band does not appear to be as saturated (low reflectance, broad minimum) as the bulk powders. The minor absorption bands below ~650 nm are also evident. The 850-1000 µm single grains still show the lowest reflectance with the smaller grain sizes showing increased reflectance. Spectral slopes beyond 1800 nm are largely unchanged.

Discussion: Olivine is a mineral that is widely present in the solar system and can be found among near-Earth asteroids that include ordinary chondrite parent bodies, primitive dark bodies, and basaltic asteroids. The single grain reflectance spectra were compared against the bulk powder reflectance spectra to verify the olivine signature in the single grains. The diagnostic olivine absorption band at 1050 nm is seen in both the bulk powder and the single grain reflectance spectra as well as additional weaker absorption bands. It was difficult to produce evident spectral features at smaller grain sizes for single grains.

The 6 mm and 12 mm distances from the sample show a clearer spectral signature when compared to the 2 mm distance. When the light source was placed 2 mm from the grain, it became more specular, resulting in a flat spectrum with high reflectance.

The sample holder was selected for single grain analysis as we are attempting to develop a process for the observation of returned asteroid materials in extremely small quantities, as there may not be the option to do bulk powder analysis.

Conclusion and future work: The results indicate that single olivine grains can produce reflectance spectra at a certain geometry and specific grain sizes that allow diagnostic spectral features to be observed. We are also exploring this and other configurations for single grain analysis of opaque minerals and meteorite samples. Our initial results indicate that the distance of the light source and pick-up probe will determine the ability to see any identifiable absorption bands, at least in olivine.

Acknowledgments: Thanks to the CSA, NSERC, CFI MRIF, and UWinnipeg for supporting this study.

References: [1] Matsumoto T. et al. (2016) *GCA*, 187, 195–217. [2] Wada K. et al. (2018) *Prog. Earth & Planet. Sci.*, 5(1), 82. [3] Lauretta D.S. et al. (2017) *Space Sci. Rev.*, 212(1), 925–984. [4] Zhang Z.H. et al. (2021) *Script. Material.*, 81, 56–59. [5] Bonal L. et al. (2015) *MAPS*, 50(9), 1562–1576. [6] Yoshikawa M. et al. (2015) *Asteroids IV, 1*, 397-418, 1. [7] Moskovitz N.A et al. (2008) *Icarus*, 198(1), 77–90. [8] Burbine T.H. et al. (2001) *MAPS*, 36(6), 761–781. [9] McSween H.Y. et al. (2012) *The Dawn Miss. to Minor Plan. 4 Vesta and 1 Ceres*, 141–174. [10] Sanchez J.A. et al. (2014) *Icarus*, 228, 288-300. [11] Cloutis E.A. et al. (2015) *Icarus*, 252, 39-82. [12] Burns R.G. (1970) *Amer. Mineral.*, 55, 1608-1632.