

## CAPABILITIES AND LIMITATIONS OF INFRARED REFLECTANCE MICROSCOPY

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Technological improvements in IR microscopy have made it an increasingly appealing tool for planetary mineralogy. Microspectroscopy presents the prospect of examining small samples nondestructively and acquiring spectra that can be related to remote sensing observations. However, complications are introduced as a target beam size is reduced, and it is critical that limitations are understood. We present the results of a series of well constrained spectroscopic measurements, linking microspectroscopic data to traditionally collected reflectance spectra and petrologic information for the same rock.

**Background:** Microspectroscopy offers opportunities beyond those provided by standard reflectance or emission spectroscopy. Individual mineral grains with diameters as small as 10-50  $\mu\text{m}$  can be targeted and measured in situ. In the midIR, where surface interactions dominate silicate spectra, samples can be measured noninvasively preserving precious extraterrestrial samples for future investigations. Slabs and thin sections can also be measured with spatial relationships preserved, allowing investigation of how spectra vary in zoned minerals or across grain boundaries [1].

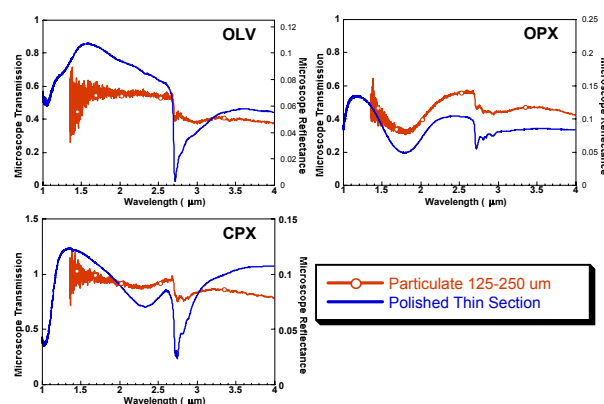
Despite the advantages, there have been limited studies applying microspectroscopy to planetary issues [1-7]. Standard thin sections cannot be studied in either transmission or reflectance in the infrared due to the opacity of glass and epoxy [7]. Microscopic reflectance measurements in the IR are limited by the geometry of the microscope optics. In the nearIR, where the extinction coefficient ( $k$ ) is near-zero, photons enter the body of a crystal; to reach the detector they must be multiply scattered off typically 10-20 interfaces. Most photons are thus scattered out of the small area viewed by the detector. In the midIR,  $k$  is high, and photons may interact with the surface and return directly to the detector.

Nevertheless, features observed in data collected by both transmission and reflectance microspectroscopy are to a first order comparable to that collected by standard laboratory spectroscopy as well as remotely sensed data [1]. To allow optimum use of microspectroscopy, we focus on documenting and analyzing systematic differences between measurements made with standard laboratory techniques and with microspectroscopy.

**Approach:** A peridotite from the Ronda massif in Spain was selected for this study based on the presence of olivine and pyroxene, common constituents of much extraterrestrial material. Additionally, hyperspectral remotely sensed AVIRIS data for the Ronda massif is available for comparison [8]. Six slabs were cut from a hand sample and the surface prepared with 400 grit. A subsample was ground to a grain size of 125-250  $\mu\text{m}$  and mineral separates of olivine, orthopyroxene and

clinopyroxene were handpicked from the bulk. Thin sections of different thickness were prepared. The most appropriate is a doubly polished section  $\sim 300 \mu\text{m}$  thick. Samples were measured in the RELAB from 0.3-2.6  $\mu\text{m}$  with the bidirectional spectrometer, from 2-25  $\mu\text{m}$  with the Pike+Nicolet diffuse reflectance system, and from 1.3- 14  $\mu\text{m}$  with the Nicolet Continuum microscope system. Polished slab measurements were ratioed with a gold mirror standard and rough (400 grit) slabs were referenced to a 400 grit diffuse gold standard to minimize detector nonlinearity issues. Olivine, orthopyroxene and clinopyroxene were microprobed to document major element abundances.

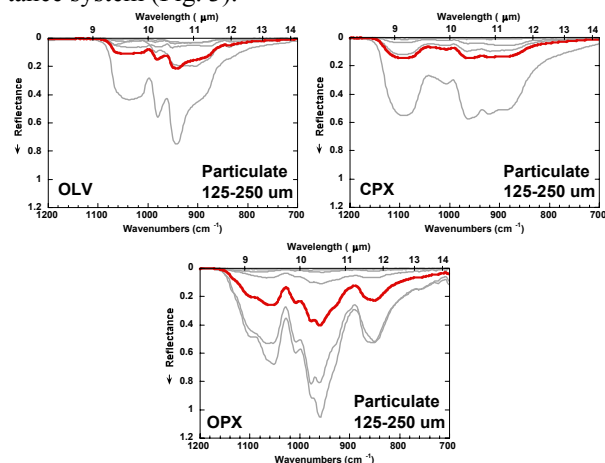
**Results: Near Infrared (1-9  $\mu\text{m}$ ).** Microscopic reflectance measurements of the six slabs produced no discernable signal above the instrumental noise. Diffuse reflectance measurements of particulate samples provide an averaged spectrum for a number of grains; thus, to obtain representative microscopic measurements and minimize artifacts due to grain orientation, several points on the mineral separates were collected. Although some diagnostic features, most notably a water absorption near 2.7  $\mu\text{m}$ , are observed, the reflectance spectra are  $\sim 10\times$  weaker than those measured by transmission of a 300  $\mu\text{m}$  thick doubly polished thin section (Fig. 1). The 2  $\mu\text{m}$  pyroxene  $\text{Fe}^{2+}$  band is discernable for both types of pyroxene, though the signal to noise and intensity of each measurement varies widely from spot to spot. The depth of features and signal strength also varies by mineral with  $\text{OPX} > \text{CPX} > \text{OLV}$ . This may be due to the nature of the mineral grains themselves, as those with the most distinct cleavage surfaces (OPX) may favorably reflect light back towards the detector.



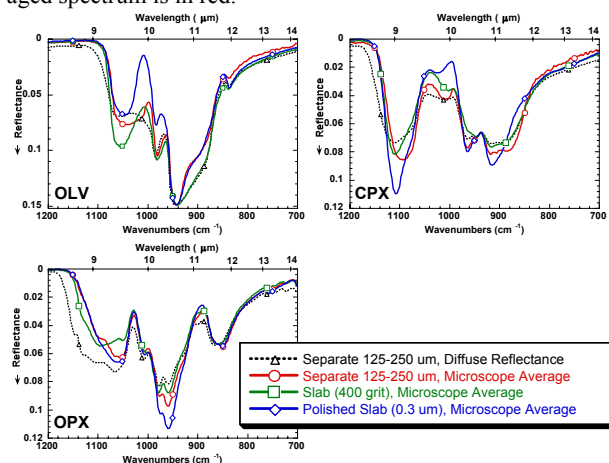
**Fig. 1.** NearIR microscopic spectra for Ronda minerals. Red: average reflectance of  $\sim 150 \mu\text{m}$  spots for a particulate mineral separates. Blue: transmission spectrum of the same mineral in a 300  $\mu\text{m}$  thick doubly polished thin section.

**Mid Infrared (8-14  $\mu\text{m}$ ).** Measurements in the midIR produce high signal to noise on particulate, polished and

unpolished surfaces. As with the nearIR measurements for the particulate samples, the intensity between spots varies greatly based on grain orientation (Fig. 2). Both the features and intensity of the averaged spectra of the mineral separates are comparable with spectra of the same bulk sample measured using the diffuse reflectance system (Fig. 3).



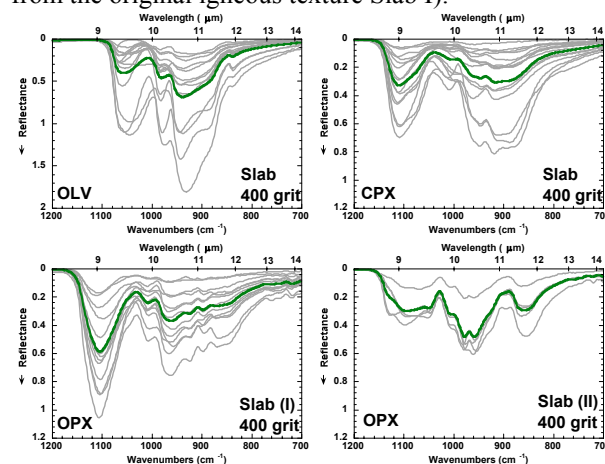
**Fig. 2.** MidIR microscopic spectra for particulate mineral separates. Individual measurements are in grey, and the averaged spectrum is in red.



**Fig. 3.** Scaled comparison of MidIR spectra of individual minerals as measured by the microscope system and the diffuse reflectance accessory.

Slab measurements produce an average signal that is 5-10x stronger than the particulate surface average although the intensity is still highly variable (Fig. 4). The shape and position of absorption bands is relatively constant for olivine and clinopyroxene. The orthopyroxene spectra, however, can be separated into two distinct groups. The majority of orthopyroxene slab measurements display a large first absorption near  $1100\text{ cm}^{-1}$  (Fig. 4, Slab I) which is not evident in either the diffuse or microscopic reflectance measurements of particulates. Further examination reveals that when a spectrum is collected perpendicular to a cleavage surface (or the c-axis), the three-peaked spectrum that is similar to that of the particulate measurements is produced (Fig. 4 Slab

II). A preferred orientation is indeed observed for the particulate orthopyroxene as grains tend to align on their cleavage surfaces due to their tabular nature. In contrast, the slabs preserve the random grain orientation from the original igneous texture Slab I).



**Fig. 4.** MidIR microscopic spectra for a series of  $150\text{ }\mu\text{m}$  slab spots. Individual measurements are in grey, and the averaged spectrum is in green. OPX spectra are separated into two distinct groups (bottom, see text for discussion).

**Conclusions:** Optical constants of samples constrain what can and cannot be measured using microspectroscopy: (1) For nearIR reflectance, where photons enter the body of a crystal and are multiply scattered by different grains, a smaller target means few photons will return to the detector from the target area. This precludes most reflectance measurements of small features in the nearIR. (2) Due to small values of  $k$  in the nearIR, transmission spectra typically require relatively thick doubly polished sections (ie. no glass or glue). (3) Microspectroscopic measurements in the midIR detect well-defined absorption features for most samples (particulate minerals; polished and unpolished slabs). Although the intensity and shapes of absorption features vary slightly, their position is consistent with diffuse reflectance measurements of bulk samples. (4) Because microspectroscopy often examines single grains, differences in spectra due to crystal orientation are accentuated, and must be considered in analyses.

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**References:** [1] Klima, R.L. and C.M. Pieters, (2004) *LPSC XXXV*, abs. #1305. [2] Morlok, A., G.C. Jones, and M.M. Grady, (2004) *LPSC XXXV*, abs. #1622. [3] Bukovanska, M., I. Nemec, and M. Solc (2000), *LPSC XXXI*, abs. #1559. [4] Palomba, E., et al. (2000), *Mem. S.A.I.*, 1139-1143. [5] Galád, A. (1999), *Meteoroids 1998*, 383. [6] Bukovanska, M., I. Nemec, and M. Solc, (1998) *Meteoritics & Planetary Science*, vol. 33, p. A25, 33, 25. [7] Dalton, J.B. and J.L. Bishop (2003), *LPSC XXXIV*, abs. #2066. [8] Launeau, P., C. Sotin, and J. Girardeau, (2002) *Bull. De La Societe Geologique De France*, 173, 491-508.