

Design, Development and Testing of an Environmental P-T Cell for InfraRed Spectroscopy Measurements.

S. De Angelis¹, M. Ferrari¹, M.C. De Sanctis¹, D. Biondi¹, A. Boccaccini¹, M. Formisano¹, A. Morbidini¹, E. Ammannito^{1,2}, T. Di Iorio^{1,3}.

¹IAPS-INAF (via del Fosso del Cavaliere, 100, 00133, Rome, Italy; simone.deangelis@iaps.inaf.it), ²ASI, Rome, Italy,

³ENEA SSPT-PROTER-OAC, Centro Ricerche Casaccia, Via Anguillarese 301, 00123, Roma, Italy

Introduction

Infrared spectra of minerals and rocks acquired in the laboratory are often dominated by the presence of absorption features due to adsorbed water. Frequently H₂O molecules from ambient water vapor, at standard P-T conditions, are subject to physical adsorption: the molecules quickly attach between the surface grains of the materials (especially powders, but also slabs) both during the preparation, storage and measuring phases. Fundamental absorption features due to OH-stretching and H-O-H bending transitions in water occur in the 3-6- μ m spectral region, while overtones and combinations occur in the 0.9-2.5- μ m region [1,2]. These absorptions can often mask diagnostic bands characteristic of the host materials, and this can also be problematic when using laboratory spectra for comparison and interpretation of data from planetary missions. In order to overcome these issues, we designed and developed an environmental cell to acquire infrared reflectance spectra of materials under controlled pressure and temperature.

1. Cell Design

The vacuum chamber was realized custom by Vacom, while other parts were realized custom at INAF-IAPS. The sample to be measured is placed in a copper sample holder, which is in contact with a ceramic heater, that allows to warm up to 400 °C. The temperature control is performed thanks to a PID controller. The cell consists of a UHV stainless steel cube: a primary diaphragm membrane pump and a secondary turbomolecular pump in the current version permit to reach a vacuum of about 10⁻⁵ mbar within the cell. A 3-mm thick transparent CaF₂ window allows acquiring spectra in the visible-

infrared range. Both the sample holder and ceramic heater are 25.4 mm in diameter, while the viewport diameter is 40 mm. The total height of the cell is about 50 mm.

2. Preliminary tests.

We performed numerous tests with various types of powder samples, mostly phyllosilicates with different hydration states, using a FieldSpec Pro spectro-photometer in the range 0.35-2.5 μ m and a QTH lamp. Test spectra were acquired in continuously-running mode by placing a sample within the cell, then pumping from ambient pressure down to 10⁻⁴-10⁻⁵ mbar, and keeping fixed the temperature at room values.

In the pumping phase the sample lost almost all the adsorbed water. We start the heating phase once that the limit pressure is reached. Further spectra were acquired at different temperatures in the range 20-300 °C. Most of the samples lost all their water above 150-200 °C. At higher temperatures (T>240-250 °C) we register the dehydroxylation process for a few phyllosilicates, in accordance with the TGA data for these minerals.

3. Example: Hectorite

The spectra of hectorite (SHCa_1) acquired during the initial pumping phase from 10³ down to 10⁻⁴ mbar are shown in figure 1. The water absorptions located at 1.4 and 1.9 μ m almost disappear during the pumping. The absorption band near 2.3 μ m, likely due to Mg-OH, remains quite unaltered. The spectra of hectorite acquired in vacuum (10⁻⁴ mbar) during the subsequent heating phase are shown in figure 2. The room P-T spectrum (red) is shown for comparison. Spectra have been acquired during the

temperature ramp 50-250 °C. The mineral experiences oxidation for $T > 240$ °C, as evidenced by the decreasing of reflectance and change in spectral shape.

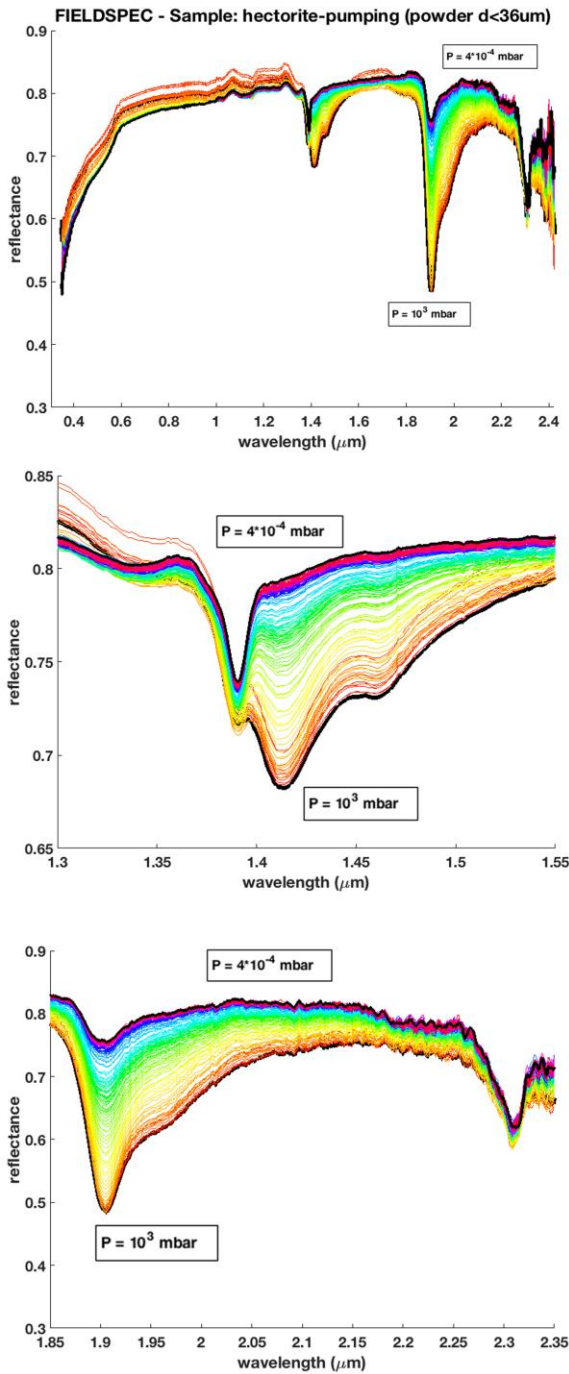


Fig. 1. Spectra of hectorite, acquired with FieldSpec Pro during the pumping phase, from 10^3 to 10^{-4} mbar. The water absorptions at 1.4 and 1.9 μm almost disappear during the pumping.

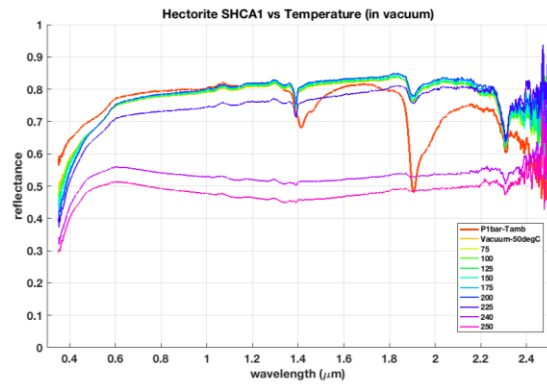


Fig. 2. Spectra of hectorite acquired with FieldSpec Pro in vacuum, during the heating phase. The starting spectrum (room P - T) is in red, for comparison. Spectra are acquired during the temperature ramp 50-250 °C.

Acknowledgements

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References

- [1] Farmer, V. C.: *The vibrations of protons in minerals: hydroxyl, water, and ammonium*, in *The infrared spectra of minerals*, edited by V. C. Farmer, pp. 137-181, Mineralogical Society, London, 1974.
- [2] Clark, R.N., et al: *High spectral resolution reflectance spectroscopy of minerals*. *Journal of Geophysical Research* 95 (B8), 12653–12680, 1990