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IR spectroscopy of ammoniated phyllosilicates at low pressure/high temperature conditions

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Introduction

Ammonium phyllosilicates are thought to be among the constituents of dwarf planet (1) Ceres surface, based on ground-based telescopic [1] and VIR-Dawn spectral data [2]. Following these findings, several works are currently trying to reproduce in the laboratory the Ceres surface composition, in terms of multi-component mineral mixtures [3,4]. Thus it is of interest to investigate the behavior and stability of ammonium compounds, when measured at pressure-temperature conditions that are different from standard laboratory values. Here we describe Visible-Infrared spectroscopic measurements of an ensemble of ammonium phyllosilicates: spectra have been acquired at various pressure-temperature conditions, by means of a P-T cell realized at INAF-IAPS laboratory.

1. Samples and experimental setup

Ammoniated phyllosilicates were produced in the laboratory starting from natural samples, following a procedure described in a series of works [e.g. 6].
Infrared spectra of five ammoniated samples (montmorillonite, SCa-3, two nontronites, NAu-1 and NAu-2, illite-smectite, ISCz-1 and hectorite, SHCa-1) were then acquired in the spectral range 0.35-2.5 μm, by using an ASD FieldSpec Pro 4 spectro-photometer equipped with a QTH lamp. The instrument is characterized by a spectral resolution of about 3-10 nm in the whole range; the spatial resolution of the setup was about 5 mm on the sample. All samples were analyzed in the form of powder, with grain size d<36 μm.
In order to acquire reflectance spectra at varying conditions, the samples were placed inside a P-T environmental cell, developed at INAF-IAPS [5]. The measurements strategy was the following: (i) acquisition at room P-T, (ii) sequence of acquisitions at room T during pumping; in this stage the pumping was first performed with only primary diaphragm pump (down to a limit of 3-4 mbar) and then also with turbo-molecular pump (down to vacuum pressure of 10^{-4}-10^{-5} mbar); (iii) acquisitions in vacuum at higher temperatures.

2. IR Spectral measurements.

Here we report, as an example, on the analyses performed on one sample, nontronite (NAu1). The spectra of NH_4-Nontronite are shown in fig.1. Data were acquired in three stages. The first spectrum (fig.1, A) is at room pressure and temperature. In panel B spectra were acquired at room T during primary pumping from ambient to 10^{-4} mbar. Finally in panel C spectra acquired in vacuum at different temperatures (50-240°C) are shown.

3. Results and Conclusions

Spectra of nontronite are characterized by Fe^{2+}-Fe^{3+} bands at 0.7-1 μm, and by OH^-/H_2O bands at 1.4 and 1.9 μm [7]. The feature at 2.3 μm is Fe-OH [7]. NH_4^+ absorption is visible at 2.12 μm in the room P-T spectrum. After pumping the adsorbed water is removed and NH_4^+ features become evident also at 1.55 and 2.01 μm: at room P-T these two features are shoulders in the 1.4 and 1.9 μm bands. After heating up to 240°C all NH_4^+ bands are visible and quite separate from hydration bands.
We can see that NH_4^+ bands remain quite unaltered both by the process of pumping, at least down to a vacuum of about 10^{-4} mbar, and by the process of heating up to 240°C. For higher temperatures the nontronite sample is subject to structural changes: all the water is removed, then ammonia and finally dehydroxylation occurs (fig. 1C).

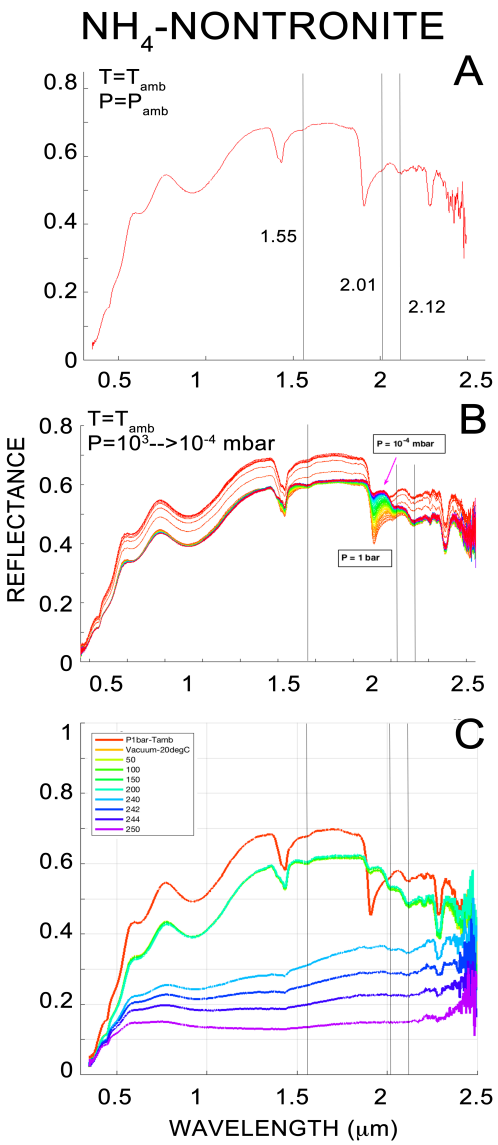

Fig.1. NH_4-Nontronite (NAu-1). A): ambient pressure and temperature; B): ambient temperature, during pumping; C): higher temperatures, in vacuum. Vertical lines indicate NH_4^+ absorptions.

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