PRELIMINARY EXPERIMENTAL VOLATILES RECOVERY FROM CARBONACEOUS ASTEROID SIMULANTS.

E. C. Unobe[1], L. S. Gertsch[1], and C. Dreyer[2], M. E. Schlesinger[1], J. Sercel[3], A. Abbud Madrid[2], R. Jedicke[4], S. Krot[4], D. Linne[5], J. Mantovani[6], [1](Missouri University of Science and Technology), [2] (Colorado School of Mines), [3](TransAstra Corp), [4](University of Hawaii), [5](Glenn Research Center), [6](Kennedy Space Center)

Introduction: Based on our current knowledge of the solar system, hydrated minerals on planetary bodies appear to possess great potential as ores from which water may be extracted and utilized for various aspects of human space exploration [1]. This study serves to bridge the gap between exploration for these resources and actual mining and recovery of useful products. We detail the setup of a laboratory system designed to extract, characterize and recover water from hydroxyl-bearing minerals present in a range of granular carbonaceous asteroid simulants. Extraction is by means of stepped vacuum pyrolysis. The evolved volatiles are characterized by a mass spectrometer and trapped as ice in a cold trap.

Experimental Setup: Fig. 1 shows a schematic of the laboratory vacuum system. Simulant samples are radiatively heated within a furnace that sits inside the main vacuum chamber (4ft diam., 6ft long), which is linked by a 6inch diam. cross-connector to a mass spectrometer and a liquefied nitrogen (LN_2) cold trap. The cold trap sits inside the cross-connecter, and is made up of ¼-inch stainless steel (SS) tubing carrying LN_2 in loops that form a cup structure. The Nitrogen gas vents upward and out from the cold trap loops and copper shimstock fins increase their frontal surface area and overall thermal conductivity. A quadrupole mass spectrometer also connected to the cross-connecter identifies molecules in the residual gas environment and outputs partial pressures of its component gasses, a value dependent on their relative concentration.

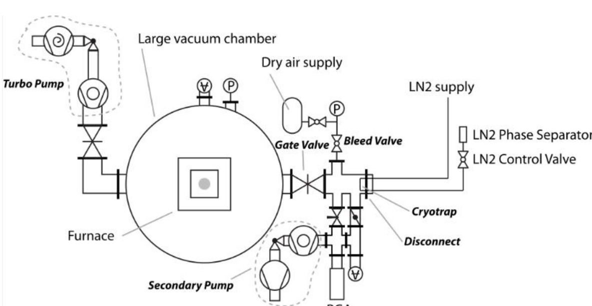

Figure 1; Laboratory Vacuum System Schematic

A series of shakedown tests to prepare and characterize the entire system have been conducted to minimize heat loss from the furnace and to improve cryotrap efficiency by installing radiation shields around the furnace and adding insulation to high heat-loss areas in the cooling sub-system.

Sample Preparation: The simulants to be tested are granular terrestrial serpentine (a major hydrated phase of carbonaceous asteroids) and olivine (a non-hydrated asteroidal component) obtained from a commercial vendor in 2-5 inch diameter pieces. Also to be tested is a simulant designed by the NASA SSERVI Center for Lunar and Atmospheric Surface Science (CLASS) to match the composition of the Orgueil meteorite. The serpentine and the olivine samples have been comminuted separately to particle sizes below 300 µm, to produce samples with similar particle size distributions to the CLASS simulant.

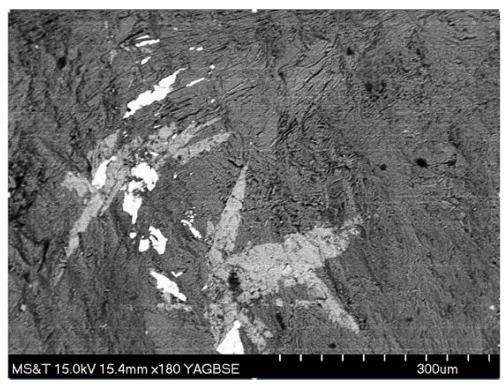

Figure 2; Backscattered image shaded to show the chemical phases present.

X-ray diffraction (XRD) analysis showed that the serpentine samples have the hydrous magnesium silicate, Lizardite [$Mg_3 Si_2 O_5 (OH)_4$] as the major phase while the anhydrous magnesium silicate, Forsterite [$Mg_2 (SiO)_4$] is the major phase in Olivine samples. Energy dispersive spectroscopy (EDS) and backscattered image analysis (Fig. 2) indicates that Nickel, Carbon, Calcium and Iron-bearing minerals exist in detectable quantities. A combined thermogravimetric-differential thermal (TG-DT) analysis was run to investigate the manner of volatile production and phase transitions to expect during heating.

For each test, a one-kg-sized sample is placed in a cylindrical 3x4 inch container made from corrosion-resistant fine-gauge 316 SS wire cloth (Fig. 4 inset). Four thermocouples carefully placed throughout the cylindrical mass (Fig. 3) yield information on the nature and progress of heat distribution during the experiments (Fig. 4).

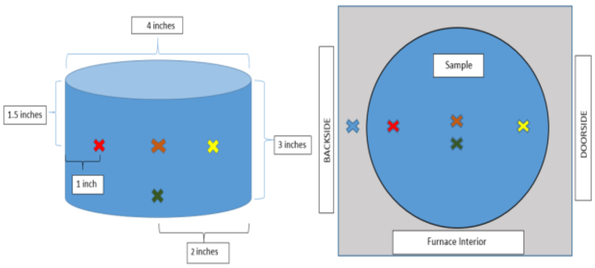

Figure 3; Schematic showing thermocouple placement in sample mass.

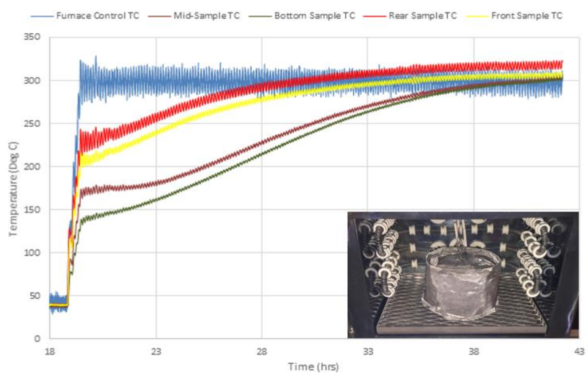

Figure 4; Heat progression and distribution within sample at 300 deg C plateau. Inset: Sample in furnace.

Experiment Procedure: Before starting each new sample test, a 12hr bakeout followed by a leak-up test is conducted to reduce and measure the contribution of background gases to the vacuum environment. Then a 1kg sample is prepared, placed inside the furnace, and heated following a stepped ramp program.

Because this study is interested in chemically bound water rather than water adsorbed from Earth's atmosphere, volatiles evolved during the first temperature ramp to 300ºC are excluded from the results. Subsequent temperature plateaus are held every 100ºC until the volatiles yield falls and the temperature equilibrates throughout the sample. When each plateau ends, the cold trap is removed, weighed, and its contents sampled. It is then cleaned out and re-attached to continue the experiment at the next temperature plateau.

The experimental procedure has been refined during the preliminary tests to maintain system cleanliness and consistency among tests, to maximize the comparability between simulants, and to reduce pump-down time and contamination from background gases such as Hydrogen, Nitrogen, CO_2, and H_2O. During experiments, these gases build up independently of the sample-derived volatile species that are the focus of this study.

Early Results and Discussion: The preliminary tests of olivine and serpentine indicate that the system can identify and successfully trap volatiles species from these basic minerals (Fig. 5 and 6). The volatiles yield is controlled in bulk samples by more than the expected heat and mass transfer rates, but also by chemical reactions that take place among the products within the sample and in the collection volume.

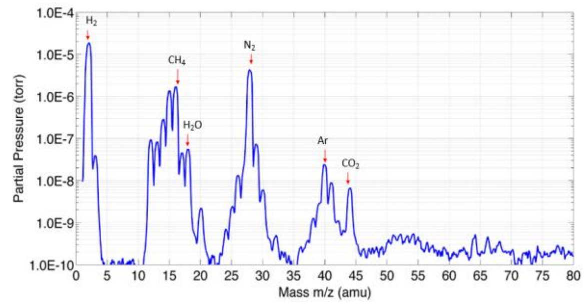

Figure 5; Typical spectra obtained during testing of serpentine at 500ºC.

Mass spectra show the volatile species produced from serpentine (Fig. 5), while water and CO_2 are the main products from olivine.

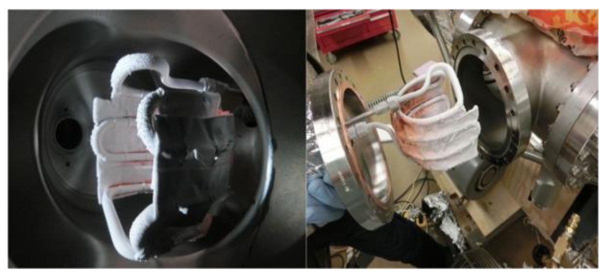

Figure 6; Left: Cryotrap operating in vacuum. Right: Cryotrap being removed with ice collected

References:

[1]Sanders et al. (2005) Results from the NASA Capability Roadmap Team for In-Situ Resource Utilization (ISRU)," ILC, 20 Sept.