THE OPTICAL PROPERTIES OF NANOPHASE IRON: INVESTIGATION OF A SPACE WEATHERING ANALOG. S. K. Noble[1] C. M. Pieters[1] and L. P. Keller[2], [1]Brown University, Providence, RI 02912, noble@porter.geo.brown.edu, [2]NASA JSC, Houston, TX 77058.

Introduction: It is known that space weathering, in particular the nanophase iron ($npFe^0$) created via vapor and/or sputter deposition, has distinct and predictable effects on the optical properties of lunar soils [1,2]. In addition to the attenuation of absorption bands, weathering introduces a "characteristic continuum" which is controlled by the amount of $npFe^0$ present [3]. The shape of this continuum may also be controlled by the size of the $npFe^0$ grains. It is thought that small $npFe^0$ grains result in reddening, while larger grains only darken the material [4]. To investigate this phenomenon we have created a lunar weathering analog by impregnating silica gel powders with $npFe^0$ following the method of [5,6].

Methods: Nanoporous silica gel chromatography substrates (courtesy of GRACEDavison) were impregnated with ferric nitrate solutions then heated. The three powders used in this study have pores with narrow gaussian distributions centered at 6 nm, 15 nm, and 25 nm. All the powders are 100-200 μm.

The gels were impregnated with ferric nitrate solutions of various normalities (0.001-1.0). This allowed us to create samples with a wide range of Fe contents. However, it is difficult to control exactly how much Fe is introduced into each sample, thus even samples created with the same normality may end up with different $npFe^0$ concentrations.

After impregnation the gels were thoroughly dried in air before calcining in air at 550°C for 60-80 hours, producing silica gel with nanophase hematite (Fe_2O_3) in the pores. Spectra of these samples compare well with similarly prepared samples of Morris et al [6].

The hematite-bearing powders were then placed in a furnace at 850°C under reducing conditions (flowing hydrogen) for 4 hours and then cooled (~500°/hr) while hydrogen flow was maintained, resulting in $npFe^0$-bearing powders. Spectra of all samples were obtained by the RELAB facility at Brown University.

Results: Figures 1-3 are the visible and near-IR spectral data that we have obtained thus far for the three silica powders. The lightest blue spectrum in each graph (SGX.0R) represents the control sample that contains no iron. Successive spectra contain increasing amounts of iron obtained by varying the strength of the ferric nitrate solution. Measurements are planned (neutron activation analysis) to ascertain the iron concentration in each of the experimental runs. The sharp peak at 1365 nm is due to OH bound to the silica host.

All three sets follow the same general trends seen in weathered lunar soils [3]. Initially there is a steep curvature in the visible while longer wavelengths remain relatively unaffected. With additional iron, the spectra become very red and increasingly linear and then begin to darken. Unlike the lunar soils however, we find that at higher iron concentrations, rather than becoming simply flat and dark, the analog powders become convex. These convexities were predicted by the modeling results of Hapke [2], and thus are probably an inherent property of $npFe^0$. This effect may be masked in natural soils by other darkening agents.

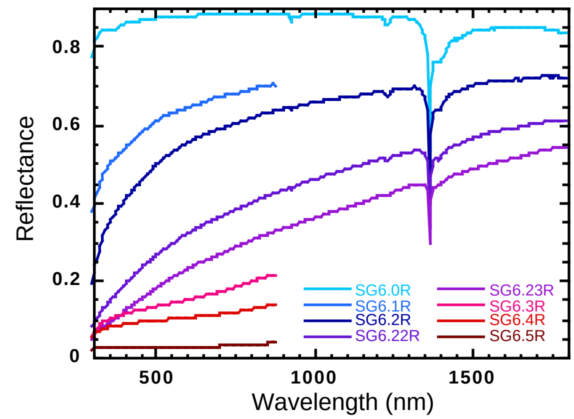

Figure 1. Reflectance spectra of 6 nm pore gel with $npFe^0$.

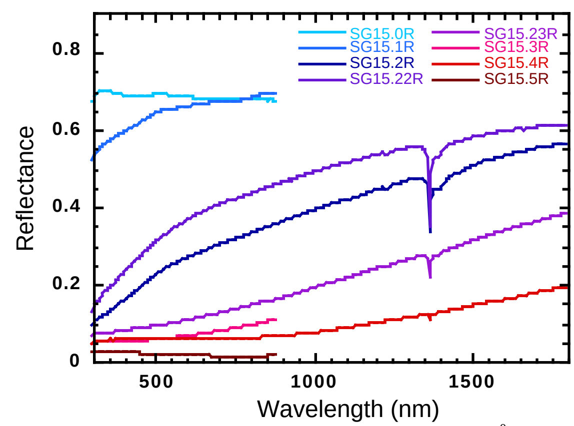

Figure 2. Reflectance spectra of 15 nm pore gel with $npFe^0$.

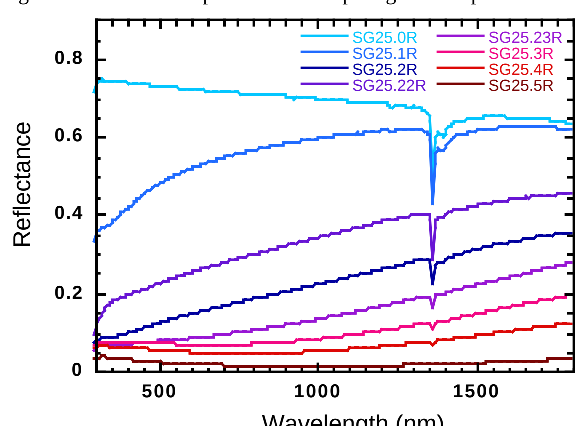

Figure 3. Reflectance spectra of 25 nm pore gel with $npFe^0$.

In figure 4 is a TEM bright field image of a lunar soil grain with a weathered rim. Compare this to figure 5, two examples of our silica gel analog. The dark spots in all three images are $npFe^0$ particles. There are several differences between the natural soil and the analog. First, in the analog, the Fe is distributed throughout the grain rather than concentrated on the outside. Second, the analog is very transparent and porous. Finally, the mean $npFe^0$ sizes created in the analogs are larger than what is found in natural weathered rims. The natural soil rims display a range of $npFe^0$ sizes from about 1 to 15 nm in diameter, though the average size is 3 nm [7]. The 6 nm pore gels contain $npFe^0$ particles ranging from about 10 to 20 nm in diameter. The 25 nm pore gels $npFe^0$ ranges from about 15 to 60 nm in diameter, though the vast majority are 30 to 40 nm.

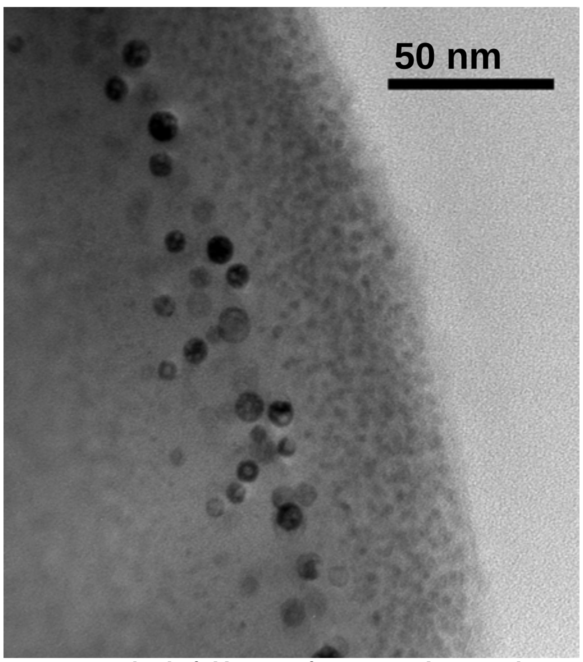

Figure 4. TEM bright field image of a mature soil grain with a complex multi-layered weathered rim.

Conclusions: It is inappropriate to compare details of the spectra for the three pore sizes without knowing the exact amount of iron in each; however, some generalizations can be made. It appears that the 25 nm pore silica gel, while following the same general trends as the smaller pore sizes, becomes linear at lower iron concentrations. It also appears to darken at lower concentrations as well. These 25 nm pore spectra nevertheless clearly display a significant amount of redness at very low iron concentrations. TEM analysis of these low iron concentration "red" spectra confirm that the average $npFe^0$ size is 30 to 40 nm. Keller et al. [4] showed that in individual lunar soil grains much of the darkening appears to result from the presence of $npFe^0$ >10 nm in diameter, whereas the reddening is only prominent in glasses and rims where the average grain size of the nanophase metal is <5 nm in diameter.

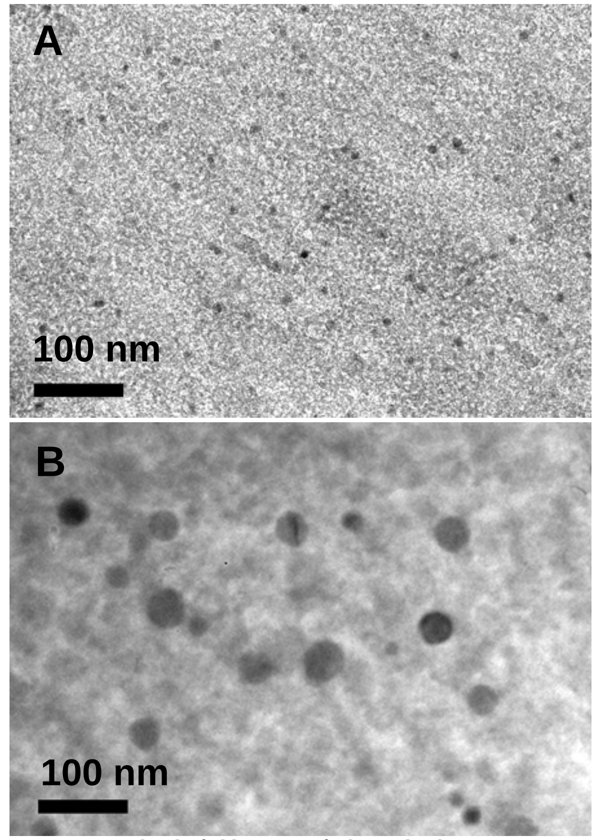

Figure 5. TEM bright field images of silica gel anlogs: A) 6 nm pore gel with iron. B) 25 nm pore gel with iron. Note that the scale is the same for both samples.

Our experiments however, suggest that other variables, most notably concentration of the $npFe^0$, also have a major effect on optical properties and needs to be factored into any consideration of space weathering of natural samples. It appears that the transition from reddening to darkening is more complex and can occur at larger $npFe^0$ sizes than previously thought. We are currently exploring ways to study both larger as well as smaller sizes of $npFe^0$ in order to better understand how the optical properties change with size and concentration of $npFe^0$.

References: [1] Pieters C. M. *et al* (2000) *MAPS*, **35**, 1101-1107. [2] Hapke B. (2001) *JGR* **106** E5 10,039-10,073. [3] Noble S. K. *et al* (2001) *MAPS.*, **36**, 31-42. [4] Keller L. P. *et al* (1998) *New Views of the Moon*, 41. [5] Allen C. C. *et al* (1996) *LPSCXXVII* 13-14. [6] Morris R. V. (1989) *JGR* **94** B3 2760-2778. [7] Keller L. P and Clement S. J. (2001) *LPSCXXXII*, 2097.

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