

Atom Probe Tomography of Reduced Phases in Apollo 16 Regolith Sample 61501,22

P. Gopon^{1,2}, M. Spicuzza², T.F. Kelly³, D.A. Reinhard³, T.J. Prosa³, D.J. Larson³, J. Fournelle²

¹ Dept. of Earth Science, University of Oxford, Oxford, Oxfordshire, UK

² Dept. of Geoscience, University of Wisconsin, Madison, Wisconsin, USA

³ CAMECA Instruments Inc., Madison, Wisconsin, USA

Nanophase iron (npFe) is relatively abundant in the lunar regolith and is widely believed to have formed by reduction of Fe during space weathering (micrometeorite/cosmic ray bombardment; [1]). Iron-silicides (Fe-Si) and native silicon require more reducing conditions than Fe⁰, and though predicted to be present in the lunar regolith have only been reported from one lunar meteorite and one lunar regolith sample [2,3,4]. Fe-Si and native silicon are only stable at extremely reducing conditions and have been proposed to form by a mechanism similar to that of metallic iron [3]. The stability of native silicon requires ten orders of magnitude lower oxygen fugacity than metallic iron at temperatures above 1600 K [5].

An early study of the soil sample (61500, [6]) suggested elevated levels of carbon, and an EPMA study of the Fe-Si from this soil sample [7] suggested that carbon might be present in the Fe-Si. Because carbon is highly reducing at the high temperatures and low pressures that would have been present during the formation of these phases, the presence of carbon was hypothesized to explain these ultra-reduced phases. In order to determine with more certainty the presence of carbon in these phases, we conducted an atom probe tomography (APT) study of Fe-Si. A representative Fe-Si bearing grain was selected and a focused ion beam (FIB) lifout of a Fe-Si rich region of that grain taken. The largest Fe-Si particle from the lifout region was previously analyzed with low keV EPMA [7], and served as a control for the data quality of the APT dataset.

Two successful APT datasets were acquired from the FIB lifout. The first dataset (M3) was from the center of the large (~2µm) Fe-Si particle that was also analyzed via EPMA. The major elemental chemistry as determined by APT of this sample was within 1 at.% for all elements, which boosted our confidence in the quality of both the EPMA and APT data. The second dataset (M9) is from the edge of one of the smaller Fe-Si particle, and contains the boundary with the host material. Interestingly, both datasets show similar levels (~60ppm) of carbon. However, while carbon (and indeed all of the trace elements) are homogeneously distributed throughout M3, dataset M9 shows enrichment of carbon, phosphorous, nickel, chromium, cobalt, and strontium near the boundary to the host.

The segregation of the trace elements towards the Fe-Si grain boundary shows that these phases did not fully quench, but does not fully constrain the timing of crystallization. The presence of carbon within the Fe-Si raises the possibility that carbon was present during the formation of these phases, and indeed could be in-part responsible for achieving the extremely low fO₂ required to form Si metal and Fe-Si.

Because the formation mechanisms for Fe-Si and npFe are hypothesized to be related, further study of npFe is warranted to investigate the similarities and differences between Fe-Si and npFe at the nano-scale. It would be interesting to see if similar trace levels of carbon or regions of trace element segregation within npFe can be visualized with APT.

References:

- [1] Hapke, B., *J. Geo. Research* **106(10)** (2001), p. 39.
 [2] Spicuzza, M.J. *et al*, 42nd Lunar and Planetary Science Conference (2011) p. 16.
 [3] Anand, M. *et al*, *Proc. of the Nat. Acad. of Sci. USA* **101(18)** (2004), p. 6847.
 [4] Nazarov *et al*, *Petrology* **20(20)** (2012), p. 506.
 [5] Essene, E.J., and Fisher, D.C., *Science* **234(4773)** (1986), p. 189.
 [6] Wszolek, P. *et al*, 4th Lunar and Planetary Science Conference (1973) p. 1693.
 [7] Gopon, P. *et al*, University of Wisconsin PhD Dissertation (2016) p. 1.

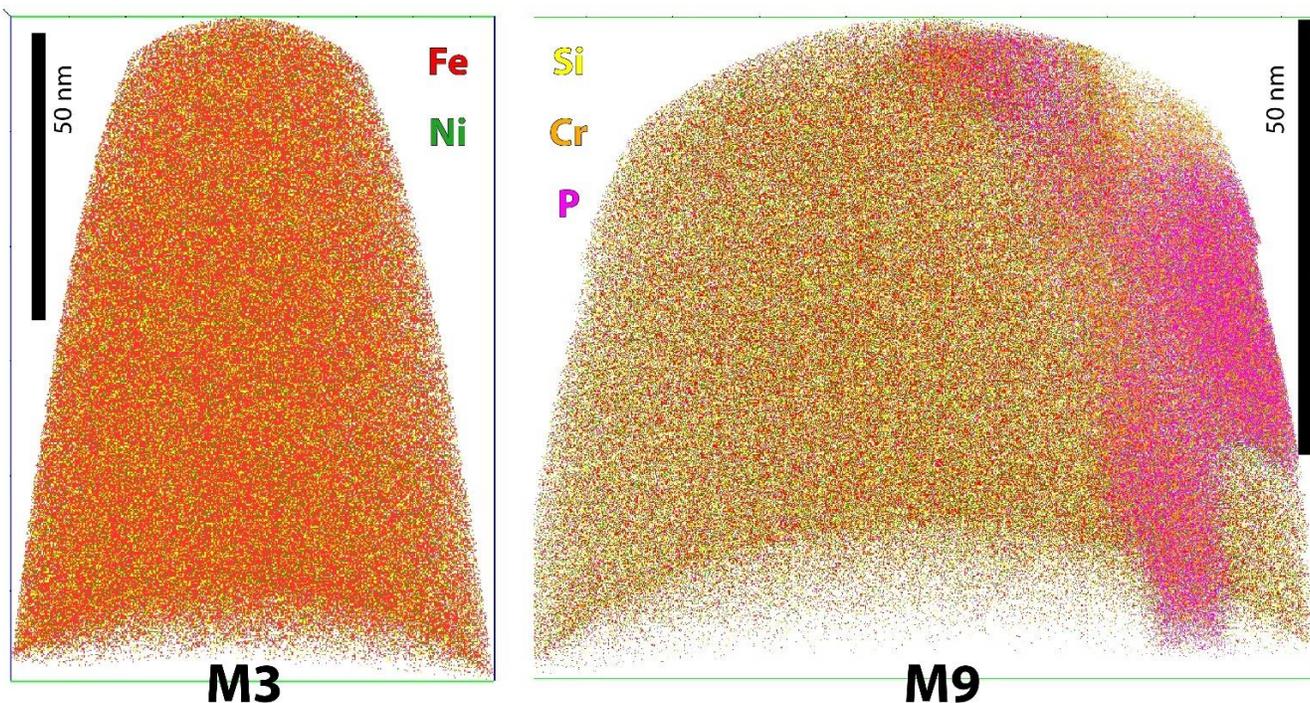


Figure 1. APT reconstructions of two needle shaped specimens by APT (side view). Each dot represents an ion detected, and the color of the dot represents its mass/charge spectral identification. Ions shown are P, Cr, Ni, Fe, Si. The colors each ion is represented with are noted in the figure, and are the same for both images. M3 (15 million ions) is from the largest suessite bleb in the liftout (correlates to EPMA analysis point 3-IV-9-4_points 1 and 2; Figure 5). Specimen M9 (35 million ions) is from a small suessite bleb. See Figure 8 for the location within the liftout where the specimens came from, as well as figure 11 for more detailed view of M9. Chemistry as determined by APT is given in Table 3.