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Complementary SEM-EDS / FIB-SEM Sample Preparation Techniques for Atom Probe Tomography of nanophase-Fe^0 in Apollo 16 Regolith Sample 61501,22

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Space weathering is defined as the processes that alter the surface of airless planetary bodies. Nanophase iron ($npFe^0$) is the most abundant product of space weathering, however it formation mechanisms are still poorly understood. On the Moon, $npFe^0$ ranges in size from a few nm to 1 µm and are widely believed to have formed by a variety of mechanisms. These include the in-situ reduction of FeO during cosmic ray bombardment, by localized heating by micrometeorites and the subsequent reduction of FeO, as well as the addition of Fe^0 from iron-nickel meteorites during micrometeorite bombardment [1], [2]. The exact nature of formation has wide ranging implications for remote spectral analysis of airless planetary bodies, as well as the cosmic ray and micrometeorite flux to the Moon. However, experiments to simulate the formation mechanisms have been unable to conclusively prove which mechanism is dominant, in no small part due to the difficulty in chemically analyzing these small, multi-phase metals. Atom Probe Tomography (APT) is the only technique currently available that is able to simultaneously acquire the major, minor, trace, and isotopic chemical information that we require to differentiate the formation mechanisms.

Here, we present novel Focused Ion Beam- Scanning Electron Microscopy (FIB-SEM) sample preparation techniques developed to characterize $npFe^0$ from an Apollo 16 soil sample (61501) using pulsed laser APT. APT provides the unique opportunity to conduct site specific analysis of these nanoscale space weathering products, and to generate spatially resolved trace element and isotopic chemical data on the near atomic scale, required to constrain their formation mechanisms. The aim is to investigate the trace element composition and the isotopic ratio of iron within the spherules, and determine if we can identify diagnostic features of either meteorites (proving the micrometeorite bombardment) or lunar material (proving the in-situ reduction by cosmic ray bombardment). However, the challenges associated with positioning an isolated <200 nm sphere into the apex of an APT needle are considerable and a combination of optimised FIB-SEM techniques and variable accelerating voltage dispersive spectrometry (EDS) mapping of the needle during sample preparation was used.

Standard site specific FIB liftout was carried out on the bulk material using SEM-EDS to locate the nanospherules [2], [3]. Larger nanospheres (greater than 500 nm) were clearly visible in secondary electron (SE) imaging and could be fabricated to be the full thickness of the APT needle through iterating imaging and milling stages combined with rotation. For nano-spherules under ~200 nm, sequential FIB polishing from all angles essentially removed the feature of interest before the outlines could be defined (Figure 1a). Instead, a combination of sequential milling and *in-situ* variable accelerating voltage EDS maps (Figure 1b) were acquired on the needle using a Zeiss Crossbeam 540 FIB-SEM with an integrated Oxford Instruments EDS detector combined with the Aztec software package. This allowed us to image the nanospherule within the bulk of the liftout prior to the final milling stages.

This paper presents the novel combination of techniques, using in-situ variable keV EDS analysis during FIB-SEM sample preparation for APT. This allowed us to position the nano-spherules within an APT needle (Figure 1c), where contrast variation from SE imaging during final stage milling was not sufficient to be able to determine the position of the nanosphere. The authors acknowledge funding from the University of Oxford Dept. of Materials and EPSRC [4].

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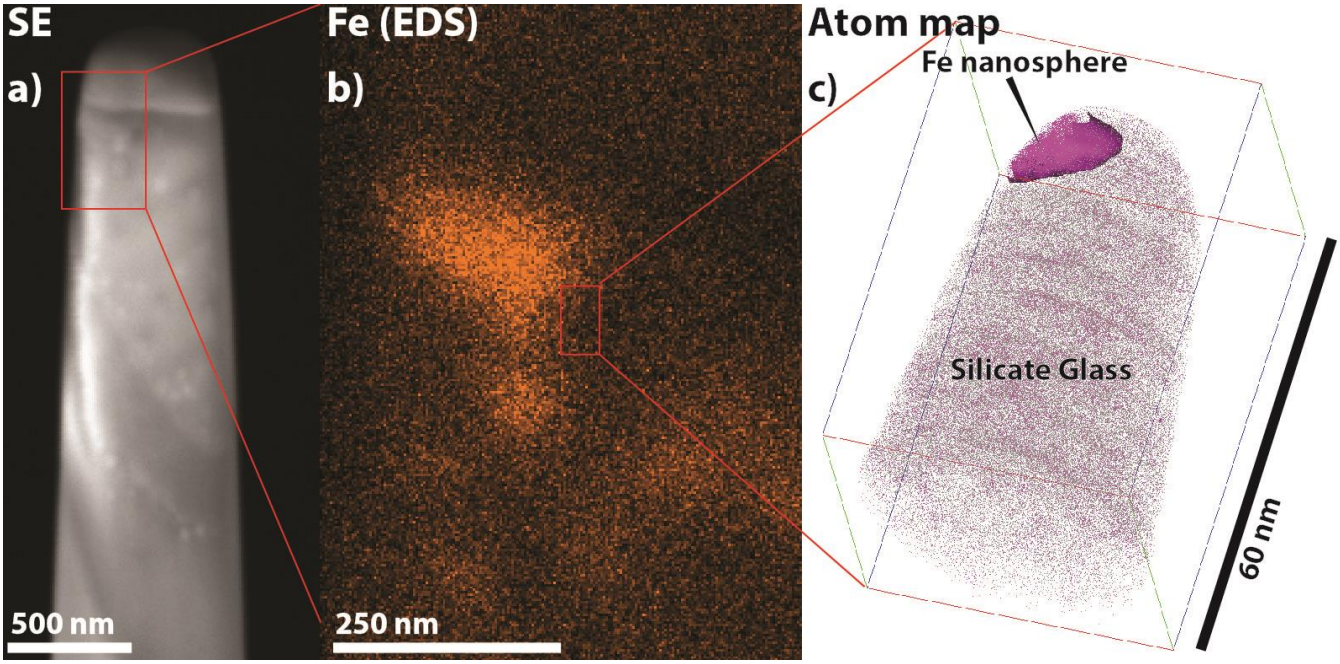

Figure 1. (a) SE image of atom probe needle prior to final polishing, red box is area enlarged in figure 1b.
(b) Iron EDS map of zoomed in view of large iron rich region previously unseen in the SE image, red box is approximate area of APT experiment.
(c) Atom map showing the edge of the nanosphere within the silicate glass matrix. Only iron and silicon ions are shown, and a 11 at. % Fe isoconcentration surface delineates around the iron nanosphere.

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