Electron Microscopy and Analysis of Martian Meteorite ALH84001 with Mochii_{ISS-NL} on the International Space Station

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Introduction: Mochii $_{ISS-NL\ (National\ Lab)}$ is the world's first and only orbital scanning electron microscope (SEM), capable of providing high-resolution imaging and elemental analyses in-situ in low Earth Orbit (LEO) on the International Space Station (ISS). The Mochii $^{\text{TM}}$ payload was delivered to ISS via Cygnus and Dragon space vehicles on NASA commercial resupply missions NG-13 and SpX-24, and has been traveling at a speed of ~ 17500 miles per hour while orbiting ~ 250 miles above the Earth's surface [1]. Mochii $_{ISS-NL}$ is part of the ISS National Laboratory [2, 3] which supports the advancement of science in space through novel in-situ microgravity scientific research and engineering in low Earth orbit (LEO) [2, 3].

The ISS is currently the only manned facility available that supports research in the unique continuous microgravity environment of LEO. ISS resources are available for use by the public – including researchers, commercial companies, and educational institutions – to perform experiments and engineering in microgravity. Mochii_{ISS-NL} provides textural, morphological, and chemical information on a wide variety of samples and is particularly useful for those samples that could be altered or destroyed during return to Earth (e.g., precipitation experiments, fragile or time-sensitive samples). Specific Mochii_{ISS-NL} applications include the following:

- o Identifying and measuring sample responses to unique extreme environments (radiation, microgravity, ionization)
- o Characterizing and documenting evolving phenomena over time in microgravity (crystal evolution, cell growth)
- O Supporting engineering needs from crew or vehicle (telemetry, safety assessment, orbital debris projectile identification)

As part of the verification of Mochii's on-orbit capabilities, we performed *in-situ* analysis on a fragment of the Martian meteorite Allan Hills 84001, designated as ALH84001, 200. This fragment was initially analyzed using a full-sized SEM and an engineering Mochii ground unit (*a.k.a*, Mochii_{Earth}) located at NASA JSC in Houston prior to launch. It was subsequently analyzed using Mochii_{ISS-NL} to compare results.

American astronaut Kayla Barron and European astronaut Matthias Maurer prepared the Mochii microscope platform and loaded the sample into Mochii_{ISS-NL} on ISS in January 2022 (Figure 1). Afterward, engineering and science teams at Voxa in Seattle and at NASA in Houston concurrently analyzed the ALH84001 sample remotely on iPads and iPhones, routing through the Voxalink cloud framework connected to NASA's command and data handling systems in Hunstville. Control latency for the ISS is about 1.5 s, and voice and



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visual communications were conducted real-time using the mobile devices while the astronauts conducted routine activities or slept on-orbit.

Martian Meteorite ALH84001: Discovered in Antarctica in 1984, ALH84001 is the oldest identified Martian meteorite which formed ~ 4.5 Ga ago [4]; it is significant in that four lines of evidence suggested the presence of past microbial life on early Mars [5]. One of those lines of evidence was the presence of unusual carbonates that comprise ~ 1 vol.% of the overall meteorite [4, 5]. These carbonates appear as circular or elliptical features ~ 10 -300 µm in size along the major axis. When viewed optically, they range in color from gold to burnt orange in their centers and are typically surrounded by a thin black-white-black rim; their three-dimensional morphology is best described as a flattened pancake or disk [5, 6]. Chemically the disk carbonates have a complex mixed cation composition with an empirical formula $Fe_xMg_yCa_zMn_{(1-x-y-z)}$ CO_3 where x + y + z = 1. The values of x, y, and z vary with radial distance in such a way that the carbonate disks can be envisioned as being composed of three concentric annular zones; starting from the center there is an inner central and outer core surrounded by a thin rim. Embedded within these carbonates are heterogeneously distributed discrete nanocrystal magnetites and thin siliceous veins tens of nm thick and up to several microns in length.

Methods & Results: Because the sample surface topography exceeded the optical depth-of-focus, through focus Z-stacks were used to generate extended depth-of-field optical images of the ALH84001 fragment that were stitched to create high resolution surface mosaics (Figure 2). This fragment was mounted on an aluminum SEM pin mount using a PelcoTM double-sided conductive C adhesive tab. The fragment surface displayed several carbonate disks, ranging from ~ 10 - 200 μm in the longest dimension, embedded in the host orthopyroxene matrix (Figure 2). A primary region of interest was identified containing two adjoining carbonate disks each with orange-colored cores (Figure 2). Following optical documentation, the sample was sputter coated with ~ 2 nm of Pt. Initial characterization was undertaken using a JEOL 7600 field emission SEM (FESEM) equipped with a Noran System VI energy dispersive X-ray spectrometer (EDS); element assays included point spectra and element maps collected at 15 kV (see Fig. 3 for spectra locations).

ALH84001, 200 was then analyzed using Mochii_{Earth} along with a collection of mineral (SPI 02753-AB 53) and metal standards. Sample locations at which EDS spectra were collected replicated those in the initial analyses with acquisition times of ~100 s. at 10 kV. The same sample was then subsequently analyzed using Mochii_{ISS-NI}. Images and spectra were collected at 10 kV with acquisition times ranging from 127.15 – 132.58 s. Locations for EDS analysis were chosen to match as close as was possible to those used in the Mochii $_{Earth}$ unit although, in a few instances, minor location variations were evident (Figure 4). Acquisition times of the Mochii_{ISS-NL} spectra were normalized to 100 s., binned and smoothed. Mochii_{ISS-NL} spectra, normalized to Mg, are shown for comparison to those collected using Mochii Earth in Figure 4. Spectra from Mochii_{ISS-NL} are a close match to those collected by Mochii_{Earth} with minor element variations between spectra due to slight mismatch in the locations of the analysis spots and sub-micron scale chemical heterogeneity of the carbonate disks (Figure 5). All spectroscopic peaks were accounted for and matched along the energy spectrum (Figure 5). Positional accuracy of the 10 kV tungsten-source electron probe was found to be better than ~ 1um on the order of the acquisition time of ~ 2 min/spectrum. There was some drift detected over longer time periods (~30 min) exceeding 1 um that could be attributed to both thermal drift and the changing magnetic field of the space environment as the space vehicle transited across the Earth's orbit (7). Mochii_{ISS-NL} was engineered to have stability better than ~ 1 um on the order of 10 minutes in the extreme environment of Earth orbit, and we found this to match nominally during on-orbit experiments.

Relative spectral peak heights were in good agreement with all elemental peaks in the ground analyses accounted for, indicating the accuracy of the tool on-orbit does match very closely with the ground

references. We found peak intensities varied more near the carbonate disk rim where composition varies dramatically, from Fe-rich to Mg-rich – on the order of tens of nanometers – making it difficult to get an exactly comparable analysis spatial position between ground and on-orbit sessions. Nevertheless, these analyses verify that Mochii_{ISS-NL} has accuracy for replicating ground-based precision elemental composition measurements in the extremes of space and provide a basis for future semi-quantitative analyses of relative composition.

<u>Summary</u>: An important area of application for SEM is in providing geological analyses for planetary exploration and astrobiology, by helping to answer questions about formation of heavenly bodies, and further for detection of trace compounds that support the search for evidence of life. Demonstrating the analysis of a sample that came to us from space and sending it back to space to analyze it there represents an important milestone supporting such studies. Mochii_{ISS-NL} sets a new benchmark enabling *in situ* analysis of regolith using miniature analyzers on other worlds, both in upcoming orbital vehicles such as NASA's Gateway and potentially on surface vehicles supporting missions such as Artemis or in future unmanned probes. Given the limited up-mass available for return to lunar orbit, future landing teams can now make better real-time selections on which samples would have the highest scientific impact to return.

Another important application area is in engineering and materials science for evaluation and identification of minerals and encapsulated compounds down to trace amounts in regolith to directly serve *in-situ* resource utilization (ISRU) needs [8, 9]. Trace element information available with SEM/EDS are expected to be critical for mining efforts needed to supply off-world bases and to identify and obtain fuels for powering surface and orbital operations. Reminiscent of the utility of "tricorder" technology from science fiction, all these areas benefit greatly from *in-situ* trace element detection using the capabilities now available in this powerful tiny (<13 kg, <100W, 20 cm) portable analytical system [8, 9, 10].



Figure 1. Astronaut Kayla Barron showing the installed Mochii_{ISS-NL} on ISS.

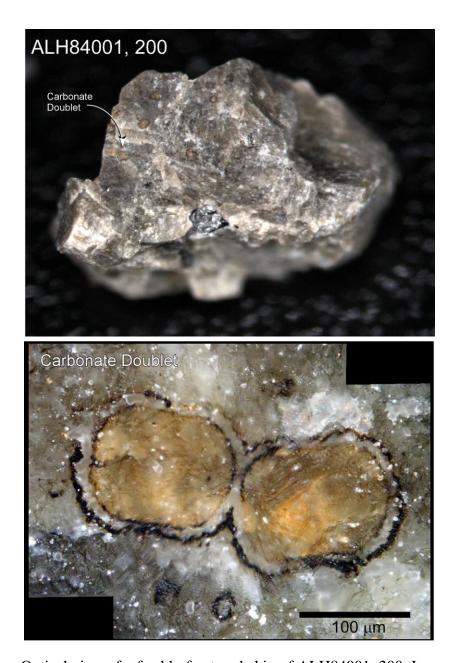


Figure 2. Upper: Optical view of a freshly fractured chip of ALH84001, 200. Lower: Optical view of carbonate doublet embedded in the orthopyroxene matrix in ALH84001, 200 (see location in upper view; arrow).

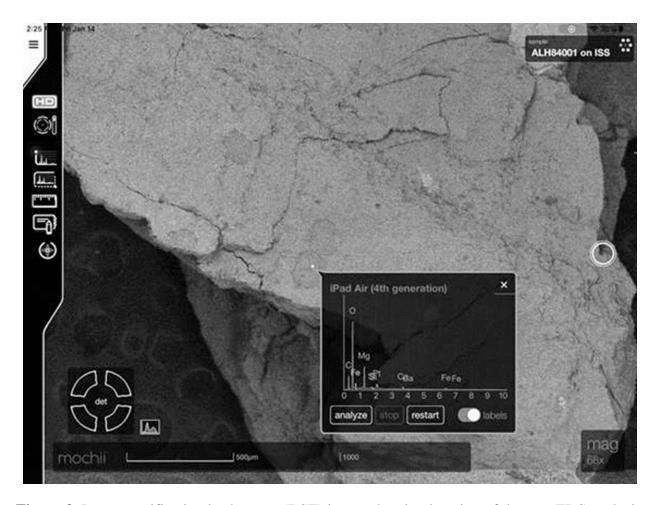


Figure 3. Low magnification backscatter (BSE) image showing location of the spot EDS analysis in ALH84001, 200. Several carbonate disks including one carbonate doublet, denoted by the EDS spectrum annotation, are present within the orthopyroxene (OPX) matrix (see Figure 2, lower view).

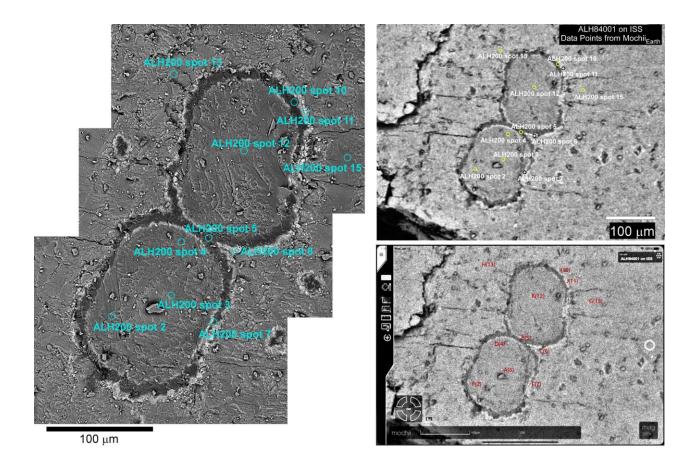


Figure 4. Carbonate disk doublet from ALH84001 grain 200 analyzed using JEOL FESEM (left), Mochii $_{Earth}$ (upper right) on-orbit Mochii $_{ISS-NL}$ (lower right). Ten sampling areas were analyzed on-orbit and subsequently compared to the ground reference spectra.

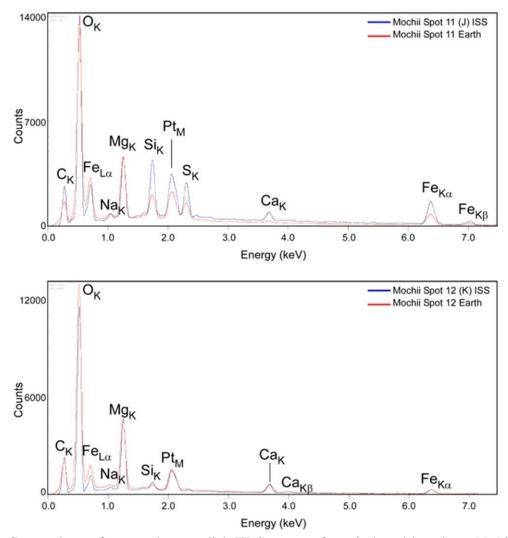


Figure 5. Comparison of two carbonate disk EDS spectra from indexed locations 11 (right corner of top disk) and 12 (center of top disk) illustrating correlation between engineering ground reference vs. an ISS on-orbit Mochii spectral performance. All elemental spectral peaks line up in the energy axis and are accounted for. In the matrix, where composition varies less over distance than near the rapid composition inversions near the disk edges, the relative intensities match

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