ADVANCES IN X-RAY INSTRUMENTS TO SUPPORT MARS SAMPLE RETURN.

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Introduction: The Mars 2020 *Perseverance* rover is currently collecting drill cores of ancient igneous and sedimentary rock in and around Jezero crater for potential transport to Earth [e.g., 1]. These samples from the martian surface will enable detailed mineralogical, geochemical, and petrological measurements to characterize ancient depositional and diagenetic environments, quantitatively age-date the samples, and identify the building blocks for life or evidence for life itself. Furthermore, these drill cores are especially precious because they may represent the most pristine samples from the martian surface and our best chance at identifying martian life, as future sample return missions may be conducted by humans that can introduce biological contaminants to the samples. Because of the importance of these samples, we must take great care in their handling, curation, and preliminary analyses so that they are preserved for scientific measurements for decades to come.

In-situ measurements by *Perseverance* have identified minerals that further warrant special treatment of the returned samples. Hydrated sulfate carbonate, swelling clay minerals, and oxychlorine salts are extremely sensitive to changes in temperature and relative humidity [e.g., 2,3]. The structures of hydrated sulfates and oxychlorine minerals, in particular, readily change when exposed to different conditions, meaning the mineral assemblage of the as-returned samples may be lost if the samples aren't handled properly [e.g., 4-6]. Characterizing the as-returned mineral assemblage, particularly of the salts, is essential for reconstructing past aqueous conditions and habitability.

To characterize the as-returned mineral assemblage, the samples must be analyzed rapidly before phase changes occur and/or under controlled conditions (e.g., within a glove box). Significant recent advances in X-ray instrumentation for robotic exploration of the solar system have resulted in high-resolution miniaturized instruments that would provide mineralogical, geochemical, and petrological information on the returned martian samples without degradation of the mineral assemblage. Here, we describe a combined X-ray diffractometer/X-ray fluorescence spectrometer (XRD/XRF), an X-ray computed tomographic (XCT) instrument, and a scanned beam XRF mapping instrument that could be used in a glove box so that the martian samples remain under controlled conditions.

XRD/XRF: The Chemistry and Mineralogy (CheMin) instrument is a miniaturized XRD/XRF on the Mars Science Laboratory *Curiosity* rover [7]. CheMin operates in transmission geometry and uses a Co source to minimize fluorescence from Fe. CheMin uses an energy-sensitive CCD to capture diffraction and fluorescence data from ~30 mg of powdered rock over the course of ~22 hours of analysis. XRF data are qualitative, and Rietveld refinement of XRD patterns provides mineral and X-ray amorphous abundances with a mineral detection limit of ~0.5-1 wt.%.

Technology developments introduced in the next-generation CheMin instrument (CheMinX) allow for substantially improved mineral detection, geochemical analysis, and speed. The inclusion of focusing optics and an array of four TimePix hybrid-pixel detectors (Fig. 1) allows for improved resolution (0.2° 2θ) and a 100X increased speed over MSL-CheMin. Increased angular resolution allows for improvements in mineral detection, including the discrimination of types of pyroxene minerals (e.g., orthopyroxene vs. clinopyroxene), not possible with MSL-CheMin. Samples could be analyzed in sealed cells such that martian materials could be prepared in a special atmosphere without being exposed to other conditions.

XCT: XCT is a non-destructive, high-resolution 3D imaging technique capable of analyzing internal features of multiphase materials (such as rock cores) and characterizing porous granular materials [10,11]. A miniaturized XCT prototype has been developed using a micro-focus X-ray source, rotating sample stage, and a scintillator film coupled to a CMOS detector with a fiber optic plate [e.g., 12]. Data collected on the flight prototype demonstrate a 30 μm spatial resolution for a 3 mm-diameter sample, allowing for the characterization of grain sizes and shapes and analysis of vesicle shape, size, and orientation (e.g., Fig. 2) [13]. A variant of this instrument could be developed for characterization of rock cores using higher energy X-rays (50-60 kV) and could be operated in a glovebox for the study of returned samples [12]. Crystal morphologies derived from XCT data would complement mineralogy determined by XRD and provide a measure of grain size distribution for the different phases.

XRF: A scanned-beam XRF instrument could allow for high-resolution elemental imaging as well as quantitative XRF analysis of rock cores or rock powder. microScanix™ is a portable µ-XRF instrument intended for art and cultural heritage studies that can scan in X,Y,Z with a resolution down to 55 µm over a range up to 300 mm. For rock sample analysis, the resolution will be improved to <30 µm and the range reduced. Quantitative XRF analyses can be obtained from 25 µm spots, or the instrument can be defocused to obtain rapid quantitative XRF data from larger areas (Fig. 3).

X-ray instruments for Mars Sample Return: X-ray instruments such as these are compact, are designed for robotic operation, and provide complimentary and synergistic data pertinent to the crystal structure, elemental composition, and morphology of returned samples. In the Sample Receiving Facility, they will provide detailed mineralogical, geochemical, and petrological information on precious samples in a minimally destructive fashion. These instruments could also be housed on a space station (e.g., ISS, Gateway) in case samples are first processed in orbit before return to Earth. The datasets collected by these instruments will provide information and context for more focused studies carried out by scientists who request and receive aliquots of returned samples for their own research.

References: [1] Simon J. I. et al. (2023) *JGR*, *128*(6), e2022JE007474. [2] Tice M. M. et al. (2022) *Sci Adv.*, *8*(47), 9084. [3] Scheller E. M. et al. (2022) *Science*, *378*(6624), 1105-1110. [4] Chipera S. J. & Vaniman D. T. (2007) *GCA*, *71*(1), 241-250. [5] Vaniman D. T. et al. (2018) *Am. Min.*, *103*(7), 1011-1020. [6] Robertson K. & Bish D. (2011) *JGR*, *116*(E7), e2010JE003754. [7] Blake D. F. et al. (2012) *SSR*, *170*, 341-399. [8] Blake D. F. et al. (2023) *LPS LIV*, 1373. [9] Allwood A. C. et al. (2020) *SSR*, *216*(134). [10] Falvard S. & Paris R. (2017) *Sedimentol.*, *64*, 453-477. [11] Thakur M. M. & Penumadu D. (2020) *Comp. Geotech.*, *124*, 103638. [12] Sarrazin P. et al. (2021) *Bull. AAS*, *53*(4). [13] Obbard R. W. et al. (2022) *LPS LIII*, 2314.

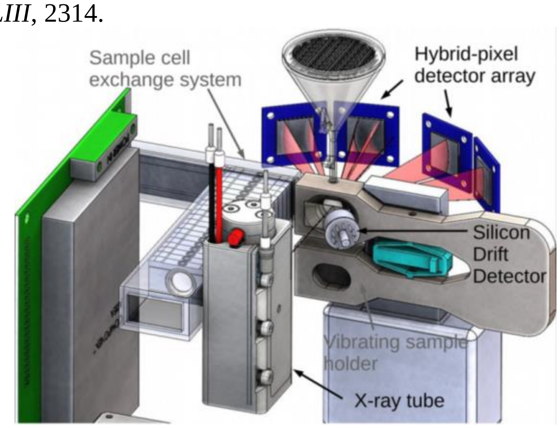

Figure 1. CheMinX design with hybrid-pixel detector array and SDD.

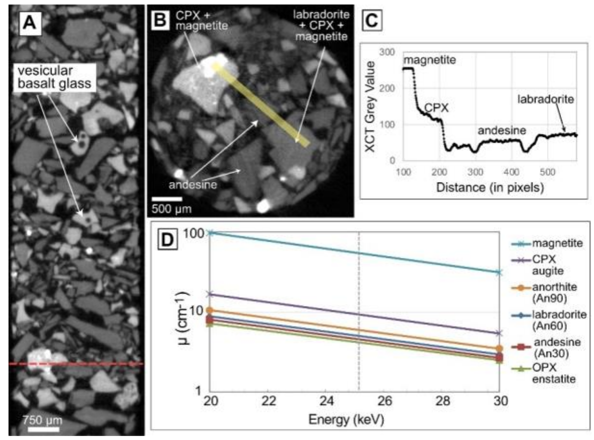

Figure 2. XCT data of gabbronorite, basaltic glass, and andesine mixture from the XCT breadboard. (A) Vertical slice showing basalt glass texture (arrows) and location of (B) (red dashed line). (B) Slice showing multimineralic gabbronorite clasts and andesine fragments. (C) Line profile highlighting different x-ray attenuations (XCT grey values) among phases. (D) Varied X-ray attenuation allows mineral discrimination in XCT data.

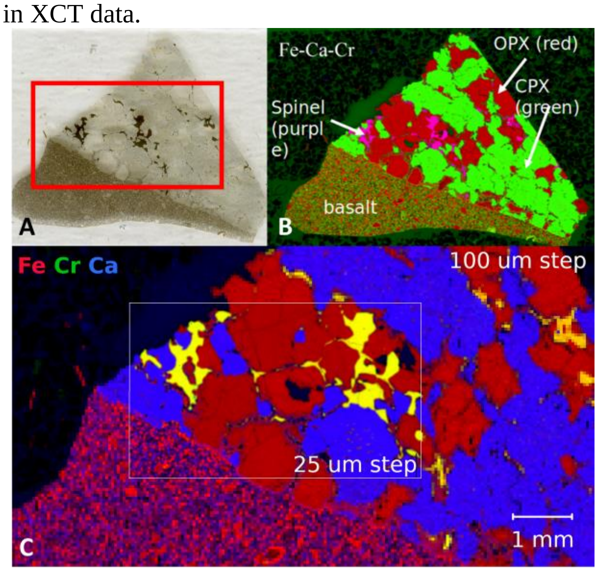

Figure 3. Xenolith compositional mapping with scanning electron microscopy energy dispersive X-ray analysis vs. microScanix. (A) Thin section of terrestrial xenolith, where the red box shows the location of compositional map by microScanix in (C). (B) Fe-Cr-Ca map for xenolith in (A) from a commercial EDAX Orbis Micro-XRF Analyzer. (C) microScanix Fe-Cr-Ca map, showing results for 25 µm vs. 100 µm step size inside and outside the white box, respectively.