

IN SITU X-RAY DIFFRACTION ON THE MOON, MARS AND ASTEROIDS. Flemming R. L.¹, McCausland P. J. A.¹, Gellert R.² ¹Dept. of Earth Sciences, U. of Western Ontario, London, ON, N6A 5B7 rflemmin@uwo.ca, ²Dept. of Physics, U. of Guelph, Guelph, ON, N1G 2W1, ralf@physics.uoguelph.ca.

Rationale: Most landed spacecraft-based instruments measure rock and soil physical properties or provide chemical information. It is desirable to deploy an XRD instrument to obtain complimentary crystal structural information, as is being built for the Mars Science Laboratory (CHEMIN) [1], or outlined for other missions [e.g. 2]. Here we advocate the development of an *in situ* XRD instrument, in which the complex tasks of sample collection and powdering would not be required.

In situ X-ray Diffraction. Micro X-ray Diffraction (μ XRD) is a versatile technique that is ideal for *in situ* studies of whole-rock specimens and powders, providing mineral identification using crystal structural parameters [3]. A key feature of this method is its ability to identify sample mineralogy with no sample preparation, an obvious advantage for spacecraft-borne observations where sample powdering for conventional

XRD analysis is problematic. Reconnaissance *in situ* XRD data provides mineralogical information complementary to the *in situ* chemical data provided by alpha particle X-ray spectroscopy (APXS) [4], including textural indications of shock metamorphism.

Current Methods: Example *in situ* XRD data were collected with the Bruker D8 Discover diffractometer at The University of Western Ontario, operating with Cu K α radiation ($\lambda = 1.5418 \text{ \AA}$) at 40 kV and 40 mA (Fig. 1). The two-dimensional (2D) General Area Detector Diffraction System (GADDS) is similar to film-based methods, enabling detection of textural features such as crystallite size, alignment, and strain-related mosaicity. Unstrained single crystals give X-ray diffraction spots, whereas randomly-oriented microcrystallites give complete and homogeneous Debye rings. Shock-related inhomogeneous strain is readily identified by partial diffraction lines, or streaks [3, 5].

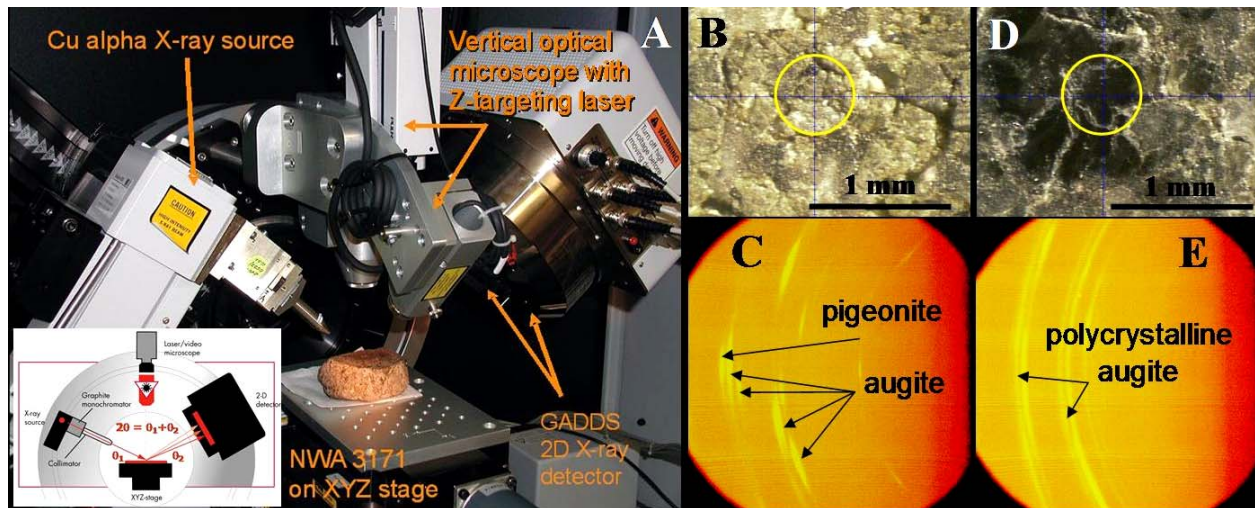


Figure 1: A) Shergottite NWA 3171 undergoing analysis on the Bruker D8 Discover diffractometer at Western (Inset: schematic image showing Theta-Theta geometry); B-E) Paired context video image and GADDS 2D X-ray image of 500 μm locations (circles) on a cut surface of NWA 3171, showing (B,C) two pyroxenes with shock-related strain, and (D,E) a melt pocket exhibiting augite with polycrystalline texture.

Discussion: Two examples demonstrate the versatility of *in situ* XRD for investigating cut surfaces of meteorites having large crystals and/or naturally polycrystalline materials. Studies of irregular surfaces are also possible [3] but here for analogy we assume a Rock Abrasion Tool-like capability.

Cut surface of martian basalt NWA 3171. Examination by μ XRD of a cut surface similar to those produced by the Rock Abrasion Tool on the Mars Exploration Rovers [6] confirms the major crystalline com-

ponents in NWA 3171 to be the pyroxenes augite and pigeonite. Maskelynite is amorphous and therefore absent from the XRD analysis, but the pyroxenes exhibit streaking, showing evidence of shock (Fig 1c). Glassy areas in NWA 3171 showed polycrystalline rings of augite only (Fig 1e).

Weathering of Diogenite NWA 2219 – Analogue for phase identification on Mars. A dry wiresaw cut through diogenite NWA 2219 exposed a weathered sulfide, surrounded by a 3 mm patch of a white pow-

derly mineral (Fig 2) which was identified by 50 μm *in situ* XRD as the Fe-sulphate szomolnokite ($\text{FeSO}_4 \cdot \text{H}_2\text{O}$) (spot 1), mantled by a yellow goethite and a dark rind of goethite and natrojarosite (spot 2).

Additional information available from *in situ* XRD: For coarse crystalline materials, data can be collected using the omega scan method [3]. For polycrystalline samples, crystal structure refinement and modal mineral analysis can be done by the Rietveld method [7].

Challenges Facing Data Acquisition on the Moon, Mars and Asteroids: Challenges for flight are restricted mass, volume, input power and robustness to launch and landing vibrations. The target mass of the XRD instrument would be a few kg, with a ~ 2 W power requirement. Additional challenges for the

Moon, Mars and asteroids include temperature extremes and abrasive dust.

Existing Lander-Based XRD: Plans to send XRD instruments to study the planets and moons have long been discussed. CHEMIN [7], a miniaturized, CCD-based, simultaneous X-ray diffraction/X-ray fluorescence (XRD/XRF) instrument is currently scheduled to go to Mars as part of the instrument payload for Mars Science Laboratory (MSL). This instrument utilizes powder X-ray diffraction, requiring sample collection and pulverization prior to analysis. Usable diffraction data have been obtained from the prototype instrument in a few minutes, and flight-instrument data collection times of 1-2 hours are predicted. The requirement for sample pulverization, however, may limit the application of the instrument.

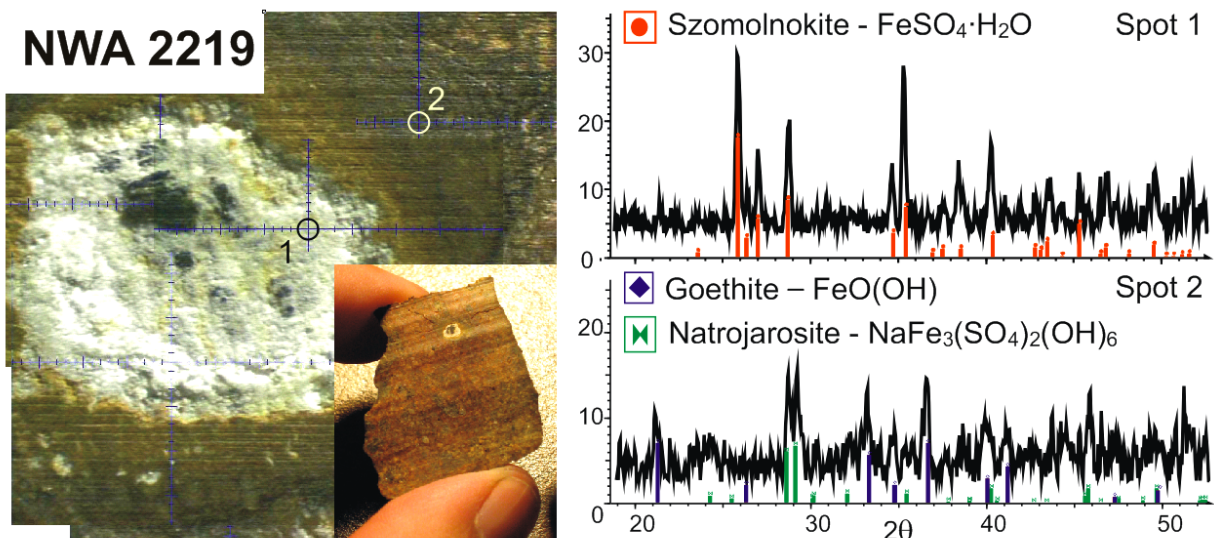


Figure 2. Diogenite NWA 2219 cut slice (inset) exhibiting a white szomolnokite powder, with a rind of goethite and natrojarosite. Data were collected using a 50 μm beam diameter (shown by circle on sample); collection time was 120 min per GADDS frame. Y axis is total counts.

Essential Modifications for *In Situ* X-Ray Diffraction on Planetary Surfaces: Many aspects of laboratory *in situ* μXRD should be retained (e.g. theta-theta geometry, laser focusing assembly, parallel beam optics) but modifications are necessary to overcome payload restrictions in power, mass and robustness as well as operational environment challenges such as temperature extremes and abrasive dust.

Small size and mass. The existing unit needs to be miniaturized, including reducing the sample to detector distance and replacing the 2D GADDS detector with CCD detectors.

No moving parts. Source and detector would ideally have no external moving parts as these could fail. A fixed bank of 2D detectors would provide flexibility and component redundancy, simplifying alignment and making the instrument more robust.

Low Power. Power reduction can be accomplished by longer data collection at low power or a larger beam diameter to reduce collection time. An increase in beam diameter from 500 μm to 1 mm would increase the analysed area by four times, greatly reducing collection time and making sample targeting easier.

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References: [1] Vaniman D. et al. (1998) *JGR-Planets* 103, 31477-31489 [2] Fortes A. D. et al. (2008) *LPSC XXXIX*: Abstract # 1183. [3] Flemming R. L. (2007) *Can. J. Earth Sci.* 44, 1333-1346. [4] Gellert R. et al. (2004) *Science* 305, 829-832. [5] Hörz F. and Quaide W. L. (1973) *The Moon* 6, 45-82. [6] Clark, B.C. et al. (2005) *EPSL* 240: 73-94. [7] Ning G. and Flemming R. L. (2005) *J. Appl. Cryst.* 38, 757-759. .