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The CheMin X-ray Diffractometer: Results from Mars and Prospects for Next Generation XRD Instruments

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Abstract

The CheMin X-ray Diffractometer (XRD) on the Mars Science Laboratory rover *Curiosity* provided the first definitive and quantitative mineralogy of the Mars surface. In its 5+ year deployment, data from CheMin revolutionized our understanding of surface processes and environments on early Mars.

CheMin follow-on instruments are being designed with improved diffraction and fluorescence capabilities and relaxed sample preparation requirements for landed missions to Mars, Earth's moon and other solar system objects.

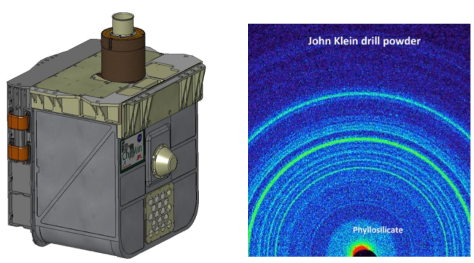

Figure 1: The MSL CheMin instrument (left) and 2D XRD data (right). Resolution is ~0.3° 2θ.

1. The CheMin Instrument

X-ray Diffraction is the reference analytical method for mineral identification and quantification in terrestrial laboratories. Data from CheMin, the first X-ray diffractometer flown in space (see Fig. 1) established the quantitative mineralogy of the Mars soil [1], characterized the first habitable environment on another planet [2], placed an upper limit on the concentration of CO_2 in the Hesperian atmosphere of Mars using the mineralogical composition of ancient lake sediments [3], and provide the first *in situ* evidence of the dessication and oxidation of early Mars [4]. All CheMin XRD patterns and mineral abundances derived using Rietveld refinement, as well as open access journal articles are available and downloadable from the Open Data Repository [5].

Powder XRD typically requires samples comprised of small grains (<10 um) packed in random orientations. In CheMin, piezo-vibrated sample cells cause the convective flow of loose powder, improving particle statistics and relaxing the requirement for fine-grained samples. This plus the use of a single photon counting X-ray CCD detector allow the collection of Debye-Sherrer XRD patterns in a small format instrument having no moving parts.

Analysis of scooped soils, or rock powders produced by a percussion drill is possible without any sample preparation other than sieving. Nevertheless, CheMin still relies on a complex sample collection and delivery system, limiting its deployment potential on smaller missions.

2. Relaxed Sample Preparation and Miniaturized XRD Geometries

Deployment of XRDs on smaller missions will require simplified sample preparation and decreased instrument size and complexity. It is also desirable to improve 2θ resolution to enhance the ability to resolve complex mineralogies, such as for example samples that contain mixtures of three pyroxenes.

2.1 ExtraTerrestrial Regolith Analyzer (XTRA)

XTRA has been proposed to analyze fines of as-received regolith from the Earth's moon without sample preparation [6]. Fine-grained regolith coats the surfaces of most airless bodies in the solar system, and because this fraction is comminuted from the rocky regolith it can often be used as a proxy for the surface as a whole. XTRA differs from CheMin in that it collects diffraction data in reflection geometry. Reflection geometry reduces sample preparation requirements because the sample can be infinitely thick relative to the X-ray beam, allowing bulk samples to be measured. Figure 2 shows a 3-D model and prototype of the XTRA instrument. Fine-grained regolith is dumped into a bulk holder having a Kapton™ window. The X-ray beam strikes the sample through the window at an acute angle (red line in Figure 2 (right)) and diffracted beams are detected by a CCD detector.

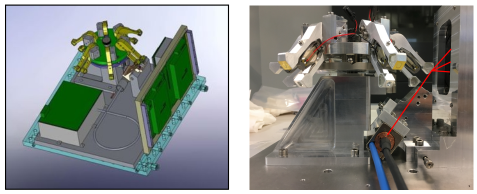

Figure 2: The XTRA reflection geometry XRD. 3-D model (left) and prototype (right) showing X-ray beam path and diffracted beams entering the detector (red lines).

2.2 Hybrid XRD

Hybrid-XRD (HXRD) is a concept under development to analyze rocks or soils without sample preparation [7]. If the material is fine-grained, a powder XRD pattern is obtained, similar to CheMin or XTRA. With coarse-grained crystals, the white bremsstrahlung radiation of the tube is diffracted into single crystal Laue patterns. Unlike typical Laue applications, HXRD analyzes the energy of each Laue spot, enabling the measurement of single crystal Bragg diffractions. Dedicated crystallographic software has been developed for identification of minerals responsible for the Laue patterns.

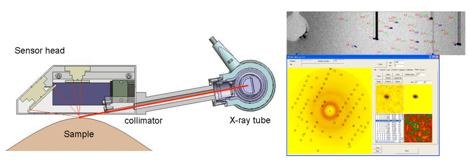

Figure 3. Arm-deployed HXRD concept (left), example data from olivine (upper right) and data reduction software (lower right).

2.3 Guinier XRD

An XRD based on Guinier geometry is under development to provide a compact high-resolution instrument. This design uses a parafocusing geometry with a curved 2D detector to cover the angular range of interest. A substantial gain in resolution has been demonstrated with a basic proof-of-concept instrument.

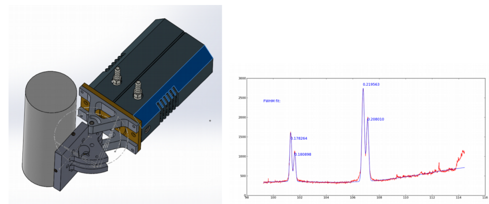

Figure 4: Guinier XRD. 3-D model with commercial camera (left) and example data (right) showing an observed resolution of 0.18° 2θ (0.13° 2θ achievable).

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