

X-ray diffraction without sample preparation

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Abstract

The author has recently invented a novel X-ray diffraction (XRD) technique which exhibits almost complete insensitivity to the sample morphology. This unique property allows XRD analysis of rocks and regolith while avoiding the need for resource-intensive and technically-challenging lander/rover sample preparation and distribution systems. The technique implements energy-dispersive XRD (EDXRD) in a back-reflection geometry. The intrinsic geometry of the method and the simplicity inherent to EDXRD enables a compact lightweight instrument design with no moving parts, suitable for deployment on a robotic arm. Details of the concept are presented here, along with a method to uncover diffraction peaks which are otherwise obscured by fluorescence peaks.

1. Introduction

XRD methods are usually applied in an angle-dispersive mode, whereby an X-ray beam with a dominant single wavelength λ is diffracted through a range of distinct 2θ scattering angles according to the Bragg equation:

$$\lambda = 2d \sin \theta \quad (1)$$

The set of crystal d -spacings, uniquely characteristic of each mineral phase, is routinely used for phase identification, quantification and structural analysis. An alternative implementation of the Bragg equation is to fix the scattering angle and use a broadband X-ray source, such as an X-ray tube, together with an energy-resolving detector. The advantages and drawbacks of EDXRD are described in [1].

For most laboratory XRD techniques, the sample must be crushed to a fine powder and presented to the instrument with a uniformly flat surface and with sub-millimetre position accuracy. In the context of XRD for planetary missions, the sample preparation requirements place a heavy burden on the limited mass and power budgets of the rover or lander, and necessitates expensive development work. The

author has recently introduced a new XRD technique with unique insensitivity to the morphology of the sample and also to the distance of the sample from the instrument, other than an intensity dependence. Specifically, EDXRD is applied in a back-reflection geometry, with $2\theta \approx 180^\circ$, described in detail in [1].

CheMin [2], a transmission XRD/XRF (X-ray fluorescence) instrument, was launched in November 2011 on NASA's Mars Science Laboratory, and is the very first planetary XRD instrument on a space mission. Mars-XRD [3] is a reflection XRD/XRF instrument employing an ^{55}Fe radioisotope source, and is included in the payload of ESA's ExoMars mission (launch 2018). Each is reliant on rover systems for sample preparation. The technique presented here is suitable for a wide range of landed planetary missions including targets such as the Moon, Mars, asteroids and even the icy moons of the outer planets. For example, on a modest-sized rover or lander lacking any sample preparation capabilities, an arm-mounted back-reflection XRD instrument could be one of the primary analysis instruments for determining the composition of surface rocks and soils. In this configuration, the ultimate instrument mass is envisaged to be below 1 kg. On a larger rover, the same instrument could perform primarily as a screening tool for selecting the most interesting samples for analysis by a series of more sophisticated instruments mounted within the rover body. A more advanced version of a back-reflection instrument could also be used within the rover body, placing virtually no constraints on the form of the processed sample.

2. Proof-of-Principle Experiments

Using a highly non-optimized set-up on an existing facility some proof-of-principle experiments were conducted. Fig. 1 shows the results using a pressed-powder pellet of quartz, mounted at two different distances from the detector, and also tilted by 45° . The high degree of consistency between the spectra demonstrates the claimed insensitivity to sample distance and, in this case, orientation. In Fig. 2, the spectra of a calcite (CaCO_3) pressed-powder pellet

and a limestone whole rock specimen are compared. The limestone consists primarily of calcite and was chosen because of its simple mineralogical composition but nevertheless clearly establishes the feasibility of analysing unprocessed rock samples.

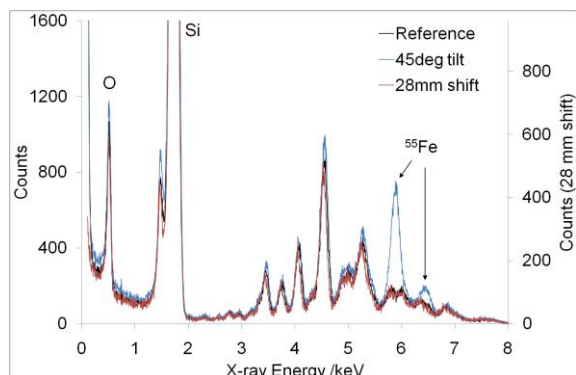


Figure 1: Spectra of a quartz sample in three different positions. The Si and O fluorescence peaks are labelled, as are two peaks due to an ^{55}Fe calibration source inadvertently exposed when the sample was tilted. All other peaks are due to quartz diffraction.

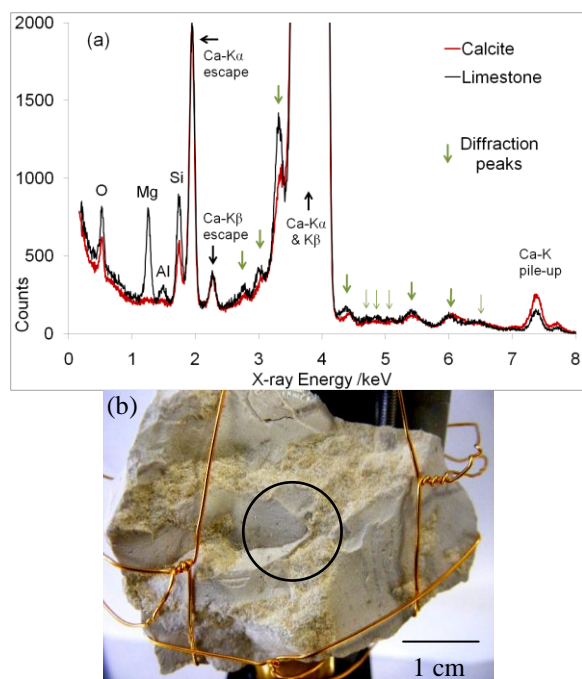


Figure 2: (a) Spectra of calcite and a whole rock limestone sample. (b) Photograph of the limestone rock. The black circle indicates the approximate size and position of the X-ray excitation beam.

3. Suppression of Fluorescence Peaks

The spectra in Figs 1 and 2 illustrate the overlap which can occur between diffraction and fluorescence peaks. There are methods available which can selectively suppress fluorescence peaks, for example by the use of filters. Suppressing fluorescence reveals underlying diffraction peaks which are often less intense by an order of magnitude or more. To demonstrate the efficacy of this method, some simulations were performed using the Monte Carlo ray-tracing program PoDFluX [4]. The results are shown in Fig. 3, in which the very strong Ca-K α and K β fluorescence peaks are suppressed, revealing a diffraction peak which is otherwise hidden.

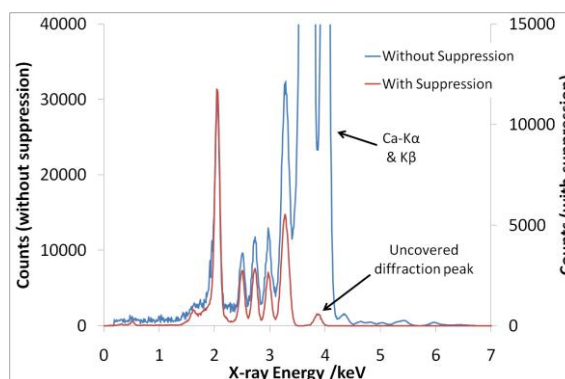


Figure 3: Simulated spectra of a calcite sample with and without suppression of the Ca-K fluorescence.

References

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