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A novel X-ray diffraction technique with almost complete insensitivity to sample morphology

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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, and also to the sample distance other than an intensity factor. The method is suitable for planetary applications, requiring no or very minimal sample preparation. 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. Details of the basic concept and of proof-of-principle experiments are presented here.

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, a unique characteristic of each mineral phase, are 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 energy-dispersive XRD are described in Ref. 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. Some methods, such as parallel beam XRD [2], have somewhat relaxed constraints on the form of the sample, but at the expense of additional complexity and mass. 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 the distance of the sample from the instrument (other than an intensity dependence). Specifically, EDXRD is applied in a back-reflection geometry, i.e. with $2\theta \approx 180°$, described in detail in Ref. 1. In the proof-of-principle experiments presented below, it is shown that XRD analysis of unprepared rock samples is feasible.

No XRD instrument has yet flown on a space mission. CheMin [3] is transmission XRD/XRF (X-ray fluorescence) instrument due for launch in late 2011 on NASA's Mars Science Laboratory, while Mars-XRD [4], a reflection XRD/XRF instrument, is part of ESA/NASA's joint ExoMars mission (launch 2018). Each is reliant on rover systems for sample preparation. The technique presented here, referred to as back-reflection energy-dispersive XRD (BR-EDXRD), is suitable for landed planetary missions with no sample preparation system. The ultimate instrument mass is envisaged to be as low as 0.5 kg.

2. Properties of BR-EDXRD

It is straightforward to show [1] that the working energy range of BR-EDXRD is approximately 0.5 – 6 keV, that the density of diffraction lines goes as the X-ray energy squared, and is greatest in the back-reflection geometry. Fluorescence lines of the common geologically-important elements mostly lie in the same energy range, such that overlaps are inevitable for many samples. The successful implementation of the method ultimately relies on overcoming these drawbacks as far as possible.

The geometry of the technique is illustrated in Fig. 1. Remarkably, there are no inherent constraints on the primary beam divergence because any diffracted X-rays reaching the detector must have scattering angles close to $2\theta = 180°$, as long as the sample distance is significantly larger than the outer detector radius. The source can, in principle, emit over 2π sr

without adversely affecting peak broadening. In practice, mutual accommodation of the source and detector, and constraints on the sample illumination area, are the limiting factors. In realistic geometries a relatively large proportion of the X-ray tube output can be utilized, enabling acquisition times as short as one minute. The detector-to-sample distance can be as small as 20 mm [1].

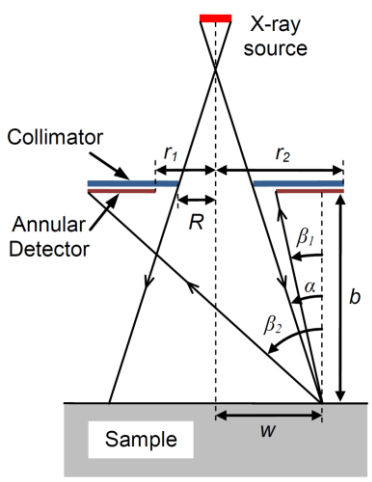

Figure 1: A schematic diagram of the essential components of a BR-EDXRD instrument.

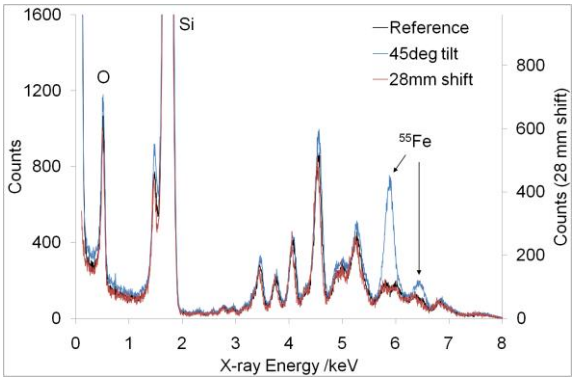

Figure 2: Spectra of a quartz sample in three different positions. The Si and O fluorescence peaks are labelled, as are two peaks due to an ^{55}Fe calibration source inadvertently exposed when the sample was tilted. All other peaks are due to quartz diffraction.

3. Proof-of-Principle Experiments

Using a highly non-optimized set-up on an existing facility (for example, the source-to-sample distance was 400 mm), some proof-of-principle experiments were conducted. Fig. 2 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. 3, the BR-EDXRD spectra of a calcite ($CaCO_3$) pressed-powder pellet and a limestone whole rock specimen are compared. The limestone consists primarily of calcite. This rock was chosen because of its simple mineralogical composition but nevertheless clearly establishes the feasibility of analysing unprocessed rock samples.

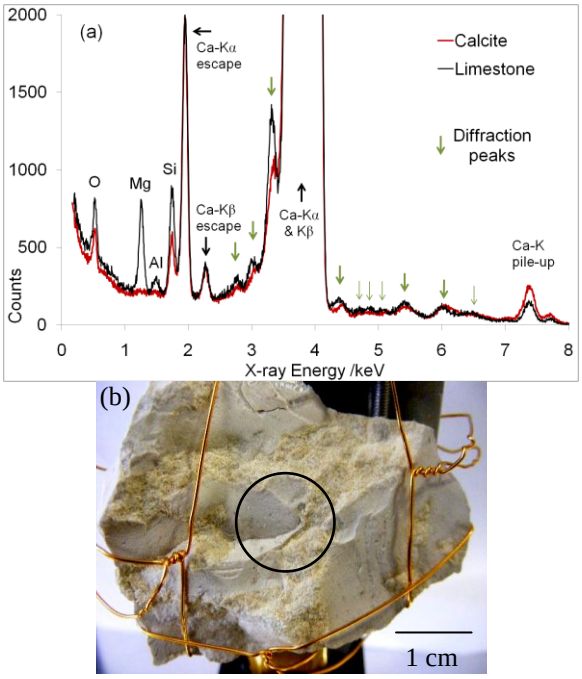

Figure 3: (a) Spectra of calcite and a whole rock limestone sample. (b) Photograph of the limestone rock. The black circle indicates the approximate size and position of the X-ray excitation beam.

References

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