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X-ray diffraction with novel geometry

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ABSTRACT

An innovative geometry for high efficiency harvesting of diffracted X-rays is explored. Further to previous work where planar samples were fixed normal to the primary axis, this work extends focal construct geometry (FCG), to samples randomly oriented with respect to the incident beam. The effect of independent sample rotation around two axes upon the scattering distributions was investigated in analytical, simulation and empirical manners. It was found that, although the profile of Bragg maxima were modified when the sample was rotated, high intensity diffraction data was still acquired. Modelling produced a good match to the empirical data and it was shown that the distortions caused by sample rotation were not severe and predictable even when sample rotations were large. The implications for this are discussed.

1. Introduction

X-ray diffraction (XRD) is a versatile analytical technique with practical applications in a broad range of scientific fields. The fundamental importance of powder XRD (PXRD) lies within its ability to characterise and identify a great variety of substances, typically polycrystalline, by obtaining structural, chemical and physical information. Even though the principles of PXRD have long been established [1–4] and applied in different disciplines [5–9], the early designs of diffractometers lacked good data fidelity and had low acquisition times mainly due to focusing deficiency [10]. A number of geometric arrangements for angular dispersive (AD) powder diffractometers have been developed since then [11–17].

1.1. Conventional geometries

Modern laboratories rely on automated diffractometers of varying arrangements mostly based on reflection mode XRD to collect scattering signatures for material analysis. In most reflection arrangements, the sample is typically a flat plate whereas in transmission arrangement the specimen is traditionally placed in a glass capillary or is in the form of a thin foil [18]. The most common reflection arrangement is the parafocusing Bragg–Brentano θ:2θ geometry with a fixed line source and an angular motion of the specimen (θ) and receiving slit (2θ) [10]. The divergence of the line source onto the sample is set by aligning the tube at a take-off angle ($\sim 6^\circ$) and controlled by a divergence slit between the sample and X-ray tube. A filter or monochromator is often employed to monochromitise the polychromatic radiation [10,18]. The radial distance between the X-ray source and the specimen is fixed and equal to the radial distance between the specimen and the receiving slit. The parafocusing arrangement of Bragg–Brentano offers high-resolution acquisition data.

In transmission mode, (Debye–Scherrer geometry), a diverging pencil beam illuminates the sample. Diffraction maxima are acquired through a 2θ rotational detector or recoding media wrapped around the sample. Modern transmission diffractometers employ a primary focusing monochromator in the form of a curved perfect crystal such as germanium [18]. Transmission arrangements are preferred for samples with low absorption and samples that are required to be sealed in glass capillaries.

1.2. Primary beam profile

The outline profile of the primary beam plays a significant role in the acquisition of high quality diffraction data. The condition of the primary beam as determined by the primary beam optics is most frequently a pencil beam (albeit diverging) in the case of transmission geometries or a fan/line beam in the case of focussing diffractometers. These primary beam forms are ubiquitous and have not been significantly modified since X-ray diffraction was first discovered. We have recently been examining the use of alternative incident beam geometries in order to maximise

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diffracted intensities without the use of relatively high cost integrating area detectors.

1.3. Focal construct geometry

An innovative beam geometry for PXRD, termed focal construct geometry (FCG) was introduced by Evans & Rogers [19] with advantageous high intensity data. FCG is based on a conventional transmission arrangement but with a novel hollow tube beam geometry. The incident beam annular collimation optic results in a post sample, pseudo-focusing of the Debye cones thus producing high intensity diffraction maxima at single locations on the primary axis (Fig. 1). These locations are referred to as condensation foci and they correspond to the Bragg maxima in conventional XRD experiments.

The intensity of the condensation foci is approximately equal to $8R_s/W_T$ when compared to a conventional pencil beam arrangement of diameter W_T [20]. In typical XRD experiments, this translates to an enhanced intensity of ~120 times. The condensation foci can be detected by translating a detector along the primary axis and their positions are used to simply calculate the corresponding 2θ using Eq. (1). Condensation rings are also formed prior and post the condensation foci due the latter's convergence and divergence, respectively. For a more detailed description of the focal construct geometry, the reader may refer to Rogers et al. [20], Evans et al. [21] and Chan et al. [22]. Several options of producing a primary beam of the desired geometry are currently being explored. These include tailored crystal optics and X-ray sources using registrable arrays.

$$2\theta = \varphi + \tan^{-1}\left(\frac{R_s}{D}\right) \tag{1}$$

This work concerns extension of the FCG approach to more generalised planar samples that have a random orientation with respect to the incident beam; rather than the special case of a sample normal to the primary axis as described above. It is also intended to show how much de-focusing is caused by the optical aberration as a result of the sample's misorientation.

2. Materials and methods

The effect random sample orientation on the focal construct geometry was investigated using analytical and simulation approaches to support an empirical study. The focal construct geometry described in Section 1.3 was utilised throughout all experiments and analysis.

2.1. Simulated data

A 3D ray tracing simulation package has been developed in-house using Matlab® to aid the optimisation of the FCG empirical system and to act as a comparative tool for experimental and simulated data. X-rays follow a path from an ideal point source through the sample volume where they are diffracted into Debye cones. Rays are emitted at a specific opening angle (φ) around the primary axis producing an annular beam of a similar beam divergence as that produced empirically with the collimation optics. A series of planar samples with the same material characteristics but different orientations relative to the primary axis, (e.g. rotations from 60° anticlockwise to 60° clockwise in steps of 20° around x and y axes), were then placed within this volume consecutively. The dimensions, position and rotation angle of the sample were specified by the user. The direction of the rays was modified by diffraction at pre-specified 2θ angles. Images are formed on a plane normal to the primary axis at any given distance from the source.

2.2. Empirical data

A conventional Zr-filtered, molybdenum X-ray tube (MoKα 0.07107 nm) was employed to produce a hollow tube beam through bespoke annular collimation optics of 17.5 mm and 18 mm inner and outer diameter respectively.

The experimental approach involved capturing standard FCG data sequences with a rotated planar sample. A 0.17 mm thick aluminium oxide (Al_2O_3) plate was employed as the sample for this set of experiments. The sample was rotated around the x-axis from 20° anticlockwise to 20° clockwise, in steps of 5° as indicated

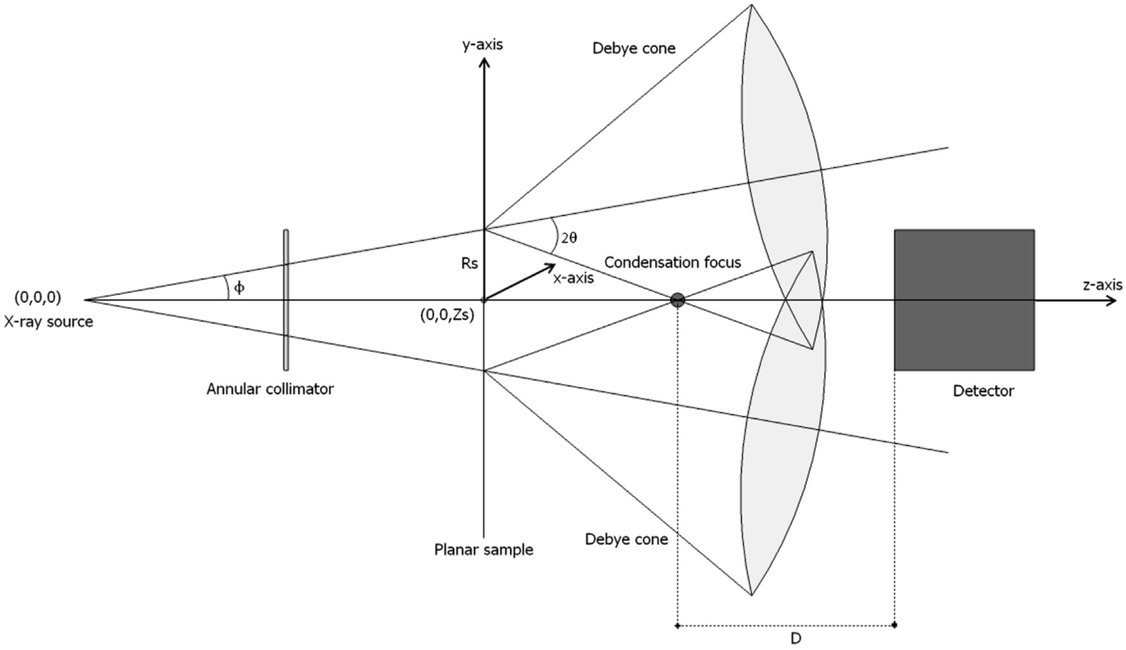

Fig. 1. A schematic diagram of the geometric arrangement of focal construct geometry including the coordinate system employed. The origin of the coordinate system is assumed to be at the X-ray source.