3.1. The optics and alignment of the divergent-beam laboratory X-ray powder diffractometer and its calibration using NIST standard reference materials

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3.1.1. Introduction

The laboratory X-ray powder diffractometer has several virtues that have made it a principal characterization device for providing critical data for a range of technical disciplines involving crystalline materials. The specimen is typically composed of small crystallites (5–30 μm), which is a form that is suitable for a wide variety of materials. A continuous set of reflections can be collected with a single scan in θ–2θ angle space. Not only can timely qualitative analyses be carried out, but with the more advanced data-analysis methods a wealth of quantitative information may be extracted. Modern commercial instruments may include features that include focusing mirror optics and the ability to change quickly between various experimental configurations. In this chapter, we discuss results from a NIST-built diffractometer with features specific to the collection of data that complement the NIST effort in standard reference materials (SRMs) for powder diffraction. While this machine can be configured with focusing optics, here we consider only those configurations that use a divergent beam in Bragg–Brentano, para-focusing geometry.

A principal advantage of the divergent-beam X-ray powder diffractometer is that a relatively large number of crystallites are illuminated, providing a strong diffraction signal from a representative portion of the sample. However, the para-focusing optics of laboratory diffractometers produce patterns that display profiles of a very complex shape. The observed 2θ position of maximum diffraction intensity does not necessarily reflect the true spacing of the lattice planes (*hkl*). While advanced data-analysis methods can be used to model the various aberrations and account for the observed profile shape and position, there are a number of instrumental effects for which there is not enough information for reliable, *a priori* modelling of the performance of the instrument. The task may be further compounded when instruments are set up incorrectly, because the resultant additional errors are convoluted into the already complex set of aberrations. Therefore, the results are often confounding, as the origin of the difficulty is problematic to discern. The preferred method for avoiding these situations is the use of SRMs to calibrate the instrument performance. We will describe the various methods with which NIST SRMs may be used to determine sources of measurement error, as well as the procedures that can be used to properly calibrate the laboratory X-ray powder diffraction (XRPD) instrument.

The software discussed throughout this manuscript will include commercial as well as public-domain programs, some of which were used for the certification of NIST SRMs. In addition to the NIST disclaimer concerning the use of commercially available resources,[1] we emphasize that some of the software presented here was also developed to a certain extent through longstanding collaborative relationships between the first author and the respective developers of the codes. The codes that will be discussed include: *GSAS* (Larson & Von Dreele, 2004), the PANalytical software *HighScore Plus* (Degen *et al.*, 2014), the Bruker codes *TOPAS* (version 4.2) (Bruker AXS, 2014) and *DIFFRAC.EVA* (version 3), and the Rigaku code *PDXL 2* (version 2.2) (Rigaku, 2014). The fundamental-parameters approach (FPA; Cheary & Coelho, 1992) for modelling X-ray powder diffraction line profiles, as implemented in *TOPAS*, has been used since the late 1990s for the certification of NIST SRMs. To examine the efficacy of the FPA models, as well as their implementation in *TOPAS*, we have developed a Python-based code, the NIST Fundamental Parameters Approach Python Code (*FPAPC*), that replicates the FPA method in the computation of X-ray powder diffraction line profiles (Mendenhall *et al.*, 2015). This FPA capability is to be incorporated into *GSASII* (Toby & Von Dreele, 2013).

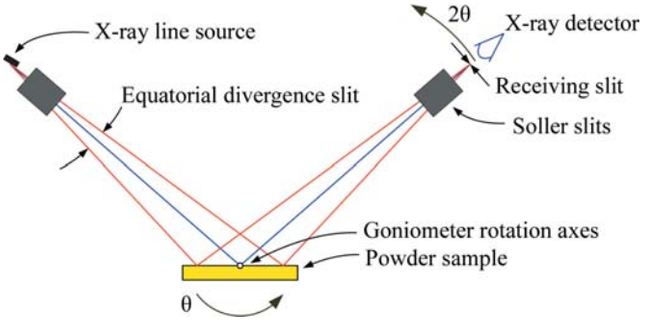

Figure 3.1.1
A schematic diagram illustrating the operation and optical components of a Bragg–Brentano X-ray powder diffractometer.

3.1.2. The instrument profile function

The instrument profile function (IPF) describes the profile shape and displacement as a function of 2θ that is the intrinsic instrumental response imparted to any data collected with that specific instrument. It is a function of the radiation used, the instrument geometry and configuration, slit sizes *etc*.[2] The basic optical layout of a divergent-beam X-ray powder diffractometer of Bragg–Brentano, para-focusing geometry using a tube anode in a line-source configuration is illustrated in Fig. 3.1.1. This figure shows the various optical components in the plane of diffraction, or equatorial plane. The dimensions of the optical components shown in Fig. 3.1.1 and the dimensions of the goniometer itself determine the resolution of the diffractometer. The divergent nature of the X-ray beam will increase the number of crystallites giving rise to the diffraction signal; the incident-beam slit defines an angular range within which crystallites will be oriented such that their diffraction is registered. One of the manifestations of this geometry is that knowledge of both the diffraction angle and

[1] Certain commercial equipment, instruments, or materials are identified in this in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

[2] See Chapters 3.6 and 5.1 for details of contributions to the profile shape from the sample.

references