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High-resolution neutron powder diffractometer SPODI at research reactor FRM II

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ABSTRACT

SPODI is a high-resolution thermal neutron diffractometer at the research reactor Heinz Maier-Leibnitz (FRM II) especially dedicated to structural studies of complex systems. Unique features like a very large monochromator take-off angle of 155° and a 5 m monochromator–sample distance in its standard configuration achieve both high-resolution and a good profile shape for a broad scattering angle range. Two dimensional data are collected by an array of 80 vertical position sensitive ^{3}He detectors. SPODI is well suited for studies of complex structural and magnetic order and disorder phenomena at non-ambient conditions. In addition to standard sample environment facilities (cryostats, furnaces, magnet) specific devices (rotatable load frame, cell for electric fields, multichannel potentiostat) were developed. Thus the characterisation of functional materials at in-operando conditions can be achieved. In this contribution the details of the design and present performance of the instrument are reported along with its specifications. A new concept for data reduction using a 2θ dependent variable height for the intensity integration along the Debye–Scherrer lines is introduced.

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1. Introduction

The high-resolution powder diffractometer SPODI (Structure Powder Diffractometer) has been built and operated by Technische Universität Darmstadt in cooperation with Ludwig-Maximilians Universität München and Technische Universität München at the research reactor Heinz Maier-Leibnitz (FRM II). During the last years the instrument underwent various instrumental upgrades and improvements of components, software and sample environment. This contribution resumes details of its current setup and present performance and lists some specifications of the instrument. More details about the design of the instrument, based on Monte-Carlo simulations by McStas software, can be found in Refs. [1,2].

1.1. Instrument layout, collimation and monochromator

The powder diffractometer SPODI has been designed to achieve both fine resolution and good profile shape [3]. In its standard configuration (highest resolution mode) SPODI uses a unique very high monochromator take-off angle of 155° along with a large monochromator-to-sample distance of 5 m. An evacuated beam tube of about 4 m in length is located between the monochromator and the sample, which controls both vertical and horizontal neutron beam divergences at the sample position. Thus the natural neutron beam divergence in the horizontal plane is 25′ only. It can be reduced down to 5′ by optional Soller collimators in front of the sample.

Complementary options for higher intensities exist at 135°, 112.5° and 90° take-off angles. An optional secondary collimation system is available for monochromator–sample distances of 2.8 m, typically installed at a monochromator take-off angle of 135°. In such a "shorter" configuration, the neutron beam divergences in both horizontal and vertical planes are determined by the slit systems. These are under remote control and allow the automatic adjustment of beam dimensions in accordance with sample size and sample environment. In this setup, the natural horizontal beam divergence at the sample position is higher (~40′) and can be reduced to 20′ or 10′ by the use of a Soller collimator changer.

The vertically focusing monochromator of 200 mm height consists of a stack of 15 germanium wafer crystals with (551) planes parallel to the surface. The crystals exhibit mosaicities of 20′ in the horizontal direction and 11′ in the vertical one. Different reflections with indices hhk can be selected by a rotation in the horizontal plane (ω-monochromator axis). For example in the standard configuration (monochromator take-off

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angle of 155°) the germanium monochromator yields wavelengths of 1.548 Å, 2.536 Å and 1.111 Å corresponding to reflections Ge(551), Ge(331) and Ge(771), respectively (Table 1). A weak $\lambda/3$ contamination persists for a neutron beam from Ge(331), but is eliminated by a tunable pyrolytic graphite filter. The monochromator crystals are mounted on a vertical focusing unit (Risoe laboratory) allowing a continuous change of the focusing distance from 1.2 (maximum bending) to ∞ (flat).

The monochromator is located at a distance of about 17.5 m from the reactor core at the thermal beam tube 8a of the FRM II reactor. A neutron guide of 14.5 m length (including a 2 m in-pile guide) is installed in front of the monochromator. Its height of 100 mm at the side of reactor vessel smoothly increases to 200 mm before the monochromator. The supermirror coatings correspond to $m_{\mathrm{horizontal}}=2$ and $m_{\mathrm{vertical}}=3$.

Examples of resolution curves for SPODI using Ge(551) orientation for the high-resolution setup and the higher flux option are shown in Fig. 1.

Table 1
List of available wavelengths at SPODI delivered by germanium stack monochromator.

Reflection	Take-off 155°, 5 m distance	Take-off 135°, 2.8 m distance
Ge(331)	2.536	2.396
Ge(551)	1.549	1.463
Ge(771)	1.111	1.050

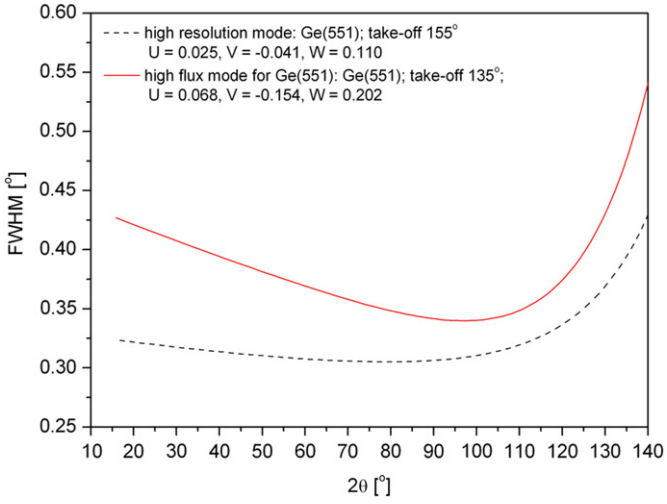

Fig. 1. Resolution function for germanium(551) orientation at the high-resolution setup (monochromator-take-off angle 155°; monochromator-to-sample distance 5 m) and the higher flux option (monochromator-take-off angle 135°; monochromator-to-sample distance 2.8 m), respectively.

2. Detector array

An angular range of 160° is covered by a multidetector consisting of 80 ^{3}He detector tubes with fixed Soller collimators (300 mm height) of 10′ horizontal divergence located in front of each detector. The ^{3}He tubes are position sensitive in the vertical direction with a resolution of about 3 mm and allow the collection of two-dimensional diffraction data. An example is shown in Fig. 2. Similar multidetector systems have also been installed at two other high-resolution powder diffractometers, namely D2B (ILL) [4] and ECHIDNA (ANSTO) [5]. Such a multidetector concept requires a data collection by stepwise positioning of the detector array, e.g. a typical stepwidth of $\Delta(2\theta)=0.05°$ corresponds to 160°/[80 ^{3}He detectors $\times$ $\Delta(2\theta)$]=40 individual steps for a collection of a full diffraction pattern in the angular range of 0–160° 2θ. This concept of a moving multidetector/multicollimator assures a good resolution over a very broad scattering angle regime. Furthermore, the resolution does not depend on the diameter of the sample. On the other hand, the measuring times are longer compared to static detectors and therefore kinetic measurements on a short time scale are not feasible.

3. Data handling

The two-dimensional diffraction data are advantageous to check sample crystallinity, alignment and possible preferred orientation effects. Moreover, high-resolution diffraction data can be derived by integration along the Debye–Scherrer rings. Prior to this stage a set of instrumental corrections is performed. In particular efficiency/intensity corrections as well as 2θ scattering angle corrections are carried out for each detector/collimator pair. The data integration along the Debye–Scherrer rings requires an analytical solution for the section of a Debye–Scherrer cone (Bragg reflection) and a cylinder (curved multidetector). On a curved detector with radius R (1.117 m for the SPODI multidetector) the Bragg peak at $2\theta_0$ and detector height $z=0$ (centre line of the detector coinciding with the centre of the sample and the beam) is detected at $2\theta_z$ (as long as $z \le R$) given by

$$\cos(2\theta_z) = \sqrt{1+(z/R)^2}\cos(2\theta_0) \quad (1)$$

This relation is generally used to transform the intensity at any position ($2\theta_z$, z) to that at ($2\theta_0$, z) thus leading to straightened Debye–Scherrer rings. The geometrical data correction incorporates a correct Lorentz factor, as the length of the Debye–Scherrer ring section within the used detector height is taken into account, rather than just its height. (Conventionally, the Lorentz-factor is approximated by the height of the Debye–Scherrer ring section.) Thus correct intensities are derived even for very low and very high scattering angles. Full diffraction patterns can then be obtained by summation over the detector height at all positions 2θ. A one to one correspondence, leading to strictly straight lines, is,

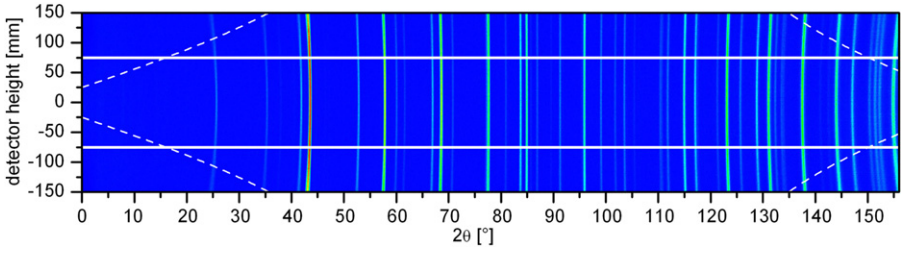

Fig. 2. Two-dimensional raw data set of a corundum reference sample. The straight white lines bound a detector height of 150 mm and the detector height 0 denotes the central line of the detector. The dotted white lines encompass the data used in the "300 mm-variable height" data set.

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