Journal of large-scale research facilities

Journal of large-scale research facilities, 1, A5 (2015) http://dx.doi.org/10.17815/jlsrf-1-24

Published: 18.08.2015

SPODI: High resolution powder diffractometer

Heinz Maier-Leibnitz Zentrum
Karlsruhe Institute of Technology
Technische Universität München

Instrument Scientists:
- Markus Hoelzel, Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München, Garching, Germany, phone: +49(0) 89 289 14314, email: markus.hoelzel@frm2.tum.de
- Anatoliy Senyshyn, Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München, Garching, Germany, phone: +49(0) 89 289 14316, email: anatoliy.senyshyn@frm2.tum.de
- Oleksandr Dolotko, Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München, Garching, Germany, phone: +49(0) 89 289 14373, email: odolotko@frm2.tum.de

Abstract: The high resolution powder diffractometer SPODI (jointly operated by the Karlsruhe Institute of Technology and the Technische Universität München) is designed for structure solution and Rietveld refinement of crystal and magnetic structural parameters on polycrystalline powders. Instrumental specification (design, flexibility, peak shape, resolution etc.) as well as a variety of specialized sample environment equipment implemented for in-situ materials characterisation make the instrument attractive for studies of complex ordering phenomena.

1 Introduction

The instrument is characterised by a very high monochromator take-off angle of 155° (standard configuration). Optionally, a take-off angle of 135° is available.

The detector array consists of 80 ^{3}He position sensitive detector tubes (300 mm active height) with fixed Soller collimators of 10' horizontal divergence. The multidetector of SPODI spans an angular range of 2θ = 160°. Each detector covers 2° corresponding to 160°/ 80 detectors. Therefore the data collection is performed via stepwise positioning of the detector array to obtain a diffraction pattern of the desired step width (typically 2°/ 40 steps resulting in $\Delta(2\theta)$ = 0.05°).

The two-dimensional raw data are evaluated to provide diffraction patterns corresponding to different detector heights ranging from 10 mm to 300 mm and variable detector height, accounting for vertical beam divergence effects. Thus, asymmetric broadenings at quite low and high scattering angles are overcome, while the full detector height in the medium 2θ regime can be used (Hoelzel et al., 2012).

CC BY

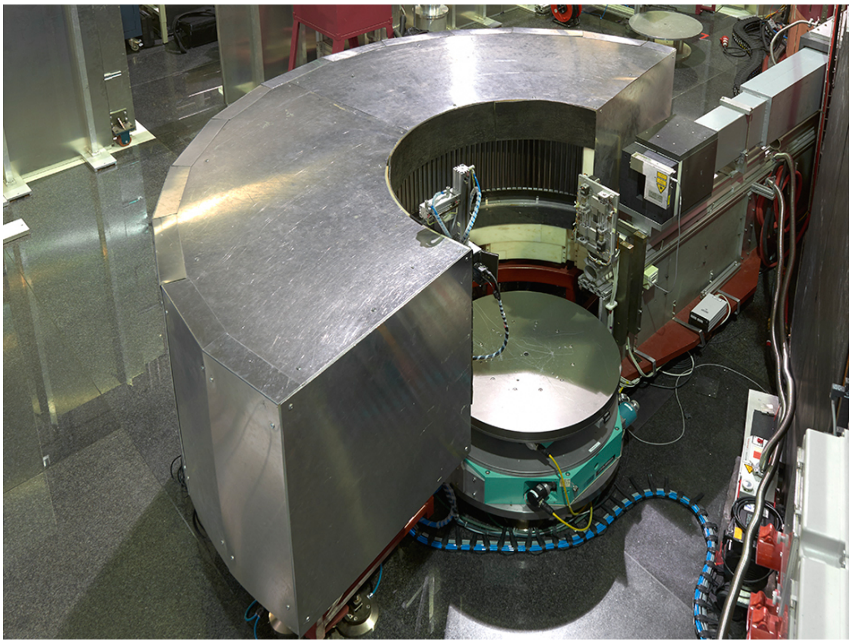

Figure 1: Instrument SPODI (Copyright by W. Schürmann, TUM).

Various sample environmental devices enable the characterisation of materials under special conditions: A rotatable tensile rig allows in-situ studies under tensile stress, compression stress or torsion while the load axis can be oriented with respect to the scattering plane. A potentiostat for charging/discharging of Lithium ion batteries is available as well as a device to apply high electric fields on ferroelectrics.

2 Typical Applications

- Determination of complex crystal and magnetic structures
- Structural evolutions and phase transformations under various environmental conditions
- Static and thermal disorder phenomena

3 Research Areas

- Ionic conductors
- Materials for lithium ion batteries
- Ferroelectrics, multiferroics
- Hydrogen storage materials
- Shape memory alloys
- Superalloys
- Correlated electron systems
- Superconductors
- Minerals

4 Sample Environment

Standard sample environment of FRM II

- Closed cycle cryostat 3 − 550 K
  (with ^{3}He insert: T_{min} = 500 mK)

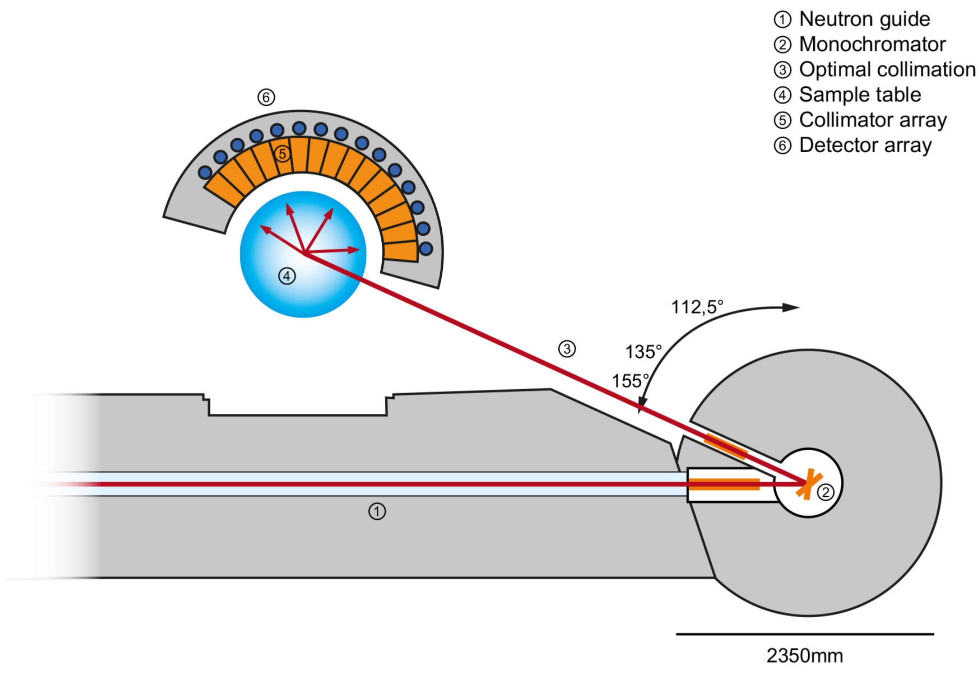

Figure 2: Schematic drawing of SPODI.

- Vacuum high temperature furnace
  T_{max} = 1900°C
- Cryomagnet
  B_{max} at SPODI: 5 T
- Sample changer (six samples, ambient temperature)

Special sample environment

- Rotatable tensile rig
  F_{max} = 50 kN, M_{max} = 100 Nm
- Device for electric fields
  V_{max} = 35 kV
- Potentiostat for electrochemical treatment of materials VMP3 and SP240

5 Technical Data

5.1 Monochromator

- Ge(551) wafer stack crystals
- standard configuration: take-off angle 155°
  Ge(551): 1.548 Å
  Ge(331): 2.436 Å
  Ge(711): 1.111 Å

5.2 Collimation

- $\alpha 1 \approx 20'$ (neutron guide)
- $\alpha 2$ = 5',10', 20', 25' nat. (for 155°)
  $\alpha 2$ = 10', 20', 40' nat. (for 135°)
- $\alpha 3$ = 10'

5.3 Detector array

- 80 position-sensitive ^{3}He tubes,
  angular range 2θ = 160°,
  effective height: 300 mm

References

Hoelzel, M., Senyshyn, A., Juenke, N., Boysen, H., Schmahl, W., & Fuess, H. (2012). High-resolution neutron powder diffractometer SPODI at research reactor FRM II. *Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment*, *667*, 32 - 37. http://dx.doi.org/10.1016/j.nima.2011.11.070