A2 - Neutron Diffraction - Instruments

A2 - O1

THE NEW D2B HIGH RESOLUTION POWDER DIFFRACTOMETER

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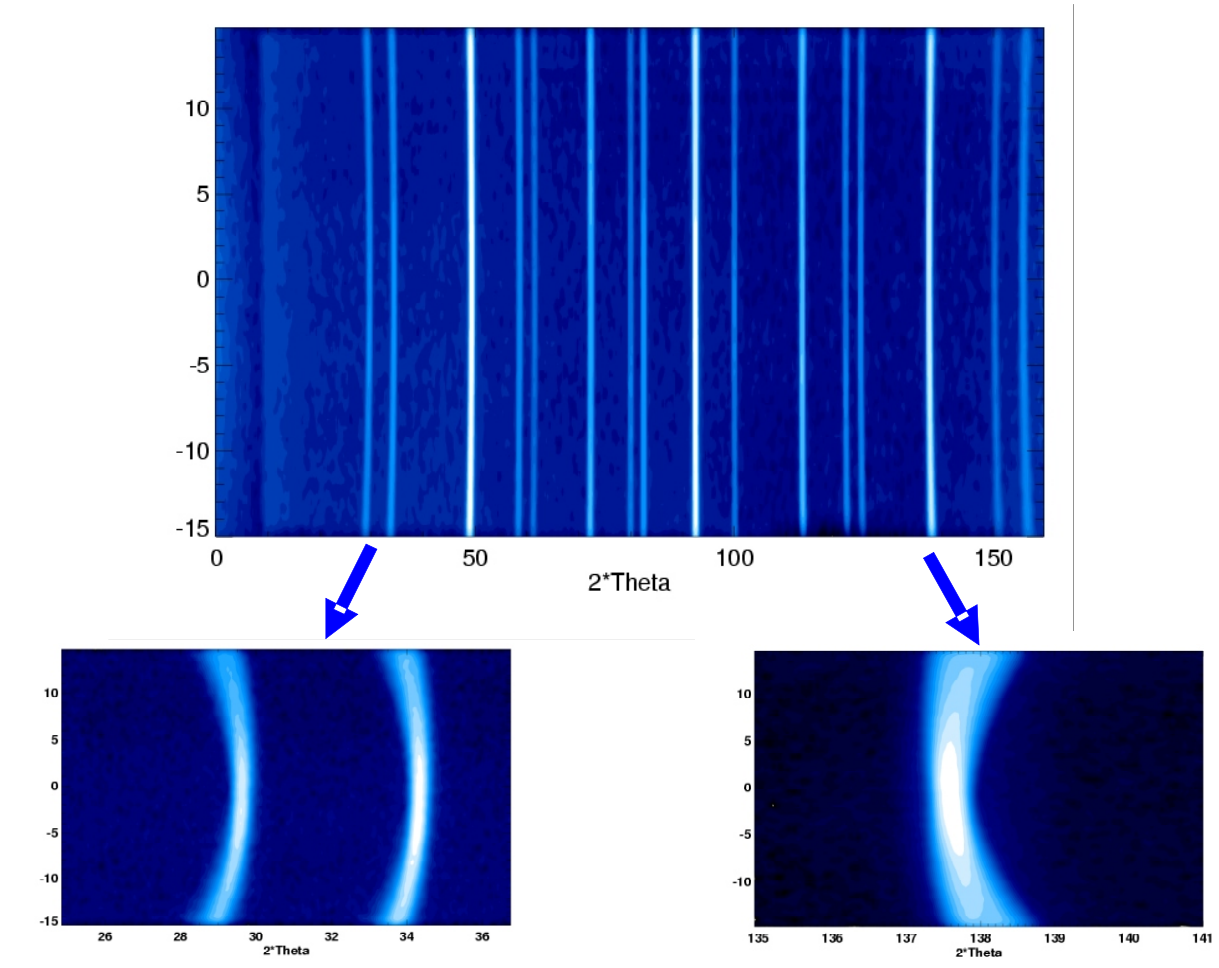
A new 2D detector was installed on D2B. This is a new type of pseudo-2D high-resolution position sensitive detector, covering 160 degrees horizontally by 14 degrees vertically. In the horizontal plane, high-resolution is obtained by scanning a bank of 128 fine 5-minute collimators. In the vertical plane lower resolution is obtained using vertical linear-wire position sensitive detectors. Since the height of the detectors is 300 mm, vertical resolution is needed to correct for the curvature of the diffraction cones. Initial problems with background and calibration were resolved, and the normal user program restarted while data reduction routines were further developed. The efficiency is about an order of magnitude greater than with the "old" detector. In the high intensity mode a pattern with good statistics can be collected in a few minutes.

A powder pattern measured on the new D2B detector, showing how the curvature of the diffraction cones depends on the scattering angle is shown below. A computer routine "straightens" and integrates these curves to produce a standard intensity vs. 2theta diffraction pattern.

A 30 mg sample of CeO2 was measured at room temperature on D2B. A special vanadium container with very thin walls was used. The data acquisition last 17 hours and the pattern obtained is perfectly refinable. Such a measurement was possible because of the high quality of the new D2B detector as well as the work done on the instrument zone to reduce the neutron background as much as possible.

We have no doubt that it should be possible to measure even smaller samples if longer counting times can be scientifically justified.



A2 - O2

FIRST TEST MEASUREMENTS AT THE NEW STRUCTURE POWDER DIFFRACTOMETER (SPODI) AT THE FRM-II

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In a collaboration of the Technische Universität Darmstadt and the Ludwig-Maximilians-Universität München, funded by german government (BMBF), we build up the new Structure Powder Diffractometer (SPODI) at the neutron source FRM-II in Garching near München (see Fig. 1). The instrument operates in the angle-dispersive mode with a previously selected wavelength. This contribution will give an overview of the status and the innovations of new components included in the concept [1, 2, 3]. Particular highlights of the set-up are for example the sophisticated supermirror neutron guides (trumpet shape) for thermal

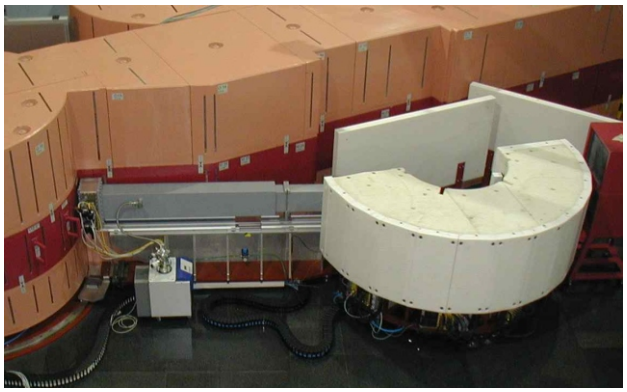

Fig. 1: Set-up of the Structure Powder DIffractometer SPODI at the FRM-II.

neutrons, the high take-off angle 155° realized with 17 slabs of an unconventional orientation (551) of the germanium monochromator (each slab consists of 37 wafers !), the 300 mm high collimators with tapered sidewalls to enlarge the effective detection area, the position sensitive detector providing 2D array detection with multifarious evaluation procedures, the space optimized shielding with different special materials (to improve the signal to background ratio) etc. The realisation of this layout aims at higher intensity, improved resolution and better profile shape. Monte Carlo simulations of the complete instrument including the sample for testing were carried out to match the components. First test measurements of single components at other neutron facilities promise many applications of SPODI.

The design and status of the new Structure POwder DIffractometer (SPODI) is reviewed. To solve complicated structures reliably by powder diffraction not only high resolution and intensity are essential, but, in particu-

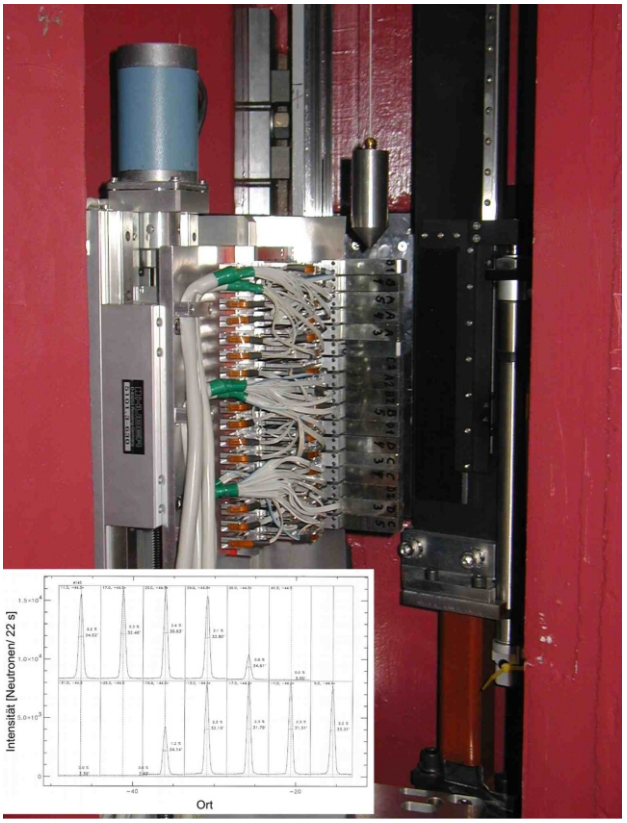

Fig. 2: Photograph of the germanium monochromator (551) installed in the monochromator drum. The inset displays rocking curves of a single monochromator slab.

lar, also good peak profiles that can be described as perfect as possible. For strongly overlapping reflections, uncertainties in the exact shape can lead to a wrong distribution of the intensities. Moreover, such knowledge virtually increases the resolution of the instrument far beyond that given by the widths of the peaks. Similar arguments hold for a good peak to background ratio which can be achieved not only by a low background, but also by narrow tails of the peaks. Finally, such good peak shapes should be maintained up to large scattering angles 2 . Based on these considerations computer simulations have been used to optimise both each single component and their interaction along the instrument. The resulting concept together with test measurements of single components at neutron sources have been described in reference [1, 2, 3,

4, 5]. The high flux at the sample position with neutrons of very low vertical divergence because of the 5m distance to the monochromator allows the unique possibility to integrate a small-angle apparatus in the whole system [6].

Currently the alignment of the single monochromator slabs is done to optimize the focusing of the beam at the sample position.

Acknowledgement
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A2 - O3

EXED – THE NEW EXTREME ENVIRONMENT DIFFRACTOMETER AT THE HAHN-MEITNER-INSTITUTE BERLIN

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The EXED instrument is a very high resolution time-of-flight powder diffractometer, which has been optimised for diffraction in extreme environments. A special focus is on high magnetic fields and thus the instrument will be equipped with a dedicated 25 T cryomagnet. The instrument is being built at the steady state reactor BERII of the Hahn-Meitner-Institut Berlin. However, its sophisticated chopper system allows the application of the time-of-flight (TOF) principle and, compared to a common crystal monochromator instrument, EXED offers a number of advantages on a continuous source: a) it can provide higher resolution, comparable to what is now achieved at synchrotron radiation sources; b) it makes small d-spacing readily accessible; c) it is more efficient in terms of neutron intensity for conventionally high resolution neutron diffraction work and d) it facilitates the use of extreme sample environment equipment by providing a full coverage of the relevant Q domain at very limited angular access in scattering angles, for instance due to the magnet geometry (see Fig. 1). The physical reason for these advantages is that at high scattering angles good resolution can be achieved without collimators.

The chopper system allows for a very flexible use of the instrument: As the repetition rate is not defined by the source, almost any d-spacing can be achieved in the forward as well as the backward scattering direction. In addition, a slewing of the chopper phases permits a continuous

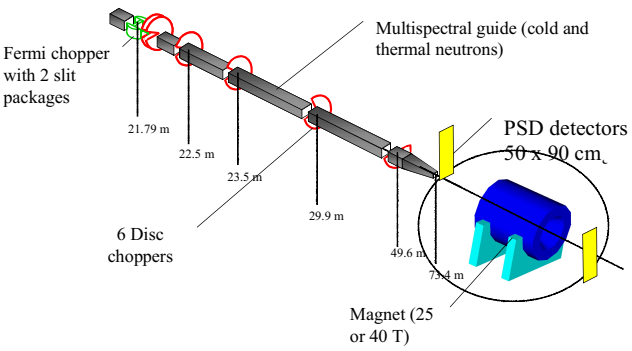

Fig. 1: Schematic view of the EXED instrument. The grey tubes correspond to a ballistic neutron guide (straight guide and the compressor [1]). The neutron pulses are produced alternatively by a Fermi chopper (in green) or by a counter rotating double disk chopper (in red), which are exchangeable. The other disk choppers (in red) are frame overlap choppers and a wavelength band chopper. The position sensitive gas detectors are presented in yellow and can be moved around the sample and the magnet.

Table 1: Various detector bank configurations. For the two first columns the symmetrical arrangement of the detector is supposed with the corresponding angular coverage of 15° on each side of the neutron beam axis, the two last columns correspond to an asymmetrical arrangement with an angular coverage of 30° on one side of the neutron beam axis.

Bank	(171 ± 7)°	(9 ± 7)°	(163 ± 15)°	(17 ± 15)°
d-spacing	0.35 - 10.1 Å	2.52 - 571 Å	0.35 - 10.4 Å	1.27 - 571 Å
Q	0.62 - 17.9 $Å^{-1}$	0.011 - 2.49 $Å^{-1}$	0.6 - 17.9 $Å^{-1}$	0.011 - 4.95$Å^{-1}$

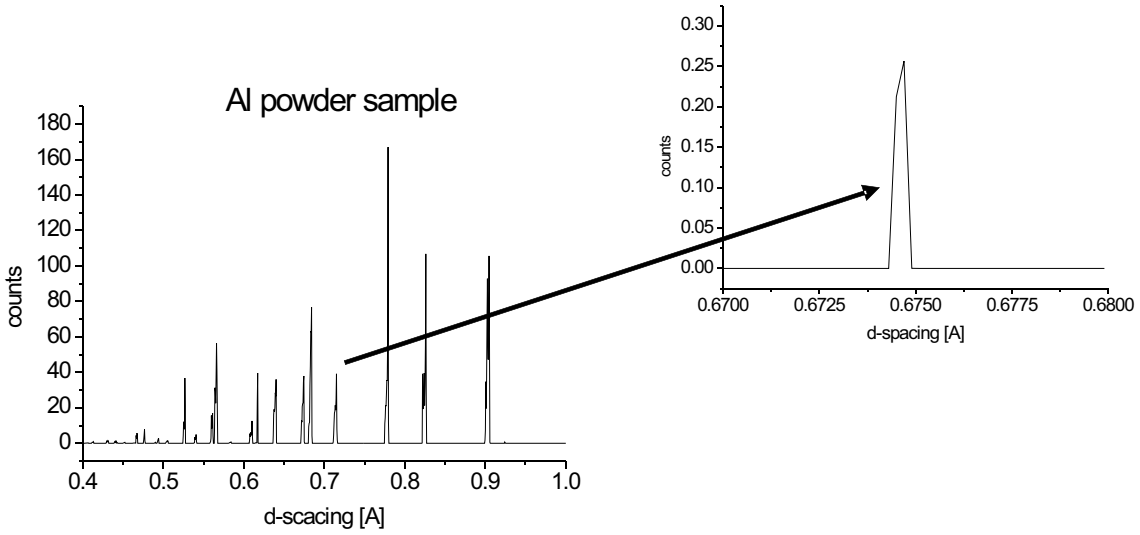

Fig. 2: Powder pattern MC simulation results for 0.7 Å < < 1.8 Å and 156 ° < 2 < 179 ° as a function of the d-spacing. At ~ 176 ° and = 1.34 Å (rhs), nearly the highest total resolution of d/d ~ 3 10^{-4} is obtained.

variation of the wavelength band. In the backward direction it can be combined with a very high resolution. The EXED beam line is unique in the sense that it has access to both the cold and the thermal moderator and takes advantage of a large wavelength band ranging from 0.7 to 20 Å with local maxima at 1.4 and 3.8 Å.

The detector banks are planned to be equipped with tubes with a diameter of 1 cm and an effective length of 90 cm, which are filled with He gas. They shall be position sensitive with a resolution of 1 cm and will cover an effective surface of 50 x 90 cm^2. It will be possible to move them around the sample position or to translate them. In the 2 m position, the two detector banks will cover a 28° scattering angle range, while in the 6 m position only 10° are covered. In the latter configuration, a movable vacuum or gas tank between the sample and the detectors will be used to reduce air scattering.

Different collimation modules can be applied to further optimise the resolution: the final interchangeable guide section either homogenises the beam whilst maintaining the divergences or further focuses it to the sample position. Or a pine hole collimator permits the fine tuning of the horizontal and vertical divergences. The d-spacings and momentum transfers Q accessible with the various detector bank configurations are listed in table 1. The instrument is designed for both narrow-bandwidth and broad-bandwidth operations, the latter achieved by repetition-rate reduction and/or chopper slewing.

First Monte Carlo simulation results show typical powder pattern diagrams for an Al sample and confirm the theoretically calculated resolution in backscattering direction of d/d ~ 3 x 10^{-4} (see fig. 2) for the wavelength range of 0.7 Å < < 1.8 Å and at scattering angles 156 ° < 2 < 179°.

Concerning the extreme environments, the most important feature is the high field steady state magnet which can create a magnetic field of up to 25 T (later on possibly 40 T). Depending on the range of scattering angle required the magnet can either be used in a symmetric or asymmetric neutron beam configuration or it can be removed from the experiment to allow for usual elastic diffraction.

Additionally to the magnet or separately further sample environment elements like cryostats creating low or high temperatures ranging from 1.5 – 700 K and pressure cells of up to 20 kbar can be employed.

In a medium-term perspective possible extensions are a small angle scattering (SANS) option with a collimator length of up to 6 m and inelastic scattering at high magnetic fields.

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A2 - O4

HIGH-RESOLUTION NEUTRON DIFFRACTION PERFORMANCE FOR STRESS/STRAIN MEASUREMENTS IN POLYCRYSTALLINE MATERIALS

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Excitation of multiple reflection effects in cylindrically bent perfect Si single-crystals which result in *Umweganregung* (Renninger effect) is in fact a simulation of forbidden basic primary reflection by a cooperative action of secondary and tertiary reflections. Schematically is this process shown on Fig. 1. Using a standard $_M$ $_D$ scan with the bent crystal in symmetric transmission geometry many strong umweg-effects could be determined as demonstrated in Fig. 2. For the present experimental studies we chose the effect observed at = 29.956° which is related to the simulation of the forbidden 222 reflection by a co-operative action of 153/1-3-1 and -31-1/513 (secondary/tertiary) reflections at = 0.156 nm. It has been already proved in the experiment with a single crystal sample depending on the crystal curvature the monochromatic beam from the umweg monochromator is highly parallel within the divergence of about (1-5) x 10^{-4} rad and has excellent resolution [1]. In the present case the monochromatic beam obtained by this multiple reflection monochromator was used in powder diffraction test carried out with a solid -Fe.polycrystalline standard sample (= 2 mm). Fig. 3 displays several diffraction profiles that clearly prove the applicability of the umweg-monochromator for high- resolution diffraction studies. *FWHM* of the diffraction profiles are however, determined by the spatial resolution of the used PSD and the width of the sample [2, 3]. Therefore, it can be considered as an upper limit.

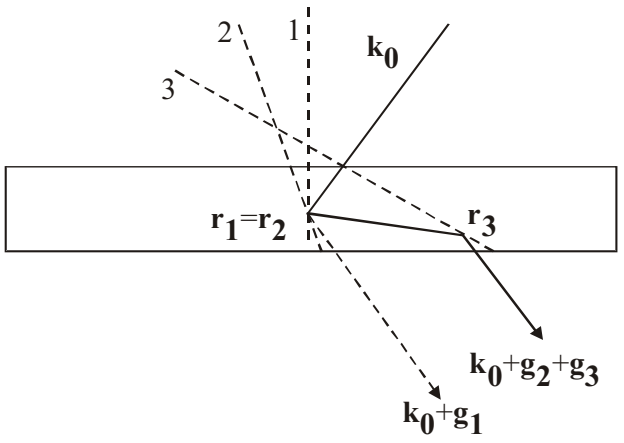

Fig. 1. Schematic sketch of a two-step multiple reflection simulating a forbidden reflection.

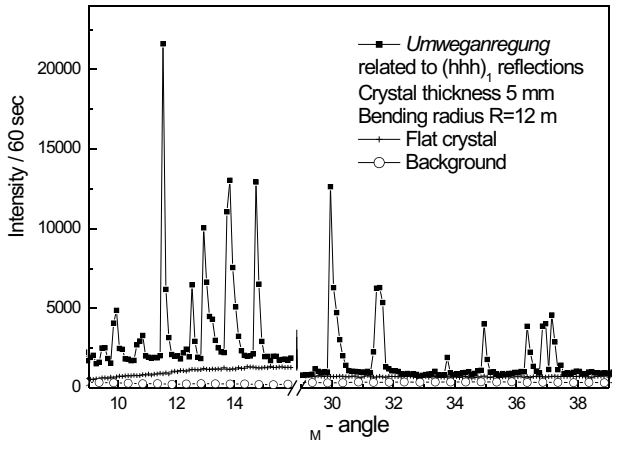

Fig. 2. A part of $_M$ $_D$ scan with the Si crystal slab (the largest face parallel to (110)) and set for $(hhh)_1$ reflections in the symmetric transmission geometry.

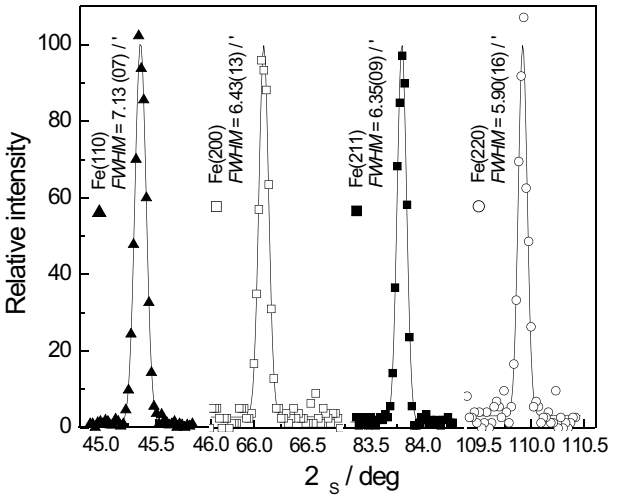

Fig. 3. Examples of the -Fe diffraction profiles taken with the umweg-monochromator.

Then, such a high-resolution diffraction performance we used for investigation of Fe-reflections in an induction hardened S45C rod (=20 mm) having different phase composition at different distances from the rod axis. For determination of gauge volume we used 2 mm input and output slits in the incident as well as diffracted beam, respectively. Fig. 4 displays the diffraction profile (black points) obtained at the distance 8 mm from the axis (2 mm

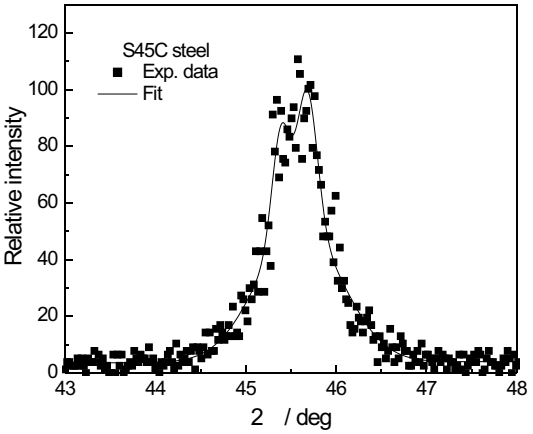

Fig. 4. Raw experimental data of a induction-hardened S45C steel taken 2 mm under the surface.

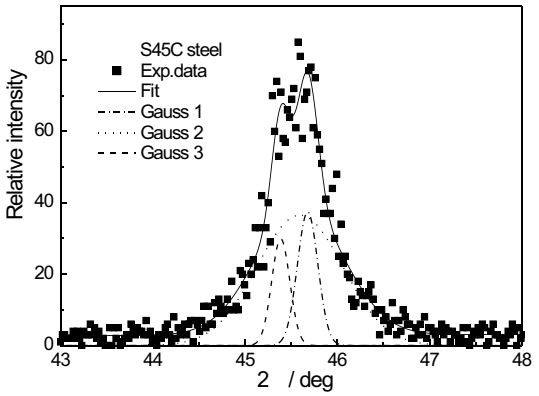

Fig. 6. Induction-hardened S45C steel diffraction profile taken at 2 mm under the surface with the fitted profiles corresponding to the perlitic, ferritic and martensitic phases

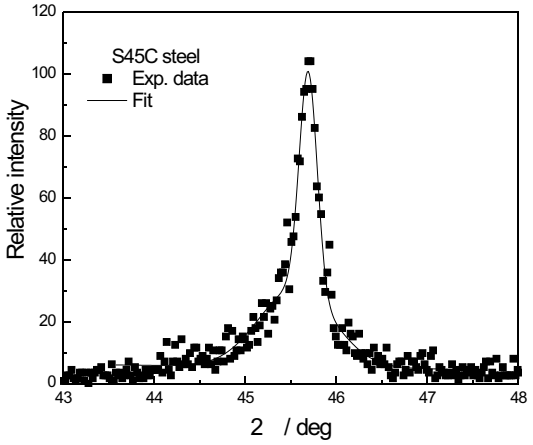

Fig. 5. Raw experimental data of a induction-hardened S45C steel taken 4 mm under the surface.

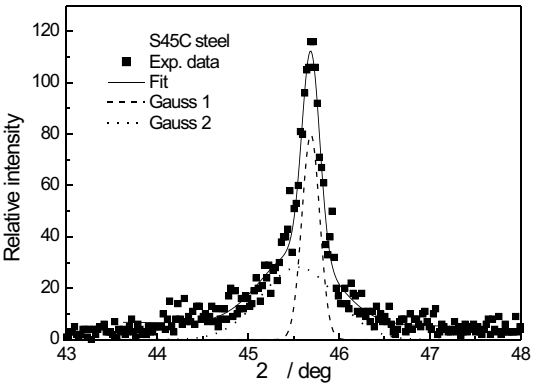

Fig. 7. Induction-hardened S45C steel diffraction profile taken at 4 mm under the surface with the fitted profiles corresponding to the ferritic and martensitic c phases.

below the surface). Similarly, Figs. 5 displays the diffraction profile (black points) obtained at the distance 6 mm from the axis (4 mm below the surface). Fitting of the experimental profiles were carried out by two (in the case of data related to Fig. 5) or three Gaussians (in the case of data related to Fig. 4) (corresponding probably to pearlitic, ferritic, and martensitic phases). The results after the fitting procedure are shown in Figs. 6 and 7. Thanks to the used high-resolution monochromatic beam, after a fitting procedure we could reliably determined contributions of the individual phases.

Diffractometers employing umweg-monochromator can provide ultra-high resolution at a rather low take-off angles. The resolution is comparable to that of back-scattering instruments. Of course, they can be efficiently used namely at high-flux neutron sources. It should be noted that in our case the resolution provided by the umweg-monochromator is higher than that presented in Fig. 3. The widths of the diffraction profile received at the standard -Fe sample are in our case determined by the spatial resolution of the position sensitive detector (1.5 mm) and the widths of input and output slits (2 mm).

Acknowledgement
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