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# FOREWORD

The international conference on neutron texture and stress analysis (NTSA) was held at Dubna (about 150 km north to Moscow) Russia from 23–26 June 1997. Idea and organisation were a joint project of the Frank Laboratory of Neutron Physics (FLNP), Dubna, Russia, the GKSS – Research Center Geesthacht GmbH, Germany, and the Technical University Clausthal, Germany. The main purpose of this conference was to bring together material scientists, geo scientists and physicists working on similar topics (texture and stress analysis of polycrystalline materials), on different materials (metals, alloys, ceramics, rocks etc.) and on identical neutron instruments but meeting on different conferences. Another point of interest was to give an overview of the Dubna instrumentation particular on the equipments for texture and strain measurements.

Neutron scattering plays an important part in the investigation of polycrystalline structures if high penetration is necessary. Among others polycrystalline structures have to be characterized by their texture (anisotrophic properties, materials history etc.) and their stresses (application profile, failure etc.). Fifty-nine participants from 11 countries had a very active competition with long intense discussions about all recent questions in texture and stress analysis (instrumentation, measurement, data evaluation and simulation). A short description of the instruments for texture and strain measurements at the FLNP, Dubna and at the GKSS – Research Center, Geesthacht is given to invite all potential candidates.

H.-G. Brokmeier

# THE SKAT TEXTURE DIFFRACTOMETER AT DUBNA

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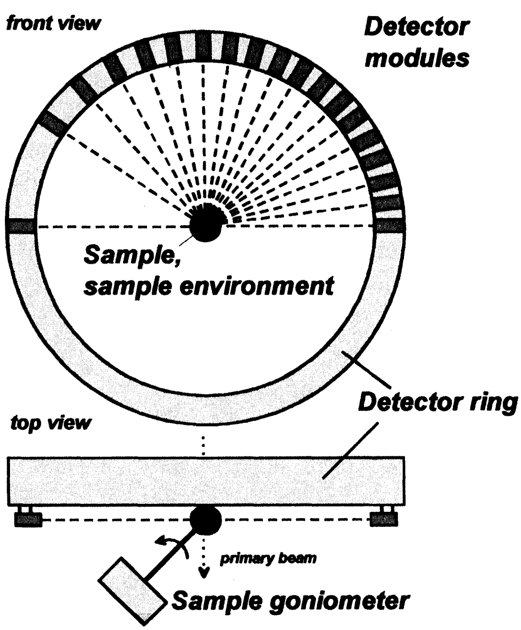
The texture diffractometer at the pulsed reactor IBR-2 is based on the time-of-flight technology which allows the recording of complete diffraction patterns by a single detector. The long total flight path of over 100 m leads to good spectral resolution of the recorded diffraction patterns, multiple detectors are used to measure several pole figure directions simultaneously. The detectors are arranged on a single Debye–Scherrer cone around the primary neutron beam at a unique scattering angle of $2\Theta = 90°$, they include an angular range of 180° (refer to the figure). This leads to the following most prominent advantages for pole figure measurements:

- The peak positions are identical for all detectors, hence, all $\lambda$ and $\Theta$ dependent corrections may be avoided.



- All pole figures can be measured simultaneously by a single sample revolution.

The sample is positioned in the center of the detector ring, and is rotated around a horizontal axis at an angle of 45° with respect to the primary neutron beam. According to this, axial symmetrical sample environment with its axis parallel to the goniometer axis can be installed with minor restrictions for the primary and secondary beam. A high pressure chamber for uniaxial load up to 150 kN and temperatures up to 1100 K is available, the maximal sample volume is 20 $cm^3$.

Beam and instrument characteristics

| | |
|---|---|
| Beam | Straight Ni-coated neutron guide (length: 92 m, evacuated: 76 m, Ar-filled: 16 m); unguided: 6 m |
| Beam cross section | $50 \times 170\,mm^2$ |
| Distance moderator-sample | 102.15 m |
| Sample-detector | 1.0 m |
| Flux of thermal neutrons | $\approx 10^6\,cm^{-2}\,s^{-1}$ |
| Range of wavelengths | 0.8–7.6 Å |
| *d*-spacings | 0.6–5.4 Å |
| Maximal sample volume | $\approx 30\,cm^3$ |
| Detector modules | $^3$He, single-tube, ⌀: 60 mm, Cd mantled, 24 available |
| Collimators | Gd-coated soller collimators, angular dispersion: 18′, cross section: $55 \times 55\,mm^2$, space: 92% |
| Resolution $\Delta d/d$, $2\Theta = 90°$, $d = 2$ Å | ≈0.5% |

## THE NEUTRON TIME-OF-FLIGHT STRAIN/STRESS DIFFRACTOMETER EPSILON

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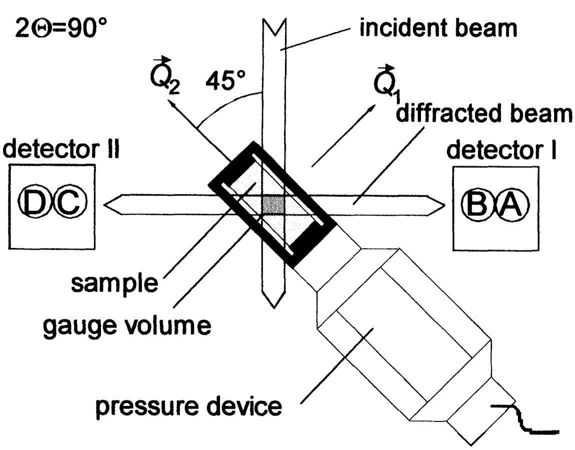
The beam line 7A at the pulsed reactor IBR-2 (JINR – Dubna, Russia) is shared by the diffractometers SKAT, which uses the upper part of the beam, and EPSILON, which is operated by the GeoForschungs-Zentrum Potsdam (Germany) and used the lower part of the beam.

The time-of-flight method allows the measurement of all Bragg reflections simultaneously. Due to the long flight path (102.38 m) a high spectral resolution is achieved and therefore this instrument is especially designed for strain measurements of micro and macro strains in materials, even of samples with low crystal symmetry (e.g., monophase and polyphase geological materials).

- The 5-axis goniometer allows the rotation about two axes and translation in three perpendicular directions and is, therefore, well suited to measure strain profiles and all six independent components of the strain tensor. High precision sample positioning is guaranteed by use of step motors.
- A deformation device for *in situ*-deformation experiments is installed. The equipment generates an uniaxial load up to 100 kN. Force, sample length and temperature are controlled *on-line*.
- Two detector units are arranged at opposite sides of the incident beam at a diffraction angle of $2\Theta = 90°$. This allows the recording of diffraction patterns for two perpendicular sample directions (characterized by the scattering vectors $\boldsymbol{Q}_2$ and $\boldsymbol{Q}_1$, cf. figure), e.g., the directions of contraction and dilatation during uniaxial deformation experiments.



Beam and instrument characteristics

| | |
|---|---|
| Beam | Straight Ni-coated neutron guide (length: 92 m, evacuated: 76 m, Ar-filled: 16 m); unguided: 5 m |
| Beam cross section | $50 \times 85\,\mathrm{mm}^2$ |
| Distance moderator-sample | 102.88 m |
| Sample-detector | 0.50 m |
| Flux of thermal neutrons | $\approx 10^6\,\mathrm{n\,cm^{-2}\,s^{-1}}$ |
| Range of wavelengths $\lambda$ | 0.8–7.6 Å |
| Lattice spacings $d$ | 0.6–5.5 Å |
| Detector units | $^3$He, ⌀: 20 mm, $2 \times 2$ tubes at $2\Theta = 90°$, shielded with Cd and $B_4C$ |
| Collimators | Soller collimators with thin mylar foils, Dy-coated, angular dispersion: 18′, cross section: 10 mm × 40 mm, transparency: 92% |
| Resolution $\Delta d/d$, ($2\Theta = 90°$, $d = 2$ Å) | $\leq 10^{-4}$ |
| Goniometer | 5-axis goniometer: $X$-, $\Psi$-rotation: 0–360°, accuracy: 0.0025° $X$-, $Y$-translation: 100 mm, $Z$-translation: 40 mm, accuracy: 0.0025 mm; |
| Uniaxial pressure device | $F = 100$ kN ($p = 150$ MPs, sample: ⌀ = 30 mm, $l = 60$ mm) |
| Maximal sample volume | $\approx 42\,\mathrm{cm}^3$ |
| Sample adjustment | 3 laser beams, 2 perpendicular to one another |

## HRFD – HIGH RESOLUTION FOURIER DIFFRACTOMETER

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High Resolution Fourier Diffractometer (HRFD) is a neutron reverse time-of-flight Fourier diffractometer intended for precise structural studies of polycrystals and residual stress investigations in bulk samples and advanced materials at a resolution level of about 0.001 or better. HRFD, the first neutron Fourier diffractometer at a pulsed neutron source, combines a high neutron flux at sample position, $\sim 8 \times 10^6\,\mathrm{n\,cm^{-2}\,s^{-1}}$, provided by the IBR-2 high flux pulsed reactor, and a high resolution over a wide range of $d$-spacings.

HRFD was constructed in the frame of cooperation between JINR (Dubna), PNPI (Gatchina), and VTT (Espoo). The main specific feature of HRFD distinguishing it from other Fourier diffractometers operating at steady state reactors, is the capability of analysis of triple correlations of signals from the neutron source, Fourier-chopper and the detector. As a result, the neutron intensity measured with HRFD is:

$$I(t) \sim R_s(t-t)R_c(t-t)\sigma(t)\,dt + cR_s(t-t)\sigma(t)\,dt + B(t),$$

where $R_c$ is the resolution function of the Fourier-chopper, $R_s$ is the source pulse, $\sigma$ is the scattering cross section of the sample, $B$ is the conventional background, and $c$ is a certain constant close to 1. The second term, called "the correlation background", is proportional not to the total detected intensity, as in the case of steady state reactors, but to the intensity measured in short time intervals equal to the width of the source pulse ($\sim 350\,\mu s$ for IBR-2). This leads to a substantial decrease of the correlation background, better quality of diffraction patterns, and permits the useful wavelength interval to be extended.

The first experiments with HRFD were performed in 1992 and the results were reported at the ICANS XII conference. Since then, the development of HRFD has continued and first of all, concerned the detectors, data acquisition system and, the data analysis procedure. In early 1994, regular experiments were started at HRFD, mainly in two directions, i.e., structural studies of new materials and residual stresses in bulk samples for industrial applications.

HRFD includes the following equipment for residual stress studies: four neutron detectors at the scattering angles of $2\theta = 90°$ (solid angles of 28 and 7 msr) and $2\theta = 152°$ (the solid angle of 80 msr each), 4-axis $(x, y, z, \omega)$ linear scanner for simple experiments, 5-axis $(x, y, z, \omega, \Omega)$ "HUBER" goniometer for total strain tensor measurements, load testing machine "TIRA" for *in situ* studies of samples under load and nitride boron slit systems for forming the incident and scattered neutron beams. Both $2\theta = 90°$ detectors are equipped by multi-slit radial collimators with gauge volume resolution of 2 mm. High neutron flux at the sample position and high resolution gives a possibility for precise strain measurements at HRFD within a reasonable measuring time.

Beam and instrument characteristics

| | |
|---|---|
| Beam | $^{58}$Ni-covered curved vacuum guide tube |
| Guide aperture | 10 mm × 100 mm, variable |
| Moderator-sample distance | 29.6 m |
| Chopper-sample distance | 20 m |
| Fourier-chopper (disk-type) | Ti–Zr alloy |
| Outside diameter | 540 mm |
| Slit width | 0.6 mm |
| Number of slits | 1024 |
| Max. speed | 9000 rpm |
| Max. beam modulation freq. | 150 kHz |
| Thermal neutron pulse width | |
| Low-resolution mode | 320 μs |
| High-resolution mode | 7 μs |
| High-resolution detectors | $^{6}$Li, time-focusing |
| Low-resolution detector | $^{3}$He, position-sensitive |
| Aperture of the detectors | |
| High-resolution 156° | 0.16 sr |
| High-resolution 90° | 0.04 sr |
| Low-resolution 0–60° | 0.006 sr |
| Wavelength interval | 0.9–8 Å |
| *d*-spacing interval | |
| High-resolution | 0.5–4 Å |
| Low-resolution | 4–20 Å |
| Flux at the sample position | $8 \times 10^{6}\,\mathrm{n\,cm^{-2}\,s^{-1}}$ |
| Sample volume | 1–2 $\mathrm{cm^3}$ |
| Resolution for | |
| $2\theta = 156°$, $d = 2$ Å | 0.001 |
| $2\theta = 90°$, $d = 2$ Å | 0.002 |

## THE NEUTRON TIME-OF-FLIGHT FOURIER SPECTROMETER FSS AT GKSS – RESEARCH CENTER

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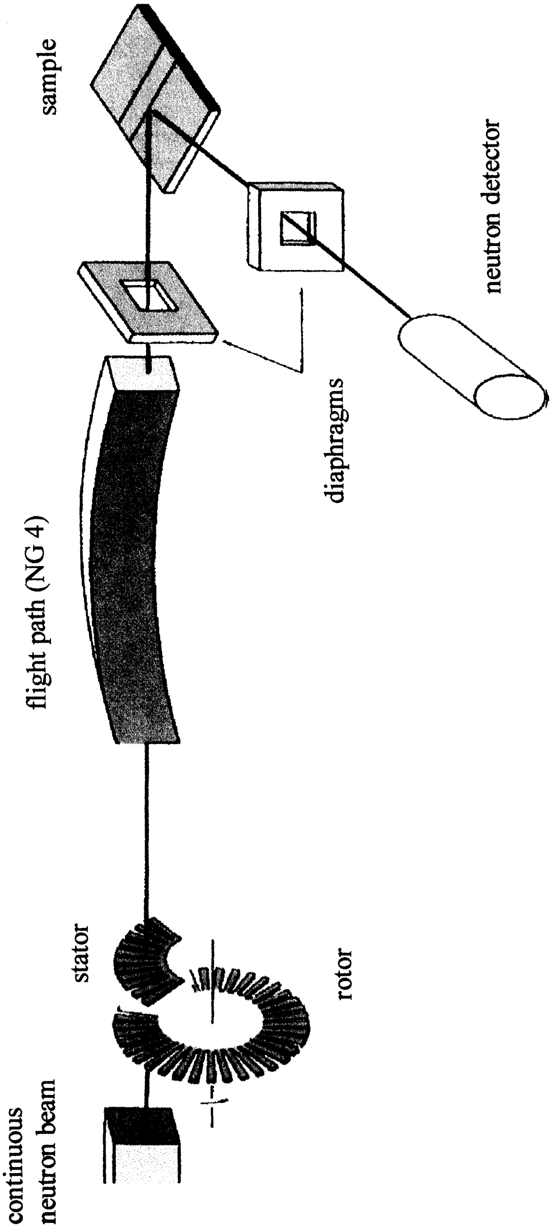
Fourier Strain Spectrometer (FSS) is a time-of-flight Fourier spectrometer with special design characteristics for non-destructive measurements of residual stresses in various materials (figure). It is jointly operated with the University of Kiel. The instrument is operated in the so-called reverse time of flight (RTOF) mode. Unlike the usual Fourier correlation method where frequencies have to be maintained precisely, RTOF uses a continuous frequency window and it is therefore very economic in terms of total measuring time for an experiment.




Schematic view of the high resolution powder diffractometer (Fourier Strain Spectrometer, FSS).

The instrument has a number of special features which demonstrate its advantages for stress determination by neutron diffraction.

- The full "white" spectrum of the thermal reactor neutrons can be used for simultaneous investigation of many reflections between 0.1 and 0.4 nm neutron wavelength.
- A scattering angle of 90° can be chosen for good definition of the scattering volume.
- Because of the fixed scattering geometry, special sample environments can easily be set up (externally applied stresses, variable temperatures, stresses under operating conditions). The weight and the size of the sample are not very crucial for the experiment.

FSS instrumental details

| | |
|---|---|
| Location at FRG-1 | Beamline 9 |
| Fourier-chopper | 1024 slits, 2000 rpm operating speed, 36 kHz maximum modulation frequency |
| Flight path | 21.15 m Ni-coated curved neutron guide<br>Radius of curvature: 3000 m<br>Characteristic wavelength: 0.183 nm |
| Beam size | $5 \times 5\,mm^2$ to $15 \times 108\,mm^2$ |
| Neutron flux | $2 \times 10^6\,n\,cm^{-2}\,s^{-1}$ |
| Wavelength range | 0.1–0.4 nm |
| Resolution | $\Delta d/d \leq 3 \times 10^{-3}$ for the quoted wavelengths range |
| TOF analyzer | VTT reverse time-of-flight correlator $6 \times 1024$ channels. 1 μs minimum channel width |
| Neutron detector | Li-6 glass scintillation detector bank in time-focusing geometry |
| Ancillary equipment | Sample positioner for strain tensor determination |

## THE DIFFRACTOMETER TEX-2 AT GKSS – RESEARCH CENTER

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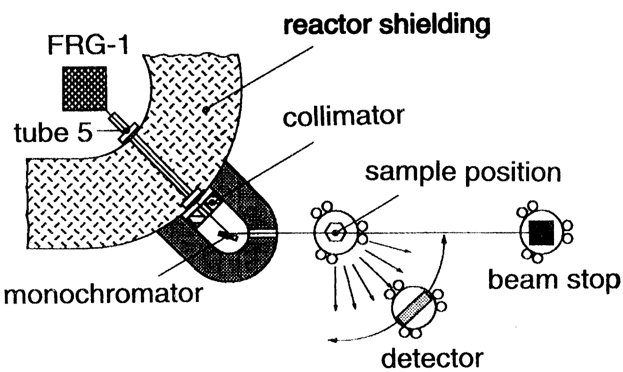
The diffractometer TEX-2 at GKSS – Research Center Geesthacht GmbH, Germany is a conventional four circle diffractometer optimized for texture investigations on different types of polycrystalline materials. Due to the excellent running circles of the 5 MW reactor FRG-1 with more than 200 days every year about 20 projects, about 150 samples and about 450 pole figures can be investigated every year. Since 1990 a broad spectrum of samples (metals, alloys, composites, intermetallics, anorganics, ceramics, superconductors and rocks) were studied in short term projects as well as in long term projects. It should be pointed out that at TEX-2 sample series of more than 50 samples have been measured. Thus one is able to compare between different deformation regimes, different compositions and the variation of subsequent annealing procedures. The following figure shows schematically the layout of TEX-2.

Looking on the instrumental details, the beam path can be optimized at different positions (primary collimation, monochromator, wavelength, sample-detector distance, diaphragms and detector collimation) and consequently one can adapt the resolution function of TEX-2 on the experimental requirements.




TEX-2 instrumental details

| | |
|---|---|
| Location at FRG-1 | Beamline 5 |
| Primary collimation | 30′, 42′, 51′ |
| Monochromator material | Cu(111), Cu(200), Ge(311), C(002) |
| Monochromator take-off-angle | 17.2°, 27.2°, 37.2°, 47.2°, 57.2° |
| Wavelength | Between 0.08 and 0.27 nm |
| Neutron flux | $0.8 - 4.0 \times 10^5\,n\,cm^{-2}\,s^{-1}$ |
| Angular range | $\varphi = -360°$ to $+360°$<br>$\chi = -360°$ to $+360°$<br>$\omega = -46°$ to $+46°$<br>$2\theta = -75°$ to $+120°$ |

TEX-2 instrumental details (*Continued*)

| | |
|---|---|
| Beam cross section | $45 \times 45\,mm^2$ |
| Standard samples for texture analysis | Cube 10 mm<br>Sphere 15–20 mm ∅<br>Cylinder 10–15 mm ∅,<br>10–15 mm high |
| Special sample holders | Rotating sample holder<br>*x–y* table<br>Tube holder for tubes up to<br>100 mm ∅ |
| Sample environment | |
| Mirror furnace | Up to 1650°C |
| Loading device | Tension up to 1.5 kN,<br>compression up to 2.0 kN |
| Detector | $^3$He-single detector, 20 mm ∅<br>38° JULIOS-PSD |
| Distance | |
| Sample – $^3$He | 40–200 cm |
| Sample – JULIOS | 70–100 cm |
| Computer for automatic control | PDP 11/23+, phytron unit |
| Texture software | ISEM, MULTEX,<br>WIMV (PC, VAX) |