Appl. Phys. A 74 [Suppl.], S86–S88 (2002) / Digital Object Identifier (DOI) 10.1007/s003390201750

Applied Physics A
Materials
Science & Processing

Neutron Fourier diffractometer FSD for internal stress analysis: first results

G.D. Bokuchava[1,*], **V.L. Aksenov**[1], **A.M. Balagurov**[1], **E.S. Kuzmin**[1], **V.V. Zhuravlev**[1], **A.P. Bulkin**[2], **V.A. Kudryashev**[2], **V.A. Trounov**[2]

[1] Frank Laboratory of Neutron Physics, JINR, 141980 Dubna, Moscow region, Russia
[2] Petersburg Nuclear Physics Institute of RAS, 188350 Gatchina, Russia

Received: 20 July 2001/Accepted: 12 March 2002 – © Springer-Verlag 2002

Abstract. At the IBR-2 pulsed reactor in Dubna a new neutron Fourier diffractometer FSD is under construction. FSD continues the development of neutron Fourier diffractometry at long-pulse neutron sources, which was started several years ago with the high-resolution Fourier diffractometer HRFD at the IBR-2. Whereas HRFD is mainly used for precise structural refinement, FSD is optimised for internal stress measurements in bulk materials. The FSD design satisfies the requirements of high luminosity, high resolution, a specific sample environment, a wide range of d_{hkl}, and fixed scattering angles $2\theta = \pm 90°$. It consists of a mirror neutron guide, a fast Fourier chopper for the neutron-beam intensity modulation, a ±90° MultiCon ZnS(Ag) ^{6}Li-loaded detector system with both geometrical and electronic focusing, a five-axis goniometer 'Huber' and loading machines, and VME-based RTOF analysers for data acquisition. Examples of the first experimental results obtained with FSD are presented.

PACS: 61.12.-q

Experiments for residual stress studies start to occupy a noticeable position in the research programs of leading neutron centres. To conduct such experiments, specialized neutron diffractometers are being constructed. The experience of application of neutron diffraction to study residual stresses with the HRFD diffractometer in Dubna [1] arouses vast interest on the side of science and industry. Therefore, the new project for construction of a neutron diffractometer (with a working name Fourier Stress Diffractometer - (FSD)) dedicated exclusively to residual stress studies started on the IBR-2 pulsed reactor in FLNP JINR. The work is conducted by joint efforts of collaborators from FLNP (Dubna), PNPI (Gatchina), IfZP (Dresden, Germany) and ISSP (Budapest, Hungary).

1 Experimental

The resolution of a TOF-diffractometer can be improved by decrease of neutron pulse duration and increase of flight path. For pulsed neutron sources with short pulses of fast neutrons a width of thermal neutron pulses of $\sim 20\,\mu\text{s}/\text{Å}$ can be achieved. An increase in flight path to 100 m gives a resolution value down to 0.001. For long pulse neutron sources (e.g. IBR-2 reactor) this way to achieve high resolution is unacceptable. Here the so called reverse time-of-flight method (RTOF) – a kind of correlation technique [2] – is of advantage. The RTOF-method assumes the application of a fast Fourier chopper for the neutron beam intensity modulation and measuring of high-resolution correlation spectra. In the RTOF-method data acquisition is conducted at the continuous changing of Fourier chopper rotation frequency from zero to some maximal frequency ω_m. The time component of the resolution function is defined by resolution function of Fourier chopper, which depends on particular frequency distribution function $g(\omega)$:

$$\Delta t_0/t \sim \int_0^{\Omega} g(\omega)\cos(\omega t)\mathrm{d}\omega \qquad (1)$$

where $\Omega = N\omega_m$ – maximal modulation frequency of neutron beam intensity, N – number of slits of Fourier chopper. At reasonable choice of ω_m the effective neutron pulse width Δt_0 equals Ω^{-1} and for $N = 1024$, $\omega_m = 100\,\text{Hz}$ (FSD parameters) gives a value $\sim 10\,\mu\text{s}$. This means that for total flight path (from Fourier chopper to detector) of ~ 6.5 m and scattering angle of $2\theta = 90°$ time component of resolution function equals $\Delta t_0/t \approx 2 \times 10^{-3}$ at $d = 2$ Å.

The FSD diffractometer is situated on beam 11a of the IBR-2 pulsed reactor with a water moderator which generates thermal neutron pulses with a frequency of 5 Hz and a duration of $\sim 320\,\mu\text{s}$. The main functional units of the FSD diffractometer are: a long curved mirror neutron guide for background suppression; a fast Fourier chopper; a straight

---

[*] Corresponding author.
(Fax: +7-09621/65882, E-mail: Gizo.Bokuchava@jinr.ru)

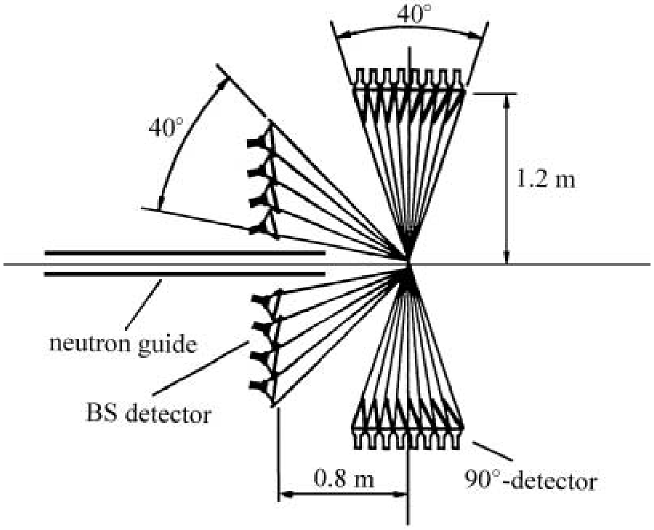

Fig. 1. The detector system of FSD diffractometer

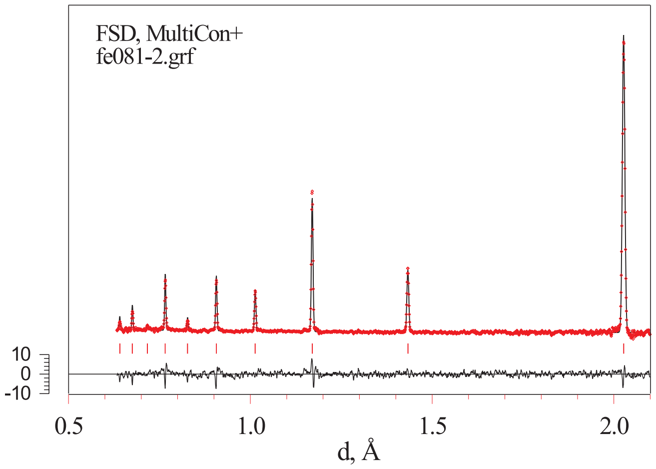

Fig. 2. Part of neutron diffraction pattern from the α-Fe standard sample measured on FSD in high-resolution mode by MultiCon$^+$ detector. Experimental points, profile calculated by the Rietveld method and difference curve are shown

mirror neutron guide to form the thermal neutron beam on the sample; a detector system; sample environment; data acquisition system, including specialised RTOF analysers. The FSD detector system includes two MultiCon detectors at average scattering angles $2\theta = \pm 90°$, at that each MultiCon detector consists of 8 elements with ^{6}Li loaded ZnS plates as a scintillator (Fig. 1). A peculiarity of the MultiCon detector is combined use of electronic and time focusing of a scattered neutron beam that allow realising fundamentally new design of a detector system with large solid angles (~ 0.16 steradians for each MultiCon detector) [3]. All 8 elements of detector are electronically focused using an appropriate channel width for each detector analyser. Thus the electronic time focusing gives the same scale d_{hkl} for all detector elements. At the same time the scintillator surface of each detector element is arranged according to the neutron time focusing condition, which gives the high-resolution value for individual detector element. Such set-up essentially increases the luminosity of FSD diffractometer (by factor of ~ 10 in comparing with actual prototype at HRFD diffractometer) at the same time providing high-resolution on interplanar spacing. Currently two first elements of MultiCon detectors are assembled and installed on FSD. Additionally backscattering BS$^-$ detector ($2\theta \approx -141°$) composed of sixteen ^{6}Li scintillation elements is installed. The spatial arrangement of BS$^-$ detector elements corresponds to a single time focusing surface for a scattered neutron beam.

2 Results and discussion

On FSD the mirror neutron guide has greater radius of curvature in comparison to the HRFD diffractometer in order to obtain appropriate neutron spectral distribution. Therefore for FSD neutron intensity spectral distribution is shifted towards shorter wavelengths by $\Delta\lambda \approx 0.2$ Å and the available wavelength range is $\lambda = 0.9 \div 8$ Å. This fits well to the FSD required characteristics since lattice parameters of most studied materials are rather small so the available d_{hkl}-range should be shifted to smaller values. The measured integral neutron flux at the sample position is $F \approx 3 \times 10^5$ neutr./cm^2/sec with Fourier chopper. The value of Fourier chopper transmission was found to be $\approx 1/4.5$. The first neutron diffraction patterns from α-Fe standard sample were measured on FSD in high-resolution mode by BS$^-$ and MultiCon$^+$ detectors (Fig. 2). The FSD resolution function was estimated from high-resolution neutron

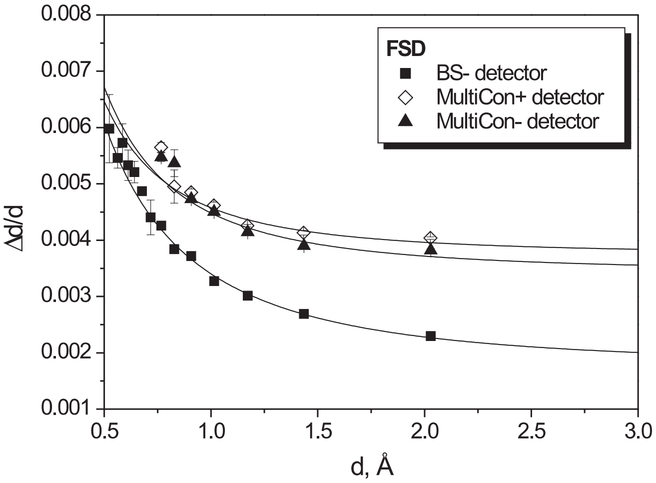

Fig. 3. FSD resolution function measured on α-Fe powder at maximal Fourier chopper speed $V_{\max} = 6000$ rpm

Table 1. Main parameters of FSD high-resolution detectors

Detector	BS$^-$	MultiCon$^+$ 1st element	MultiCon$^-$ 1st element
Scat. angle 2θ,°	140.864	107.5	107.5
d_{hkl} - range, Å	0.51 – 5.39	0.63 – 6.71	0.63 – 6.71
$\Delta d/d$ ($d = 1$ Å)	3.4×10^{-3}	4.6×10^{-3}	4.5×10^{-3}
$\Delta d/d$ ($d = 2$ Å)	2.3×10^{-3}	4.0×10^{-3}	3.7×10^{-3}
Solid angle Ω, str	0.054	0.02	0.02

diffraction spectra measured on annealed $\alpha-\mathrm{Fe}$ powder by all three detectors at maximal Fourier chopper speed $V_{\max} = 6000$ rpm (Fig. 3). Results of experiments show that all detectors have sufficiently high-resolution and a wide enough range of d_{hkl} (Table 1): $\Delta d/d \approx 2.3 \times 10^{-3}$ for BS$^-$ detector and $\Delta d/d \approx 4 \times 10^{-3}$ for both first elements of MultiCon$^+$ detectors at $d = 2$ Å. Furthermore the dependence of resolution function and diffraction peak shape on maximal Fourier chopper speed $V_{\max}$ was measured. In the RTOF-method the intensity, amplitude and width of diffraction peaks depend on the maximal Fourier chopper speed $V_{\max}$. According to theory the effective neutron pulse width decreases as $1/V_{\max}$, reaching the value of $\sim 10\,\mu$s at $V_{\max} = 6000$ rpm (Fig. 4). Thus this specific feature of the RTOF-method gives the possibility to select the optimal regime of FSD operation depending on the required resolution and intensity.

3 Conclusion

Recent experiments have shown that the FSD parameters confirm the fitness of the diffractometer for residual stress studies with required accuracy. In 2001 physical experiments program was started on FSD diffractometer. Auxiliary equipment (loading device, mirror furnace, Huber goniometer, etc.) allows one to vary broadly the experimental conditions. Further work on diffractometer construction will include development of the detector system, radial collimators as well as electronics and software.

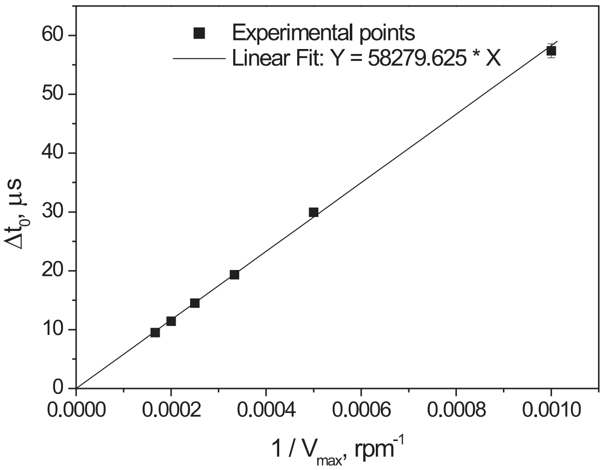

Fig. 4. Effective pulse width Δt_0 dependence versus maximal Fourier chopper speed $V_{\max}$ in high-resolution mode

Acknowledgements. This work was supported in the framework of an agreement between BMBF (Germany) and JINR.

References

1. V.L. Aksenov, A.M. Balagurov, V.G. Simkin, A.P. Bulkin, V.A. Kudrjashev, V.A. Trounov, O. Antson, P. Hiismaki, A. Tiita: J. Neutron Res. **5**, 181 (1997)
2. P. Hiismaki, H. Poyry, A. Tiitta: J. Appl. Cryst. **21**, 349 (1988)
3. V.A. Kudryashev, V.A. Trounov, V.G. Mouratov: Physica B **234–236**, 1138 (1997)