ICANS-XIII
13th Meeting of the International Collaboration on
Advanced Neutron Sources
October 11-14, 1995
Paul Scherrer Institut, 5232 Villigen PSI, Switzerland

SETUP AND OPERATION MODE OF A PULSED WHITE BEAM ANGLE-DISPERSIVE TOF-TEXTURE-DIFFRACTOMETER

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ABSTRACT

The paper reports on the setup and texture operation mode of the redesigned ROTAX instrument at ISIS. Pulsed white beam time-of-flight diffraction, a one-circle sample goniometer and a linear position-sensitive detector are used to assemble the neutron texture diffractometer. Sets of complete pole-figures can be measured in a minimum of different sample orientations due to the fact that the conventional pole-distance scanning of the sample becomes unnecessary. Test results on copper specimens are reported. Data collection times are reduced up to a factor 50 compared to those at a continuous reactor source with monochromatic neutrons.

1. Introduction

Despite the advantages of using neutrons for volume texture analysis, e.g. the high penetration capability of neutrons and the possibility of using rather large samples, those of irregular shape and coarse grained material, the measurement of neutron diffraction pole-figures often fails because of the time consumptive experimental procedure and the demand of beam time. This is due to the fact that Bragg intensities have to be measured for a large number of different sample orientations for adequate pole-figure resolution. When using monochromatic neutrons, the sample scanning is usually performed on a two-circle Eulerian cradle with rotation axes ϕ and χ for azimuth and pole-distance scanning, respectively. Depending on the crystalline constitution, i.e. mainly on the grain size of the crystallites, the number of different sample orientations varies roughly between 100 and 1000 to stepscan one hemisphere of the sample. Depending on the scattering power of the sample, i.e. mainly on size, composition and structural symmetry, measuring times of about 1 to 10 min per pole-figure data point are necessary; this is experienced, when working with monochromatic neutrons at a conventional reactor diffractometer (see e.g. [1]). Thus, the total measuring time for a set of pole-figures lies between approximately half a day for high-symmetry metallic samples and one week for low-symmetry multiphase geological samples.

Keywords: TOF-diffraction, Texture diffractometer, Pole-figure scanning, ROTAX

It is the merit of position-sensitive detector (psd) installations to obtain several or a multitude of individual pole-figures simultaneously during one pole-figure scan [2]. The so-called 'blind area' arising for those pole-figures, which are registered under non-bisecting scattering conditions at the outer parts of the psd, can be overcome by a few additional sample orientations [3]. When using the linear JULIOS-psd of 70 cm sensitive length, for instance, one needs only one additional sample orientation to fill the blind area [4]. When installed in the horizontal scattering plane, the psd saves measuring time only with respect to the measurement of more than one pole-figure, but it cannot reduce the number of individual sample orientations during the pole-figure scanning procedure. When installed under $2\Theta=90°$ in the vertical scattering plane, the psd allows a reduction of different sample orientations, but only one hkl pole-figure can be measured during one sample scan. [5].

This situation will change, when using polychromatic neutrons at a pulsed spallation source. Here, the psd fulfils two objects simultaneously: (1) the simultaneous measurement of a multitude of hkl pole-figures <u>and</u> (2) an essential reduction of sample orientations in the course of the pole-figure scanning. Furthermore, angle-dispersive TOF-measurements will reduce the measuring time per data point because of the better exploitation of the primary neutron spectrum [6]. Method, instrumental setup and experimental tests will be described in the following.

2. Scheme of pole-figure scanning

The angle-dispersive, white beam pole-figure scanning is based on the fact that Bragg reflection conditions are fulfilled for different wavelengths at different positions of the linear position-sensitive detector. One and the same Bragg reflection,

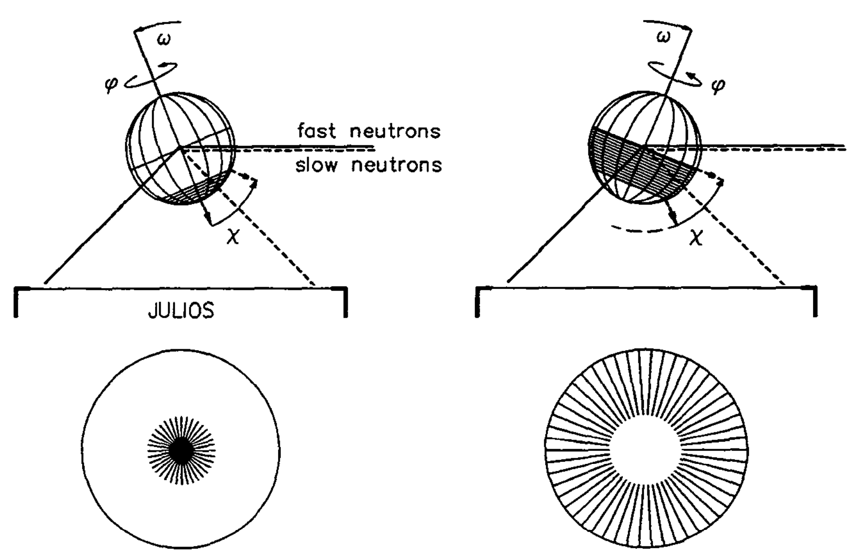

Fig. 1: Reference pole-spheres around the sample mounted in two different ω-positions (top: left and right resp.). The limiting scattering vectors (arrows) of one Bragg plane are shown for the shortest and the longest neutron wavelength diffracting at the left (solid line) and right hand side (dashed) of the linear JULIOS detector, resp.; accessory Δχ-coverages in stereographic projection (bottom), when applying Δφ step scanning in steps of 10° (left) and 7.2° (right). Pole-distances from 0° to 45° (left) and 45° to 90° (right) are covered simultaneously.

which appears along the detector, originates from differently oriented crystallites each. Thus, different orientations of a Bragg plane within a sample can be registered simultaneously in a fixed sample position. This is illustrated in Fig. 1. The angular width of different cystallite orientations expressed by the width of the pole-distance $\Delta\chi$, which can be registered simultaneously, depends on the linear extension of the position-sensitive detector and its simultaneous 2Θ-coverage. A $\Delta 2\Theta$ of 180° corresponds to a $\Delta\chi$-coverage of 90°; this means the complete coverage of one hemisphere of the sample in one fixed ω-setting with respect to the primary neutron beam. The pole-figure scanning procedure is reduced to a $\Delta\phi$ azimuth stepscanning, the grid of which is selected according to the desired pole-figure resolution. The complete $\Delta\chi$-scanning, indispensible for complete pole-figures when using monochromatic neutrons, is substituted by means of the position-sensitive detector and the exploitation of the white neutron spectrum. The $\Delta\lambda$-window accessible by the instrument determines the d-spacing coverage and thus the number of hkl pole-figures registered simultaneously.

Obviously, this method of pole-figure scanning is not confined to the ideal conditions of a 180°-scattering angle detector system. When using detector installations of smaller extensions, however, more than one fixed ω-setting of the sample is needed to cover the complete pole-distance of one hemisphere around the sample. Two ω-settings are sufficient for a 90°-detector instrument. A $\Delta 2\Theta$-coverage of 90° corresponds to a $\Delta\chi$-coverage of 45°. First and second ω-position are selected according to individual scattering geometry realizing that the two settings correspond to pole-distance coverages $\Delta\chi$ from 0° to 45° and from 45° to 90° (see Fig. 1). The shorter the detector dimensions or the simultaneous 2Θ-coverage, respectively, the more different ω-settings are necessary, where the 360° $\Delta\phi$ scanning procedure has to be performed each time. In case of using an arrangement of several counting tubes instead of a psd, each counting tube position defines one fixed χ-value and measures one concentric ring of the pole-figures [7].

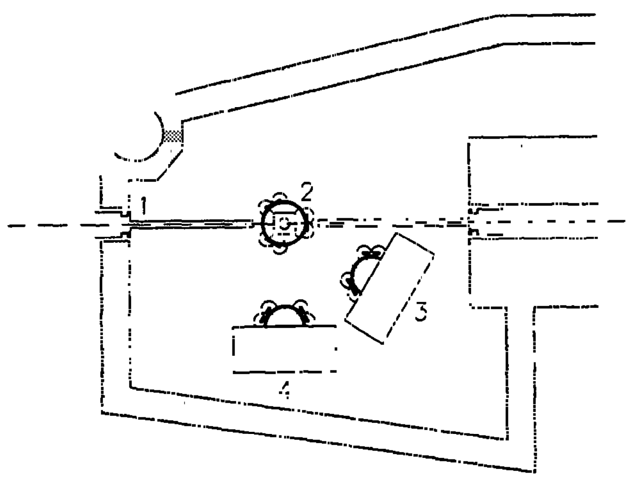

Fig. 2: The ROT/DIFF-setup in the ROTAX-blockhouse (shaded); primary beam (1), sample position and goniometer support module (2), JULIOS detector units to be positioned on air pads (3 and 4)

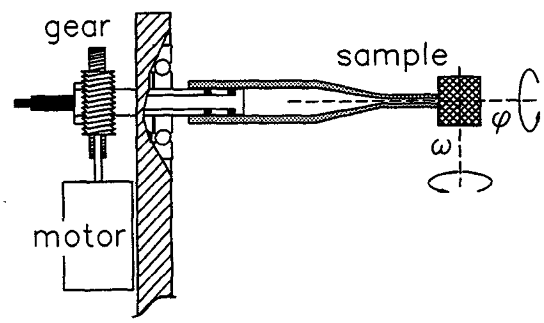

Fig. 3: One-circle goniometer with horizontal ϕ-axis and sample to be centered in the vertical ω-axis of the turn-table (see (2) in Fig. 2)

3. ROT/DIFF instrumental setup

The instrumental setup of a pulsed white beam angle-dispersive time-of-flight texture diffractometer is being realized at the ROTAX instrument at ISIS. The ROTAX spectrometer [8] has been redesigned into a versatile neutron powder diffractometer ROT/DIFF [9], which is characterized by the flexible installation of several linear psd-units of the JULIOS-type [10] (Fig. 2). The detector positions can be varied with respect to different 2Θ-settings and with respect to variable distances between sample and detector. One detector unit of 682 mm sensitive length and 2.3 mm spatial resolution covers a Δ2Θ-section of about 38° in 100 cm distance. In minimum distance the Δ2Θ-coverage is approximately 90°, thus allowing the scanning of complete pole-figures in two ω-settings of the sample as is shown in Fig. 1. The wavelength band used is from 0.4 Å to 4.0 Å. The d-spacing coverage of the instrument is from 0.2 Å in backscattering to about 60 Å at small scattering angles. The neutron flight path from the moderator to the sample is 14 m.

In its operation mode for pole-figure measurements, the instrument is equipped with an additional goniometer device, which is installed on the sample-table of the diffractometer. A stepping motor drive at the turn-table allows different ω-settings of the goniometer with respect to the primary beam. The goniometer itself (Fig. 3) is constructed with a horizontal rotation axis to perform the $\Delta\phi$ pole-figure scanning. The ϕ-axis is driven by a stepping motor combined with a reduction gear of 100 steps per one degree rotation. The texture sample can be glued on a glass-capillary at the end of the rotation axis. Flexible lengths are adjustable to enable the centering of samples of different size within the vertical ω-axis of the turntable.

4. Test measurements and results

Pole-figure test measurements have been performed during the reconstruction of the ROTAX-instrument by transferring one unit of the linear JULIOS-detector and the sample goniometer to the TESTbeam facility at ISIS. The provisional experimental setup allowed flexible ω-settings, $\Delta\phi$-stepscanning and a JULIOS installation of 90° 2Θ-coverage. The neutron flight path from the moderator to the sample was 12 m at TEST; i.e. 2 m shorter than at ROTAX with the consequence of improved primary intensity and slightly reduced d-spacing resolution.

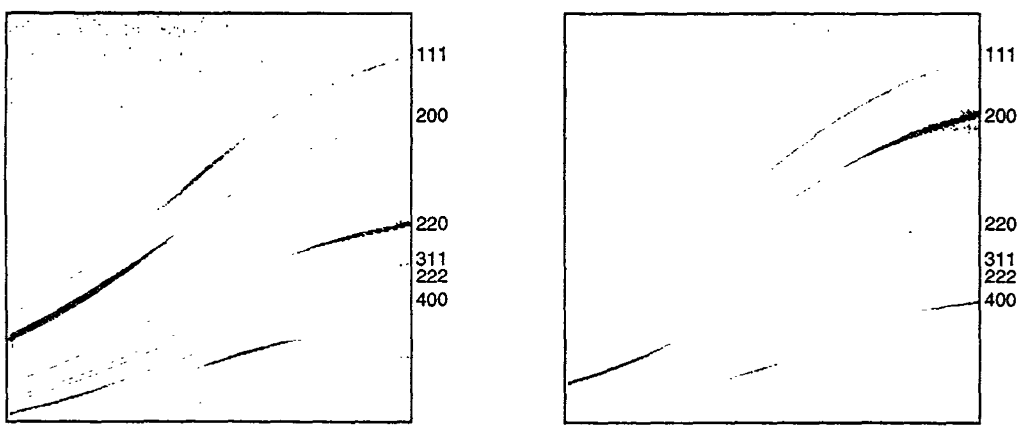

Fig. 4: Images of the detector matrix with 2Θ-channels (horizontal) and λ-channels (vertical) showing traces of simultaneously registered Bragg reflection intensities of the recrystallized copper specimen for sample orientations ω_1 = +22.5°, ϕ_1 = 0° (left) and ω_2 = −22.5°, ϕ_2 = 0° (right).

Pole-figure scans have been performed on cube-shaped copper specimens. The samples had been prepared by cutting plates of 0.9 mm thickness into pieces of 1 cm^2 and glueing them on top of each other in the same orientation. The one specimen was made from cold rolled copper sheets, the other from recrystallized ones. [8]. The central detector position during the angle-dispersive time-of-flight measurements was $2\Theta = 90°$, the wavelength band used was from 0.6 Å to 4.2 Å resulting in a simultaneous experimental d-spacing coverage from about 0.4 to 5.0 Å, where all relevant Bragg-reflections of the fcc-structure of copper are contained. According to the 90° $\Delta 2\Theta$-coverage of the detector the pole-figure scanning was performed in two ω-settings of each sample: $\omega_1 = +22.5°$ to cover pole-distances $\Delta\chi$ from 0° to 45° and $\omega_2 = -22.5°$ for $\Delta\chi$ from 45° to 90°. The grid of the 360°-azimuth stepscanning was $\Delta\phi_1 = 7.2°$ and $\Delta\phi_2 = 10.0°$ for the ω_1- and ω_2-setting, respectively. Fig. 4 shows the variation of Bragg intensities for two different (ω, ϕ)-orientations of one texturized sample. The real counting time for one sample orientation was 40 sec; the total experimental time, i.e. positioning and counting, was approximately 1 h for one sample.

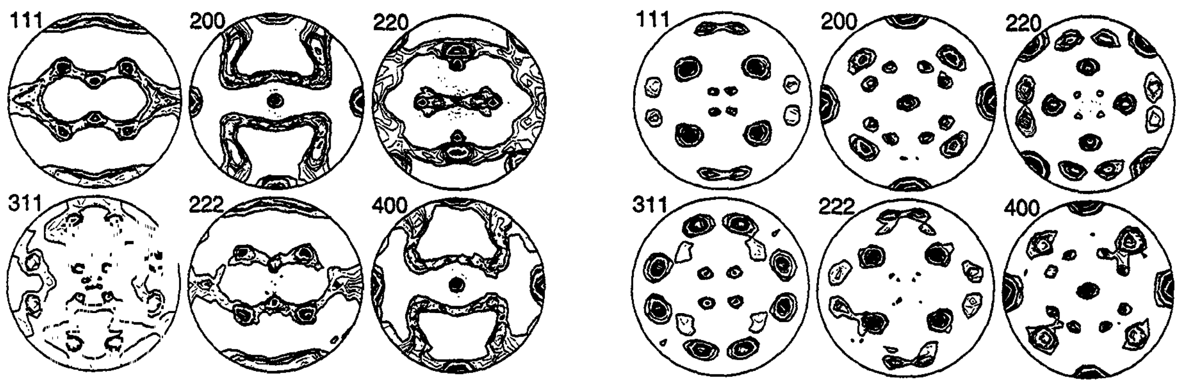

Fig. 5: Experimental pole-figures of the rolled copper specimen (left) and of the recrystallized copper specimen (right).

A total of 86 individual angle-dispersive time-of-flight diffraction patterns per sample have to be analysed for pole-figure representation. The experimental data, which are stored in (2Θ, λ)-channels, have to be rearranged for a proper allocation of (ϕ, χ)- and hkl-parameters. The orientation dependent hkl intensity variations are plotted as usual as hkl pole-figures in stereographic projections of the sample hemisphere. A selection of the simultaneously measured pole-figures is shown in Fig. 5. The pole-figures show the typical appearence of rolling and recrystallization textures.

5. Discussion and prospects

The copper specimens have been chosen for this methodical test of white beam angle-dispersive pole-figure scanning, because the identical samples had already been measured in conventional constant-λ technology at the JULIOS-equipped diffractometer SV7 at the FRJ-2 reactor [8]. The identical material and the identical detector system form a favourable basis for a direct comparison of both technologies. The quality of the pole-figures, which is defined on the one hand by the resolution width and the density of the mesh of data points and on the other hand by the measurement statistics, is found almost the same for both measurements.

This is revealed by a direct comparison of the reactor and spallation source pole-figures [9]. The constant-λ $\Delta\phi$- and $\Delta\chi$-pole-figure scanning had been performed with a total of 800 sample orientations and 3 min measuring time each. The total experimental time was about 48 h per sample. Thus, a gain factor of roughly 50 in experimental beam time can be stated in favour of the angle-dispersive time-of-flight technology at ISIS. This gain may be splitted into a factor 10 obtained by the reduction of different sample orientations (86 instead of 800) and a factor 5 resulting from the general advantage of pulsed white beam time-of-flight diffraction and its exploitation of the primary neutron spectrum (compare [6]).

The substantial reduction of neutron beam time is of special interest in geological texture analysis. The low structural symmetry of most natural mineral constitutents and the multiphase character of many geological specimens cause complex diffraction patterns and the distribution of Bragg intensities over a multitude of comparatively weak reflections. This involves rather long measuring times to guarantee adequate statistics in the diffraction patterns during pole-figure scanning. Furtheron, geologists are interested in the study of texture variations involving large series of many individual samples to be analysed. The decisive factor of beam time, which often prevents people from doing neutron diffraction texture measurements, becomes less important when using the technology described here.

Acknowledgement This work has been funded by the German Federal Government (BMBF) under contract-nos. 03-WI4BO2 and 03-WI4BO3.

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