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APPLICATION OF NEUTRON TIME-OF-FLIGHT DIFFRACTION TO TEXTURE STUDIES

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Introduction

Thermal neutron diffraction is known to be a powerful tool especially for volume texture analysis because of the low absorption of neutrons by the majority of substances. At stationary reactors the angle dispersive diffraction technique is applied to preferred orientation studies for more than two decades (1). Modern high flux pulsed sources such as the pulsed reactor IBR-2 (JINR,Dubna) and the spallation neutron source SNS (RAL,Harwell, U.K.) provide an efficient application of the neutron time-of-flight (TOF) diffraction for texture analysis. This energy dispersive method allows one to record a complete Bragg diffraction pattern simultaneously at a constant scattering geometry. This means that not a single pole figure as in the angle dispersive technique, but a large number of pole figures can be obtained at one and the same time using also the information on overlapping reflections. Furthermore each TOF diffraction spectrum is equivalent to the inverse pole figure of the given sample position, assuming an ideal resolution of the experimental equipment.

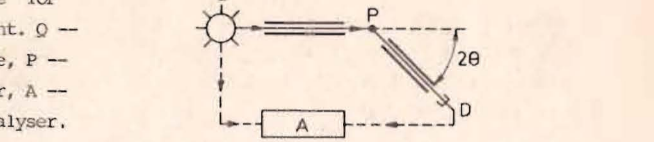
Alternatively to the conventional angle dispersive technique the TOF texture investigations are especially convenient for the solution of problems requiring a great number of pole figures like preferred orientation studies in low symmetric substances or high symmetric materials containing more than one phase. Furthermore, the TOF technique can be applied to observe texture formations caused by external influences performing in-situ experiments due to its constant scattering geometry.

Time-of-Flight Diffraction

The TOF diffraction is a typical method for experiments using a pulsed polychromatic neutron beam emitted by a pulsed neutron source like a nuclear pulsed reactor or a spallation source. The scheme of the TOF diffractometer is shown in fig. 1. If the source emits at t_0 a polychromatic pulse, the neutrons of different wavelengths corresponding to this pulse arrive at the specimen at different times flying from the source to the sample along a long flight path QP. If the grain orientations satisfy the Bragg's law the reflected neutrons are recorded by the detector at a fixed angle 2ϑ. The connection between the wavelength λ and the total time of flight $t - t_0$ from the source to the detector QPD is given by the following relation

$$t - t_0 = \frac{m \cdot \lambda}{h}\, \overline{QPD} = \beta \cdot \overline{QPD} \cdot \lambda \qquad \beta = 2{,}528 \cdot 10^{6}\, s \cdot m^{-2}. \qquad (1)$$

Fig. 1. Scheme of the TOF diffraction experiment. Q -- pulsed neutron source, P -- sample, D -- detector, A -- multichannel time-analyser.



The detector signals are stored in a multichannel time analyser. Because of the wavelength spectrum of the primary beam a great number of different reflections are recorded simultaneously by the TOF diffraction. Fig. 2 shows the TOF diffraction pattern of copper powder. For more details on the TOF method see for example (2).

Comparison of TOF and conventional texture studies

In the conventional neutronographic texture analysis (1) the experimental technique allows one to determine one pole figure after another by the variation of the Bragg angle. The detector aperture is so poor that all the integrated intensity of the chosen Bragg reflection is recorded in one measuring point. To cover the pole figures with experimental values, the specimen has to be rotated in the texture goniometer using equal angular , equal area, helical or other scans. In this way the experimental expense in the conventional method depends on the number of required pole figures. On the other hand the number of pole figures which can be determined is limited to the number of nonoverlapped reflections. If there is any overlapping, the measured intensity of $(h_1 k_1 l_1)$ reflection does not contain any information on the contribution of the $(h_2 k_2 l_2)$ peak. Fig. 3 illustrates the situation.

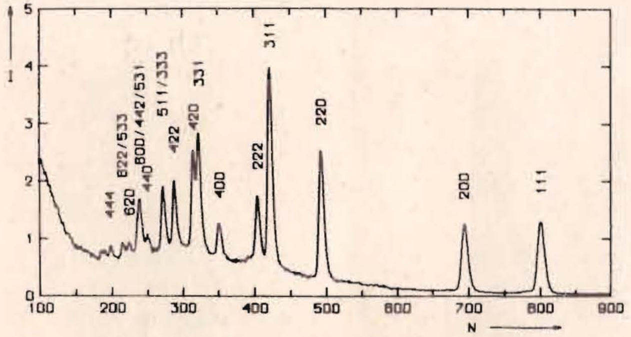


Fig. 2. TOF diffraction spectrum of copper powder.

In the TOF diffraction a great number of Bragg reflections is recorded simultaneously. Therefore, contrary to the angle dispersive method each specimen position corresponds to one point in all considered pole figures. In this way the experimental expense is independent of the required number of pole figures, i.e., the TOF method becomes more efficient if the number of necessary pole figures increases. Separating the overlapped reflections by means of a computer fit program (3) the information on peaks like those shown in fig. 3 can be used also. Furthermore, this line profil analysis averages the statistical errors of individual experimental points and allows one to take into account the background in a very accurate way, especially in the range of nonoverlapped reflections.

On the other hand the time required to record one TOF spectrum is not less than 10 minutes. Thus it is more convenient to investigate the texture of cubic or hexagonal materials by the angle dispersive method.

Of special interest is the fact that the information about one TOF spectrum is equivalent to the inverse pole figure of the chosen sample position. Unfortunately, neither the available number of reflections nor their distribution over the inverse pole figure range allows one to apply the mathematical texture analysis. Nevertheless, the constant scattering geometry makes the TOF diffraction well-suited for the observation of texture component formation in the in-situ experiments.

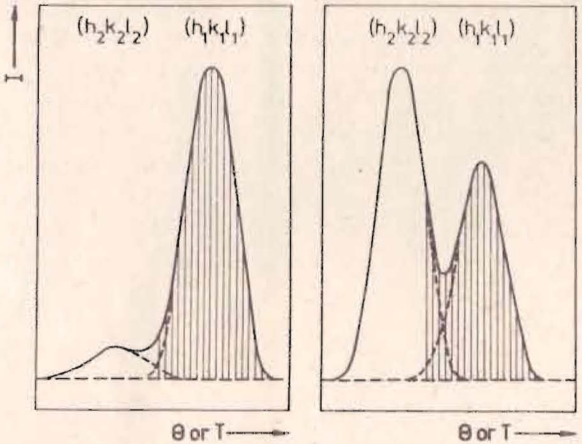


Fig. 3. Intensity relations of two overlapped Bragg reflections for different positions of a texturized specimen with respect to the scattering vector. The angle dispersive method records the shaded area.

Experimental equipment

As a result of the preceding considerations the TOF diffractometer was started into operation at the high flux pulsed reactor IBR-2 of the JINR (4). Schematically it is shown in fig. 4. At present the first flight path determining the time resolution of the spectrometer is 32.5 m. The necessary rotation of the specimen is carried out by an automatical texture goniometer having three perpendicular independent axes. The goniometer can be handled by hand or by a program tape.

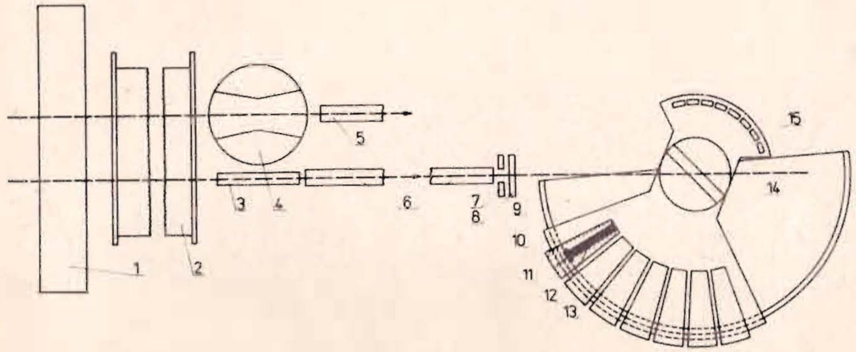


Fig. 4. Scheme of the TOF texture diffractometer: 1 -- reactor shielding, 2-- evacuated neutron guide tube, 3,6 -- mirror guide tubes, 4,5 -- equipment on the neighbouring beam, 7 -- diaphragma, 8 -- monitor, 9 -- basic platform, 10 -- moveable platform, 11 -- detector, 12 -- slit collimator, 13 -- detector shielding, 14 -- texture goniometer, 15 -- other equipment.

In the future the spectrometer will be installed at a distance of 100 m from the reactor. The neutron mirror guide tube is necessary to minimize the loss of intensity. A fully automatical minicomputer controlled regime of measurement is in the stage of preparation. The parallel measurement with several detectors allows one to determine the corresponding number of points in each pole figure simultaneously. The geometrical conditions and corrections for different Bragg angles are described in (4).

Experiment

At the former pulsed reactor IBR-30 a number of texture investigations has been carried out. They are representative for different applications using a total flight path of about 32 m. All mathematical texture analysis procedures have been done using the series expansion method (5). The Euler angles are chosen by the notation of Matthies (6).

Firstly, the texture of a rolled sheet of microduplex steel consisting of bcc (α) and fcc (γ) iron phases has been measured (7). Fig.5 shows the parts of the TOF spectra corresponding to normal, transverse and rolling directions. The texture effects can be seen from comparing the relative peak intensities of one spectrum. By means of the fit program ORION (2) seven reflections could be separated from each phase in all diffraction patterns.

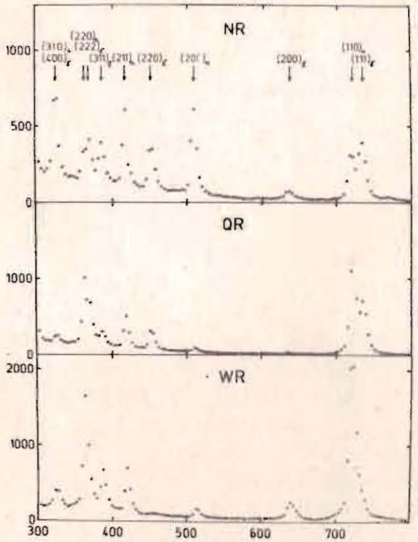


Fig. 5. TOF spectra of microduplex steel (Counter pulses versus time channels) ND=NR, RD=WR, TD=QR.

In another experiment the formation of recrystallization texture in copper has been studied (8). The sample was composed from 96 per cent deformed axisymmetric copper bars 5 mm thick. Because of the relatively low counting rate, the recording time for one TOF spectrum was 2 hours. Therefore, the recrystallization temperature was chosen to be 225°C only. According to microhardness measurements the recrystallization process was over after 35 hours. The inverse pole figures recorded after various annealing times at the steady state reactor of the CINR Rossendorf are represented in fig.6. The initial texture consists of a strong ⟨111⟩ and ⟨100⟩ component. During recrystallization the ⟨111⟩ component decreases in favour of the ⟨100⟩ one.

The texture analysis on the tetragonal τ -phase of MnAl bars has been carried out (9). The material is of interest because of its hard-magnetic properties (10). By the TOF technique 13 pole figures could be determined. They are shown in fig. 7. The main texture components in the inverse pole figure (fig. 7) do not coincide with the easy magnetization direction [001]. The pole density variations are weak. This result explains the nonoptimal magnetic properties of the investigated material.

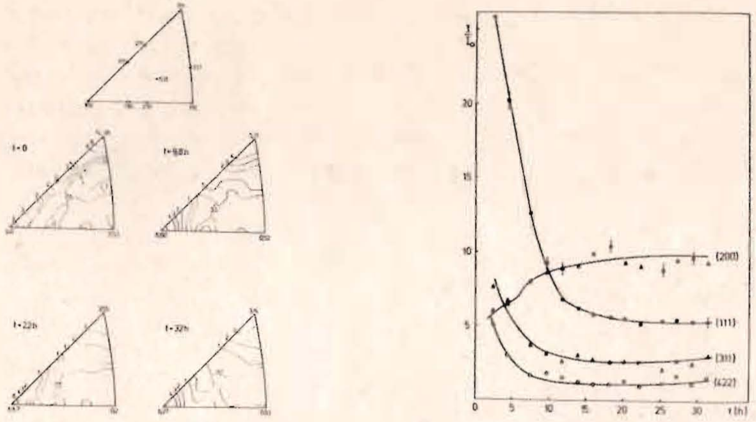


Fig. 6. Inverse pole figures of Cu for different annealing times (left). Reflection intensities in dependence on annealing time.

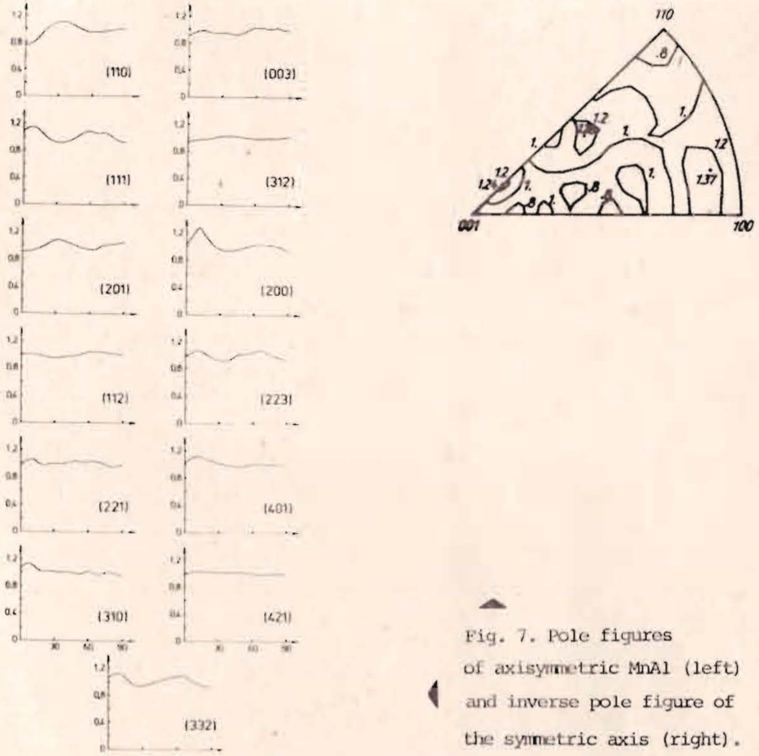


Fig. 7. Pole figures of axisymmetric MnAl (left) and inverse pole figure of the symmetric axis (right).

A relatively large beam cross-section (diameter 5 cm) and a good transmission ability of neutrons for a majority of substances allow one to investigate not only fine-grained materials by means of the TOF diffraction. Therefore, the proposed method is well suited for the texture analysis of rocks and minerals. Preferred orientations of quartz crystallites in granulite have been studied (11). The full texture analysis up to ODF has been performed with the series expansion cut-off at $l = 14$. The specimen symmetry was assumed to be monoclinic. For texture analysis 6 pole figures were taken into account (see fig.8). Other pole figures were uncertain because of the high content (~ 50 %) of other phases, especially feldspars, in the specimen. The selection criterion was the coincidence of C_2^{11} coefficients for single pole figures.

The (0003) pole figure of quartz is not measurable (11), but of special interest for the interpretation. In Fig. 9 this pole figure has been calculated as well as the inverse pole figure for the three coordinate axes ND, direction of lineation DD and TD. Finally in fig. 10 the ODF of the quartz part of granulite is represented. In the ODF a high intensity range in the neighbourhood of $\beta = 0$ can be seen which is almost independent of α and γ. In complience with ODF high pole density is concentrated in the centre of (0003) pole figure at the 0001 point of the inverse pole figure. Up to the present stage of the work no ghost corrections could be taken into account.

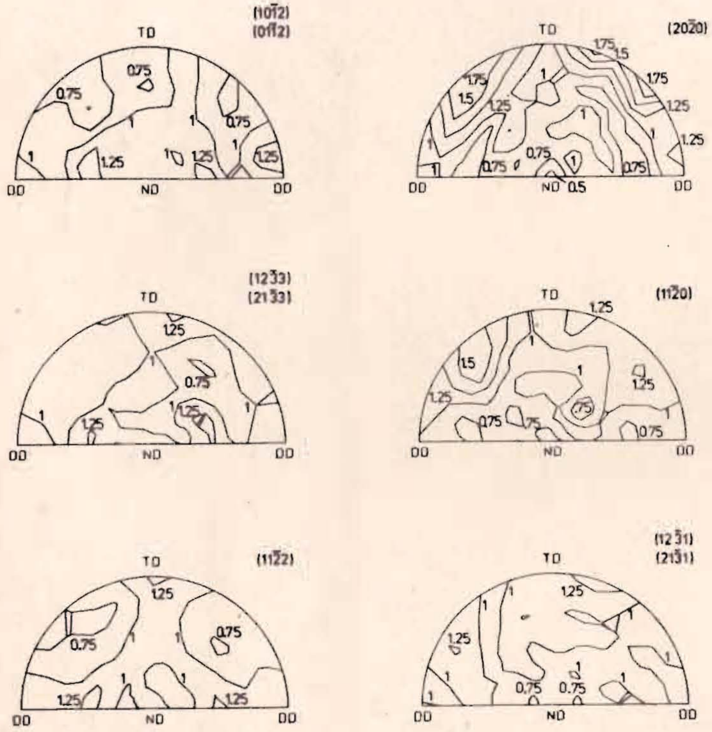


Fig. 8. Experimental pole figures of quartz crystallytes in granulite.

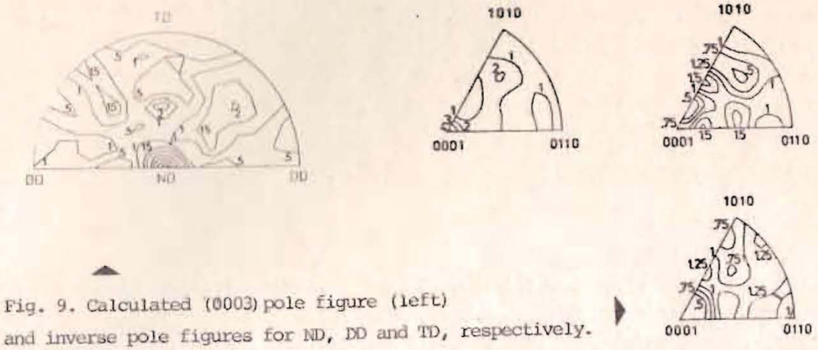


Fig. 9. Calculated (0003) pole figure (left) and inverse pole figures for ND, DD and TD, respectively.

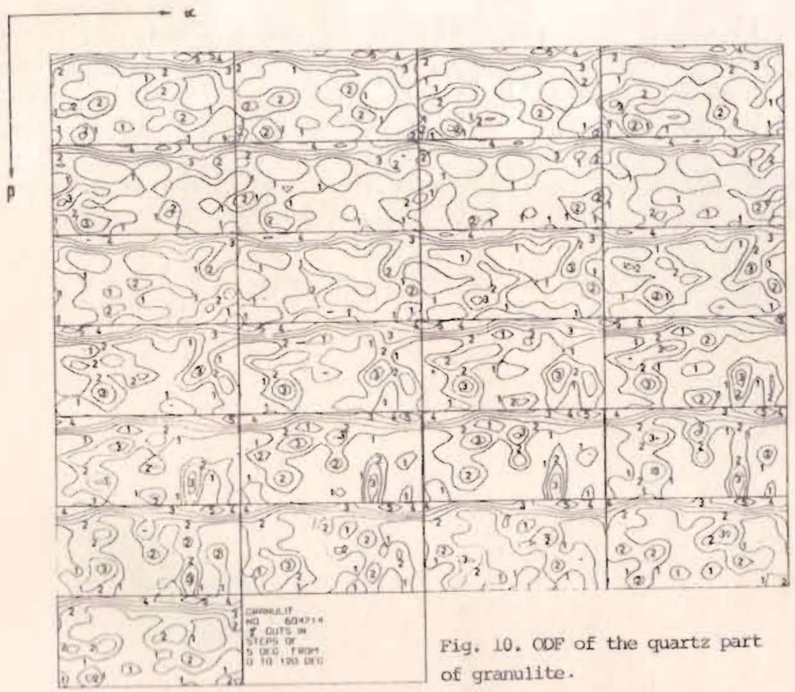


Fig. 10. ODF of the quartz part of granulite.

Summarizing, the proposed TOF texture analysis allows one to solve a wide range of volume texture problems. Because of the simultaneous measurement of all pole figures, the method is especially suited for investigation of low symmetric or multiphased substances. Furthermore, the constant scattering geometry of the TOF method enables to observe the texture formations caused by external influences, immediately.

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Использование нейтронной дифракции по времени пролета
для текстурных исследований

В данной работе описывается использование нейтронной дифракции по времени пролета для исследований текстур в поликристаллических твердых телах. Подробное сравнение со стационарным методом показывает преимущества, а также недостатки предложенной техники. Представляется текстурный дифрактометр НСВР, который вошел в строй на импульсном реакторе ИБР-2. Несколько выбранных экспериментов на металлических и геологических образцах демонстрируют хорошие возможности нейтронографического текстурного анализа по времени пролета.

Работа выполнена в Лаборатории нейтронной физики ОИЯИ.

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Feldmann K. E14-85-284
Application of Neutron Time-of-Flight Diffraction
to Texture Studies

In this paper the application of neutron time-of-flight diffraction to texture investigation on polycrystalline solids is described. A composition with the stationary method shows the efficiency but also some disadvantages of the proposed technique. The texture diffractometer NSWR is described, which was going into operation at the pulsed reactor IBR-2. Selected experiments on metallic and geological specimens demonstrate the efficiency of neutron texture analysis using time-of-flight technique.

The investigation has been performed at the Laboratory of Neutron Physics, JINR.

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