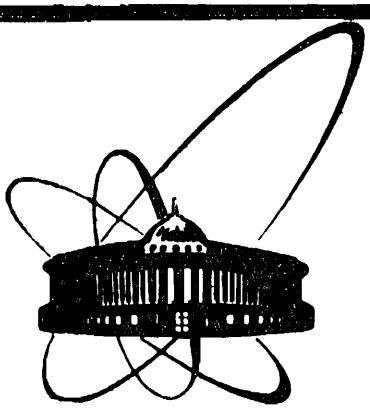

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E14-87-662

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TEXTURE INVESTIGATION BY NEUTRON DIFFRACTION

Submitted to VIII International Conference on Textures of Materials ICOTOM 8, Santa Fe, USA, 20-25 September, 1987.

1987

Introduction

Modern quantitative texture analysis is based mainly on spectroscopic methods like X-ray or thermal neutron diffraction. The low absorption of neutrons by the most of isotopes makes them well suited for bulk texture studies in large specimen volumes up to 10 cm^3 and more. Texture inhomogeneities are completely averaged. Therefore, neutron diffraction permits to investigate relatively coarse grained aggregates like those often found not only in petrofabric problems, but also in selected metallic materials. Furthermore, complete pole figures can be determined without special preparation techniques combining reflection and transmission geometry measurements.

At present, texture investigation facilities at various neutron sources are in operation. Different variants of the conventional angle dispersive neutron diffraction at stationary reactors are widely used, which are similar to the well-known diffraction of monochromatic X-rays. In the previous five years powerful pulsed neutron sources were put into operation (1,2). On this base the energy dispersive neutron time-of-flight diffraction could be demonstrated to broaden the spectrum of problems for texture investigations in the direction of low symmetric and multiple phase materials (3).

In the present paper a review is given of the commonly used experimental techniques for neutronographic texture studies. Some non-standard applications of neutron diffraction for preferred orientation measurements as well as potential directions of further development are discussed too.

Remarks on Neutron Diffraction

In this chapter some aspects of neutron scattering should be remembered, which may be important in connection with texture investigations. For more detail see monographs (e.g. (4,5)).

Neutron scattering takes place due to the interaction of neutrons with nuclei, but also of neutron spins with magnetic moments of atomic electron shells in the investigated matter. The differential elastic scattering cross-section consists in general of nuclear and magnetic coherent components as well as an incoherent part. For texture studies information can be obtained from the coherent scattering only. The incoherent part as well as inelastic effects contribute to the background.

For the most of isotopes the incoherent scattering is of low intensity. It is extremely predominant for hydrogen. Therefore, neutron diffraction measurements including texture studies at materials containing hydrogen like polymers and biological substances are very complicated. These difficulties can be avoided using deuterated specimens.

Magnetic diffraction can be expected only if there is a magnetic ordering of any kind in the studied material. In many cases the atomic and magnetic lattices coincide. Then, it is difficult to separate the nuclear from the magnetic component of the Bragg reflection. On the other hand a non-coincidence of magnetic and conventional lattice texture may lead to errors in the texture analysis.

Neutron diffraction is determined by the Bragg's law

$$\lambda = 2 d_{hkl} \sin \theta . \qquad (1)$$

There are two possibilities to satisfy this equation for discrete lattice spacings d_{hkl} leading to two different experimental techniques:

- The incident beam is monochromatic. A Bragg angle variation is carried out (conventional angle dispersive method).

- A polychromatic beam consisting of a certain wavelength spectrum is used at constant scattering angle 2θ (TOF method).

The absorption of neutrons by the most of isotopes is lower than that of X-rays by a factor of typically 10^3 to 10^4 (,6). Therefore, thick samples up to an order of centimeter can be investigated even in transmission geometry. By neutronographic methods the averaged volume texture is studied. Surface effects in general produce no influence. Consequently, the requirements to specimen preparation are very low.

Generals on Neutronographic Texture Analysis

In neutronographic texture studies complete pole figures can be determined combining transmission and reflection geometry measurements. Eulerian cradles or triple axis goniometers having one vertical and two mutually perpendicular axes are used for pole figure scanning. The point net may be an equal angle, an equal area or even any other one.

Although the absorption for neutrons is low, pole figure data have to be corrected for beam weakening and effectively irradiated volume at every sample position of the pole figure scan in general. This procedure may be avoided without loss in accuracy using spherical specimens (6,7). The absorption coefficient can be calculated correctly, if the sample is bounded by parallel planes like rolled metal sheets. In (8) the texture analysis errors are shown to be significantly less for neutron than for X-ray diffraction.

Furthermore, the detector opening should be large enough to accept the full diffraction intensity. Defocusing effects due to specimen rotations with respect to the primary and scattered beam give rise to strong correlations between detector aperture, area of the incident beam and permissible sample dimensions (9).

The large dimensions of neutron beam cross-sections and penetration depths permit to investigat the bulk texture of volumes up to several dozens of cubic centimeters. Thus, with neutron diffraction material having relatively large grain sizes, like transformer sheets or geological samples, can be measured and correlated with bulk properties.

Texture Analysis by Monochromatic Beam Methods

In Fig. 1 the lay-out of a normal angle dispersive diffraction experiment is shown. The monochromator crystal M selects a monochromatic neutron beam (narrow wavelength band) from the Maxwell-like energy spectrum outcoming from the moderator of the source. The energy and wavelength of the neutron are correlated by

$$\lambda = 2\pi h/\sqrt{2mE}, \qquad (2)$$

where m is the neutron mass and h -- the Planck's constant.

The maximum of the wavelength distribution is near to 1.5 Å for commonly used moderators. The chosen wavelength of monochromatic neutrons hitting the sample S determines the scattering angle 2θ at which the diffracted neutrons of the studied lattice spacing d_{hkl} are recorded by the detector.

For texture analysis the specimen is kept in a goniometer rotating it with respect to a pole figure scan. Thus, in such experiment the pole figures are measured point for point and one after another. The number of Bragg reflections, i.e. pole figures, being measurable in this method are confined by

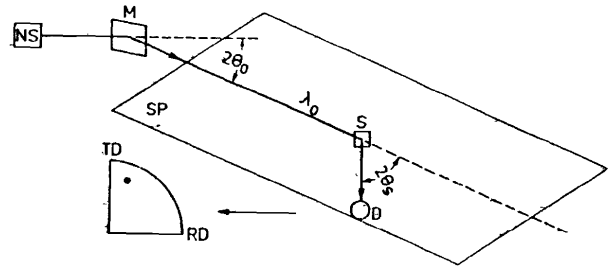

Figure 1. Lay-out of a diffraction experiment using the monochromatic beam technique. M-monochromator, S-sample, D-detector.

the neutron wavelength (Fig. 2). Equ. 1 can be satisfied only for reflections intersected by the const.-λ-line.

The detector opening is dimensioned in a way to encompass the full investigated intensity of the diffracted Bragg peak from the whole specimen area. However, no intensity from neighbouring reflections may be detected. A partial overlapping of peaks leads to errors which are out of control. This condition causes a further confine of the number of measurable reflections all the more, since the peaks broaden strongly with increasing Bragg angle (10). This may be understood from the decreasing angle between the λ =const. line and the curves representing Bragg reflections in Fig.2. The $\sin\theta/\lambda$ dependence known from X-ray diffraction is not operating in neutron-nucleus interaction.

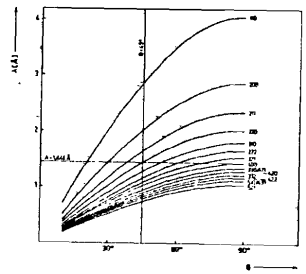

Figure 2. Bragg condition for a bcc-lattice. The horizontal line represents the angle dispersive and the vertical one - the TOF diffraction experiment.

Therefore, the described method is well-suited for texture analysis of high symmetric single phase materials requiring a relatively low number of pole figures. Especially convenient is the immediate measurement of pole density values (unnormalized). The exposition time for one pole figure point at a medium reactor (RFR 10 MW, CINR Rossendorf, GDR) varies from 10 sec to about one minute in dependence on scattering characteristics of the sample and on the pole figure under investigation.

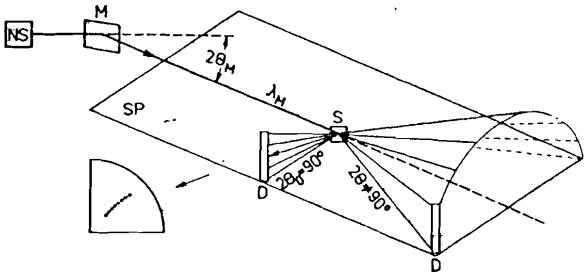

Figure 3. Intensity distribution measurement along a (hkl) Debye-Scherrer cone using a vertical PSD.

In practice the required time for a complete texture analysis measurement of a cubic metal sheet having orthorhombic specimen symmetry (3 or 4 single quadrant pole figures) is of an order of several hours. This is too long for selected problems. Efforts have been made at the Risø National Laboratory to fasten the measurements using a linear position sensitive detector (PSD) (11). In Fig. 3 the lay-out is represented. The PSD is situated perpendicular to the basic scattering plane, i.e. each detector increment forms its own scattering plane. The Bragg angle is exactly 45° for every increment. In such a geometry the Debye Scherrer cone degenerates to a plane perpendicular to the incident beam. Therefore, each detector section records neutrons diffracted at the same angle but representing different pole figure points (see Fig. 3). If $2\theta \neq 90°$, the diffraction cone bends away from the linear PSD. The data from different detector increments are not comparable. The Bragg condition is satisfied by wavelength variation of the incident monochromatic beam. This technique permits the very rapid determination of a one quadrant pole figure in the range of 7-15 minutes depending on the scattering cross section of the sample material. This speed has been used to study recrystallization kinetics and other texture forming processes (11).

In materials with low crystal symmetry having a large amount of peaks in the diffraction pattern the conventional angle dispersive method is not able to determine the necessary number of pole figures for ODF calculation because of multiple peak overlappings. This difficulty has been avoided at the ILL in Grenoble using a PSD which is situated in the scattering plane (see Fig. 4) (12). With such an equipment a section of the complete Bragg pattern is recorded simultaneously. Now a line profile analysis has to be carried out to determine integrated peak intensities being proportional to pole density values. Several pole figures are measured at the same time. Overlapped reflections can be separated. Totally coinciding pole figures are considered as superpositions in the pole figure inversion procedure (13).

But each Bragg reflection in one pattern corresponds to another diffraction vector, i.e. to another point position in its pole figure. If the pole figure corresponding to ϑ_m in Fig. 5 is completely scanned, all other pole figures contain blind areas like shown in Fig. 6. For these ranges additional scans have to be carried out.

Nevertheless the described method has opened the possibility for quantitative neutronographic texture analysis in low symmetric materials. Because of the simultaneous measurement of several pole figures the method is much more rapid, than the conventional angle dispersive technique.

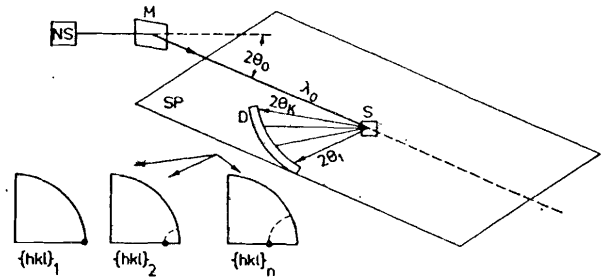

Figure 4. Application of a horizontal PSD to measure the range of the diffraction pattern.

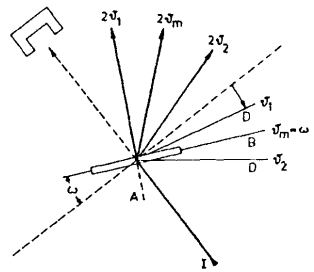

Figure 5. Incident and diffracted beams and the specimen position with respect to the diffraction vectors.

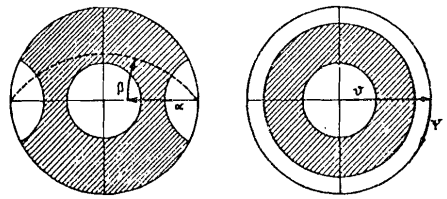

Figure 6. Blind areas in the pole figure for the Eulerian cradle (left) and the triple axis goniometer.

Texture Analysis by TOF Diffraction

The Bragg condition can be satisfied for various lattice spacings d_{hkl} at a fixed scattering angle, if the incident beam consists of polychromatic neutrons. Since there are no wavelength or energy sensitive neutron detectors, the time of flight to go a certain distance is measured to determine the energy of a given neutron. Pulsed beams must be used to do this. In Fig. 7 the lay-out of a TOF diffraction experiment is shown. If a neutron

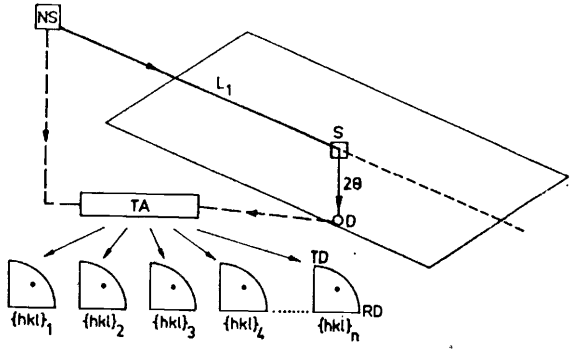

Figure 7. TOF diffraction experiment. TA-multichannel time analyzer, L_1-flight path of the primary beam.

with energy E starts from the source at the moment t_o, it is recorded in the detector at time t

$$t - t_0 = T = (L_1 + L_2)\sqrt{2m/E}. \qquad (3)$$

The detector signal is stored in a multichannel time analyzer (TA) which is started synchronously with each pulse emission. Thus, the spectrum of recorded counts is built up in dependence on neutron flight time. The relation between the time of flight, total flight path and wavelength is given with respect to equ. 2.

$$\lambda = T/[\beta\cdot(L_1+L_2)] \qquad \beta = 2.528\times10^{6}\ \mathrm{sec/m^2}. \qquad (4)$$

Considering the vertical constant - Θ line in fig. 4 representing a TOF experiment, all nonforbidden diffraction peaks are recorded at fixed Bragg angle simultaneously. The low index reflections are favoured for investigation, because the measured intensity is proportional to λ^4 in the TOF method. Moreover, the resolution of peaks improves with increasing wavelength.

In texture investigation each sample setting results in one point at the equivalent position in every pole figure under consideration (see Fig. 7). The pole density values are determined by the line profile analysis of the corresponding Bragg reflections. Overlapped peaks may be separated, where the number of separable reflections is strongly influenced by the resolution of the experimental equipment being determined mainly by the width of the neutron pulse and the length of the flight path. Collimation effects are of less importance.Moreover, the number of separable pole figures is restricted by the efficiency of the available fit program and the lattice symmetry of the investigated sample.

In the TOF method pole figures from all deconvoluted reflections are

measured simultaneously by only one scan. Therefore, the experimental expense is nearly independent of the required quantity of pole figures, i.e. the TOF technique becomes more efficient, if the number of necessary pole figures increases. Consequently, the method is especially suitable for preferred orientation studies in samples having low lattice symmetry or in multiple phase materials like those often found in geological specimens (14).

The representation of reflections as well as background by several points increases the statistical reliability of the pole figure data. This advantage can be used to provide very accurate texture investigations.

In TOF diffraction extended area detectors and large cross section beams are available to investigate relatively coarse grained materials. At the JINR Dubna a 5 x 17 cm^2 beam is to go into operation, especially, for preferred orientation studies in metamorphic rocks.

For the determination of pole density values a computer fit of Bragg reflections has to be carried out. To do it the diffraction patterns must be normalized with respect to wavelength independent neutron flux. The wavelength distribution of the primary beam is reflected by the spectrum of an incoherent scatterer like vanadium. If the incoherent cross section of the studied specimen is not too low, in TOF texture analysis the incoherent part of each spectrum can be used for normalization. In this case the influences of weakening and effectively irradiated volume as well as possible detector and source fluctuations are eliminated also. Multiple diffraction effects, which are increasing path of neutrons in the sample, remain uncorrected because the incoherent scattering does not depend on them. If this procedure is applicable, the requirements to sample preparation are decreased furtherly.

Comparing with monochromatic beam methods the TOF texture analysis is less convenient concerning the amount of experimental data and its handling. For each sample setting in the order of 10^3 points of the corresponding spectrum have to be stored. Pole density values are determined only via line profile analysis of diffraction patterns. On the other hand TOF diffraction may be applied to solve a very wide spectrum of preferred orientation problems.

At the present texture diffractometer of the IBR-2 pulsed reactor in Dubna (15) the exposition time for one sample setting varies from some minutes for metals like iron and copper to about one hour for complex low symmetric metamorphic rocks. To fasten the measurements similar considerations may be done as in monochromatic beam techniques. Using the geometry equivalent to that one in Risø the detectors have to be situated on a circle around the primary beam to avoid flight path differences. The application of discrete counters on the circle segment instead of quasi-continuous PSD diminishes the amount of experimental data. Points are measured like shown in Fig. 3 at equivalent positions in all considered pole figures. Therefore, the detector positions should be chosen with respect to the point net on the pole figure. The pole figure tilt angle depends on the angle between detector and horizontal scattering plane in a nonlinear way. Consequently, the counter areas have to be changed strongly from one to another to ensure equal pole figure windows. Up to now, this geometry for TOF texture facilities is under discussion only.

A further possibility to fasten the measurement is the application of several detectors situated in the scattering plane at different Bragg angles. In opposite to the PSD in Fig.4 every counter records the complete diffraction pattern. Therefore, the detector positions may be determined with respect to the pole figure point net in a way to rotate the specimen only around the vertical goniometer axis and the specimen normal for a com-

plete pole figure scan. This geometry avoids the strong defocusing at tilting around a horizonal axis. Pole figure points at different tilt angles are corresponding to various Bragg angles. Therefore, a mutual calibration of them is necessary. At the JINR two detectors are used at present (16). In the nearest future the measurements will be carried out with six counters simultaneously.

Inverse Pole Figure Measurement

A TOF diffraction pattern consists of all nonforbidden Bragg reflections of the investigated sample measured at constant scattering geometry. This means, the information of one TOF spectrum is equivalent to that of the inverse pole figure for the corresponding specimen position. Unfortunately, the available number of separable reflections and their inhomogenous distribution over the inverse pole figure range complicate sufficiently the procecure of mathematical texture analysis. The majority of experimental points is situated on symmetry lines.

For certain crystal symmetries like the trigonal lattice the measurement of inverse pole figures is not possible at all. Reflections of the type {hkil} and {khil} are not symmetrically equivalent, although they have the same lattice spacing. They coincide completely in powder diffraction patterns.

Nevertheless, the constant scattering geometry makes the TOF diffraction well-suited for the observation of texture component formation in in-situ experiments. Fig. 9 shows the behaviour of four low index reflections of copper during the recrystallization process (17). The exposition time of 30 minutes per spectrum is too long to observe technically relevant processes in general. Recently, times of less than 1 minute have been reported to observe the reaction kinetics of hydration of $Ca_3Al_2O_6$ at the IBR-2 reactor (18). Such experiment seems to be promising for in-situ texture investigations as well.

Other Applications

The line profile analysis is applied to determine pole figure values in the TOF technique and the monochromatic beam method combined with a PSD in the scattering plane. The necessary computer program may be used to fit the position, the height and the line width of the studied peak too. Therefore, these methods include the principal possibility to extract information on residual stress anisotropy of the investigated sample besides the texture data from diffraction patterns. Special efforts have to be made to ensure sufficient resolution parameters of the experimental equipment.

A non-destructive method to measure texture at different depths using a masked off technique with neutron absorbing foils (Gd, Cd) has been proposed in (19). In (6) a critical discussion has been provided. No further applications of this method are known.

Attempts have been made to apply neutron diffraction for texture investigations of the magnetic sublattice in magnetically ordered materials (20). there are two methods to separate nuclear and magnetic peaks:

- The magnetic intensity decreases with increasing $\sin\theta/\lambda$, i.e. the difference in various order pole figures is expected to represent the pure magnetic texture part. Corresponding careful experiments have been done without satisfactory results. Extinction effects seem to be the reason.
- The magnetic diffraction can be suppressed completely, if the specimen is magnetized up to its saturation with magnetization direction parallel to the diffraction vector. The difference in one pole figure measured without and with the field, respectively, represents its magnetic component. Of course, such experiments are not reproducible. Furthermore, a diamagnetic goniometer must be used to avoid strong forces in the saturation field.

Conclusions

Thermal neutron diffraction is shown to be an efficient tool for bulk texture analysis via pole figure measurements. The method is applicable for the most of materials with the exception of hydrogen containing substances, e.g. of polymers. Neutron diffraction is not an adequate technique to investigate surface textures and texture inhomogeneities.

At present, a number of different experimental methods is in operation. The conventional angle dispersive technique is widely used and most convenient in data handling. Its main drawback is the restriction on the consideration of high symmetric materials. This method permits to solve a wide range of texture problems in metallurgy and metal physics as well as a number of questions in petrofabric analysis. The application of one dimensional position sensitive detectors may fasten the number of measurements significantly. If it is situated in the scattering plane, low symmetric materials can be investigated, too. Some inconveniences in pole figure scanning and a more expensive data handling procedure have to be accepted.

The neutron time-of-flight diffraction can be widely used , especially to investigate low symmetric and multiple phase materials up to rather complex sample compositions. It avoids the pole figure scanning drawbacks of the angle dispersive technique with PSD. The application of discrete multidetectors decreases the time for measurements without difficulties in pole figure scanning.

Very short exposition times in both methods are promising to observe the kinetics of texture formation caused by external influences immediately.

Magnetic texture studies are confronted by methodical difficulties up to now. Further progress remains to be waited for.

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Фельдманн К.
E14-87-662

Определение текстуры с помощью нейтронной дифракции

В обычной угло-дисперсивной дифракции нейтронов используются монохроматические нейтроны. Полюсные фигуры сканируются друг за другом. Стандартный вариант этого метода ограничен исследованием неперекрывающихся в дифрактограмме отражений. Обсуждается использование позиционно-чувствительных детекторов. В нейтронной дифракции по времени пролета импульсный белый пучок нейтронов позволяет измерить одновременно все брэгговские отражения при постоянном угле рассеяния. Описываются основные характеристики этого метода. Время-пролетная дифракция является адекватным методом для исследования низкосимметричных или многофазных образцов, в частности, геологических материалов, требующих большого числа полюсных фигур для математического текстурного анализа. Одновременное измерение всех брэгговских отражений соответствует информации обратной полюсной фигуры. При условии малых времен измерений этот подход можно использовать для исследования развития текстур во внешних взаимодействиях. С помощью магнитного момента нейтрона изучаются магнитные анизотропии в материалах. Обсуждаются два различных метода.

Работа выполнена в Лаборатории нейтронной физики ОИЯИ.

Препринт Объединенного института ядерных исследований. Дубна 1987

Feldmann K.
E14-87-662

Texture Investigation by Neutron Diffraction

In the conventional angle dispersive neutron diffraction a monochromatic neutron beam is used. The pole figures under investigation have to be scanned one after another. The commonly applied angle dispersive method is limited to the consideration of Bragg reflections being isolated in the diffraction pattern. The application of multidetectors or position sensitive detectors is discussed. In the neutron time-of-flight (TOF) diffraction a white pulsed neutron beam allows one to satisfy the Bragg law for all lattice spacing at a fixed scattering angle. The main characteristics of the TOF diffraction experiment are shortly outlined. In this method all non-forbidden Bragg reflections are recorded in one pattern simultaneously. The TOF technique is well-suited to study low symmetric or multiphased specimens, especially geological materials, requiring a large number of pole figures for mathematical texture analysis. Multidetector systems can be used to shorten the necessary time for experiments.

The registration of all Bragg reflections of fixed scattering geometry is equivalent to the information of the inverse pole figure for the corresponding sample position. Having short exposition times this approach can be applied for in-situ investigations. The magnetic moments of neutrons can be used to study magnetic anisotropies in materials. Two different techniques are discussed.

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

Preprint of the Joint Institute for Nuclear Research. Dubna 1987