JOURNAL OF MATERIALS SCIENCE 19 (1984) 3467–3476

Review
Texture studies using neutron diffraction

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Various aspects of the application of neutron scattering methods to texture studies are reviewed. The neutron method is compared with other methods of texture measurement and techniques of neutron diffraction registration of texture are discussed.

Examples are presented of the use of neutron scattering for texture examination in materials having various grain sizes and degrees of structural inhomogeneity. It is also demonstrated that the information about texture can be used in the discussion of the deformation and recrystallization processes in metals.

Neutron diffraction results are shown to be helpful in the examination of the influence of texture on the anisotropy of physical properties in materials. The possibility of neutron diffraction measurements of magnetic texture is reported. Finally the accuracy of texture measurements using the neutron method is discussed.

1. Introduction

The fiftieth anniversary of the discovery of the neutron by Chadwick is an occasion for discussion of its consequences. Its discovery initiated many new disciplines of science, whose development has provided us with many new experimental techniques. Amongst these is neutron scattering, developed almost 40 years ago, which is providing us with new information about the structure and dynamics of matter. Materials science has also profited from neutron scattering, and it is the application of neutron diffraction to texture studies that would like to be discussed here.

There is another anniversary connected directly with texture – thirty years ago (1953) the first neutron diffraction texture experiments were made by Brockhause [1]. Nothing more was done in texture studies using neutrons until 1968 when systematic texture investigations were started [2–7]. At present the neutron diffraction method is well established and is used to achieve a better understanding of deformation and recrystallization, and also to study the relationship between texture and anisotropy of various physical properties.

However, there are still other important goals which might be achieved using neutron diffraction in studying texture. Dynamic studies of texture changes under the influence of external parameters such as strain, temperature and magnetic field have yet to be explored and the TOF technique or position sensitive detector technique are especially suitable for such experiments. Also the use of polarized neutrons may provide important information about the changes in the statistical distribution of magnetic domains.

The word texture has different meanings in the different sciences of metallurgy, geology and biology. In this paper the metallurgical definition, where texture can be described by a crystal orientation distribution function (ODF), is the one that will be used.

In order to define the crystal orientation distribution function we have to choose two reference frames, one being the crystal reference frame, the other, the specimen's reference frame (Fig. 1). The orientation distribution function $f(g)$ can be defined as:

$$\frac{dV}{V} = f(g)\,dg \qquad (1)$$

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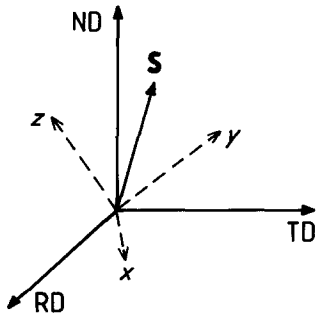

Figure 1 The definition of the crystal and specimen reference frames.

where dV is the volume fraction of the material in which crystallites are oriented in the interval $g, g + \Delta g$ in the Euler's angle space. The vector **S** originates from the origin of both reference frames. We use this vector in order to define the two-dimensional distribution function which can be measured. If the orientation of this vector is fixed in the crystal frame then the distribution of crystallographic direction in the frame of the specimen, the so-called pole figure, can be measured. If, however, this vector is fixed in the frame of the specimen, the distribution of the specimen direction in the frame of the crystal, i.e. the inverse pole figure, is defined. The three-dimensional ODF cannot be directly measured and usually has to be derived from measured two-dimensional functions, that is the pole figures or the inverse pole figures.

2. What neutron diffraction can offer in texture studies

Texture can be quantitatively described when there is reliable statistical information about the orientation of a sufficient number of grains. Usually the knowledge of orientation from volumes which contains 10 000 grains is sufficient. The ODF represents the relative volume of crystallites of a particular orientation referred to the reference frame of the specimen.

A knowledge of texture can be derived from experiments where the orientations of single grains can be measured, for example the Laue method for large grains or the Kossel and Kikuschi method, which can be used to measure orientations of small grains or subgrains. The use of methods of selective measurements of grain orientation can only seldom be justified. The preparation of specimens is difficult and the interpretation of results time consuming. Additionally, the time of collection of the necessary number of measurements of the grains' orientations is excessively high. The X-ray method is most often used in texture studies, but encounters difficulty when measuring a coarse grained specimen.

Neutron diffraction was introduced into texture studies mainly because the neutrons offer unique possibilities for measuring bulk specimens. The linear attenuation coefficients for neutrons are 100 to 1000 times smaller than for X-rays in the majority of materials, and because the cross-section of the neutron beam is much higher than that of an X-ray beam the volume investigated can be 10^3 to 10^4 times greater. This characteristic of neutron experiments is an advantage in a variety of texture studies which will be discussed later.

Another advantage of neutron diffraction over the X-ray method is the relative ease of performance of kinetic experiments. There are practical advantages, for example the fact that the neutron diffractometer is larger than an X-ray diffractometer, which allows stress–strain equipment or a furnace to be accommodated. This is a particular advantage when time-of-flight (TOF) or position sensitive detector registration is used. Also, most of the constructional materials are relatively transparent to neutrons.

Texture studies using the TOF technique will profit enormously if the Spallation Neutron Sources (SNS) or pulse reactor comes into operation. The use of several neutron counters can be envisaged, and since each provides us with the neutron diffraction pattern characterizing the different inverse pole figure, texture data can be measured and used for ODF determination. Such information can be registered in a short time (order of seconds) and this makes it possible to follow texture transformation. Another obvious advantage of measuring texture using SNS is its wide range of energies. The typical energy spectra of slow neutrons produced by the moderator, depending on its temperature, is between 0.001 and 1 eV, which makes it possible to register a high number of diffraction maxima.

Speculation about the application of the SNS can be extended even further if we consider the use of position sensitive detectors (PSD) in such studies. Such detectors using the fibre optic coded scintillation technique may have a spatial resolution of 3 mm. The detector which is designed for the powder diffraction instrument to be built at the Institut Laue Langevin in Grenoble has

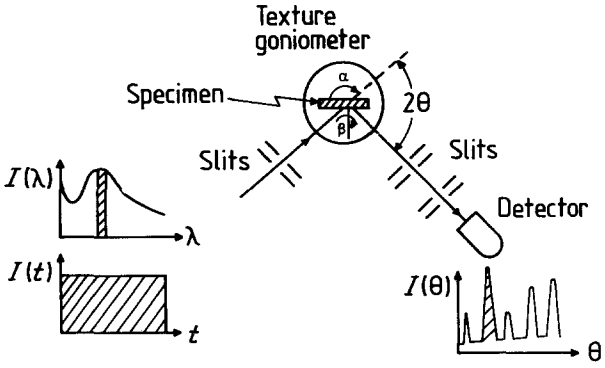

Figure 2 Principle of the classical pole figure measurements.

100 elements, 3 mm x 150 mm on a radius of 1800 mm, and will have 1600 elements.

A position sensitive detector working in the TOF mode will solve all the problems of fully quantitative dynamic texture analysis, even in multi-phase materials. The information about texture can be gathered simultaneously in the reference frame of the specimen and in the reference frame of the crystal. This is because the data may be gathered in the reference frame of the specimen, since the scattering vector changes its orientation in the reference frame of the specimen for the registration at various positions of PSD.

Additionally, TOF registration provides, for each scattering vector fixed in the specimen reference frame, information which characterizes the distribution of the specimen direction in the reference frame of the crystal. This data, when analysed, provides an enormous amount of information about texture. Additionally, the statistical sampling of the grains can be improved by rotating or oscillating the specimen. When this is done the geometry of the experiment does not change while the beam scans different areas of the specimen.

When applying neutron diffraction in texture examination one has to remember that X-ray methods are the most frequently used in such studies. The choice of the method depends on the structure of the investigated material and on the problem which is being studied. In this sense a coarse grain structure precludes the use of X-ray methods, but for studies of the problem of texture inhomogeneity in rolled materials, the X-ray method is more useful.

3. Present methods of registration of texture data

There are several possible means of recording diffraction maxima. There are three variables in the Bragg equation and two of them (scattering angle and wavelength) can be changed continuously while the interplanar spacing is a discrete variable.

The angular dispersive method is applied to measure pole figures as illustrated in Fig. 2. The scattering vector coincides with the vector **S** which is fixed in the crystal reference frame, i.e., the registration of diffraction at constant θ_{hkl} is made. By rotation of the specimen the orientation distribution of the $[h\,k\,l]$ crystal direction in the frame of the specimen is measured.

Various experimental techniques are used in the measurement of the pole figure of the rolled material; the combined reflection transmission technique [5], the spherical sample method [2], and the back reflection method [8]. The principle, of the data collection is illustrated in Fig. 3.

In the case of the spherical sample method the entire specimen is in the path of the neutron beam and the attenuation of the radiation is low. The spherical specimen can be substituted by a cylindrical specimen. It has been demonstrated [9] that using various shapes of specimens – cylindrical, spherical, composite and plates – one obtains about 30% change in the maximum of the ODF.

Recently methods for pole figure measurement using a position sensitive detector have been described. Two possible uses of the detector have been reported [10, 11].

One possibility [10] is illustrated schematically in Fig. 4. The detector is used here to record the diffraction spectrum, and several diffraction maxima can be examined simultaneously. This allows one to separate the overlapping peaks and measure the intensity in a number of pole figures simultaneously. The intensity obtained from a diffraction pattern for a specific orientation of the specimen corresponds to different places on the pole figures. A reduction of the time necessary for pole figure measurements is thus achieved.

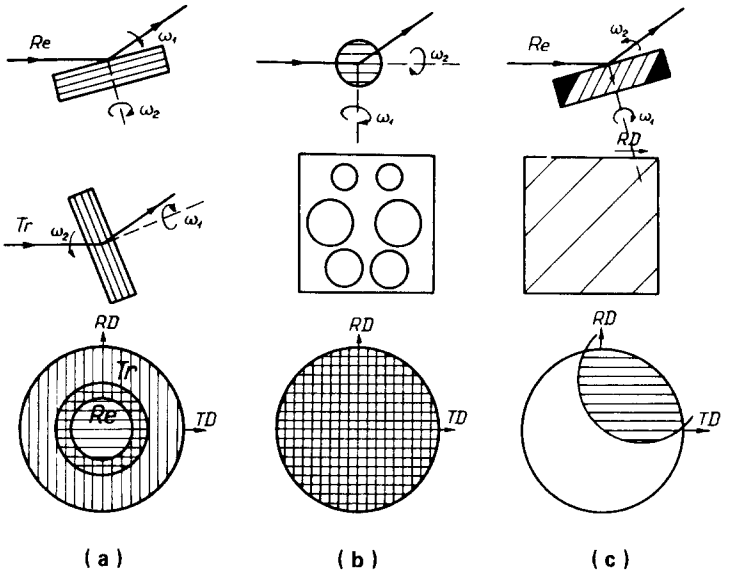

Figure 3 The different experimental techniques used in neutron diffraction measurements of sheet texture. (a) Transmission–reflection method; (b) spherical sample method; (c) back-reflection method. Parts of the pole figure measured using the given experimental techniques are marked accordingly.

The other means of measurements is presented in Fig. 5. The wavelength is selected by a curved pyrolitic graphite crystal monochromator. The diffracted beam is registered by a linear position sensitive detector which is placed with its axis vertical to the incident beam. An appropriate wavelength is selected which allows a measurement of the intensity distribution along the Debye–Scherrer cone at $2\theta_{hkl} = 90°$.

A set of measured intensities along the detector corresponds to a part of a small circle in the pole figure as shown in Fig. 5. By rotating the sample a complete pole figure can be obtained. The main advantage of the method is in limiting the number of rotations necessary to cover the area so that the time necessary for the measurement of the pole figure is shorter. The possibilities of studies of certain slow kinetic processes are envisaged.

Since the first application of the time-of-flight method in texture studies [7] little has been done. However recently [12] more systematic studies of texture using TOF have started in Dubna with the aid of a pulse-reactor.

In TOF measurements where specimen and counter do not move [13], the vector **S** is fixed in the specimen reference frame and consequently the information about texture can be obtained when a fixed direction of the specimen coincides with the scattering vector. Such data represent an inverse pole figure (Fig. 6).

If the specimen is rotated while the pole figure is measured the simultaneous recording of several pole figures may be possible [13]. The whole diffraction pattern is recorded simultaneously and if necessary the separation of overlapping reflections can be made. This is an advantage over

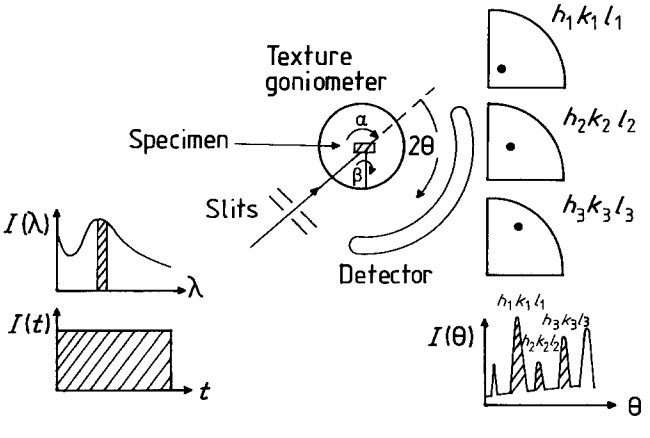

Figure 4 Schematic view of texture measurements using a position sensitive detector. Diffraction spectrum as a function of 2θ is measured.

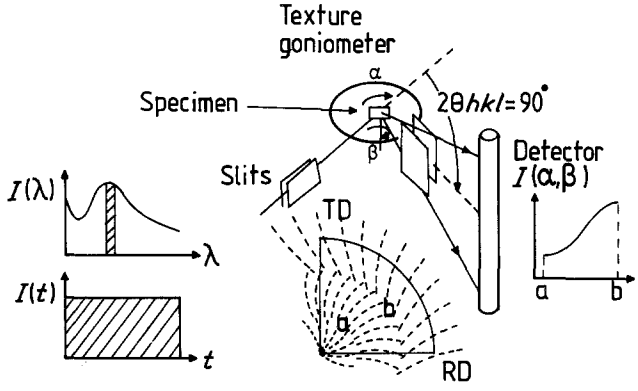

Figure 5 Schematic view of texture measurements using a position sensitive detector. Scanning over a circle in an $\{h\,k\,l\}$-pole figure.

the traditional angular dispersive registration where such reflections in a measured pole figure cannot be separated.

The determination of the ODF requires data about several pole figures, and the TOF method provides this. However, most interesting is the constant geometry for the inverse pole figure measurements. Various geometries of texture measurements for the TOF method have been discussed [13]. With a constant geometry it is easy to investigate the kinetics of texture changes with time and temperature and such heavy equipment as magnets, cryostats and compression equipment can be easily installed.

4. Examples of neutron diffraction texture of various materials

The most attractive property of the neutron diffraction method is to offer much better statistical results due to the increased number of grains involved. This advantage may be illustrated by experiments made on transformer steel. The maximum grain size in the first specimen in Fig. 7 is 20 mm. In spite of such a large grain dimension the pole figure has been determined using a combined transmission–reflection technique. The pole figure shows the strong Goss texturing of this sample. In the next specimen (Fig. 7b), one observes the coarse grain size and the colonies of small grains. Neutron diffraction is superior to the X-ray method in such a case. Traditionally only the Laue technique can be used to determine the orientation of each grain separately. Discussing these results one has to remember that the ODF represents the volume of crystallites of a particular orientation in Euler space, thus the information about texture should represent data about the volume of crystallites having a particular orientation. The Laue technique or other selective measurements on single grains gives results which are not suited to the ODF calculation, especially in the case of a second specimen.

Another advantage of using neutron diffraction is in the examination of weak textures. The difference between maximum and minimum intensity in a specimen of copper–zinc which was cold-rolled and annealed (Fig. 8) is only 0.5 in random units. The mean grain size was 1 mm, which means

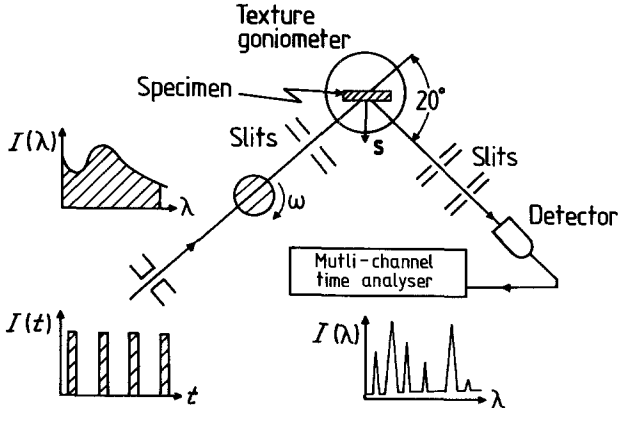

Figure 6 Principles of TOF texture measurements.

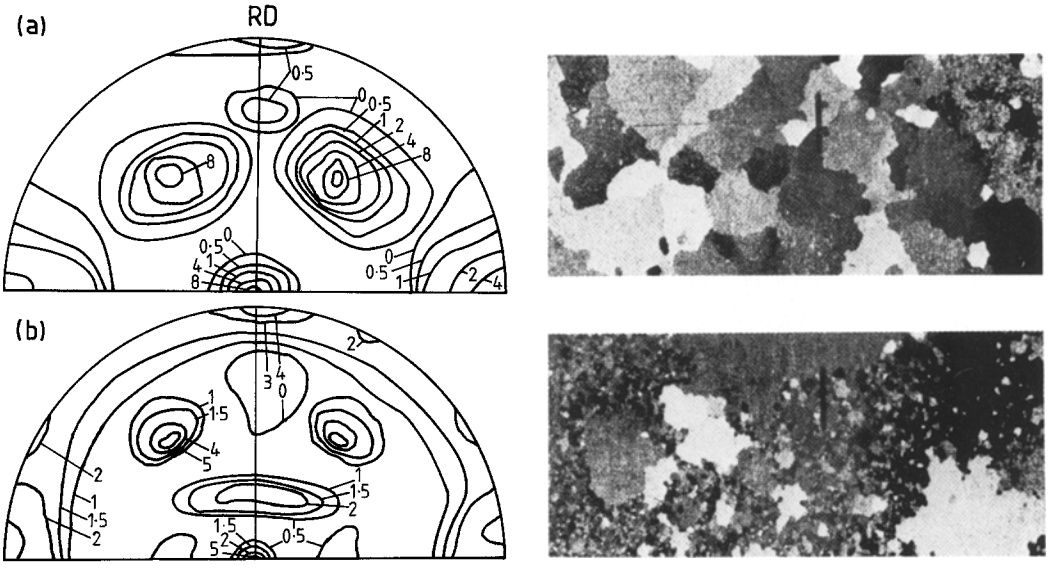

Figure 7 {1 1 0} pole figures obtained for iron–silicon steel sheets [8].

that no other technique of measurement can give reliable results.

Studies of materials having inhomogeneous structure can also be made. For such materials the measurement of bulk specimens having a volume of a few cubic centimetres are of crucial importance. Fig. 9 shows the pole figure obtained for an industrial graphite [14] in which well graphitized particles of coke are joined together and the whole structure is cut across by pores and inclusions. The porosity for this specimen is 35%.

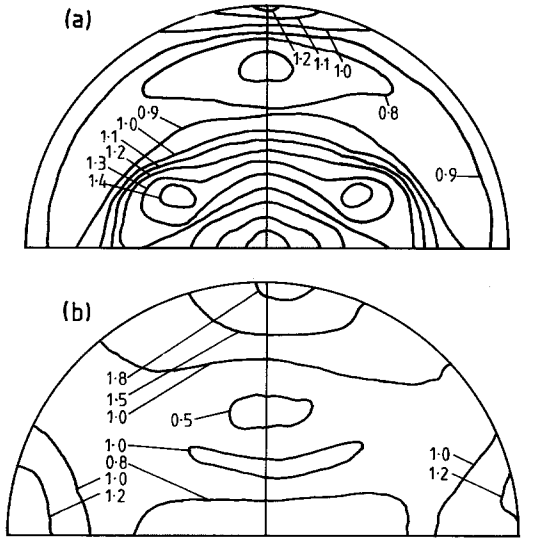

Figure 8 {1 1 1} pole figures for (a) brass Cu–Zn 20, (b) copper.

Another obvious application of the neutron diffraction technique is the measurement of mean texture in materials where texture does change. At the present stage of understanding of the correlation between texture and anisotropy the mean information is often more suitable. The results of studies of texture inhomogeneity in rolled aluminium [15] are presented in Fig. 10. The average texture A measured by the author is much less strong (4 units) than texture in the middle layer of the specimen measured by X-ray method (15 units).

These examples illustrate that among the

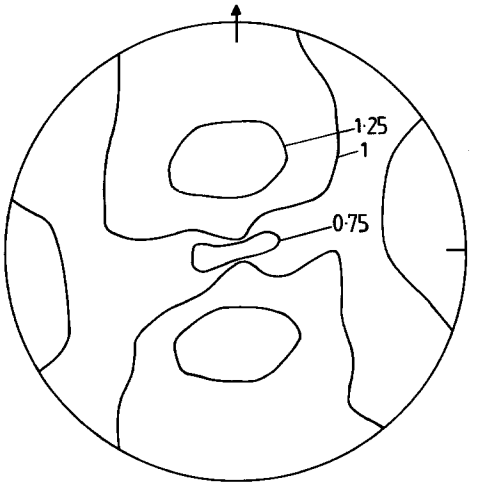

Figure 9 {0 0 2} pole figure for the graphite electrode.

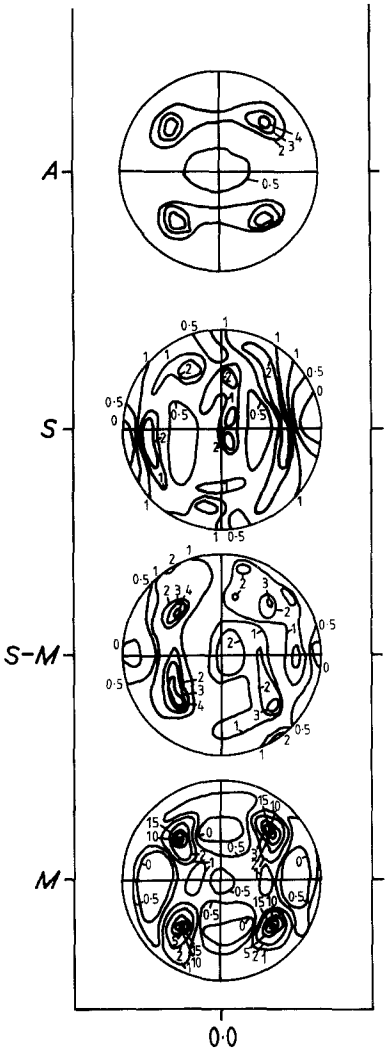

Figure 10 Studies of texture inhomogeneity in rolled aluminium by the X-ray method as shown by Truszkowski *et al.* [15]. S – texture in the surface layer; M – texture in the middle layer; A – the average through-thickness texture obtained by neutron diffraction [8].

variety of structures in polycrystalline materials there is a class of materials in which neutron diffraction texture studies are recommended.

Several investigations of the systematic texture changes due to cold-rolled deformation or recrystallization have been made using neutron diffraction [16, 17]. The advantage of using neutron diffraction in the study of deformation is that it gives better statistics for the number of grains. It also gives the possibility of acquisition of through-thickness texture in the materials which can later be investigated using other methods.

The disadvantage of using neutron diffraction in such studies is the fact that the information about systematic texture variation is lost. For example, the information about surface texture in cold-rolled sheets is suppressed by the mean information.

Neutron diffraction seems to be the most convenient method for studying recrystallization texture. This is partly because the recrystallization usually means that the material investigated will have a coarse grain structure and partly because it is technically easy to accommodate a furnace around the specimen. This makes it possible to study the kinetics of recrystallization by *in situ* texture measurements [18].

Neutron diffraction measurements are well adapted to provide the results about texture that are essential for studies of texture–anisotropy correlation. Also the non-destructive method of studying bulk samples offers the possibility of using the same specimen for further testing. It is appropriate to compare the through-thickness average information with the anisotropy of mechanical properties, because the mechanical test is made on specimens of the same thickness. For this reason diffraction was used [8] in studies of texture influence on the anisotropy of elastic properties and plasticity. Also, neutron diffraction enables the quantitative studies of the correlation between the anisotropy of the magnetocrystalline energy and the texture in iron–silicon steel [19]. No other method can offer the information necessary for the ODF calculation in materials where the mean grain size is of the order of millimetres.

Neutron diffraction was also used in texture studies [20] because the through-thickness average of the texture was required for the comparison with ultrasonic measurements.

5. Magnetic textures

When studying texture in magnetic materials the question arises whether the measured texture due to the nuclear scattering of the neutrons is the same as the texture due to magnetic scattering. It might be that, because of the internal strain in the investigated material, the magnetic texture is different from the crystalline texture. This may produce a maximum error of the order of a few per cent in texture results. A positive aspect of such an effect is that neutron diffraction provides a unique means of studying the statistical distribution of the magnetic domain vectors. An attempt to separate this magnetic texture from the crystal-

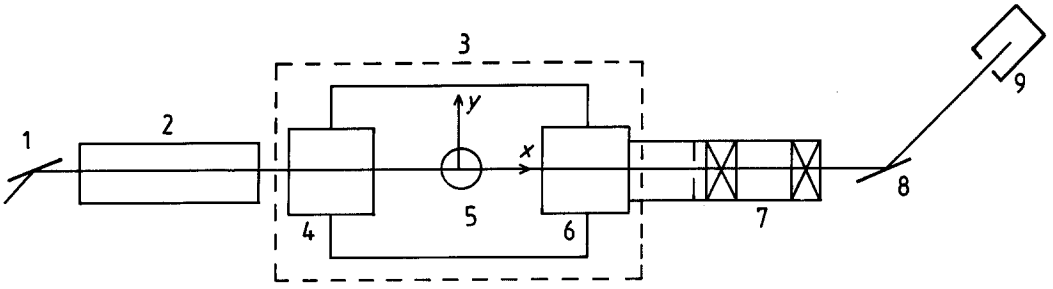

Figure 11 A diagram of the experimental apparatus as used by Akselrod *et al.* [22]. (1) Polarizer, (2) guide field, (3) magnetic screen, (4) input rotator, (5) sample unit, (6) output rotator, (7) spin flipper, (8) analyser, (9) detector.

line texture by comparing the results obtained for different orders of reflection [7], was unsuccessful. Neither did measurements of texture in magnetic fields [21] show significant changes in neutron intensity.

New possibilities of measuring magnetic texture exist using polarized neutrons [22]. The depolarization of neutrons depends on the anisotropic properties of the sample and can be used to study its magnetic texture. We will discuss here the results obtained by Akselrod *et al.* [22] for uniaxial magnetic anisotropy.

The neutron beam, which is polarized along its propagation direction (polarization vector P_0), falls on the thin sample at an angle ψ with respect to the magnetic anisotropy axis, which is characterized by a direction **n**. The depolarization of the beam is related to the direction of the magnetic anisotropy and to the parameter ξ characterizing its distribution.

According to [22] the depolarization of a beam is expressed by:

$$\Delta P = A\left\{\left[\frac{1+2\xi}{3} - \tfrac{1}{2}(\xi + C)\sin^2\psi\right] \times P_0 + (nP_0)\left[\tfrac{1}{2}n_\perp(\xi + C) - \xi n\right]\right\} \quad (2)$$

where A and C are numerical constants. If the x-axis coincides with the direction of the propagation of the neutron beam, the components of the vector ΔP are given by the following:

$$\Delta P_x = AP_0\left[(1-\xi)/3 + (\xi - C)/2\sin^2\psi\right] \quad (3)$$

$$\Delta P_y = AP_0\left[\cos\psi\, n_y(C-\xi)/2\right] \quad (4)$$

$$\Delta P_z = AP_0\left[\cos\psi\, n_z(C-\xi)/2\right] \quad (5)$$

These components change when the direction of the sample anisotropy is changed. For these changes it is possible to determine n_y and n_z which are the direction cosines of the magnetic anisotropy vector. The specimen may, for example, be rotated around the x-axis, the value ΔP_x will be constant, but the transverse components n_y and n_z of the ΔP vector varies. ΔP_z becomes zero for the vector **n** in the x–y plane and so if the specimen is rotated around the z-axis the ΔP_y component can be measured.

Consecutive rotations around the x- and y-axes makes it possible to orient the magnetic anisotropy axis of the sample parallel to either the x- or y-axis, and thus to determine the direction of the vector **n**. The parameter ξ is defined as

$$\xi = \tfrac{1}{2}(3\cos^2\theta - 1),$$

where θ is the angle between the magnetization direction and the anisotropy axis. This can be used to determine the distribution of magnetic moments around the magnetic easy axis of the specimen.

This parameter may vary between 1 and 0. An isotropic distribution of magnetic moments corresponds to $\cos^2\theta = \frac{1}{3}$. Estimation of this parameter gives the information about distribution of magnetic moments. The experimental set-up for the experiment shown in Fig. 11 was designed in order that the neutrons could be polarized either parallel or perpendicular to the beam. The component vector of the polarization of the transmitted neutrons could subsequently be analysed. For ferrites Ni_2W, the changes in value of the depolarization, ΔP, with rotation are shown in Fig. 12. The point A in Fig. 12 corresponds to the position of the sample when the texture axis lies in the x–y plane.

The method has been used for specimens showing uniaxial anisotropy and enables quantitative measurements of texture to be obtained. Neutron depolarization is also used [23] to obtain information about mean magnetization, mean

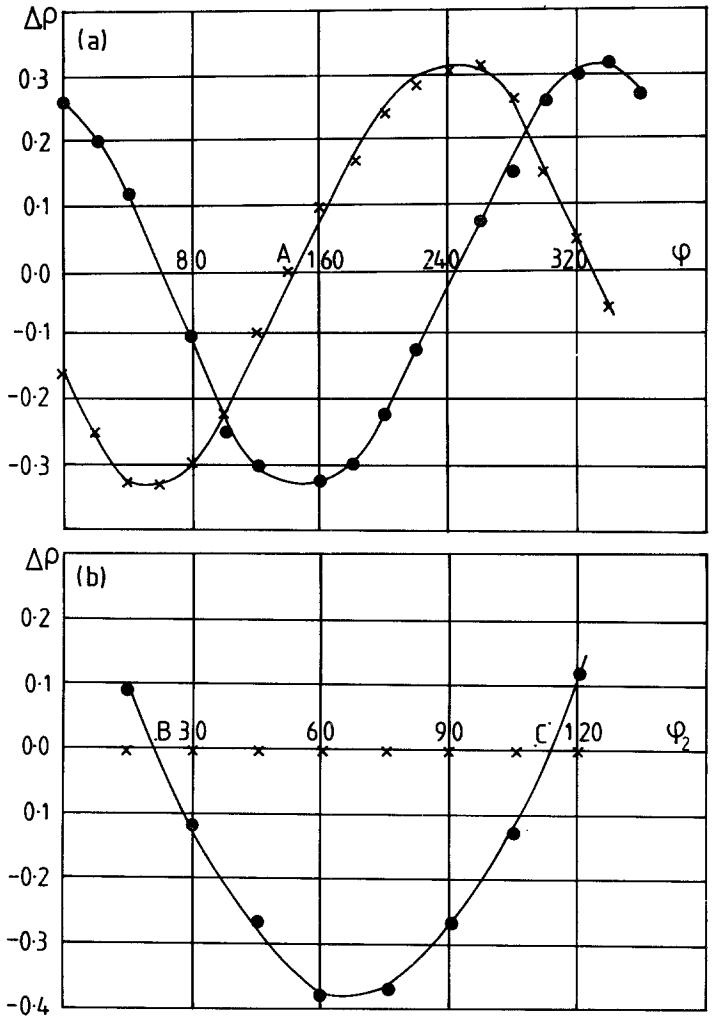

Figure 12 Variation of the transverse component ΔP with the angle of rotation of the sample. (a) The sample was rotated around the x-axis; (b) the sample was set in position "A", then rotated around the z-axis (dots: ΔP_y, crosses: ΔP_z) [20].

domain size, the mean square direction cosines of the inner magnetization, etc.

6. Discussion of the accuracy of neutron diffraction texture experiments

The accuracy of the neutron diffraction experiments on texture have to be discussed for various geometries of measurements and various techniques of registration. Accuracy might also be affected by the structure of the investigated materials.

Using traditional diffractometry in the examination of pole figures we observe changes of intensity of the diffraction maxima as a function of the specimen rotation. It is recommended that a considerable part of such maxima are registered, which means that good collimation is not required. We remember that when the diffraction from an anisotropic specimen is measured there is always a possibility that there exists anisotropy of internal stresses, stacking faults, inclusions, etc.

In transmission or reflection measurements of the pole figure the beam diffracted by the specimen changes its width while the specimen is rotated and a broadened diffracted beam might not be entirely registered by the counter. Also, absorption corrections are necessary if this method of pole figure registration is used. These absorption corrections are calculated in a similar manner to those for X-ray measurements. The linear attenuation coefficient is the sum of the linear absorption coefficient and the linear scattering coefficient. The coherent part of the linear scattering coefficient does change with the specimen position, and so correction formulas should be used [24]. The use of spherical specimens simplifies the corrections. However primary extinction corrections might be necessary in the case of anisotropic grain shape with high perfection of grain structure. Often, however, the rotation of a spherical specimen around the scattering vector might be used

to find these corrections experimentally [21]. A slight rolling reduction of iron–silicon sheets is also helpful [7]. Such a reduction does not change the texture but destroys the perfection of the grain structure. The problem of correction to experimental data and its accuracy should be discussed, bearing in mind the accuracy of the mathematical formulation that is used for quantitative texture description.

Serious difficulties were discovered [25] that affect the accuracy of the calculation of the ODF from the pole figures. The experiment is partly blamed for these difficulties. The series expansion of the texture function should contain terms of odd order which are blotted out, in the diffraction experiment, by Friedel's law in non-centrosymmetric crystals or by the presence of the inversion centre in the crystal symmetry. A suggestion was made [26] that anomalous scattering might be used for pole figure measurements. Friedel's law, requires $F_{\overline{hkl}} = F_{hkl}$ to be broken in the vicinity of an absorption edge. Such an experiment may allow determination of the odd order coefficients only for very rare classes of crystals.

7. Conclusions

The application of neutron scattering to texture examination has proved that the method can be recommended in studies of coarse grained materials, porous materials in bulk specimens and in all materials which have an inhomogeneous structure or show a low degree of texture development.

The neutron diffraction method was used to measure the through-thickness average of the texture in specimens. Such information is often required in studies of the influence of texture on the anisotropy of various physical properties.

Neutron diffraction texture experiments can be performed under external stress and a furnace, cryostat or magnet can easily be accommodated around the specimen. The kinetics of the recrystallization or deformation can be examined. The TOF method is particularly suitable for such studies.

Neutron scattering offers the possibility of measuring the statistical distribution of magnetic domains, the so-called magnetic texture.

Acknowledgement

I have the pleasure of thanking S. Barnett and M. Hill for correction of the manuscript.

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Received 30 June
and accepted 1 November 1983