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GEOMETRY OF TEXTURE MEASUREMENTS FOR DISPERSIVE METHODS

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Abstract: The application of energy-dispersive neutron and X-ray methods in texture studies is briefly reviewed and discussed. New geometries of texture measurement using energy-dispersive methods with stationary counters and specimen are suggested. Depending on the number of counters the experimental data can be sufficient for the orientation distribution function (ODF) or the ideal orientations determination. Coarse-grained materials can be studied not only by neutron diffraction but also by using the energy-dispersive X-ray method. Studies of texture changes under the influence of temperature, time and external stresses can also be conducted conveniently. The energy-dispersive X-ray method may readily be adapted to industrial texture control.

INTRODUCTION

Unlike angular-dispersive diffraction methods which have been utilized in texture studies for a long time, energy-dispersive methods are not very well known. The main purpose of this paper is to stress the particular advantages of using energy-dispersive methods in texture examination.

The first energy-dispersive experiments for texture studies were made in 1968 by Szpunar et al.,[1] who used neutron diffraction. The energy-dispersive X-ray experiments, suggested by the author[2] have a more interesting history. The X-ray method was used by Laine[3] to measure preferred orientations in splat-cooled Cadmium. Szpunar, Ojanen and Laine[4] used this method to obtain information about inverse pole figures in a rolled sheet. Next, the method was used by Gerward, Lehn and Christiansen[5] to demonstrate its applicability to the *in situ* studies of texture changes during the tension test. This was followed by Laine, Kivila and Lahteenmaki,[6] who investigated the preferred orientation on integrated X-ray intensities in powder specimen. And most

recently, Szpunar and Gerward[7] demonstrated that the method can be used for simultaneous recording of several pole figures in Alpha and Beta Brass.

ENERGY DISPERSIVE METHODS

The main difference between angular-dispersive methods and energy-dispersive methods is, by definition, the difference in the manner in which the diffracted radiation is registered. It is well known that in the Bragg diffraction condition there are three variables, two of which can change continuously (θ and λ), and the third one (d_{hkl}), changes discretely, depending on the crystal structure. In order to fulfill the diffraction condition, usually one of the continuously changing variables is fixed. The wavelength, λ, is fixed in the traditional angular-dispersive method and the Bragg angle, θ, in the energy-dispersive method; and the intensity of radiation is registered as a function of θ or λ, respectively.

The principle is the same for X-ray and neutron diffraction. The experimental set-up is illustrated in Figures 1

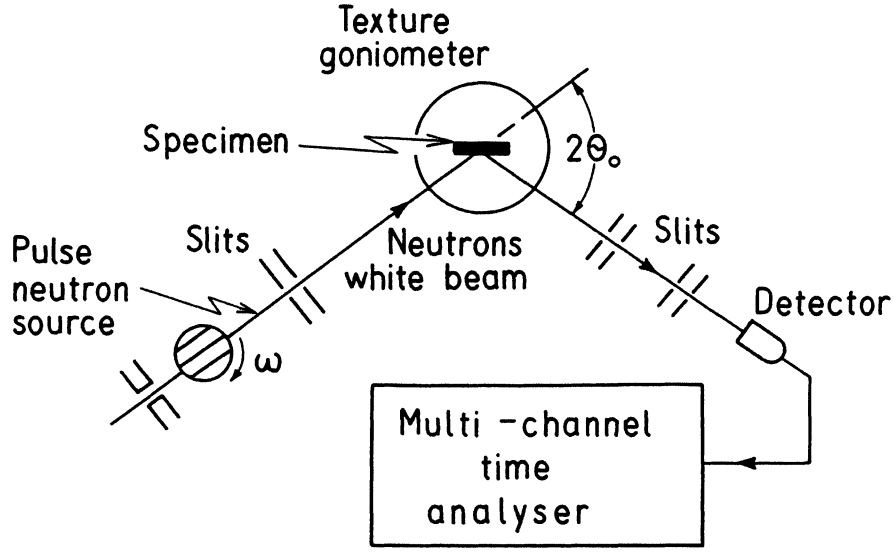

Figure 1. Principles of the time-of-flight neutron diffraction method.

and 2. In neutron diffraction experiments, an inherent pulsed source, i.e., pulsed reactor, linear accelerator or steady state nuclear reactor with a beam chopped by a conventional chopper, a statistical chopper or a Fourier chopper, are used. The time of flight of neutrons is registered in a multichannel analyser, and the wavelength and the energy of neutrons can be determined. Using some new neutron sources such as the pulse reactor in Dubna or the Harwell Linac, the neutron diffraction pattern can be gathered in a few seconds.

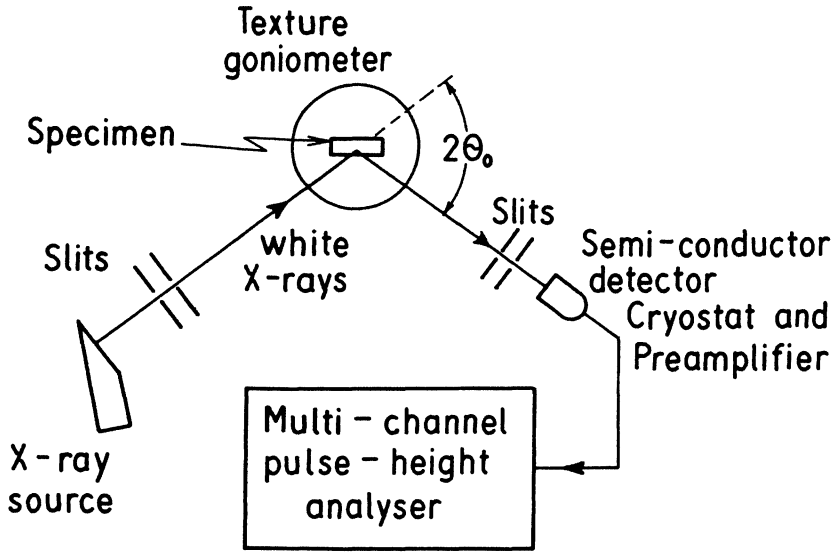

Figure 2. Principles of the X-ray energy-dispersive method.

In Figure 2, we show the principle of X-ray energy-dispersive measurements. Usually, the bremsstrahlung (continuous or white radiation) of an X-ray tube is used. The whole spectrum of X-ray energy irradiates the sample. The diffracted intensity is measured with a semiconductor detector and registered in a multichannel analyser. A very sensitive point of the energy-dispersive X-ray method is the energy resolution of the semiconductor detector. The conditions for obtaining optimum resolution were discussed by Buras, Nimura and Staun-Olsen.[8] The resolution of the energy-dispersive method is not as good as the resolution of the angular-dispersive method. The uncertainty of the position of the diffraction maxima on the energy scale depends not only on the quality of collimation but also on the resolution of the detector. For the usual collimation of $(10^{-3}-10^{-4})$ radians, using Silicon or Germanium detectors in the whole θ range, the full width at half-maximum in the d scale is about 10^{-3} Å.

This resolution is sufficient for purposes of structure and texture examinations. With X-ray tubes which were used up to now in texture studies, 5-10 minutes were required for one diffraction pattern to be measured. This time will be shorter with high-power generators and X-ray generators with rotating anodes. New interesting experiments can be made using synchrotron radiation. The time of the data collection for one diffraction pattern is of the order of seconds or even shorter, but a more sophisticated detection system is required in order to make use of the high intensity of synchrotron radiation. Another advantage of using synchrotron radiation in texture studies is the wide range of useful energies in the interval from 0.01 keV to 100 keV. Synchrotron radiation has a smooth spectrum and is free of undesirable characteristic radiation. The wide range of energy makes

it possible to register much higher numbers of diffraction maxima in one diffraction pattern, as shown in Figure 3.

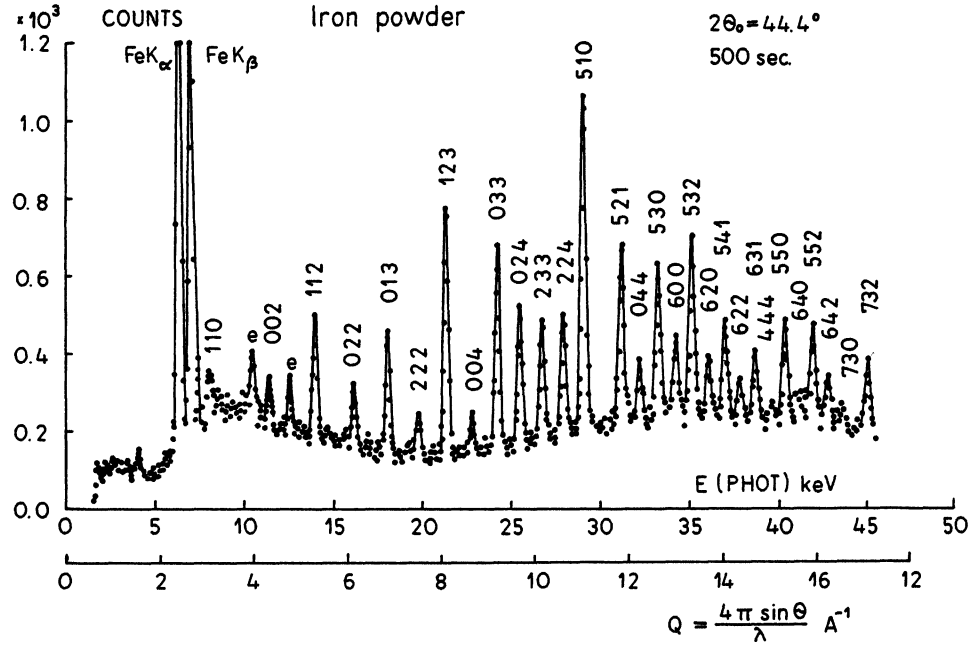

Figure 3. Diffraction spectrum of iron powder, obtained using synchrotron radiation.[9]

TEXTURE MEASUREMENTS

In principle and in practice energy-dispersive methods can be used to determine simultaneously the pole figures and the inverse pole figures, i.e., the statistical experimental information about the texture.

It has been demonstrated experimentally[7] that using this method one can determine simultaneously several pole figures which can then be used to calculate ODF for two-phase materials. The complete diffraction spectra are registered at each point of the pole figure and overlapping reflections can be easily separated. In the case of angular-dispersive texture measurements, the separation of the effect caused by the overlapping reflections is much more complex.

Application of the energy-dispersive method is however most advantageous for the determination of inverse pole figures. In previous experiments with the use of an X-ray tube it has been demonstrated that around 15 points can be measured in the space of the inverse pole figure. With synchrotron radiation this number can be much higher. The suggested geometrical arrangements for measuring inverse pole figures are presented in Figures 4, 5 and 6. In Figure 4 a collimated white beam impinges onto three planes. These planes represent the rolling plane, the plane perpendicular

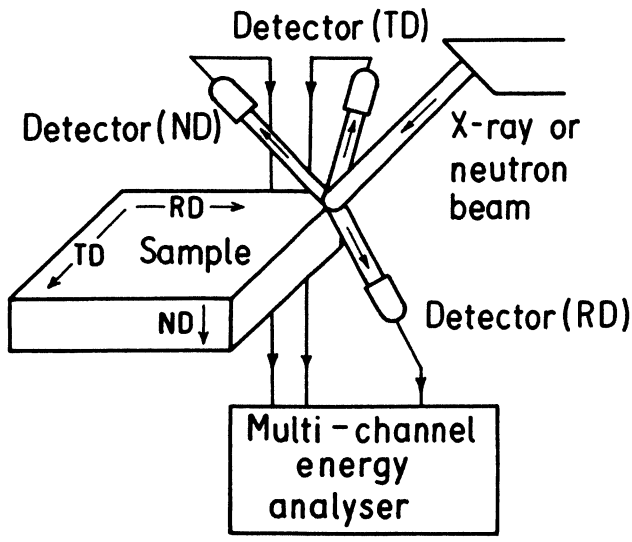

Figure 4. Principles of the measurements of the inverse pole figures for RD, TD and ND using energy-dispersive X-ray or time-of-flight method.

to the rolling direction and the plane perpendicular to the transverse direction. The vectors perpendicular to the three planes coincide with scattering vectors for each counter. The counters must be placed at correct positions in order to register the inverse pole figures corresponding to rolling, transverse and the normal directions. The inverse pole figures obtained in this manner provide us with information about preferred orientations of crystallites. Measurements can be made within a few minutes.

More accurate data about the texture can only be obtained from a higher number of inverse pole figures. A greater number of counters would have to be used. We then need to answer the question: how many counters do we need to collect enough experimental data to determine the orientation distribution function. The answer depends on the symmetry of the crystal structure, on the interval of energy at which the spectrum is registered, on the sharpness of texture and on other less important factors. For a typical X-ray tube and cubic structure of tested material, 10 counters should be sufficient. The information collected with these counters will roughly correspond to the information contained in the two pole figures measured at 10 degree intervals of both polar angles. The determination of the orientation distribution function from the inverse pole figures has not as yet been accomplished, but the general method of calculation of ODF from points arbitrarily chosen in space of the pole figure and the inverse pole figure has already been proposed by Bunge.[10]

Quantitative information about the ideal orientation and the ODF can also be obtained in another way. The schematic arrangement is shown in Figure 5. A sheet while being

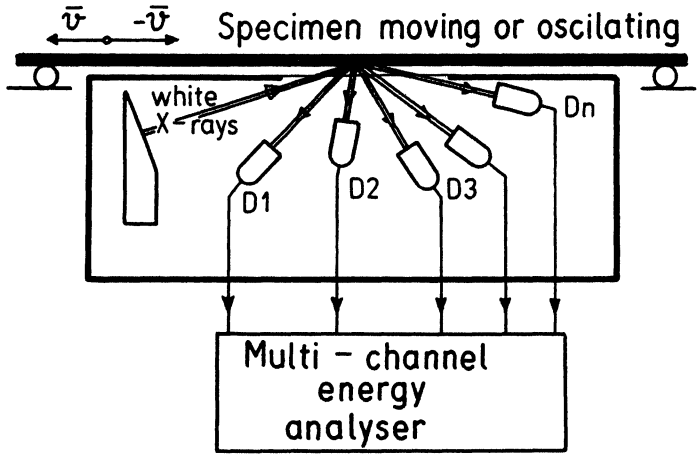

Figure 5. Principles of geometry of the inverse pole figure measurements in reflection using X-ray energy-dispersive method. Specimen moving or oscillating.

rolled, or a sheet specimen, are irradiated by an X-ray beam and several inverse pole figures are registered in reflection geometry. Depending on the number of counters the experimental result can be translated to more or less extensive quantitative information about the texture. Averaged information about the texture in a larger volume is collected. The moving sheet or the moving specimen continuously introduce new grains into the diffraction position; hence the statistics improve. The X-ray energy-dispersive method can thus be used to measure texture in coarse-grained specimens.

Several other geometries suitable for measuring inverse pole figures can be introduced. The specimen can be illuminated by two beams of X-rays. Such a situation is, e.g., illustrated in Figure 6. Here the only reason for using two beams is the high absorption of X-rays in metals and the advantage of a reflection technique in texture examination. When using neutron diffraction it will be possible to use the reflection and the transmission techniques simultaneously. Thus, there is no reason for applying two neutron beams.

DISCUSSION AND CONCLUSION

The application of the energy-dispersive method enhances the range of texture examinations. Angle-dispersive and energy-dispersive methods can be used with both neutron and X-ray diffraction. A question arises: which radiation and which method is the most suitable? The choice of the method depends on the structure of the investigated material (i.e., grain size) and on the texture problem studied.

The most important differences between texture studies using X-rays and neutrons are given in Table I. A comparison between the various applications of the angle-dispersive and the energy-dispersive methods is presented in Table II.

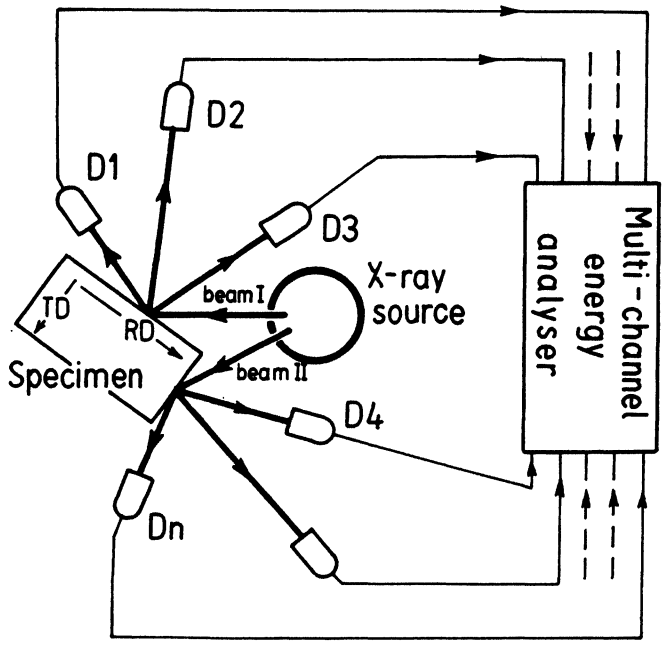

Figure 6. Principles of geometry of the inverse pole figure measurements in reflection using X-ray energy-dispersive method. Two beams of X-ray are used simultaneously.

TABLE I

Comparison of X-ray and Neutron Diffraction Methods for Texture Studies

Property	Neutrons	X-ray	Remarks
Investigated volume (relative units)	10^5	1	
Specimen thickness	1-20 mm	10^{-2}-10^{-1} mm	
Specimen preparation	Easy	Difficult	
Possibility of introducing texture distortion during specimen preparation	Not possible	Possible	Depending on technique of specimen preparation
Recommended experimental technique	Transmission, reflection, spherical sample	Reflection technique	
Correction for absorption and change in geometry	Rather easy	More difficult	Depends on experimental technique
Extinction coefficient	Rather difficult	Practically unnecessary	Necesary only in some experiments

TABLE I (Continued)

Property	Neutrons	X-ray	Remarks
Influence of surface layers	Low	Large	
Separation of reflection	Good, sufficient for texture experiments	Very good	Change of X-ray tube or neutron λ possible (if necessary)
Possibility of measuring many pole figures simultaneously	Yes	Yes	
Accuracy of experiment	Good	Moderate	
Time necessary for pole figure measurements	1 unit	1 unit	Depending on experimental conditions
Necessary apparatus	Reactor, diffractometer, texture specimen holder, electronic equipment	X-ray tube, diffractometer, texture specimen holder, electronic equipment	X-ray diffraction equipment is cheaper; for neutron measurements a reactor is necessary
Method is recommended for	Studies of coarse-grained materials; information on average texture	Selective texture measurements; small-grained materials	

TABLE II

Differences Between Angular Dispersive Method and Energy Dispersive Method

	Angular Dispersive Method	Energy Dispersive Method	Remarks
1	2	3	4
Scanning	over θ	over λ	Scanning over θ and λ is also possible at the same time
Intensity	depending on θ	depending on λ and on spectral distribution of energy	

TABLE II (Continued)

	Angular Dispersive Method	Energy Dispersive Method	Remarks
1	2	3	4
Resolution	better for higher d value	better for lower d value	
Absorption corrections	θ dependent, and dependent on changing geometry	energy dependent	
Extinction correction	dependent on θ and on material structure	dependent on λ and on material structure	
Limits of the precision of the measurements		The necessity of the measuring of the spectral distribution	In practice the intensity ratio; textured specimen to powder specimen is measured
Possibilities of the simultaneous measurements of several reflections	not possible	possible	
Geometry of experiment	changing	not changing	
Facilities for changing temperature, high pressure, magnetic field and external stresses	difficult to install	easy to install	
Dynamic studies	difficult	easy	
Type of information about texture	pole figure, inverse pole figure	inverse pole figure, pole figure	
Time of measurements (relative units)	1	1	in dispersive measurements time of collecting information can be much shorter than in angular dispersive (synchrotron, pulse reactor)

TABLE II (Continued)

	Angular Dispersive Method	Energy Dispersive Method	Remarks
1	2	3	4
Recommended applications in texture studies	standard pole figure measurements	inverse pole figure measurements, dynamic studies, multiphase materials, low symmetry structure	

The choice of a given method of texture examination is also influenced by more practical reasons—the source of radiation at our disposal. For this reason, the X-ray tube is more often used in texture examination and texture control. The use of other sources, i.e., pulse reactor or synchrotron can only be justified when studying more sophisticated, time-dependent phenomena. The energy-dispersive methods can facilitate both texture examination and texture control. They can be easily applied even for on-line control and for coarse-grained materials. The preparation of the specimen is not difficult.

The most interesting capability of energy-dispersive methods arises from the constant geometry of inverse pole figure measurements. It is very easy to examine texture and structure changes under the influence of external parameters, such as temperature, time and external stresses. Heavy equipment can be easily installed, and only the inlet and outlet channels for the collimated white radiation need to be provided.

In the discussion of the geometry of the energy-dispersive measurements it was not always indicated whether X-ray or neutron sources are used. The geometry of experiment is very similar in both methods. Another problem is the correction of experimental data. In the energy-dispersive method, the absorption corrections are energy-dependent while in angular-dispersive measurements the corrections depend on the value of the scattering angle. The correction problems were discussed earlier.[7,11] When measuring texture using neutron diffraction, we have to consider extinction corrections. The present author has discussed this problem elsewhere.[11]

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