

ENERGY DISPERSIVE DIFFRACTOMETRY FOR QUANTITATIVE TEXTURE STUDIES

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(Received December 10, 1988; in final form January 10, 1989)

Energy dispersive diffractometry is becoming a useful tool for texture measurements. In this work we demonstrated that the intensity measured at points in four inverse pole figures for cold-rolled steel can be used to calculate the orientation distribution function (ODF) with an accuracy sufficient for the determination of about 12 series expansion coefficients. A pole figure generated from such a selective experiment agrees with the pole figure measured by neutron diffraction.

KEY WORDS Energy dispersive diffractometry, inverse pole figures, ODF-calculation.

INTRODUCTION

Energy dispersive X-ray diffractometry offers new possibilities of texture studies and some applications of this technique have already been examined. Szpunar *et al.* (1974) applied the technique to determine the inverse pole figures in textured specimens and later Szpunar and Gervard (1980) used the method for the simultaneous recording of several pole figures in Alpha and Beta Brass. The possibilities of this technique for texture measurements have been discussed previously by the author, specifically for geometries for on-line texture control (Szpunar, 1981) and the studying of texture transition under the influence of external parameters. Kopineck (1986) has already applied the semi-conductor detectors for on-line measurements of certain texture information.

In this work we would like to demonstrate that the inverse pole figures obtained using energy dispersive diffractometry can be used to calculate the orientation distribution function (ODF). This is a new approach to quantitative texture measurement and analysis which can be used in the future for the examination of the texture transition and on-line determination of the ODF.

ANGLE DISPERSIVE AND ENERGY DISPERSIVE METHODS FOR TEXTURE ANALYSIS

Standard X-ray methods used in texture studies make use of a monochromatic beam. The counter position is fixed to receive $\{hkl\}$ reflections and the specimen rotates so that all planes of chosen type $\{hkl\}$ will satisfy the diffraction condition.

The energy dispersive method is based on the use of a white X-ray beam. The semiconductor detector is fixed at a constant scattering angle and can be used to

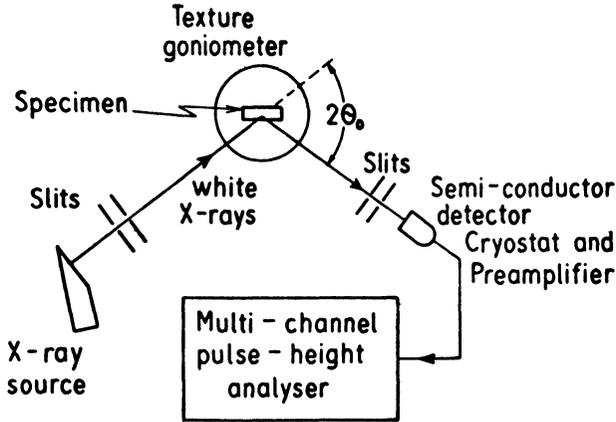
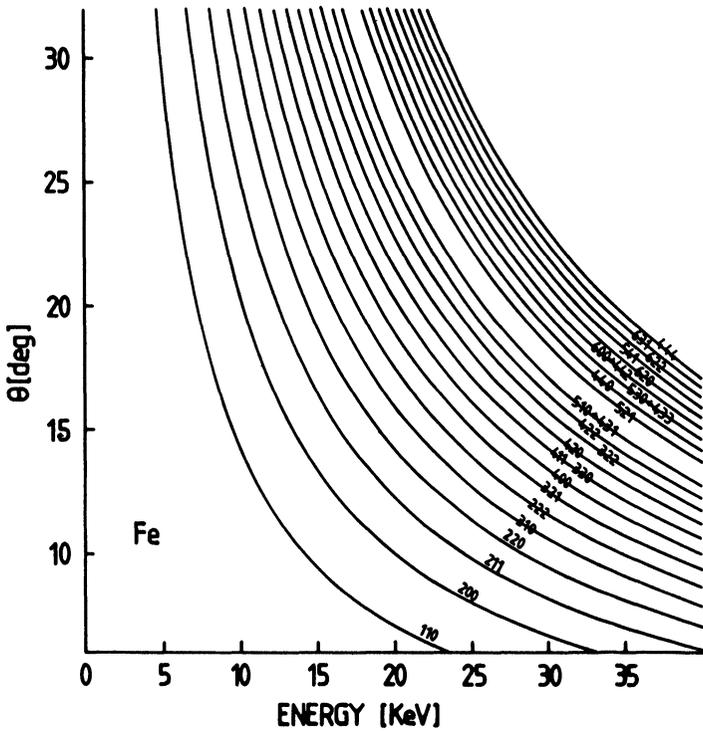


Figure 1 Schematic illustration of the measurements of the inverse pole figure.



record several diffraction maxima. These maxima represent points in the space of the inverse pole figure determined for that direction of the specimen which coincides with the scattering vector. The schematic geometry of this texture experiment is illustrated in Figure 1. The detector, by changing its position, registers the experimental data in various inverse pole figures. An obvious advantage of this geometry of measurement is the possibility of keeping the position of the specimen constant during the texture measurements. Such constant geometry measurement facilitates the installation of additional equipment for thermal and mechanical treatment of the sample. Texture measurements can be performed at the same time and therefore it is possible to follow texture changes as a function of temperature, time or mechanical treatment. The time of data collection is short, and this allows, in some cases, a recording of the texture transition during the deformation and recrystallization processes.

In order to use the experimental points on the inverse pole figures to calculate the ODF, an attempt should be made to maximize the number of points in the inverse pole figure. Also, several inverse pole figures have to be measured. The number of the experimental data points depends on the interval of the energy which can be measured and also on the selected values of the diffraction angle θ . The number of registered reflections will also be affected by the crystal symmetry and the dimensions of the unit cell. An illustration of the diffraction law for steel is presented in Figure 2.

METHOD OF CALCULATING THE ORIENTATION DISTRIBUTION FUNCTION

Full pole figures are required in order to calculate the series expansion coefficients of the pole figures by integration (Bunge, 1982). The ODF is calculated by solving a system of linear equations relating the coefficients of expansion of the pole figures and the coefficients of the ODF. It is expressed by the following formula.

$$f(g) = \sum_l \sum_{\mu} \sum_{\nu} C_l^{\mu\nu} \dot{T}_l^{\mu\nu}(g) \quad (1)$$

Where $\dot{T}_l^{\mu\nu}$ are harmonic functions and each function is multiplied by an appropriate series expansion coefficient, $C_l^{\mu\nu}$.

In our experiments, the intensities at points which are irregularly spaced on the pole figure are measured. It is therefore impossible to use the integration to calculate the series expansion of the coefficients of the pole figure. A different technique of ODF calculation must then be used (Bunge, 1977) which does not require integration at all. For this technique, a fit between the theoretical ODF and the isolated experimental points is suggested. The experimental points can be chosen arbitrarily in both the pole figure and the inverse pole figure. The solution of the problem requires the minimization of the following expression:

$$\sum_i \sum_j (P_{ij} N_j - P_j^{\text{th}}) W_{ij} \Rightarrow \min \quad (2)$$

where P_{ij} are measured intensities at point i of the pole figure j , and N_j are normalization factors which are used to express the pole density values in the

units corresponding to random density. W_{ij} are weighing factors. Our experimental points can therefore be considered as measured at the same points on several pole figures. This technique of calculation was used by us previously to determine the ODF from partial pole figures (Szpunar and Hinz). In the present work the calculation will be modified. Instead of using the N_j normalization constants for each crystallographic reflection separately, only one constant will be used. Such an approach is justified, because the experimental results were always compared to results obtained for a suitable powder specimen which does not show any texture and therefore texture intensities are expressed in the same units. The least square minimization condition will now be expressed by:

$$\sum_i \sum_j [P_{ij}N - P_{ij}^{\text{th}}]W_{ij} \Rightarrow \min \quad (3)$$

where the theoretical pole figures are given by the following expression:

$$P_{ij}^{\text{th}} = 1 + \sum_l \sum_\mu \sum_\nu C_l^{\mu\nu} \frac{4\pi}{2l+1} k_l^\mu(j)k_l^\nu(i) \quad (4)$$

Here k_l^μ and k_l^ν are spherical harmonics being invariant with respect to the crystallographic and the specimen symmetry.

EXPERIMENTAL RESULTS

The steel-sheet specimen examined was hot rolled at 850°C annealed for 30 minutes at 1200°C and then cold rolled with the reduction of 90%. The experimental results of these measurements are published by the author with Ojanen and Laine (1974). All experimental data are presented in Table 1. There are four inverse pole figures measured and there are 9 points on each pole figure. There are therefore 36 experimental points. According to our previous experience and analysis (Szpunar and Hinz), this number of experimental data points allows us to determine a number of expansion coefficients of the ODF which is about 3 times less. The ODF coefficients were calculated for I_{max} 8, 10 and 12; Table 2 lists the values of the coefficients for I_{max} 10 and 12. The {110} pole figure which was calculated from these coefficients (Figure 3a) is compared with the experimental pole figure measured by neutron diffraction (Figure 3b).

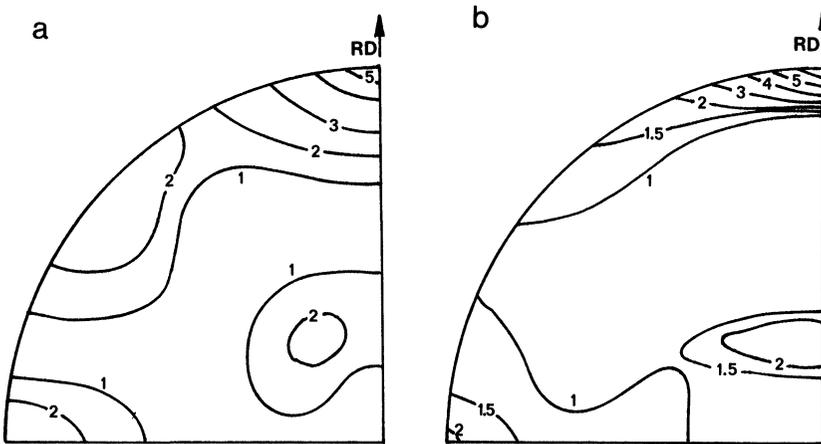
Table 1 The intensities measured on the inverse pole figure.

<i>hkl</i>	<i>RD</i>	30°	60°	90°
110	11.83	3.90	2.43	2.69
200	0.37	0.50	0.50	0.34
211	0.67	0.74	1.0	0.61
310	0.34	0.82	1.0	0.17
222	0.61	0.82	0.91	1.48
321	1.35	1.10	1.35	4.86
210	0.61	0.74	0.67	1.64
323	0.41	0.37	0.82	3.61
521	0.61	0.91	0.74	0.61

Table 2 The ODF's coefficients calculated for $I_{\max} = 10$ and 12.

		Coefficients for $I_{\max} = 10$					
l	μ	= 1	2	3	4	5	6
4	1	-2.613	0.603	-0.667			
6	1	4.806	-2.961	1.615	-0.480		
8	1	2.606	-2.627	0.556	-0.363	1.188	
10	1	-1.940	2.842	0.959	0.389	-0.057	1.856

		Coefficients for $I_{\max} = 12$					
l	μ	= 1	2	3	4	5	6
4	1	-2.268	-0.702	-0.357			
6	1	4.323	-2.609	1.570	-0.493		
8	1	4.038	-0.944	0.726	-0.207	0.739	
10	1	-2.690	5.041	3.484	0.525	-2.068	-3.218

**Figure 3** $\{110\}$ pole figure calculated from: (a) ODF coefficients, $I_{\max} = 12$. (b) $\{110\}$ pole figure measured using neutron diffraction.

The intensity of the pole figure measured by the neutron diffraction compares favorably with the intensity on the pole figure calculated from ODF coefficients.

Acknowledgments

The author would like to thank D. C. Hinz for computer calculations.

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