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X-ray diffraction analysis of substructures in plastically deformed BCC materials

P. KLIMANEK

Freiberg University of Technology, Department of Materials Science, 9596 Freiberg/Sa., Germany

ABSTRACT

A procedure for line-shape analysis of broadened X-ray (or neutron) diffraction peaks is presented and specified for b.c.c. materials, which takes into account the effect of interfaces, internal stress of the 2nd kind and a dislocation distribution with weak defect correlation. Application of the technique is demonstrated by the estimation of dislocation densities in plastically deformed α -iron and steel. The results confirm that X-ray line-broadening analysis is suitable for integrated substructure characterization, but it becomes also evident that local structural inhomogeneity and the texture of the sample material must carefully included into the interpretation of experimental data.

INTRODUCTION

Substructure analysis (i.e. characterization of the concentration and the spatial arrangement of lattice defects as dislocations, stacking faults etc.) in polycrystalline materials with higher defect concentrations as occurring e.g. after plastic deformation can be performed by evaluation of the broadening of X-ray (or neutron) diffraction peaks. However, the interpretation of experimental results may be difficult and requires the introduction of physically realistic microstructure models.

In the present paper a methodology of integrated substructure analysis from the Fourier coefficients of broadened diffraction peaks is briefly treated and specified for the case of b.c.c. materials, which takes into account theoretical work of Krivoglaz et al. [1 - 3] and Wilkens [4 - 7] concerning the estimation of dislocation densities. The application of the technique is demonstrated by experimental results obtained with plastically deformed α -iron of technical purity and high-alloy ferritic steel X8CrTi17.

METHODOLOGICAL BACKGROUND

Evaluation of Fourier coefficients

Shape analysis of diffraction peaks $F(x)$ ($x = 2\Delta \sin\theta/\lambda$) of polycrystals encloses, in general, the following steps:

- 1) separation of the instrumental line shape $g(u)$ due to the nonideal conditions of the diffraction experiment and the physical line profile $f(x)$ due to the lattice disorder of the crystallites,

- 2) identification of the types of the lattice disorder being present and separation of the corresponding contributions to the physical line broadening, and
- 3) quantitative estimation of substructure parameters as dislocation densities, stacking-fault probabilities or subgrain sizes.

The first problem can approximately be solved on the base of the Fourier transformation by means of the Stokes correction [8]. For this purpose the instrumental line shape $g(u)$ is usually measured by means of a standard sample, the effect of whose lattice disorder is negligible, and the Fourier coefficients $A(L)$ of $f(x)$ are then calculated from those of the line profiles $g(u)$ and $F(x)$ (e.g. [9 - 12]).

The evaluation of the physical line broadening is based on the fact that the line shape $f(x)$ is a convolution of partial profiles related to the statistically independent components of lattice disorder. Obviously, in plastically deformed polycrystals the following components of line broadening should always be taken into consideration:

- particle-size broadening due to interfaces (subboundaries) or small crystallite size,
- strain broadening due to internal stresses of the 2nd kind which are constant within a crystallite or a subgrain, and
- line broadening due to dislocations.

Accordingly the Fourier coefficients of the physical line shape $f(x)$ can be defined as the products ($a_p(L)$ - particle-size effect, $a_\epsilon(L)$ - strain effect, $a_d(L)$ - dislocation effect)

$$A(L) = a_p(L) a_\epsilon(L) a_d(L) \quad (1)$$

Introducing here results of the kinematical theory of X-ray and neutron scattering by imperfect crystals as presented in [13,14, 1-7] the relationships

$$A(L) \approx \exp(-L/T) \exp(-K\langle\epsilon^2\rangle L^2) \exp(-B \ln(L_0/L) L^2) \quad (2)$$

and

$$-\ln A(L) = L/T + \{K\langle\epsilon^2\rangle + B \ln(L_0/L)\} L^2 \quad (3)$$

are obtained, whose symbols have the following meaning:

- $L = nd(hkl)$ - measuring length perpendicular to the scattering lattice planes (hkl)
- $T = T(hkl)$ - effective particle size,
- $B = B(hkl)$ - factor proportional to the mean total dislocation density N_d
- L_0 - length proportional to the outer cut-off radius R of the strain field of a dislocation,
- $\langle\epsilon^2\rangle = \langle\epsilon^2(hkl)\rangle$ - mean square strain due to internal stress of the 2nd kind

Equation (3) leads to the function $(K=2\pi^2(h^2+k^2+l^2)/a_0^2)$; a_0 - lattice parameter)

$$\phi(L) = -\ln A(L)/L = 1/T + K\langle\epsilon^2(L)\rangle L \quad (4)$$

the graphic representation of which has been called a Warren - Averbach plot [18,19] because it can formally be interpreted in terms of the well-known substructure model of these authors [13,14]. The plot allows the determination of an effective reciprocal particle size $1/T$ and an effective mean square strain $\langle\epsilon^2(hkl)\rangle$ from the relations

$$\Phi(0) = 1/T, \quad (d\Phi/dL)_{L \rightarrow 0} = K \langle e^2 \rangle \quad (5)$$

On the other hand, using the work of Wilkens [4,5] and Krivoglaz [2,3] concerning diffraction-line broadening by dislocation arrangements with weak defect correlation, from (3) the function

$$\Psi(L) = -\ln A(L)/L^2 = 1/TL + (K\langle e^2 \rangle + B \ln L_0) - B \ln L \quad (6)$$

is obtained, whose graph was called a Krivoglaz-Wilkins plot [18,19]. $\Psi(L)$ should have a linear branch at higher L , from which the quantities B and L_0 can be derived via the relationships

$$d\Psi/d(\ln L)_{L \gg 1} = B, \quad (K\langle e^2 \rangle + B \ln L_0) - B \ln L = 0 \quad (7)$$

An example of the plots is given in Fig. 1.

Fig. 1 : Krivoglaz-Wilkins plots of (211) reflections of α -iron after different tensile deformation

Estimation of dislocation densities

In order to estimate dislocation densities N_d from (6,7) the parameter $B(hkl)$ must be known explicitly. For dislocation arrangements in elastically isotropic cubic crystal it is defined [1,2] by (b - amount of the Burgers vector of the dislocations, $h = 2\sin\theta/\lambda = 1/d(hkl)$)

$$B(hkl) = B_0(hkl) N_d \quad [nm^{-2}]; \quad B_0(hkl) = (\pi/2) (bh)^2 \chi(hkl) \quad (8)$$

with

$$\chi(hkl) = \chi_0 + \chi_1 \Gamma(hkl); \quad \Gamma(hkl) = (h^2 k^2 + l^2 h^2 + k^2 l^2) / (h^2 + k^2 + l^2)^2$$

The quantity $\chi(hkl)$, the general form of which was discussed in [15], is called orientation factor because it depends on the orientations bet-

ween the the slip sytems $\{hkl\}\langle uvw \rangle$ and the Burgers vectors b , respectively, of the dislocations and the operating diffraction vector h .

Table 1 specifies the components of $\chi(hkl)$ for the case of a b.c.c. polycrystal with randomly oriented grains, where the slip systems are statistically equivalent. Assuming then that only dislocations with Burgers vectors $b = 1/2 \langle 111 \rangle$ are present, the numerical values of Table 2 can be calculated for $\chi(hkl)$ and $B(hkl)$, respectively.

Table 1: Components of the mean orientation factors $\chi(hkl)$ in elastically isotropic b.c.c. polycrystals

Screw dislocations	Edge dislocations
$2 \chi_0 = -\chi_1 = 4/9$	$\chi_0 = \frac{25 - 68\mu + 72\mu^2}{144 (1 - \mu)^2}$
μ - Poisson's ratio	$\chi_1 = \frac{19 - 44\mu + 24\mu^2}{144 (1 - \mu)^2}$

Table 2: Factors for the estimation of dislocation densities in b.c.c. materials from Krivoglaz-Wilkins plots

Reflection	$\Gamma(hkl)$	χ_s	χ_e	B_{os}	B_{oe}
110	1/4	0.111	0.185	0.262	0.436
200	0	0.222	0.157	1.047	0.740
211	1/4	0.111	0.185	0.785	1.309
220	1/4	0.111	0.185	1.047	1.746
310	9/100	0.182	0.167	2.147	1.976
222	1/3	0.074	0.194	1.047	2.751
321	49/256	0.137	0.178	2.262	2.946
400	0	0.222	0.157	4.189	2.960

s - screw dislocations, e - edge dislocations

RESULTS AND DISCUSSION

The application of the equations (4) and (6) for substructure analysis in plastically deformed b.c.c. materials is illustrated by Fig. 2 and 3. The X-ray diffraction peaks were measured with a powder diffractometer HZG3 (Freiberger Präzisionsmechanik GmbH) by means of $CoK\alpha$ radiation. In order to reduce the background and the instrumental line broadening, soller slits and secondary-beam monochromatisation (graphite) were used. Moreover, the Fourier coefficients $A(L)$ of the physical line profile $f(x)$ were calculated from the Fourier transforms of carefully fitted profiles $F(x)$ and $g(u)$, which is a requirement for the evaluation of Krivoglaz-Wilkins plots.

Fig. 2 presents dislocation densities N_d in technical α -iron after tensile deformation at room temperature as estimated by TEM [16] and from the integrated line widths or the Fourier coefficients of the reflections (220), (200) and (211) [17].

Fig. 3 informs about the changes of the dislocation density N_d in tensile deformation of high-alloy ferrite X5CrTi17 (Results obtained after compression by neutron and X-ray diffraction are presented in [21]).

Fig.2: Dislocation densities N_d in tensile deformed α -iron

Fig.3: Dislocation densities N_d in high-alloy ferrite X8CrTi17 after tensile deformation at room temperature

The experimental results can be characterized as follows :

- * The averaged dislocation densities N_d estimated from the Fourier coefficients for α -iron are in good agreement with the TEM results.
- * After equal tension ϵ the dislocation densities of the α -iron and the ferritic steel X8CrTi17 are quite similar, and in both cases a nearly linear increase of N_d is observed.
- * As illustrated by Fig.2, dislocation densities estimated from the physical line - widths of the reflections are usually higher than those determined from the Fourier coefficients. Moreover, the distances L_0 obtained from the Krivoglaz-Wilkins plots are frequently too large. Both phenomena can be easily explained as an indication of stresses of the 2nd kind, but in order to separate the corresponding strain component quantitatively from that of the dislocations, further development of the methodology of profile analysis is necessary.
- * Fig. 3 presents dislocation densities $N_d(hkl)$ estimated from various reflections of the steel X8CrTi17, which were measured perpendicular to the tensile axis. The results indicate that the defect content of crystallites with various orientations seems to be different. Because the tensile specimens had a significant $\{110\}$ annealing fibre texture,

the defect densities $N_d(220)$ should be representative for the majority of the crystallites, and the relationship $N_d(220), N_d(200) < N_d(211)$ may be due to the influence of slip system selection on the orientation factor $\chi(hkl)$. A more detailed interpretation has also to take into account the elastic anisotropy of the sample material.

- * Fig. 2 shows relatively large scattering of the dislocation densities $N_d(\epsilon)$ estimated from the reflections (220) and (200) especially for low tensions. One reason for the effect may be the uncertainty of the Krivoglaз - Wilkens plot, which, of course, increases with decreasing defect content, but more important is insufficient averaging over the crystallite ensemble due to the influence of the grain size on reflections with low multiplicity factor. Thus the measurements illustrate the problem of local structural inhomogeneity as treated in [18-20].

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