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Theoretical Considerations for Dislocation-Density Determination by Neutron Scattering Extinction Measurements

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Abstract. It is intended to establish the theoretical basis to evaluate dislocation densities in pure metals by measurements of integrated intensities for Bragg-Peaks.

Under the assumption that randomly distributed dislocations divide a polycristalline metal in ideal domains where perfect periodicity is conserved, and distorted domains containing the dislocations, the observed integrated intensity of a Bragg-Peak for neutrons is calculated as the sume of a dynamic and a kinematic contribution. Supposing furthermore that the average form of the ideal domains is a sphere, the dynamic contribution is calculated from the Zachariasen's General Theory of X-Ray Diffraction in Crystals.

It is estimated that from a measurement of the Cu (111)-reflection dislocation densities lower that or equal to 10^8 cm^{-2} could be determined.

Resumen. Se pretende establecer la base teórica para la evaluación de densidades de dislocaciones en metales puros por medio de la intensidad integrada de picos de Bragg.

Bajo la suposición de que las dislocaciones distribuidas aleatoriamente dividen un metal policristalino en dominios ideales donde la periodicidad perfecta se conserva y dominios deformados conteniendo las dislocaciones, se calcula la intensidad integrada de un pico de Bragg para neutrones como la suma de una contribución dinámica y una cinemática. Suponiendo además que la forma promedio de los dominios ideales es esférica, la contribución dinámica se calcula mediante la teoría general de la difracción de rayos X en cristales, de Zachariasen.

Se estima que a partir de una medida de la reflexión (111) en Cu, se podrían determinar densidades de dislocaciones en el rango de 10^8 cm^{-2} o menores.

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1. Introduction

Measurements of intensities in pure metals with neutron scattering, deliver values smaller than those predicated by the kinematical theory of diffraction [1], suggesting that dislocations should affect extinction in an observable range. This idea is further supported by extinction affects observed from texture studies with neutron diffraction [2]. On the other hand, the reduction in intensity is not due to inelastic scattering because this is several orders of magnitude smaller. It is expected therefore that from extinction measurements dislocation densities could be determined. In fact, determinations of dislocation-density from measurements with X-rays have been reported by Ivanov *et al.* [3,4]. The present work intends to present a neutron diffraction treatment.

In agreement with Ivanov *et al.*, it is assumed here that the diffraction volume consists of distorted domains around the dislocation lines, whose contribution to the Bragg-reflection is well described by the kinematical theory; and ideal domains, whose contribution should be calcualted with the dynamical theory. As we know, the kinematical theory does not take into account the weakening of the beams, *i.e.* extinction, and it is succesfully applied to crystals with some degree of imperfection. Dynamical diffraction on the other hand is applied only to perfect crystals. It is also assumed that diffracted rays from individual domains are incoherent. Coherence length amounts only to about 3/4 of the domains diameter.

In this paper it is proposed to consider the ideal domains in average as spheres with mean diameter *d*. Their contribution is then calculated from the General Theory of X-Ray diffraction proposed by Zachariasen [5]. The application of this theory to neutron scattering is straightforward, provided that proper structure factor and constants are used.

2. The Extinction Factor

According to the assumptions mentioned above, we consider the observed integrated intensity I as

$$I = i_k V_k + i_d (V - V_k), \tag{1}$$

where i_k and i_d are the kinematic and dynamic diffracted intensities per unit volume respectively, V_k the distorted domain volume and V the total irradiated volume. It is well known from the kinematic diffraction theory [6,12] that for a powder sample

$$i_k = N(\lambda) Q^{\theta}, \tag{2}$$

where $N(\lambda)$ is the monochromatic incoming intensity with wavelength λ and

$$Q^{\theta} = \frac{z\lambda^3 \|F_N(\boldsymbol{\tau})\|^2}{4 v_0^2 \sin\theta}. \tag{3}$$

Here $F_N(\boldsymbol{\tau})$ is the structure factor corresponding to the planes with reciprocal vector $\boldsymbol{\tau}$, v_0 the unit cell volume, θ the Bragg-angle and z the multiplicity factor of the reflection.

We suppose that the whole dynamic domain volume $V - V_k$ consists of n spheres with radius $d/2$.

According to Zachariasen's general theory of X-ray diffraction the extinction factor Y_d for an average spherical ideal domain with radius $d/2$ is

$$Y_d = \left\{ 1 + \frac{3}{4} \frac{d^2 Q^{2\theta}}{\lambda} \right\}^{-1/2}, \quad Q^{2\theta} = \frac{\lambda^3 \|F_N(\boldsymbol{\tau})\|^2}{v_0^2 \sin 2\theta}, \tag{4}$$

from which its contribution to the reflected intensity results $i_k^{2\theta} v Y_d$, being v its volume $\pi d^3/6$, and

$$i_k^{2\theta} = N(\lambda) Q^{2\theta}.$$

The total contribution from n ideal domains, under the assumption that the scattering from them is incoherent and neglecting secondary extinction, is therefore

$$i_k^{2\theta} v Y_d n f = i_k^{2\theta}(V - V_k) Y_d f = i_k Y_d (V - V_k), \tag{5}$$

where f accounts for the fraction of domains which actually happen to be in the Bragg orientation, so that $i_k^{2\theta} f = i_k$ and

$$I = i_k \left\{V_k + Y_d(V - V_k)\right\}. \tag{6}$$

The extinction factor Y is then obtained dividing I by the integrated intensity $i_k V$ as calculated by the kinematical theory. This gives

$$Y = \frac{V_k}{V} + Y_d\left(1 - \frac{V_k}{V}\right). \tag{7}$$

Klimanek and Hanisch [7] calculated V_k under the assumption of interpenetrating cylinders of radius r around the dislocation lines:

$$\frac{V_k}{V} = 1 - e^{-\pi G r^2 \rho}, \tag{8}$$

with G the fraction of dislocations for which $\boldsymbol{r} \cdot \mathbf{b} \neq 0$, being $\mathbf{b}$ the dislocation Burgers vector and ρ the dislocation density considered as the total length of dislocation lines in unit volume.

The breaths of dislocation images found by X-ray topography of nearly perfect crystals [8,9] lead to an estimation of the radius r in Eq. (8) [4]:

$$r = K_1 K_2 \frac{t_e}{2\sqrt{2}\,\pi} \sqrt{\boldsymbol{r} \cdot \mathbf{b} \cot\theta}, \tag{9}$$

where K_1 and K_2 are factors which account for different displacements in different directions and for the relative fractions of edge and screw components in dislocations respectively, and t_e is the extinction length. G, K_1 and K_2 could be known from dislocation theory. In practice $G \approx 0.5$, $K_2 \approx 1.5$ and $K_2 \approx 1.3$.

Combining Eqs. (4) and (8), Eq. (7) is

$$Y = 1 - e^{-\pi G r^2 \rho}\left[1 - \left\{1 + \frac{3}{4}\frac{d^2 Q^{2\theta}}{\lambda}\right\}^{-1/2}\right], \qquad (10)$$

with r given by (9).

So far we need only an estimation for d. Williamson and Smallman [10] proposed the relation

$$\rho = \frac{12}{d^2}, \qquad (11)$$

which seems to be satisfactory at least for the region of [11] $10^6\ \mathrm{cm}^{-2}$.

3. Self Consistence and Observability Range

It the dislocation density is very large the exponential in (10) tends to zero, but also d becomes very small so that Y tends to one. That is expected because for this case almost every region is distorted and the diffraction is kinematic, so there is no extinction.

When ρ is very small, the exponential tends to one and

$$Y \approx \left(1 + \frac{3}{4}\frac{d^2 Q^{2\theta}}{\lambda}\right)^{-1/2},$$

which is the expresion (5), valid for one domain free from dislocations.

The validity of Eq. (5) limits that of Eq. (10). According to Zachariasen, if the integrated intensity can be measured to an accuracy of 2%, then

$$\frac{3}{8}\frac{d^2 Q^{2\theta}}{\lambda} < 0.02,$$

which for $\lambda = 1.08$ Å and $Q^{2\theta}_{(111)} = 1.6 \times 10^{-2}\ \mathrm{cm}^{-1}$ for Cu, gives

$$d/2 < 1.18 \times 10^{-4}\ \mathrm{cm},$$

which according to Williamson and Smallman corresponds to

$$P_{\max} \approx 3 \times 10^8 \ \mathrm{cm}^{-2}. \tag{12}$$

4. Discussion

The principal approximations contained in Eq. (10) are: *1)* Ideal domains scatter incoherently; *2)* There is no secondary extinction; *3)* Eq. (8) consideres randomly distributed dislocations. Approximation 1) is reasonable if the dislocation density is not so large, *i.e.* if the number of ideal domains is not extremly large so that multiple scattering remains low. A certain amount of secondary extinction could be expected if grains are small. To correct this effect would require a whole similar treatment. It is also common to observe dislocation clusters and this effect could be accounted for in Eq. (8) by a factor in the exponent as suggested by Klimanek and Hanisch.

The fact that Eq. (7) does not contain details of the ideal domain geometry leads us to think that this is actually not very important. It is nevertheless taken into account by the expression for Y_d.

5. Conclusion

Through a simple model we have arrived at some expressions which should enable us to determine dislocation densities lower than 10^8 cm^{-2} with neutron scattering. This should be advantageous specially because of its non-destructive character.

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References

1. U. Buchenau, private communication.
2. J. Szpunar, *Atomic Energy Review* **14**(2) (1976) 199.
3. A.N. Ivanov, Yu.A. Skakov, and E.I. Fomicheva, *Ind. Lab.* **48**(9) (1982) 893.
4. A.N. Ivanov, P.I. Klimanek, and Yu.A. Skakov, *Sov. Phys. Crystallogr.* **28**(1) (1983) 58.
5. W.H. Zachariasen, *Acta Cryst.* **23** (1967) 558.
6. W. Marshal, and S.W. Lovesey, *Theory of Thermal Neutron Scattering*, Clarendon Press, Oxford (1971).
7. P. Klimanek, and K.H. Hanisch, *Crystal Res. & Technol.* **18**(3) (1983) 361.
8. U. Bonse, *Z. Physik* **153** (1958) 278.
9. M. Wilkens, and F. Meier, *Z. Naturforschg.* **18**a (1963) 26.
10. G.K. Williamson, and R.E. Smallman, *Proc. Phys. Soc.* **B68** (1955) 577.
11. G.M. Vorob'ev, and L.I. Kotava, *Sov. Phys. Crystallogr.* **12**(4) (1968) 548.
12. G.E. Bacon, *Neutron Diffraction*, Clarendon Press, Oxford (1975).