About secondary extinction in textureless polycrystals

J. Palacios Gómez* and L.R. Macías Betanzos
Gerencia del Reactor, Instituto Nacional de Investigaciones Nucleares
Salazar, Edo. de Méx., México

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Abstract. A brief calculation is made to estimate the amount of secondary extinction in polycrystals with no preferred orientations. To this end, use has been made of the Zachariasen's general theory of X-ray diffraction in crystals, together with corrections introduced by Cooper and Rouse. It was found, that secondary extinction is negligible, so that any extinction observed is primary in origin, which is related to the dislocation density.

Resumen. Se hace un breve cálculo para estimar la cantidad de extinción secundaria en policristales libres de direcciones preferenciales. Para esto se hizo uso de la teoría general de difracción de rayos X de Zachariasen, junto con correcciones introducidas por Cooper y Rouse. Se encontró que la extinción secundaria es despreciable, de tal manera que toda extinción observada es de origen primario, la cual está relacionada con la densidad de dislocaciones.

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1. Introduction

Although secondary extinction has received considerable attention from crystallographers, it has been mainly devoted to real "single" crystals. That is because extinction is generally a source of variations of the diffracted intensity in studies of crystal structure, and which should be corrected. Nevertheless, since extinction is related to crystal imperfections, its measurement has been proposed to determine dislocation densities [1, 2, 3]. On the other hand, primary extinction relates to details within crystalline domains and secondary extinction is caused by the domains contribution, which is not related to dislocations. Therefore it is important to clearly identify the rate of each extinction type present in an experiment. This communication intends to estimate the secondary extinction by a textureless polycrystal.

2. The secondary extinction factor

In a classical paper [4] Zachariasen has demonstrated that the secondary extinction factor Y is

$$Y = Q^{-1} \int \bar{\sigma}(\epsilon_1)\phi(\bar{\sigma})\, d\epsilon_1, \tag{1}$$

*On sabbatical leave from Escuela Superior de Física y Matemáticas, IPN, and also fellow of COFFA.

where $\bar{\sigma}$ is the reflectivity averaged over the orientations distribution of the mosaic, ϵ_1 is the deviation from the Bragg angle Θ, $\phi(\bar{\sigma})$ is the function

$$\phi(\bar{\sigma}) = \frac{1}{1+\bar{\sigma}\bar{T}}, \tag{2}$$

with $\bar{T}$ the mean path length through the whole sample, and finally Q is the conventional crystallographic quantity, which for a neutron 2Θ scan is

$$Q = \frac{\lambda^3 \left|F(\bar{\tau})\right|^2}{v_0^2 \sin 2\Theta}, \tag{3}$$

with λ the neutrons wavelength, $F(\bar{\tau})$ the structure factor corresponding to the reflection $\bar{\tau}$, and v_0 the unit cell volume.

Expression (2) is an approximation which does not contain any angle dependence. Cooper and Rouse [5] have pointed out that unless $\sigma\bar{t} \ll 1$, $\phi(\bar{\sigma})$ may have a significant angle dependence and they gave a better expression, which for the whole crystal of cylindrical shape is

$$\phi(\bar{\sigma}) = 1 - \bar{\sigma}\bar{T} + \frac{25}{24}\left(\bar{\sigma}\bar{T}\right)^2 f(\Theta) - \frac{25}{27}\left(\bar{\sigma}\bar{T}\right)^3\left[f(\Theta)\right]^2 + \frac{45}{64}\left(\bar{\sigma}\bar{T}\right)^4\left[f(\Theta)\right]^3 + \cdots \tag{4}$$

and $f(\Theta) \simeq 1 + \frac{1}{3}\sin^{2.5}\Theta$. $\bar{\sigma}$ is further obtained by

$$\bar{\sigma}(\epsilon_1) = \int W(\Delta)\sigma(\epsilon_1 + \Delta)\, d\Delta, \tag{5}$$

with $W(\Delta)$ the orientation distribution function, and σ the reflectivity for a single domain. For our case, where no texture is present,

$$W(\Delta) = \frac{1}{2\pi}, \tag{6}$$

because as a weight factor is should satisfy the condition

$$\int_0^{2\pi} W(\Delta)\, d\Delta = 1.$$

Furthermore, also calculated by Zachariasen for a parallelepiped is

$$\sigma(\epsilon_1) = Q\alpha\frac{\sin^2 \pi\alpha\epsilon_1}{(\pi\alpha\epsilon_1)^2}, \tag{7}$$

where α is the ratio of the mean thickness of the domain normal to the incident beam, and λ, over the crystal volume. Zachariasen has also mentioned that for a symmetrically shaped crystal, Eq. (7) will be approximately correct. Unfortunately the expression for

$\sigma(\epsilon_1)$ becomes very complicated for a crystal of arbitrary shape, so it is very difficult to have a value of the error by using Eq. (7).

Using Eqs.(6) and (7) in (5), we obtain

$$\bar{\sigma}(\epsilon_1) = \frac{Q}{2\pi^2} \int_{X_1}^{X_2} \frac{\sin^2 X}{x^2} \, dX, \tag{8}$$

with $X_1 = \pi\alpha(\epsilon_1 - \pi)$ and $X_2 = \pi\alpha(\epsilon_1 + \pi)$. ϵ_1 is always much less than π and α is large because the average domain thickness amounts to some hundreds or thousands of Angstroms and the wavelength is of the order of 1 Angstrom, so the absolute value of X_1 and X_2 is large. Furthermore, the integrand of Eq. (8) decreases rapidly to zero, so we can replace the limits by $-\infty$ and $+\infty$ respectively, and Eq. (8) is

$$\bar{\sigma} = \frac{Q}{2\pi}. \tag{9}$$

Thus, we see that the ϵ_1 dependence vanishes, and so $\phi(\bar{\sigma})$ is also independent of ϵ_1. Eq. (1) is then

$$Y = \phi(\bar{\sigma}). \tag{10}$$

For reflection (111) of pure copper, with $\lambda = 1.08$ Å, $Q = 1.06 \times 10^{-2}$ cm^{-1} [6], and a small sample for which $\bar{T} \simeq 0.4$ cm,

$$\bar{\sigma}\bar{T} = \frac{Q\bar{T}}{2\pi} \simeq 6.7 \times 10^{-4},$$

and thus, according to Eqs. (4) and (10)

$$Y \simeq 1.$$

Reflection (111) is one of the strongest reflections of copper, so secondary extinction is negligible also for other reflections in copper, provided the sample has no texture.

3. Discussion and conclusions

It is important to compare this result with the known analysis of secondary extinction from real "single" crystals. Actually we could consider the real "single" crystal as a strong textured polycrystal with the usual crystal orientation distribution function

$$W(\Delta) = \eta^{-1}\sqrt{2\pi}\,\exp(-\Delta^2/2\eta^2), \tag{11}$$

with η the standard deviation of the mosaic blocks [7].

As is well known, secondary extinction in that case is the most important. Unfortunately there is no value of η in Eq. (11) which reproduces Eq. (6), nevertheless, it is well established that the broader the standard deviation of the mosaic blocks the smaller the secondary extinction [8], in consistence with our treatment.

Along this treatment, no consideration of absorption was made. Because X-rays are strongly absorbed, they do not penetrate much into the sample and thus no much chance exists to produce secondary extinction. This is even valid for the real "single" crystals. This treatment is therefore only interesting for neutrons.

Texture affects measurements in two ways: the intensity of Bragg reflections itself is sensitive to texture, and secondary extinction could be large. Nevertheless, texture can be reduced through a slight deformation followed by a recrystallization. Furthermore, the first effect could be neutralized if the sample spinns during the measurement. When the textureless condition is satisfied, the only extinction measured is primary and from it dislocation density can be reliably determined.

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