Extinction in textures: Nullifying the extinction effect

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Accounting for secondary extinction (SE) in a crystallographic direction, a straightforward approach is devised for XRD characterizations of textures by nullifying the extinction effect. To this end, a proper reconsideration of the nature of the extinction coefficients is carried out. It is shown that whereas the SE coefficient $g$ is proportional to the product of pole density $P$ and incident-beam intensity $I_0$, the empirical extinction coefficient $k$ is independent of the ratio $g/P\times I_0$. Based on the invariability of the $k$-coefficient with respect to $g/P\times I_0$, the extinction effect is nullified by equating two its expressions defined by intensities of a reflection measured at a series a levels interaction whose variation is controlled by $P$ and $I_0$. Techniques representing extended versions of this approach are developed for (i) reliability-evaluation of the controlled variation of the levels of interaction by using instrumental variable (generator current) and, hence, to test the capability of the XRD apparatus to collect accurate and precise data, and (ii) determination of extinction-free data of the pole density that is a fundamental physical parameter. The experimental results are discussed in terms of the influence of extinction coefficient $g$ on the accuracy in the determination of the pole density in ideal $<100>$ direction of nickel texture.

Key words: extinction, texture, pole density.

1. INTRODUCTION

Extinction was introduced to account for the reflecting power of a real crystal with respect to the power described by kinematical diffraction [1]. Extinction in a mosaic structure is power loss caused by the production of the diffracted beam. Depending on the block size, one has to distinguish between primary extinction, which is extinction within a single crystal block, and secondary extinction, which occurs when a ray reflected by one mosaic block is subsequently reflected by another block with the same orientation.

To overcome the deficiencies in the theoretical estimates of extinction correction factors, based on simplified parameterization of the extinction effect across the scan of reflection, devoted experimental procedures were applied to decrease as well as cancel the extinction effect. In this respect, Schneider (1976) designed $\gamma$-ray diffractometry experiments where extinction is only in the order of 10% or less [2]. Analyzing the process of the X-ray scattering and the level of interaction between radiation and crystal medium, Mathieson substantiated an approach for derivation experimental structure-factor values, which are free from extinction effects [3]. The approach involves “(i) determination of integrated reflectivity at a series of levels of interaction (attained by controlled variation of a suitable physical parameter) and (ii) extrapolation of an appropriate function of the measurements to zero level of interaction as identified by zero diffracted power”.

The procedures to experimental realizations of the null-intensity (extinction-free) limit were illustrated in the paper of Mathieson & Mackenzie [4] as well. To attain a true zero-extinction kinematic limit value, the question of extrapolation to zero extinction in case of wavelength in the $\gamma$-ray region has been discussed from different point of views in the literature [5–7]. In this connection, the $\gamma$-ray data used in each of these investigations have been fitted by using polynomials of different types corresponding to the particular conceptions of the authors for attainment of the true zero-extinction kinematic limit value.

The present study outlines an alternative approach for nullifying the extinction effect. In this respect, careful considerations are carried out of
the process of the scattering of X-rays and the level of interaction between the radiation and the crystal medium. Our concern here is essentially with mutual connection between extinction coefficients and their exact relationship with diffraction at a series of levels of interaction attained by controlled variations of suitable physical parameters.

2. BASIC DEFINITIONS OF EXTINCTION
THERY AND TEXTURE ANALYSIS

The formalism considered here is valid for the symmetrical Bragg geometry with a plane-parallel plate sample appearing infinitely thick to the X-rays. According to theory [1, 8–14] and experiment related with it [2, 5–7], the extinction decreases the measured intensity \( I_m \) of a reflection with a factor \( y \), the extinction factor, defined by

\[
I_m = yI_{kin}
\]

(1)

Here, \( I_{kin} \) is the intensity that a Bragg reflection would have if kinematic theory would apply exactly to the system being examined. Therefore, the intensity \( I_{kin} \) delimits an imaginary (physically non-attainable) level of interaction of the diffraction process. In the symmetrical Bragg geometry, \( I_{kin} \) has to be expressed as

\[
I_{kin} = P I_0 QS / 2\mu
\]

(2)

where \( I_0 \) is the intensity of the incident beam, \( S \) is the cross section of the beam, \( Q \) is the reflectivity per unit crystal volume, \( \mu \) is the ordinary linear absorption coefficient, and \( P \) is the pole density. It is defined by the volume fraction \( dV/V \) of crystallites whose \( <hkl> \)-poles fall into a (infinitely small) space-angle element \( d\Omega \) (Bunge [15, 16]):

\[
(dV/V) / d\Omega = P.
\]

(3)

The factor \( P \) connects (2) with the well known formula

\[
I'_{kin} = I_0 QS / 2\mu,
\]

(4)

which is derived under assumption for random distribution of crystallites, i.e. \( P=1 \). In the case of pure SE, Chandrasekhar gave an expression for the extinction factor \( y \) [17]:

\[
y = \mu / \mu_e,
\]

(5)

where \( \mu_e \) is an effective absorption coefficient. In the symmetrical Bragg geometry with a plane parallel plate sample one should use the effective absorption coefficient as a first order approximation for the SE correction \( \varepsilon \) [9]:

\[
\varepsilon = \mu + gQ \left( \frac{p_2}{p_1^2} \right).
\]

(6)

Here \( g \) is the SE coefficient, which is a dimensionless quantity [1]. The symbol \( p_n \) denotes the polarization factor for incident X-ray beam [9]:

\[
p_n = \left[ 1 + \cos^2 \left( 2\theta_0 \right) \cos^2 \left( 2\theta_0 \right) \right] / \left[ 1 + \cos^2 \left( 2\theta_0 \right) \right]
\]

(7)

where \( n = 1, 2, \ldots \), \( \theta_0 \) is the Bragg angle of reflection. From (6) a formula follows for the SE correction \( \varepsilon \), which has been derived by Darwin [1], and later the polarization \( p_2 / p_1^2 \) of the incident X-ray beam has been incorporated in \( \varepsilon \) by Chandrasekhar [17] and Zachariasen [9]:

\[
\varepsilon = gQ \left( \frac{p_2}{p_1^2} \right).
\]

(8)

3. ANISOTROPY AND BEHAVIOUR
OF THE EXTINCTION COEFFICIENTS

Bragg et al. [18] deduced the SE correction empirically, whereas Darwin [1] deduced it theoretically. To this end, the authors have supposed respectively that both the empirical extinction coefficient \( k \) and the SE coefficient \( g \) are constants for the crystal, that is, parameters independent of crystallographic direction. Moreover, Darwin [1] had assessed that the definition, deduced by Bragg et al. [18], was correct to the first order approximation for the SE correction alone. Consequently, in the discussed frames, the two definitions for the SE correction have to be equivalent. In order to account for the crystal and textural anisotropy, the nature of \( k \) and \( g \) is reconsidered here. Replacing \( Q \) by its corresponding expression from (2) transforms (8) into

\[
\varepsilon = kI_{kin} \left( \frac{p_2}{p_1^2} \right).
\]

(9)

where the expression

\[
k = 2g\mu / P I_0 S
\]

(10)

shows that \( k \) and \( g \) are mutually connected. Reforming (10) yields the expression for

\[
g = kP I_0 S / 2\mu.
\]

(11)

Evidently, (2) and (11) reveals that whereas \( I_{kin} \) defines the upper limiting value of the diffraction process, \( g \) defines the extinction-induced weakening of the level of interaction of the same process. Depending on \( P, g \) and \( k \) are anisotropic coefficients. The anisotropy of \( P \) comprises the crystallographic, microstructural and textural anisotropies. Acting together for all crystallites contributing to reflection, anisotropy parameters such as size, shape, dislocation substructure, crystallographic orientation and crystallographic arrangement (Bunge [19]) synthesize...
the resulting anisotropy of \( g \) and \( k \). The coefficients show different behaviour with respect to the level of interaction of the diffraction process that is controlled simultaneously by \( P \) and \( I_0 \) under otherwise equal conditions. At lower limiting values of either both \( P I_0 \rightarrow 0 \) or one of them makes it evidently that in the only case of no diffraction (\( I_0 \rightarrow 0 \)) (2), there is no extinction (\( g \rightarrow 0 \)) (11). This is in accord with Mathieson’s statement that ‘extinction is only zero, in absolute sense, when diffracted power is identically zero’ [3]. Moreover, whereas \( g \) is proportional to the product \( P I_0 \), the coefficient \( k \) is proportional to the ratio \( g/P I_0 \). Therefore, by virtue of the interdependence between \( g \) and \( P I_0 \), any change of either one, \( P \) and \( I_0 \), or one of them does not cause change of the ratio \( g/P I_0 \) and, hence, \( k \) is independent of the product of both \( P > 0 \) and \( I_0 > 0 \).

3.1. Definitions for \( k \) independence of the levels of interaction

Suppose the incident beam changes its intensity from \( I_{0\_i} \) to \( I_{0\_r} \) under \( P = \) const. Here \( i \) and \( r \) denote the values of the generator current. Then, by analogy of (10), we shall have

\[
k_i = 2\mu g_i / P I_{0\_i} S,
\]

\[
k_r = 2\mu g_r / P I_{0\_r} S,
\]

(12a)

where it is accounted that \( g_i \) and \( g_r \) are proportional to \( I_{0\_i} \) and \( I_{0\_r} \), respectively, i.e.

\[
\frac{g_i}{I_{0\_i}} \left/ \frac{g_r}{I_{0\_r}} \right. = 1.
\]

(13)

Dividing (12) and (12a) with accounting for (13) yields

\[
k_i = k_r,
\]

(14)

that \( k \) is independent of the level of interaction. Second, suppose the pole density changes from \( P \) to \( P' = 1 \) under \( I_0 = \) const. Following (12), \( k_i \) is then transformed into \( k_i' \), i.e.:

\[
k_i' = 2\mu g_i' / I_0 S.
\]

(15)

Here it is accounted that \( g_i' \) is proportional to \( P' \) corresponding to random distribution of crystalline orientations. Dividing (12) and (15) with taking into account for \( P = g/g_i' \) yields

\[
k_i = k_i'.
\]

(16)

Thus, (14) and (16) constitute conditions for the invariability of \( k \) from the level of interaction of the diffraction process. Then, one can utilize \( k \) for nullifying the extinction effects by equating two its expressions that are defined by two different couples of intensities of a reflection measured at a series of levels of interaction.

To analyze what a constant is \( k \), let us throw look at Fig. 1, which designs the diffraction condition in terms of the reciprocal (scattering) space (Ewald [20]). The coefficient \( k \) has dimension of reciprocal volume (see (10)). It corresponds to the scattering space. Actually, this is the scanned volume inside the node \( P_{hkl} \). Due to the relationship between real and scattering space, the reciprocal quantity of \( k \) \( \left[ 1/k = (S/2\mu) (P I_0/g) \right] \) corresponds to the real space. Then, one may consider \( 1/k \) as consisting of two terms of different range. For an infinitely thick sample, the term, \( S/2\mu \), represents the irradiated crystalline volume that is a constant for all reflections of the XRD pattern, whereas \( P I_0/g \) is a constant inherent for any particular reflection. The last is due to the interdependence of these three parameters since \( I_0 \) scales \( g \) by means of \( P \) (see (11)). The quantity \( 1/k \) defines a volume in the real space where the X-radiation, interacting with crystal medium, produces the measured intensity \( I_0 \).

3.2. Expressing the SE coefficient \( g \) and SE correction \( e \) in case of polycrystalline materials

Due to fine-crystalline structure and high density of imperfections [21], reflection broadening in textures is about two orders of magnitude larger than that one in single crystals, which amounts to a
few ten of seconds of arc [5, 7]: for the same reason, the textures exhibit pure SE as well [22, 23]. This reflects in the observed reflection broadening recorded by conventional diffractometry. Actually, it is a superposition of physical (microstructural) and instrumental broadening. Since the determination of the SE coefficient $g = 1/2\eta \sqrt{\pi}$ is based on the crystal-mosaic distribution alone [8]), it is not justified to expect that this definition would adequately account for the microstructural properties of polycrystalline materials. (Here $\eta$ is the standard deviation of the Gaussian function). The only way to account adequately for the anisotropic effects of the pole density $P$ on the SE coefficient is to determine $g$ using quantities corresponding to reflection whose profile synthesizes all microstructural effects. Thus, if $k$ and $I_{kin}$ are known, from (8) and (9) one obtains:

$$g = kI_{kin}/Q.$$  (17)

Replacing $I_{kin}$ with its corresponding expression obtained by reforming (1) in succession with (5), (6) and (9), transforms (17) into

$$g = k\mu I_{m}/Q\left[\mu - k I_{m}(p_2/p_1^2)\right].$$  (17a)

By analogy, for the SE correction $\varepsilon$ defined by (9), one writes

$$\varepsilon = \mu k I_{m}(p_2/p_1^2)/\left[\mu - k I_{m}(p_2/p_1^2)\right].$$  (17b)

These definitions account implicitly for parameters describing as a whole the crystallographic, textural and microstructural anisotropy in the probing direction of the sample as well as the measurement conditions. Moreover, in the Appendix A is shown that the basic source of the $g$-coefficient anisotropy is the loading density (the number of atoms per unit area of $\{hkl\}$ system of net-planes [24]). This constitutes that $g$ is greater for a denser atomic net-plane system. In general, reducing the loading density of net-planes, the lattice imperfections diminish thus the coefficient $g$.

### 4. PARAMETERIZATION OF THE RELATIONSHIP BETWEEN LEVELS OF INTERACTION CONTROLLED BY $I_0$-INTENSITY

The levels of interaction of the diffraction process at a series of measurements of a reflection are defined by respective change of the incident X-ray beam intensity $I_0$. The controlled variation of the $I_0$-intensity can be caused by ether transmission factor of a thin foil crossed by the incident beam or stepwise reduction of the generator current of the XRD apparatus [26]. Meanwhile, the last type of the procedures may supply information that is representative for the internal consistency of the XRD apparatus, i.e. for its capability to collect precise and accurate data. Below this idea is depicted.

#### 4.1. Expressing the relationship $R$ between a couple of levels of interaction controlled by generator current

Suppose a reflection is measured in succession at intensities, $I_{0,i}$, $I_{0,2i}$, $I_{0,3i}$, and $I_{0,4i}$, of the incident beam, where the generator tension $V$ is constant, and the reduction of the values of the generator current $i$ is dependent on geometric progression ($i=2^{i=4}i^{i=8}$). In the equations listed inside of Fig. 2, $A$ is a constant, $V_k$ is the critical excitation

Fig. 2. Parameterization of the relationship between couples of levels of interaction of the diffraction process. Since diffraction and extinction are indissolubly linked (see (2) and (11)), $I_{kin}$ and $g$ define in the same way any level of interaction and, hence, the relationship $R$ between couples of neighbor levels of interaction (18), (22) and (23).
potential of the $K\alpha$ radiation, and $n \approx 1.5$ [27]. The intensity $I_0$ quantifies any level of interaction of the diffraction process simultaneously by quantities both the kinematical intensity $I_{\text{kin}}$ (2) and the SE coefficient $g$ (11). Then, in the frames of the straight proportionality between $I_0$ and $i$ ($I_0 = Ai(V - V_0)$), the parameter $R_{i,p}$ defines the relationship between the first couple of levels of interaction:

$$I_{0,i}/I_{0,i^*} = I_{\text{kin},i}/I_{\text{kin},i^*} = g_i/g_{i^*} = i/i^* = R_{i,i^*}$$  \hspace{1cm} (18)

The intensities, $I_{\text{kin},i^*} \leftrightarrow I_{\text{kin},i^*}$, defining the first and second levels of interaction (Fig. 2) can be expressed respectively by reforming (1) in succession with (5), (6) and (9), i.e.:

$$I_{\text{kin},i} = \left\{\mu \left[ \mu - k_i I_{m,i} \left( p_2 / p_1^2 \right) \right] \right\} I_{m,i^*},$$  \hspace{1cm} (19)

$$I_{\text{kin},i^*} = \left\{\mu \left[ \mu - k_i I_{m,i^*} \left( p_2 / p_1^2 \right) \right] \right\} I_{m,i^*^*}$$  \hspace{1cm} (20)

Solving (19) and (20) for $k_{i,i^*} = k_i = k_{i^*}$, with taking into account for $R_{i,i^*}$ from (18), yields

$$k_{i,i^*} = \frac{\mu \left[ R_{i,i^*} - \left( I_{m,i} / I_{m,i^*} \right) \right]}{I_{m,i} \left( p_2 / p_1^2 \right) R_{i,i^*} - 1}.$$  \hspace{1cm} (21)

Hereafter any coefficient of the type $k_{i,i^*}$ will be denoted with indices $i$ and $i^*$ corresponding to the values of the generator current applied to the measurement of the respective couple of intensities, $I_{m,i} \leftrightarrow I_{m,i^*}$, used for its expressing. With a view to expressing the parameters $k_{i,i^*}$ and $R_{i,i^*}$ by using measured intensities alone, one needs additional data. Then, by analogy of (18), the parameters $R_{i,i^*}$ and $R_{i,i^*,i^*}$, corresponding to the first-neighbour levels of interaction, are defined respectively with

$$I_{0,i}/I_{0,i^*} = I_{\text{kin},i}/I_{\text{kin},i^*} = g_{i}/g_{i^*} = i/i^* = R_{i,i^*},$$  \hspace{1cm} (22)

$$I_{0,i^*}/I_{0,i^*^*} = I_{\text{kin},i^*}/I_{\text{kin},i^*^*} = g_{i^*}/g_{i^*^*} = i^*/i^{*^*} = R_{i,i^{*^*}},$$  \hspace{1cm} (23)

where the value of any of the parameters

$$R_{i,i^*} = R_{i^*,i} = R_{i,i^{*^*}}$$  \hspace{1cm} (24)

has to be equal to any of the respective ratios defined by the values of the generator current

$$i/i^* = i^*/i^{*^*} = i^{*^*}/i^{***} = 2.$$  \hspace{1cm} (25)

By virtue of (14), $k$ is constant for any level of interaction under otherwise equal condition, i.e.

$$k_i = k_{i^*} = k_{i^{**}} = k_{i^{***}}.$$  \hspace{1cm} (26)

Then, the intensities, $I_{\text{kin},i^*} \leftrightarrow I_{\text{kin},i^*}$, are expressed by analogy of (19) and (20), respectively:

$$I_{\text{kin},i^*} = \left\{\mu \left[ \mu - k_{i,i^*} I_{m,i^*} \left( p_2 / p_1^2 \right) \right] \right\} I_{m,i^*};$$  \hspace{1cm} (27)

$$I_{\text{kin},i^{*^*}} = \left\{\mu \left[ \mu - k_{i,i^{*^*}} I_{m,i^{*^*}} \left( p_2 / p_1^2 \right) \right] \right\} I_{m,i^{*^*}}.$$  \hspace{1cm} (28)

Solving (27) and (28) for $k_{i,i^*} = k_{i^*} = k_{i^{**}}$, with accounting for $R_{i,i^*}$ from (23) yields

$$k_{i,i^*} = \frac{\mu \left[ R_{i,i^*} - \left( I_{m,i} / I_{m,i^*} \right) \right]}{I_{m,i} \left( p_2 / p_1^2 \right) R_{i,i^*} - 1}.$$  \hspace{1cm} (29)

Now, solving (21) and (29) for $R_{i,i^*} = R_{i,i^{*^*}}$, under nullifying the extinction effect by equating $k_{i,i^*} = k_{i,i^{*^*}}$, yields

$$R_{i,i^*} = \frac{I_{m,i} I_{m,i^*} \left( I_{m,i} - I_{m,i^*} \right)}{I_{m,i} I_{m,i^{*^*}} \left( I_{m,i} - I_{m,i^{*^*}} \right)}.$$  \hspace{1cm} (30)

By analogy, one can derive the relationship between the second-neighbour levels of interaction assuming that $R_{j,j^*} = R_{j^*,j}$ and $R_{j,j^{**}} = R_{j^{**},j}$ (see Figure 2) as well. The coefficients $k_{j,j^*}$ and $k_{j,j^{**}}$ are then expressed with the couple of intensities $I_{\text{kin},j^*} \leftrightarrow I_{\text{kin},j^{**}}$ and $I_{\text{kin},j} \leftrightarrow I_{\text{kin},j^{*^*}}$, (see (19)↔(27) and (20)↔(28), respectively:

$$k_{j,j^*} = \frac{\mu \left[ R_{j,j^*} - \left( I_{m,j} / I_{m,j^*} \right) \right]}{I_{m,j} \left( p_2 / p_1^2 \right) R_{j,j^*} - 1},$$  \hspace{1cm} (31)

$$k_{j,j^{**}} = \frac{\mu \left[ R_{j,j^{**}} - \left( I_{m,j} / I_{m,j^{**}} \right) \right]}{I_{m,j} \left( p_2 / p_1^2 \right) R_{j,j^{**}} - 1}.$$  \hspace{1cm} (32)

At the end, solving (31) and (32) for $R_{j,j^*} = R_{j,j^{**}}$, under nullifying the extinction effect by equating $k_{j,j^*} = k_{j,j^{**}}$, yields

$$R_{j,j^*} = \frac{I_{m,j} I_{m,j^*} \left( I_{m,j} - I_{m,j^*} \right)}{I_{m,j^{**}} I_{m,j^{*^*}} \left( I_{m,j} - I_{m,j^{*^*}} \right)}.$$  \hspace{1cm} (33)

Variation of the levels of interaction, and hence of $I_{\text{kin}}$ and $g$, with control has an additional advantage in that it allows for the capability for internal experimental checks that are based on using of instrumental variables. Actually, the generator current $i$ is incorporated implicitly in this technique as referent parameter. For instance, the capability of the measurement tool to collect accurate and precise
data is controlled by the degree of approximation of \( R \) to the ratio \( \frac{i}{i^{*}} \).

5. EXPRESSING THE EXTINCTION-FREE POLE DENSITY \( P \)

By definition, the extinction-induced systematic error of the pole density is expressed with the difference \( \Delta P_{m} \) between \( P \) and \( P_{m} \), i.e.

\[
\Delta P_{m} = P - P_{m},
\]

(34)

\[
P = I_{\text{kin}}/I_{\text{kin}}^{r},
\]

(35)

\[
P_{m} = I_{m}/I_{m}^{r}.
\]

(36)

Here \( P \) and \( P_{m} \) are defined by using the kinematical intensities from (2) and (4) and the measured intensities of the textured sample, \( I_{m} \), and powder standard, \( I_{m}^{r} \), respectively. To express \( P \) by measured intensities, a proper procedure is designed to data collection (Fig. 3). In this respect, the same reflection of textured sample and powder standard is measured at a series of the incident beam intensities \( I_{0i}, I_{0i^{*}}, I_{0i^{**}}, \) and \( I_{0i^{***}} \) caused by stepwise reduction of the values of the generator current, i.e. \( i, i^{*}, i^{**}, \) and \( i^{***} \).

Any of the particular levels of interaction is characterized by respective kinematic, \( I_{\text{kin}} \), intensities, the pole densities, \( P \) and \( P_{r} \), and the coefficient \( k \). According to the definitions (14) and (16), \( k \) is the same for each of the levels of interaction and \( P_{r} \) is equal to unit. Then, \( P_{r} \) is expressed by the kinematic intensities corresponding to the first couple of levels of interaction measured at the intensity \( I_{0i}^{r} \):

\[
P = I_{\text{kin}}/I_{\text{kin}}^{r}.
\]

(37)

where

Fig. 3. Data collection procedure that is designed by the levels of interaction of the diffraction process. The same reflection of textured sample and random standard is measured at a series of the incident beam intensities \( I_{0i} \) caused by variation of the generator-current values \( i \).
Solving the system of equations (19) and (38) for \( k_1 = k_1^* \) (see (16)) yields:

\[
I_{kin,i} = \left\{ \frac{\mu}{\left[ \mu - k_1^* I_{m,i}^* \left( p_2 / p_1^2 \right) \right]} \right\} I_{m,i}^* \tag{38}
\]

Further, by analogy of (37), we write for the second couple of levels of interaction measured at the intensity \( I_{0,i} \):

\[
P_{i*} = I_{kin,i*} / I_{kin,i*}^* \tag{40}
\]

where

\[
I_{kin,i*} = \left\{ \frac{\mu}{\left[ \mu - k_1^* I_{m,i}^* \left( p_2 / p_1^2 \right) \right]} \right\} I_{m,i}^* \tag{41}
\]

Solving the system of equations (20) and (41) for \( k_{i*} = k_{i*}^* \) (see (16)) yields:

\[
k_{i*} = \frac{\mu \left[ P_{i*} - I_{m,i}^*/I_{m,i}^* \right]}{I_{m,i}^*/p_2/p_1^2 \left[ P_{i*} - 1 \right]} \tag{42}
\]

At the end, solving (39) and (42) for the unknown extinction effects \( k_i = k_{i*} \) under nullifying the extinction effects \( k_i = k_{i*} \) yields an extinction-free value for the pole density corresponding to the first and second couples of levels of interaction:

\[
P_{i*,*} = \frac{I_{m,i} / I_{m,i}^* \left( I_{m,i}^* - I_{m,i}^* \right)}{I_{m,i}^* / I_{m,i}^* \left( I_{m,i}^* - I_{m,i}^* \right)} \tag{43}
\]

Therefore, starting from kinematic definitions (37) and (40) for the pole density, an operative formula (43) that is in exact accord with the kinematic theory is derived using measured intensities. Following the same procedure, extinction-free data for the pole density \( P_{i*,i**}, P_{i*,i***}, P_{i*,i****}, \) corresponding to respective combination of the other couples of levels of interaction are achieved as well. Once determined, the empirical extinction coefficient \( k \) can be employed for calculation of the kinematic intensities, the SE coefficient \( g \) defined by (17a) and the SE correction from (17b).

6. EXPERIMENTAL, RESULTS AND DISCUSSION

As a model, an electrodeposited nickel coating (Ni38) was used. It represents fiber texture with a main <100> component. The 200 reflection was measured with conventional goniometer using CuKα radiation separated by graphite focusing monochromator. The divergence slit was 1/2°, and receiving one 0.05 mm. The four step measurement procedure, shown in Figure 2, was carried out in such a way to compensate the stepwise decrease of the generator current from \( I \) to \( I^{**} \) (\( i=2i^*\equiv4i^**\equiv8i^***\)) by respective increase of the data collection time per scanned step from \( \tau \) to \( \tau^{***} \), i.e. \( i\tau=i^*\tau^*+i^**\tau^**+i^***\tau^*** \). The compensative condition ensures the same statistical errors in the respective points of the measured profiles.

Fig. 4 illustrates the proportionality between coefficient \( g \) and incident beam intensity \( I_0 \). The ordinate axis represents the values of the coefficient \( g \) determined at different levels of interaction of the diffraction process. The abscissa axis shows that the \( I_0 \)-reduction is dependent on geometric progression, i.e. \( I_0/R^n (n=0,1,2,3) \) where \( R \) is the ratio between neighbor levels of interaction. Figs. 4a and 4b

\[ \image \]

\[ \text{Fig. 4. a) Plot of the SE coefficient } g^n \text{ vs. incident beam intensity } I_0 \text{ whose stepwise reduction is dependent on geometric progression } I_0/R^n (n=0,1,2,3), \text{ where } R = 2.00213 \text{ is the ratio between neighbor levels of interaction. The measurement of the 200 reflection of Ni38 sample representing sharp } <100> \text{ texture } (P=35) \text{ is carried out by CuKα-radiation. b) Analogical plot for the SE coefficient } g' \text{ of random standard: the conditions of its measurements are the same.} \]
show the degree of extinction effects measured at the textured sample (Fig. 4a) and random standard (Fig. 4b), respectively. The scale between $g$ values, corresponding to the textured sample and random standard is equal exactly to $P = \frac{g_{\text{tx}}}{g_{r}}$.

In Table 1, results are listed about the pole density. The first column shows that the stepwise reduction of the incident beam intensity is dependent on geometric progression, $I_{0}R^{-n}$ ($n=0,1,2,3$), where $R$ is the ratio between neighbor levels of interaction caused by the generator current. The second column contains extinction-free data, and third column contains data affected by extinction. While the extinction-free data represent constant values, the extinction-affected data suffer systematic errors $\Delta P$ of different values. The systematic error is highest at the highest level of interaction and lowest at the lowest level of interaction. The percentage errors vary from about 6% to less than 1%.

7. CONCLUDING REMARKS

The main advantage of this approach is to gain accuracy and, hence, physical reality of the data. Accuracy can only be gained by care in the design and implementation of an experiment. Actually, this approach offers a possibility for existing methods both to be re-considered and improved for texture characterizations and to develop new ideas. In general, one can state that no problem related to using the integral intensities can be correctly solved if secondary extinction effects are not nullified.

Since diffraction and extinction are indissolubly linked, the formalism developed here is valid for single crystals, textures and crystalline powders. The loading density (number of atoms per unit area of a net-plane) that is the source of the crystal anisotropy unifies the general behaviour of these three structural forms.

APPENDIX A

Source of the coefficient $g$ anisotropy

To find the source of the anisotropic nature of the coefficient $g$, one has to resort to $P$. Since $P$ comprises as a whole the crystallographic, textural and microstructural anisotropy, the resulting anisotropy of the coefficient $g$ is synthesized by their contributions to the probing direction. Suppose the space-angle element $d\Omega$ covers the range of the probing direction and the volume element $dV$ contains very many crystallites [16]. Following (3), one can write an extended expression for the factor $P$:

$$
\frac{(dV/V)}{d\Omega} = P = \vartheta m.
$$

Here $m$ is all the number of crystallites per unit volume of the sample, and $\vartheta$ is the average volume of the crystallites. If the crystallite size $D$ is defined as the cube root of the average crystallite volume, $\vartheta^{1/3}$, one can rewrite (44) as

$$
P = D \vartheta^{2/3} m.
$$

where $\vartheta^{1/3}$ is the area of the crystallographic plane contributing to reflection. Replacing $P$ with its corresponding expression from (45) transforms (11) into

$$
g = D \vartheta^{2/3} mG,
$$

where $G = kl_S S/2\mu$ is a number. Further, one can represent the crystallite size as:

$$
D = Nd,
$$

where $(N + 1)$ is the number of the $(hkl)$ atomic planes having net-plane spacing, $d$. Kleber (1970) showed that $d$ is directly proportional to loading density $L$ (the number of atoms per unit area of a net-plane):

$$
d = V_c L,
$$

where $V_c$ is the volume of the primitive cell. By virtue of (47) and (48), from (46) an expression follows that connects the coefficient $g$ with the loading density $L$:

$$
g = LNV_c \vartheta^{2/3} mG
$$

Evidently, the coefficient $g$ depends on the crystallographic direction by means of the loading density. This outcome qualifies $L$ as the source of the extinction anisotropy and constitutes that the coefficient $g$ is grater for a denser atomic net-plane system under otherwise equal conditions. In general, reducing the loading density of net-planes, the lattice imperfections diminish thus the coefficient $g$.

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ЕКСТИНКЦИЯ В ТЕКСТУРИ: АНУЛИРАНЕ НА ЕКСТИНКЦИОННИ ЕФЕКТИ

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(Резюме)

Отчитайки вторичната екстинкция (ВЕ) в едно кристалографско направление, аналитичен метод е описан за рентгеново дифракционно характеризиране на текстури чрез анулиране на екстинкционни ефекти. За тази цел е извършено подходящо преразглеждане на природата на екстинкционните коефициенти. Показано е, че докато коефициентът на ВЕ $g$ е пропорционален на произведението от полюсната плътност $P$ и интензитета $I_0$, то емпиричният екстинкционен коефициент $k$ е независим от отношението $g/PI_0$. На основата на неизменността на коефициента $k$ по отношение на $g/PI_0$, екстинкционният ефект се анулира чрез изравняване на два негови израза, дефинирани чрез интензитетите на едно отражение, измерено при серия от нива на взаимодействие, чието изменение се контролира от $P$ и $I_0$. Техници, представляващи разширени версии са разработени за (i) оценка на достоверността на контролираното изменение на нивата на взаимодействие чрез използване на инструментална променлива (силата на генераторния ток) и, оттук следва да се тества потенциалната възможност на рентгеновия апарат да събира точни и прецизни данни, и (ii) определяне на свободни от екстинкция данни на полюсната плътност, която е основен физичен параметър. Експерименталните резултати са дискутирани посредством влиянието на екстинкционния коефициент $g$ върху точността на определяне на полюсната плътност в идеалното $<100>$ направление на никелови текстури.