NULLIFYING THE EXTINCTION EFFECT IN XRD CHARACTERIZATION OF FIBRE TEXTURES

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To gain accuracy and, hence, physical reality of the data acquired by XRD measurements of fibre textures, a technique is elaborated to achieve experimental values, which are free of extinction effects. Its elaboration is based on combining basic definitions of the extinction theory and texture analysis. This technique is applicable to characterization of metal coatings that appear infinitely thick for X-rays. A nickel sample representing <100> + <221> texture components is used as a model. Resultant derived series of data on pole-density distribution of the {200} diffraction pole figure shows that the data corresponding to the main <100> texture component are strongly affected by extinction. On the contrary, due to definitions that require reduction of the intensity distribution to multiples of random density, the extinction-free values of the volume fraction of texture components do not differ substantially from those calculated by standard methods. Evidently, any of the standard methods for volume fraction measurements provides reasonable data if secondary extinction is even disregarded.

Key words: extinction, extinction-free data, fibre texture, pole density, volume fraction, X-ray diffraction

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I. INTRODUCTION

In order to evaluate pole-figure measurements quantitatively, one needs the normalization factor, which reduces measured intensity values to multiples of random density. For the case of fibre textures of electrolytically deposited layers, it has been shown that the normalization factor and hence the volume fraction of the texture components are calculated in a rather simple way requiring only one, incompletely measured, diffraction pole figure (Tomov et al., 1977; Tomov and Bunge, 1979). This method is here extended by a technique for analytical nullifying the extinction effects that is applied to measurements of pole density and volume fraction of texture components. To realize the purpose of this study, our concern is essentially with an exact relationship between diffraction and extinction at any of the levels of interaction between X-radiation and crystal medium.

II. BASIC DEFINITIONS OF EXTINCTION THEORY 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 (Sabine, 1988; Sabine, 1992; Zachariasen, 1967), the secondary extinction (SE) decreases the measured intensity I_m of a reflection and, hence, the level of interaction of the diffraction process by a factor of y, the extinction factor being defined by

$$I_m = yI_{kin} \ . \tag{1}$$

(Except for I_m intensity, further on subscript m will be used to denote either intensity quantities suffering SE or structural parameters that are affected by extinction-induced systematic errors). Here, I_{kin} is the intensity that a Bragg reflection would have if kinematical theory would apply exactly to the system being examined:

$$I_{kin} = PI_0SQ/2\mu \ . \tag{2}$$

Here, 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, μ is the ordinary linear absorption coefficient, and P is the pole density representing a volume fraction dV/V of crystallites whose <*hkl*> poles fall into an (infinitely small) space-angle element $d\Omega$ (Bunge, 1982):

$$(dV/V)/d\Omega = P \ . \tag{3}$$

In the case of pure SE, an expression is given for the extinction factor y (Chandrasekhar, 1960; Zachariasen, 1963):

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$$y = \mu / \mu_\varepsilon = \mu / \left[\mu + gQ\left(p_2 / p_1^2 \right) \right] ,$$ (4) wher μ_ε

is an effective absorption coefficient including a first order approximation for the SE correction ε (Zachariasen, 1963) as well:

$$\varepsilon = gQ\left(p_2 / p_1^2 \right) . \tag{5}$$

Here, g is the SE coefficient derived theoretically by Darwin (1922) as a constant for a sample defined by FWHM of a respective reflection, and the polarization p_2 / p_1^2 of the incident X-ray beam has been incorporated in ε by Chandrasekhar (1960) and Zachariasen (1963):

$$p_n = \left[1 + \cos^{2n}(2\theta_B) \right] / 2 , \tag{6}$$

where θ_B is the Bragg angle of reflection, and n=1,2.

III. HOW TO NULLIFY THE EXTINCTION EFFECT

To nullify the extinction effect by XRD characterization of a crystal medium and, particularly, of a texture, one of the general disadvantages of the conventional (Darwin-Zachariasen) approach to treating the problem of extinction (Darwin, 1922; Zachariasen, 1963 and 1967) needs to be properly reconsidered. This disadvantage concerns a discrepancy between the theoretical models of the inner morphology of the crystal and their experimental evidence. So that examining the relationship between diffraction and extinction at any of the levels of interaction, our attention was focused to account for the global effect of the crystal, textural and micro-structural anisotropies. As a relative volume fraction of crystallites contributing simultaneously to the diffraction process, the pole density P comprises all sources of these anisotropies that are inherent of any particular sample. Whereas the crystal anisotropy is defined by the loading density L_{hkl}, i.e. the number of atoms per unit crystal area (Kleber, 1970) of {*hkl*} net-plane system, textural and micro-structural anisotropies includes such sources as size, shape, dislocation substructure, crystallographic orientation and crystallite arrangement (Bunge, 1988). Hence, by its very nature P controls any particular reflection of the XRD pattern. In this context, accounting for P in the interpretation of experimental results, the general disadvantage of the conventional approach to treating the problem of extinction could be eliminated. Thus, combining diffraction equation (2) used in texture analysis with SE correction ε (5) given by Darwin (1922) and Zachariasen (1963) yields an expression that is *a theoretically derived version* of the SE correction ε introduced *a posteriori* by Bragg *et al*. (1921):

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$$\varepsilon = kI_{kin}\left(p_2/p_1^2\right) . \tag{7}$$

Here, k defines a relationship between diffraction and extinction:

$$k = 2g\mu/PI_0S . \tag{8}$$

Thus reconsidering the above mentioned authors, the SE corrections (5) and (7) are constrained to be equal in value, and their coefficients are enriched with a clear physical meaning. Since g is originally introduced as a constant (Darwin, 1922; and James, 1965), we would like to analyse how it is related to the diffraction process and especially to its level of interaction controlled by PI_0. In this respect, rewriting (8) as

$$g = PI_0S/2\mu k , \tag{9}$$

and supposing the lowest limiting value of the product ($PI_0 \rightarrow 0$) it becomes clear that, in the only case without diffraction ($I_{kin} \rightarrow 0$), there is no extinction ($g \rightarrow 0$). Hence, as I_{kin} is proportional to the factor PI_0, g should also be proportional. Evidently, at any level of interaction of the diffraction process, the measured intensity I_m is affected to a different extent by extinction.

To treat the behaviour of k with respect to the variation of the level of interaction, we suppose that for I_0=const the pole density changes from P to random density, i.e. P^r=1 and then (8) has to be rewritten as:

$$k^r = 2\mu g^r/I_0S . \tag{10}$$

Here, it is also considered that g^r is proportional to P^r. Dividing (8) by (10) and bearing in mind $P=g/g^r$ one yields the identity

$$k \equiv k^r . \tag{11}$$

The constancy of k is a precondition for nullifying the extinction effect. Such an intention is practically realized by equalizing a pair of its expressions that are defined by the intensities measured at a series of levels of interaction.

IV. QUANTIFYING THE INCOMPLETELY MEASURED MULTI-COMPONENT DIFFRACTION POLE FIGURE

Prior to consider the procedures for deriving extinction-free data on a diffraction pole figure, here we shall mention again earlier results on quantitative characterization of multi-component fibre texture (Tomov and Bunge, 1979). In this context, we suppose that the texture consists of c components whose crystallites are oriented with their $\langle uvw\rangle_c$ crystal direction parallel (within a

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few degrees) to the fibre axis. Any of these <*uvw*>$_c$ components is characterized by its relative volume fraction M_m^c that is defined by

$$\sum_c M_m^c = 1 \ . \tag{12}$$

To calculate M_m^c, one has to use a diffraction pole figure $I_m(\phi)$, that is assumed to be incompletely measured, i.e. it is available only in the range $0 < \phi \leq \phi_{\max} < 90^{\mathrm{o}}$. For such a type of pole figure it is found that the normalized area (intensity) $S_{m,j}^c$ of the *c* texture component contributing to the pole-figure peak number *j* has to be expressed by:

$$S_{m,j}^c = \int_{\phi=\phi_{j1}^c}^{\phi_{j2}^c} I_m(\phi)\sin\phi \ \ \mathrm{d}\phi = N_m \frac{z_j^c}{z} M_m^c \ . \tag{13}$$

Here, ϕ is an angle of the spherical polar coordinate system fixed to the specimen in such a way that the direction $\phi = 0$ coincides with fibre axis, [ϕ_{j1}^c, ϕ_{j2}^c] defines the range corresponding to the peak *j*, *z* is the number of the crystallographic equivalent <*hkl*> directions contributing to the pole figure, z_j^c is the number of the equivalent <*hkl*> directions of the *c* texture component contributing to the pole-figure peak number *j*, and N_m is the normalization factor that connects the pole density $P_m(\phi)$ with the intensity $I_m(\phi)$ measured at ϕ-angle, i.e.

$$I_m(\phi) = N_m P_m(\phi) \ . \tag{14}$$

From (13), an expression follows for determination of the volume fractions:

$$M_m^c = \frac{z}{z_j^c} \frac{S_{m,j}^c}{N_m} \ . \tag{15}$$

Since the volume fractions M_m^c are independent of the number of the specific peak *j*, the S_j^c / z_j^c ratio is also independent of *j*. Hence, the normalization factor can be determined using any one peak of each texture component and summing up over *c* that is over all texture components, yields:

$$N_m = z \sum_c \frac{S_{m,j}^c}{z_j^c} \ , \tag{16}$$

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where j denotes one of the peaks belonging to the texture component c. Once the factor N_m is known, the volume fractions M_m^c as well as the pole density distribution of the $\{hkl\}$ pole figure $P_m(\phi)$ can be obtained from (15) and (14), respectively.

V. NULLIFYING EXTINCTION EFFECTS BY CALCULATION OF POLE DENSITIES AND VOLUME FRACTIONS OF TEXTURE COMPONENTS

To define extinction-free pole density P or volume fractions of texture components, a proper procedure is designed for data collection (Figure 1). To this end, the same diffraction pole figure of textured sample is twice measured by change of the incident beam intensity from $I_{0,i}$ to $I_{0,i*}$ caused by a stepwise decrease of the generator current values from i to $i*$ (i=2$i*$) according to:

$$I_0 = Ai(V - V_k)^n \ . \tag{17}$$

Here, A is a constant, V_K is the critical excitation potential of the K_α radiation, and n=1.5 (Guinier, 1956). As a whole, the measurement procedure is carried out at constant generator tension V. Figure 1 shows that any pair of the particular levels of interaction measured at the same ϕ angle of the diffraction pole figure is characterized by: (i) kinematical intensities, $I_{kin,i}(\phi) \leftrightarrow I_{kin,i*}(\phi)$; (ii) respective normalization factors, $N_{kin,i} \leftrightarrow N_{kin,i*}$; (iii) extinction-free pole densities, $P(\phi) \leftrightarrow P^r$ (P^r=1 (Bunge, 1982)) and the coefficient $k = k_i = k_i^r = k_{i*} = k_{i*}^r$ that according to (11) is the same for each of the levels of interaction (Tomov, 2011).

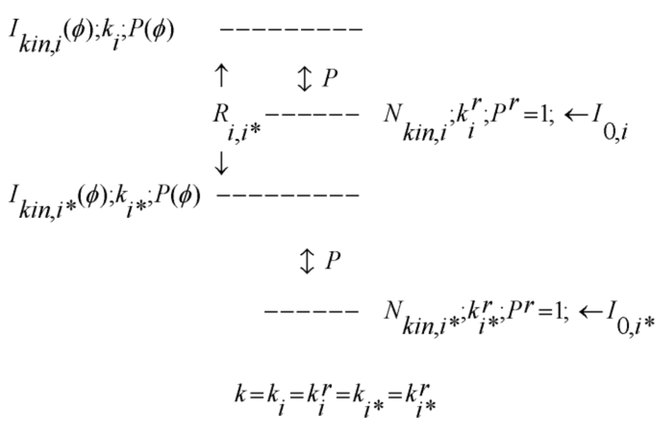

Figure 1. Prescription for data collection procedure based on controlled variation of the levels of interaction of the diffraction process. These levels were quantified using parameters within the kinematical approximation. The relationship $R_{i,i*}$ between a pair of levels of interaction controlled by the incident beam intensities $I_{0,i}$ and $I_{0,i*}$ is caused by variation of the generator current from i to $i*$ (i=2$i*$), respectively. The angle ϕ is defined to the sample normal direction.

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In case of the diffraction pole figure measured at *i* current density, we write:

$$P(\phi) = I_{kin,i}(\phi)/N_{kin,i} \quad . \tag{18}$$

Using (1), (4) and (7), the kinematical intensity from (18) is expressed by the respective measured intensity I_m:

$$I_{kin,i}(\phi) = \{\mu/[\mu - k_i I_{m,i}(\phi)(p_2/p_1^2)]\} I_{m,i}(\phi) \ . \tag{19}$$

Since the $N_{kin,i}$ factor represents intensity reduced to multiples of random distribution, it has to be expressed by analogy of (19):

$$N_{kin,i} = \{\mu/[\mu - k_k^r N_{m,i}(p_2/p_1^2)]\} N_{m,i} \ . \tag{20}$$

Solving the system of equations (19) and (20) for $k_i = k_i^r$ (see (11)) yields the expression:

$$k_i = \frac{\mu[P(\phi) - (I_{m,i}(\phi)/N_{m,i})]}{I_{m,i}(\phi)(p_2/p_1^2)[P(\phi) - 1]} \ . \tag{21}$$

The second measurement of the same pole figure, carried out at *i** current density, defines the set of expressions for $P(\phi)$, $I_{kin,i*}(\phi)$, $N_{kin,i*}$, and k_{i*}, accordingly:

$$P(\phi) = I_{kin,i*}(\phi)/N_{kin,i*} \ , \tag{22}$$

$$I_{kin,i*}(\phi) = \{\mu/[\mu - k_{i*} I_{m,i*}(\phi)(p_2/p_1^2)]\} I_{m,i*}(\phi) \ , \tag{23}$$

$$N_{kin,i*} = \{\mu/[\mu - k_{i*}^r N_{m,i*}(p_2/p_1^2)]\} N_{m,i*} \ , \tag{24}$$

$$k_{i*} = \frac{\mu[P(\phi) - (I_{m,i*}(\phi)/N_{m,i*})]}{I_{m,i*}(\phi)(p_2/p_1^2)[P(\phi) - 1]} \ . \tag{25}$$

Solving (21) and (25) for $P(\phi)$ under nullifying the extinction effect by equalizing the coefficients k_i=k_{i*} yields extinction-free value of the pole density corresponding to the ϕ angle:

$$P(\phi) = \frac{I_{m,i}(\phi) I_{m,i*}(\phi)(N_{m,i} - N_{m,i*})}{N_{m,i} N_{m,i*}(I_{m,i}(\phi) - I_{m,i*}(\phi))} \ . \tag{26}$$

This procedure is applied for any measured ϕ angle of the diffraction pole figure.

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To calculate extinction-free volume fraction M^c of the c component, the normalized intensity (area) $S^c_{m,j}$ of the c component contributing to the pole-figure peak number j has to be corrected for extinction. Under XRD measurement conditions defined by $I_{0,i}$ and $I_{0,i*}$ (see Figure 1), (13) is respectively rewritten as:

$$S^{c,j}_{kin,i} = \left\{\mu / \left[\mu - k^r_i S^{c,j}_{m,i}\left(p_2/p^2_1\right)\right]\right\} S^{c,j}_{m,i} = N_{kin,i}\frac{z^c_j}{z_{hkl}} M^c \ . \tag{27}$$

$$S^{c,j}_{kin,i*} = \left\{\mu / \left[\mu - k^r_{i*} S^{c,j}_{m,i*}\left(p_2/p^2_1\right)\right]\right\} S^{c,j}_{m,i*} = N_{kin,i*}\frac{z^c_j}{z_{hkl}} M^c \ . \tag{27a}$$

Replacing $N_{kin,i}$ with its corresponding expression from (20) in (27), one obtains:

$$S^{c,j}_{kin,i} = \left\{\mu / \left[\mu - k^r_i S^{c,j}_{m,i}\left(p_2/p^2_1\right)\right]\right\} S^{c,j}_{m,i} = \left\{\mu / \left[\mu - k_i N_{m,i}\left(p_2/p^2_1\right)\right]\right\} N_{m,i}\frac{z^c_j}{z} M^c \ . \tag{28}$$

Solving the terms on the right hand side of (28) for $k_i = k^r_i$ yields

$$k_i = \frac{\mu\left[M^c - \left(S^{c,j}_{m,i}/N_{m,i}\right)\left(z/z^c_j\right)\right]}{S^{c,j}_{m,i}\left(p_2/p^2_1\right)\left[M^c - \left(z/z^c_j\right)\right]} \ . \tag{29}$$

By analogy, combining (24) and (27a) under $k_{i*} = k^r_{i*}$ yields for k_{i*} an expression similar to that of (29):

$$k_{i*} = \frac{\mu\left[M^c - \left(S^{c,j}_{m,i*}/N_{m,i*}\right)\left(z/z^c_j\right)\right]}{S^{c,j}_{m,i*}\left(p_2/p^2_1\right)\left[M^c - \left(z/z^c_j\right)\right]} \ . \tag{30}$$

At the end, solving the system of equations (29) and (30) for M^c under nullifying the extinction effect k_i=k_{i*} yields an extinction-free value for the volume fraction of texture components:

$$M^c = \frac{z}{z_j}\left[\frac{S^{c,j}_{m,i} S^{c,j}_{m,i*}\left(N_{m,i} - N_{m,i*}\right)}{N_{m,i}N_{m,i*}\left(S^{c,j}_{m,i} - S^{c,j}_{m,i*}\right)}\right] . \tag{31}$$

By rule, the nullification procedure starts with kinematical definitions of the quantity under study and finishes with a solution of a system of two independent equations of k_i=k_{i*} that yields the operative formulae (26) and (31). Despite that these formulae contain only the series of measurement data affected differently by extinction, the value of any of the studied quantities ($P(\phi)$ and M^c) is constrained by its kinematical definitions to be extinction-free.

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VI. EXPERIMENTAL, RESULTS AND DISCUSSION

An electrodeposited nickel coating (Ni2), appearing infinitely thick to X-rays, was studied. It represents a fibre texture with a main <100> and twin-related <221> components. The {200} diffraction pole figure was measured twice within the interval $0 \le \phi \le 70^{\circ}$ using conventional texture goniometer and Ni filtered CuK_{α} radiation (Figure 2). The two scanned versions $I_{m,i}(\phi)$ and $I_{m,i^*}(\phi)$ of the {200} diffraction pole figure were carried out under a compensative condition $(i\tau = i^*\tau^*)$. This condition is experimentally realized by reducing the incident beam intensities from $I_{0,i}$ to I_{0,i^*} caused by a decrease of the generator current from i to i^* (i=2i^*) and a respective increase of the data collection time per scanned step from τ to τ^*. Under such a condition the $I_{m,i}(\phi)$ and $I_{m,i^*}(\phi)$ intensity distribution curves would be overlapped one with another if there is no extinction. Actually, because the two curves are affected differently by extinction, their course in the range of a few degrees around the ideal [001] direction of the pole figure is apparently distinguished. Each of the observed differences, at a given ϕ angle, is controlled by means of g coefficient dependence on the level of interaction PI_0 defined by (9) that predetermines the magnitude of the extinction correction (5). Thus, the experiment performed under a compensative condition predetermines the extinction effect to be such as it is, which reveals that the intensity distribution $I_{m,i}(\phi)$ is more strongly extinction-affected than $I_{m,i^*}(\phi)$. Finally, the compensative condition ensures (nearly) the same statistical errors for any pair of intensities measured at the same ϕ angle of both $I_{m,i}(\phi)$ and $I_{m,i^*}(\phi)$ curves.

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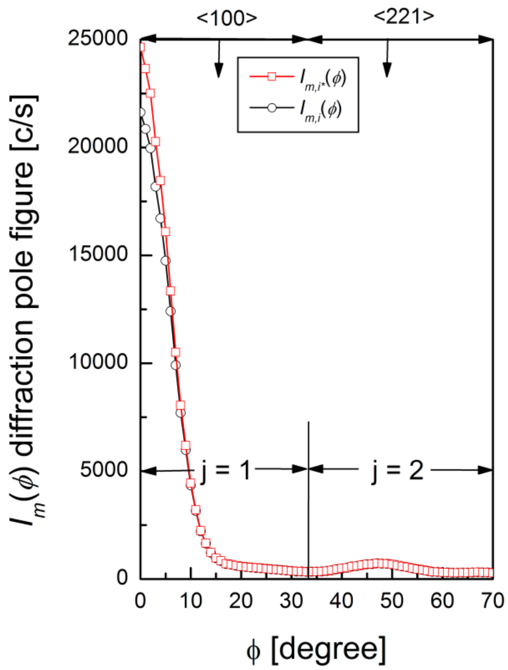

Figure 2. Two recorded variants $I_{m,i}(\phi)$ and $I_{m,i^*}(\phi)$ of the {200} diffraction pole figure of an electrodeposited nickel (Ni2) coating whose measurement was carried out under a compensative condition $(i\tau = i^*\tau^*)$ by using a texture goniometer and Ni filtered CuK_α radiation.

Further, the apparently distinguished differences between the two recorded versions $I_{m,i}(\phi)$ and $I_{m,i^*}(\phi)$ of the 200 diffraction pole figure are quantitatively illustrated in Figure 3. Except the pole density distributions $P_{m,i}(\phi)$ and $P_{m,i^*}(\phi)$ (shortly $P_m(\phi)$), Figure 3 displays the extinction-free $P(\phi)$ distribution as well. Using the $P(\phi)$ distribution as a reference, the percentage extinction-induced systematic errors $\Delta P_{m,i}(\phi)\%$ and $\Delta P_{m,i^*}(\phi)\%$ (shortly $\Delta P_m(\phi)\%$ are defined by

$$\Delta P_m(\phi)\% = 100\{\Delta P_m(\phi)/[P(\phi)+P_m(\phi)]/2\} . \qquad (32)$$

A variation of these errors, $\Delta P_{m,i}(\phi)\%$ and $\Delta P_{m,i^*}(\phi)\%$, is given in a range of the main <100> texture component, where the pole densities are rather high (Figure 4). Depending on the variation of the levels of interaction, these errors illustrate the extinction effects caused by particular variations of both values $P(\phi)$ and I_0. Evidently, the $P_m(\phi)$ distributions corresponding to the main <100> suffer extinction-induced systematic errors of different values.

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The errors are highest in the ideal direction (ϕ=0) of the fibre texture where the errors $\Delta P_{m,i}(\phi)\%$ and $\Delta P_{m,i*}(\phi)\%$ amount to 20.8 and 11.3%, respectively.

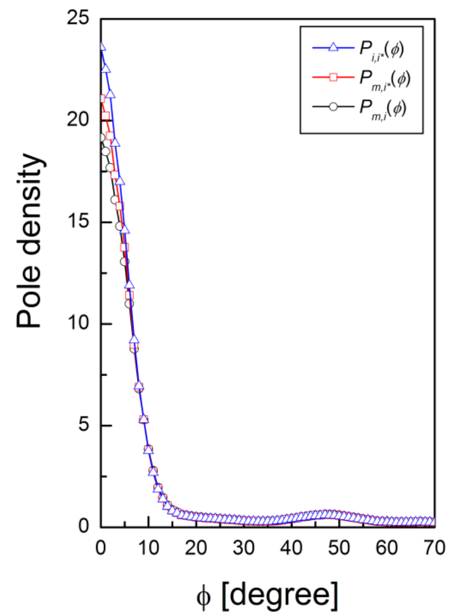

Figure 3. <100> pole density $P_{m,i}(\phi)$ and $P_{m,i*}(\phi)$ distributions of a nickel (Ni2) coating that were calculated directly by the measured intensities (see (14)), whereas $P(\phi)$ was calculated under nullifying the extinction effects (see (26)).

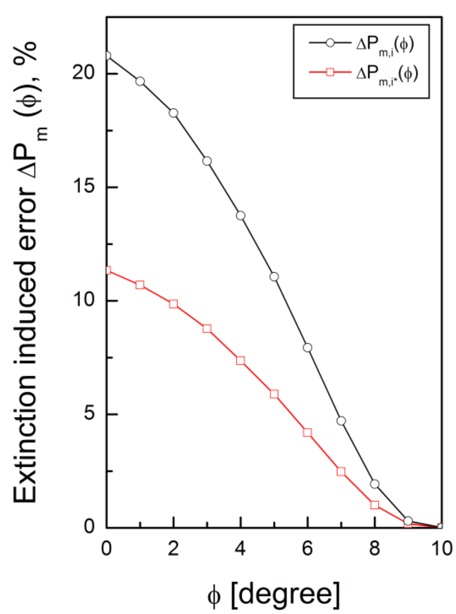

Figure 4. Variation of the percentage extinction-induced systematic errors of $\Delta P_{m,i}(\phi)\%$ and $\Delta P_{m,i*}(\phi)\%$ of the <100> pole density $P_{m,i}(\phi)\%$ and $P_{m,i*}(\phi)\%$ distributions, respectively, in a range where they are apparently distinguished.

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In contrast to the $P_{m,i}(\phi)$ and $P_{m,i*}(\phi)$ distributions corresponding to the <100> component that are strongly affected by extinction (see Figures 3 and 4), the volume fractions $M^c_{m,i}$ and $M^c_{m,i*}$, calculated by using the two measured versions of the same pole figure, are weakly affected by extinction. In this particular case, the 'weakening' of the extinction effects is artificially presupposed by the normalization procedure used for the calculation of $S^j_{m,i}$, $S^j_{m,i*}$, $N_{m,i}$, and $N_{m,i*}$ quantities. Any of these normalized quantities reduces simultaneously to multiples of random density both the measured intensity values and the corresponding extinction effects. Each of the resultant reductions is ϕ dependent, i.e. the contribution to the random density is proportional to the normalization term $\delta I_m(\phi)\sin\phi$, where δ [rad] is the scan step (13). Therefore, the volume fractions $M^c_{m,i}$ and $M^c_{m,i*}$ are calculated under implicitly reduced extinction effects comparable to those inherent of the random densities. That is why the absolute values of the percentage errors in the volume fractions $M^c_{m,i}$ and $M^c_{m,i*}$ deviate from the extinction-free volume fractions M^c by less than 3% (Table I).

Table I Volume fractions $M^c_{m,i}$ and $M^c_{m,i*}$ of the <100> and <221> texture components affected differently by extinction. These fractions were calculated according to (15) from the diffraction pole-figure versions $I_{m,i}(\phi)$ and $I_{m,i*}(\phi)$. The M^c volume fractions of the same components, calculated under extinction-free conditions, deviate by less than 3% from the extinction-affected ones.

<uvw>	$M^c_{m,i}$	$M^c_{m,i*}$	$M^c_{i,i*}$
<100>	0.702	0.711	0.723
<221>	0.298	0.289	0.277

VII. CONCLUDING REMARKS

The main advantage of this approach is to gain accuracy and, hence, physical reality of the data. The pole density distribution corresponding to the main <100> texture component is strongly affected by extinction.

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Due to definitions that require reduction of the intensity distribution to multiples of random density, any of the standard methods for volume fraction measurements provides reasonable data if the secondary extinction is even disregarded (Tomov *et al.*, 1977; Tomov and Bunge, 1979).

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