Phenomenon of primary and secondary extinction in textured material

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A new X-ray diffraction method is proposed for a more exact calculation of pole figures to determine the crystallite orientation distribution function (CODF) in textured materials, by the introduction of a correction of the integrated intensity of the diffracted beam due to the phenomenon of extinction. Besides, for the case of a symmetrical Bragg reflection from an "infinitely thick" flat parallel plate textured sample a simple solution is developed for the problem of separation and determination of primary and secondary extinction parameters, which can be present simultaneously. The determination of these parameters gives additional information about crystallite structure and allows us to evaluate the average subgrains size and their disorientation, respectively [1]. In this work, according to the dynamic diffraction theory, it is shown that the extinction length, which is directly connected with the phenomenon of primary extinction, in the Bragg geometry for σ polarization (perpendicular) is independent on the wavelength used for a given reflection. On the other hand, the additional contribution from secondary extinction depends on X-rays wavelength due to the change of effective absorption coefficient. Considering this fact, the calculations of pole density and the parameters of primary and secondary extinction are performed using the same strong reflection for two different wavelengths and, for one of these wavelengths, a second order of reflection. For confirmation of the proposed method a partially cold rolled aluminum sample and an aluminum powder standard sample were measured. The corrected pole densities, the values of primary and secondary extinction and the average sizes of perfect coherent areas and average angle of disorientation of subgrains in the selected directions were obtained. The obtained results show that the precise X-ray diffraction characterization of textured materials requires the consideration, in general, of primary as well as secondary extinction phenomenon.

Keywords: Primary and secondary extinction.

Se propone un nuevo método de difracción de rayos X para el cálculo más exacto de figuras polares para la determinación de la función de distribución de orientaciones de cristalitos (CODF) en materiales con textura, introduciendo una corrección para la intensidad integrada del haz difractado debida al fenómeno de extinción. Además, para el caso de la difracción simétrica de Bragg de una muestra texturada en forma de placa de "espesor infinito", se desarrolla una solución simple para la separación y determinación de los parámetros de extinción primaria y secundaria, las cuales pueden estar presentes simultáneamente. La determinación de estos parámetros da información adicional de la estructura de los cristalitos y nos permite evaluar el tamaño promedio de los subgranos y su desorientación respectivamente [1]. De la teoría de la difracción dinámica se demuestra que la longitud de extinción, que está directamente relacionada con el fenómeno de extinción primaria, en la geometría de Bragg para la polarización σ (perpendicular), es independiente de la longitud de onda usada para una reflexión dada. Por otra parte, la contribución adicional de la extinción secundaria depende de la longitud de onda de los rayos X debido al cambio del coeficiente de absorción efectiva. Considerando este hecho, se realiza el cálculo de la densidad de polos y los parámetros de extinción primaria y secundaria usando la misma reflexión fuerte para dos longitudes de onda diferentes y en el caso de una de estas longitudes de onda, para una reflexión de segundo orden. Para confirmar el método propuesto fueron medidas una muestra de aluminio parcialmente laminado en frío y una muestra de polvo de aluminio. Ahí fueron obtenidos: la densidad corregida de polos, los valores de la extinción primaria y secundaria y los tamaños promedios de los dominios coherentes y el ángulo promedio de desorientación de los subgranos en las direcciones seleccionadas. Los resultados muestran que la caracterización precisa por difracción de rayos X de materiales con textura requiere la consideración en general del fenómeno de extinción primaria así como de la secundaria.

Descriptores: Extinción primaria y secundaria.

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1. Introduction

In the kinematical theory of X-rays scattering it is considered that a crystal consists of small mosaic blocks randomly distributed. At calculation of the integrated intensity scattered by a whole crystal volume, we simply have added the reflected by individual blocks intensities, instead of amplitudes. Consequently, the task to compute the integrated intensity is reduced to counting the integrated intensity scattered by small size crystals. The amplitude of the waves, scattered by such crystal is small in comparison with the amplitude of the incident wave, and therefore, their interaction can be neglected [2-4]. However, if the crystal size is rather large and perfect in its whole volume, such neglect becomes un-

allowable. When the amplitudes of the scattered waves are summed up in a condition of interference, the amplitude of the scattered wave becomes comparable with the amplitude of the incident wave. Such case takes place already for a number of scattering layers of about $10^3 - 10^4$ (the size of crystal is about $10^{-4} - 10^{-5}$ cm) and it is necessary to take into account the interaction between the amplitudes. This phenomenon depends not only on the size of a crystal, but also on the type of reflection, in such way that a strong reflection requires a much less amount of layers than for a weak one to be observed.

This circumstance was clear already at the construction of the kinematical theory. For a series of reflections from some crystals the measured intensities essentially differed from the ones calculated according to the kinematical theory [1, 2]. The measured intensities were less, so the phenomenon of extinction due to power losses of energy in the diffracted beam was observed. In this connection, adequate corrections have been introduced into the kinematical theory [2, 5- 7]. Simultaneously with the kinematical theory, Darwin and Ewald created the foundation of the dynamic theory [2, 8] and later Zachariasen [6,7] considered the interaction between incident and scattered waves. The geometry of diffraction pattern in both theories practically matches and the results mainly differ at evaluation of intensities. So, if the mosaic structure consists of relatively large perfect blocks and there is essential interaction between amplitudes of incident and diffracted beams, the phenomena of extinction takes place within such blocks and is called primary extinction [1].

The model of a real crystal represents it as consisting of slightly disorientated, optically independent blocks [2]. If some of these blocks have their planes exactly parallel, the integrated reflection will be reduced not only by ordinary absorption, but also due to a further reflection by these blocks, away from the reflected beam of certain fraction of the radiation, which passes through them. The twice-reflected radiation from any blocks can be deflected into the reflected beam and again a certain fraction of this radiation will be deflected back into direction of the incident beam. But there is an essential difference between this process and the one, which takes place in large perfect crystal. Since the blocks are optically independent, there is no regular phase relation between the reflected radiation and in such a phenomenon we speak about summation of beams intensities [4]. The phenomenon of the power losses due to the fact that the incident as well as the diffracted beams are constantly reflected when they traverse the blocks that are in diffraction condition, is called secondary extinction. Both phenomena lead to an integrated intensity decrease but characterize different properties of mosaic crystal.

In many samples both phenomena can be present simultaneously, particularly, in good annealed textured materials and epitaxial layers [9]. The influence of extinction on the correct determination of the crystallite orientation distribution function (CODF) may be very strong and gives large errors [9]. There exist some complicated methods for separation of such

phenomena, however only for mosaic crystals [10, 11]. Unfortunately for textured materials the problem is more complex and not completely solved.

The aim of our work is to propose a comparatively simple solution to separate and determine the coefficients of primary and secondary extinction, as well as to correct pole density according to the determined extinction parameters, and from the coefficients of primary and secondary extinction, as an application, to evaluate the crystallite structure of a partially cold rolled aluminum sample.

2. Introducing the general terms for extinction

The phenomenon of primary extinction can be explained within the framework of dynamic theory in terms of block thickness l and extinction length Λ [1, 12]. For the Bragg case the extinction length Λ is

$$\Lambda = \frac{2\sqrt{\gamma^0 \left|\gamma^g\right|}}{k_0 \left|C\right| \left|\chi^g\right|}; \tag{1}$$

with

$$\chi^g = -\frac{1}{\pi} r_0 \lambda^2 \frac{F^g}{v} \tag{2}$$

and

$$k_0 = \frac{2\pi}{\lambda}, \qquad \gamma^g = \sin\left(\varphi - \theta_0\right), \qquad \gamma^0 = \sin\left(\varphi + \theta_0\right),$$

where: χ^g is the Fourier series components of polarizability, λ is the radiation wavelength, θ_0 is the diffraction angle, φ is angle between crystal surface and atomic planes, F^g is the structure factor, v is the unit cell volume, r_0 is the electron orbit radius, C is the polarization factor and k_0 is the wave vector of the incident beam.

The integrated coefficient of reflectivity depends on the value of the product $l\Lambda^{-1}$ as [12]:

$$R^g = \frac{\lambda \left|\gamma^g\right| th\left(l\Lambda^{-1}\right)}{\sin 2\theta_0 \Lambda}. \tag{3}$$

For a very thin crystal $l \ll \Lambda$ we obtain the well-known result of kinematical theory

$$R_{kin} = \frac{\pi k_o C^2 \left|\chi^g\right|^2 l}{2\sin 2\theta_0 \left|\gamma^0\right|}. \tag{4}$$

For a very thick crystal $(l \gg \Lambda)$ the $th(l\Lambda^{-1}) \cong 1$, so the integrated coefficient of reflectivity is

$$R^g_{dyn} = \frac{\lambda \left|\gamma^g\right|}{\sin 2\theta_0 \Lambda}. \tag{5}$$

For the case of not polarized radiation the integrated coefficient of reflectivity is [12]:

$$R_{dyn}^g = \frac{\pi}{\gamma^0 \sin 2\theta_0} \frac{1 + \cos 2\theta_0}{2} \left(\gamma^0 \left|\gamma^g\right| \chi^g \chi^{-g}\right)^{1/2}. \quad (6)$$

If we divide Eqs. (3) by (4) and use the definition for the extinction length Λ (1), we get

$$R^g / R_{kin} = \left(l/\Lambda\right)^{-1} th\left(l/\Lambda\right) = \varepsilon. \quad (7)$$

This ratio gives a method to determine the thickness of a coherent scattering volume, and thus the size of mosaic blocks. In other words, the ratio in Eq. (7) gives the correction for the primary extinction.

The effect of a secondary extinction on the integrated intensity is equivalent to an increment in effective absorption coefficient at a reflection angle and can be allowed to use a good approximation simply by an appropriate increase in μ_0 the absorption coefficient in the equation for the integrated reflectivity from the mosaic crystal. The phenomenon of secondary extinction depends on the degree of blocks varying orientations, which are randomly distributed with a depth [1,6,7].

In the case of a flat parallel thick plate for the symmetrical Bragg reflection the kinematical theory, with consideration of absorption, gives a simple equation for reflectivity of the diffracted beam [1] as

$$R_{kin} = \frac{Q_{kin}}{2\mu_0}, \quad (8)$$

where Q_{kin} is the kinematical integrated reflectivity per unit volume.

The correct form for the integrated reflectivity coefficient due to secondary extinction, if Q_{kin}^2/μ_0 can be neglected, as it is mainly observed for real mosaic crystals, and for σ - polarization (because the corrections for the two types of polarizations differ somewhat [13]), is given by the equation [1,6,7]:

$$R = \frac{Q_{kin}}{2\left(\mu_0 + gQ_{kin}\right)}, \quad (9)$$

where g is a constant of the mosaic crystal and depends on the characteristic of blocks distribution over the angles and is determined by the equation

$$gQ_{kin}^2 = \int_{-\infty}^{\infty} G^2(u)\, du, \quad (10)$$

where u is the angle of deviation of the normal of the diffracting planes of blocks from an average direction over the crystal and $G(u)$ is the fraction of blocks whose glancing angle of incidence on the crystal face is $\theta + u$ [1,11].

When both phenomena of extinction are present in a real mosaic crystal, the use of R^g instead of R_{kin}, with the assumption that blocks are all of the same average size, makes the correction for primary extinction. So Eq. (8) now will be

$$R_m = \frac{Q^g}{2\left(\mu_0 + gQ^g\right)} = \frac{Q_{kin}\varepsilon}{2\left(\mu_0 + gQ_{kin}\varepsilon\right)}. \quad (11)$$

If we separate these phenomena and determine the parameters of primary and secondary extinction we can get information about average size of blocks and average angle of their disorientation, respectively.

3. Evaluation process

Quantitative texture analysis belongs to the fundamental methods of structural characterization of materials and films, which give us the understanding of the interrelation of both structure and growth process or treatment as well as structure and properties. In practice, the indirect measurements of diffraction pole figures by means of intensities of X-ray diffracted radiation contain a reproducibility problem [14,15]. For texture analysis intensity measurements should come first and then data handling for pole figure representation, CODF calculation and interpretation of results. In these operations we must take into account different sources of errors and their influence on the result. For example, the tests on texture measurements and CODF calculations obtained from the same samples by different laboratories have shown considerable differences [15]. There are some mathematical and physical problems for calculation and interpretation [16], but the starting position is the measured integrated intensity of X-ray diffraction. In some reports it was shown that for textured samples the low-index diffraction lines and those related to main texture component are strongly affected by extinction, which causes a large systematic error in pole-density measurements [17]. The magnitude of this error is always negative, so it reduces the density of poles. In the proposed method [17] for correction of pole density it was assumed that only secondary extinction is present. Really both types of extinction can be present and all relations become more complicated and the solution of the problem will not be so simple.

As it was mentioned above the phenomenon of primary extinction can be explained by Eq. (7), in such a manner that an additional coefficient in the kinematical integrated reflectivity arises. This coefficient depends only on the ratio between l and Λ (see Eq. (7)). Let us use the Eq. (2) to represent the extinction length (1) as

$$\Lambda = \frac{\sin\theta v}{|C|\, r_0 \lambda F^g} = \frac{1}{2d} \frac{v}{|C|\, r_0 F^g}. \quad (12)$$

Taking into account that $sin\theta/\lambda = 1/2d$ and that the structure factor F^g for given reflection depends only on

$sin\theta/\lambda$ and is constant for different wavelengths, we will see that $\Lambda = constant/|C|$ for a given reflection regardless of X-ray radiation wavelength in the Bragg case. Thereby the coefficient of primary extinction ε according to Eq.(7) is independent on wavelength for the σ polarized radiation used and differs very small for nonpolarized radiation according to $(1 + \cos 2\theta)/2$ for small angle reflections with low indexes.

At the same time the coefficient of secondary extinction, as it was mentioned above, is the characteristic of the blocks distribution [1,6,7,10]. In textured materials for the given direction $\langle hkl \rangle$ the coefficient of secondary extinction for higher order reflections, since they belong to the same direction, must be constant. At different wavelengths diffractions for given direction the coefficient of secondary extinction is also constant as characteristic of the blocks distribution. At once, for different wavelengths the absorption coefficients μ_0 and the kinematical integrated reflectivities per unit volume Q_{kin} will change essentially and not identically. The kinematical integrated reflectivity per unit volume Q_{kin} for different reflections also will change essentially. Thus, the ratio between coefficients in effective absorption in Eq. (11) also will change.

The kinematical integrated intensity of an "ideal mosaic" crystal for a flat parallel thick plate is related to the kinematical integrated reflectivity Q_{kin} as [1]:

$$I_{kin} = \frac{Q_{kin}}{2\mu_0} I_0 \qquad (13)$$

where I_0 is the intensity of the incident beam. The intensity per unit ring length for a reflection from crystalline powder sample will differ from (13) by some coefficient K, which depends on a type of plane and reflection, distance from crystal to counter and wavelength [1].

In textured materials the value of the pole density P for a given direction $\langle hkl \rangle$ is defined by the ratio $P_m = Q_m^T / Q_m^P$, where Q_m^T and Q_m^P are the integrated reflectivity per unit ring length for the same reflection from textured and powder standard samples of the same substance, respectively. If in the investigated textured sample both types of extinction and pole density P affect the diffracted integrated intensity, this can be expressed as:

$$I_m^T = \frac{P Q_{kin}\varepsilon}{2(\mu_0 + gQ_{kin}\varepsilon)} K I_0 \,. \qquad (14)$$

For the selected reflection and radiation wavelength the values of kinematical reflectivity Q_{kin} and absorption coefficient μ_0 can be calculated. Using monochromatic incident beam, its partial polarization must be taken into account. As the integrated intensity measurements are made in a relative scale, measurements of textured and powder standard samples must be carried out at fixed all geometrical conditions, intensity of incident beam and density of textured and powder standard samples. In this case the value I_0 at measurements of power standard sample according to Eq. (13) can be represented as:

$$I_0 = \frac{2\mu_0 I_m^p}{K Q_{kin}}. \qquad (15)$$

Now we have, that for the given reflection, wavelength and all geometrical conditions fixed for measurements of textured and powder standard samples the ratio I_m^T/I_m^P contains three unknown values: P, ε and g, which are constant for it. All other terms in Eq. (16) can be calculated or determined experimentally. Thus, we need a system of three independent equations for a textured sample.

Since the theory of extinction [5-7] was developed for mosaic crystal, the statement that the parameter of secondary extinction g is constant for such a kind of crystalline structure is quite correct [1]. But as it was marked in Ref. 18 it is not yet widely appreciated that extinction depends on the orientation of the plane of incidence. This extinction anisotropy was shown, for example, for quartz in [19]. There are two possibilities for anisotropic extinction. The first one is due to anisotropy in coherent-domain shape (primary extinction). And the second one is due to anisotropy in mosaic spread (secondary extinction) [18]. In textured materials we have just such crystalline model and in consequence of different grains space distribution, the statement of a constant parameter of secondary extinction g in textured materials may be called into question and needs additional testing. Since the parameter of primary extinction determines the average coherent-domain shape, in another word the size of perfect area for a given direction, it also can differ for different directions. So, there is more reason to prepare these equations and carry out the measurements for the same direction $\langle hkl \rangle$ that allows us not only to correct the pole density according to phenomena of extinction, but also to determine the parameters ε and g for different directions.

For the same low-index reflection but with different wavelengths and for a high order reflection for one of the wavelengths and with the assumption that the primary extinction is absent for it, which is justified since for such high order reflection the extinction length Λ, which characterizes the area of coherent scattering for the appearance of the primary extinction phenomenon, increases essentially, we will have the following system of three equations:

$$\stackrel{hkl}{\frac{I_m^T}{I_{m(\lambda 1)}^p}} = A = \frac{P\mu_1\varepsilon}{(\mu_1 + gQ_{kin1}\varepsilon)},$$

$$\stackrel{hkl}{\frac{I_m^T}{I_{m(\lambda 2)}^p}} = B = \frac{P\mu_2\varepsilon}{(\mu_2 + gQ_{kin2}\varepsilon)}, \qquad (16)$$

$$\stackrel{n(hkl)}{\frac{I_m^T}{I_{m(\lambda 2)}^p}} = D = \frac{P\mu_2}{(\mu_2 + gQ_{kin3})}.$$

This system can be solved analytically and its solution gives us three parameters for textured material: the value of corrected pole density P, parameters of primary extinction ε and of secondary extinction g:

$$P = \frac{ABD\,(\mu_2 Q_{kin1} - \mu_1 Q_{kin2})}{AB\,(\mu_2 Q_{kin1} - \mu_1 Q_{kin2}) + D\mu_1 Q_{kin3}\,(A-B)},$$

$$\varepsilon = \frac{AB\,(\mu_2 Q_{kin1} - \mu_1 Q_{kin2}) + D\mu_1 Q_{kin3}\,(A-B)}{D\,(A\mu_2 Q_{kin1} - B\mu_1 Q_{kin2})}, \quad (17)$$

$$g = \frac{(B-A)\,D\mu_1\mu_2}{AB\,(\mu_2 Q_{kin1} - \mu_1 Q_{kin2}) + D\mu_1 Q_{kin3}\,(A-B)}.$$

These coefficients of extinction can be used for the evaluation of the average size and disorientation of domains.

4. Experimental procedure

We investigated an aluminum sample after partially cold rolling using Cu and Mo radiations. It has some texture and different diffraction directions $\langle 100\rangle$, $\langle 110\rangle$ and $\langle 111\rangle$ were tested. For a given direction the first and second order reflections were measured. An aluminum powder standard sample was measured also. From metallography it was observed that the sizes of grains were about 100 μm with some amount of defects in these grains (Fig.1).
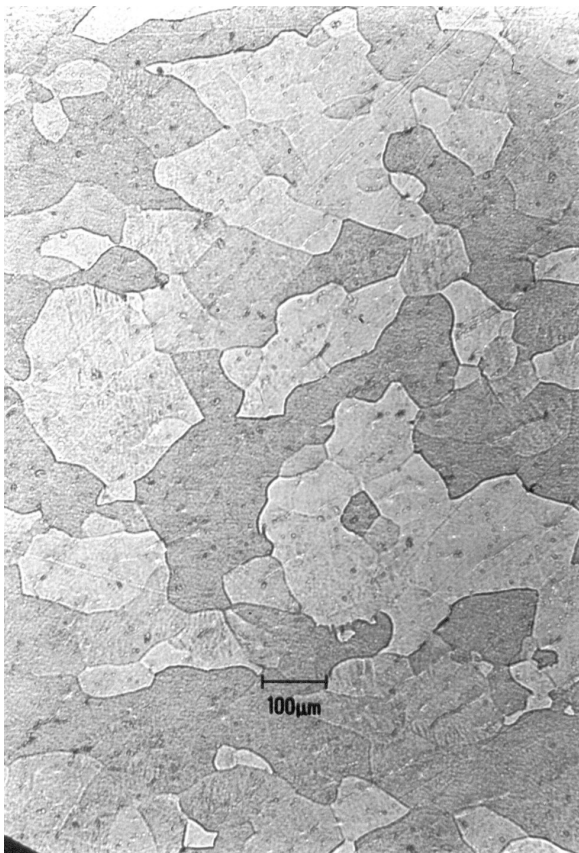

FIGURE 1. Micrograph of a partially cold rolled aluminum sample.

For such large grain size and preferred orientation the phenomena of primary and secondary extinction can take place. To compare the values of corrected pole densities, obtained by the proposed method with the same one, which can be obtained with the assumption that only secondary extinction is present the calculations according to [17] were done.

The absorption coefficients of aluminum for Cu - and Mo K_α- radiations are $\mu_{Cu} = 131,44 cm^{-1}$ and $\mu_{Mo} = 14,3 cm^{-1}$, respectively [20]. The results of calculations of main diffraction parameters and pole density for aluminum calculated directly from measured intensities for textured and standard powder samples according to the relation $P_m = I_m^T / I_m^P$ are presented in Table I.

TABLE I. Calculated parameters of diffraction in aluminum: Λ_{Cu}, Λ_{Mo} - extinction lengths, Q_{Cu}, Q_{Mo} - the kinematical integrated reflectivity per unit volume and pole densities $P_m^{Cu} P_m^{Mo}$ - for Cu and Mo K_α - radiations, respectively.

hkl	$\Lambda_{Cu}\mu m$	$\Lambda_{Mo}\mu m$	$Q_{Cu}10^2 cm^{-1}$	$Q_{Mo}10^2 cm^{-1}$	P_m^{Cu}	P_m^{Mo}
111	1.59	1.48	9.276	2.166	3.4	2.9
200	1.96	1.79	6.718	1.625	5.9	4.7
220	3.9	3.2	2.408	0.646	8.3	7.3
222	6.85	4.36	1.184	0.313	4.8	5.3
400	8.74	5.91	0.752	0.163		7.6
440		11.64		0.020		10.7

From Table I we see that pole densities calculated directly from measured intensities for textured and powder standard samples, as it is done commonly, which must be constant for the given direction are very different for different order of reflection or for the different wavelength used. Pole densities are somewhat less for Mo radiation and increase essential for high order reflections. The low-index reflections affected by extinction that leads to a reduction of a pole density. Such results indicate that an extinction phenomenon is present. For the second order reflections the difference between Λ_{Cu} and Λ_{Mo} is large due to the polarization factor and thus these reflections do not meet the assumption of equality of primary extinction. But they were used with the assumption that the phenomenon of primary extinction is inessential for them according to the large value of Λ. At once the pole density P and coefficient of secondary extinction g in the given direction are the same for both radiations for low-index as well as for high-index reflection.

The coefficient of primary extinction can be also represented as a function of the amount p of atomic layers in the coherent perfect volume scattering in the diffracted direction and the amplitude q, scattered by a single atomic plane for σ - polarization, as [11]:

$$\varepsilon = \frac{th\,(pq)}{pq}, \quad (18)$$

and

$$q = nd\lambda F r_0 \frac{1}{\sin\theta}, \qquad (19)$$

where n is amount of unit cells per unit volume and d is the spacing of the lattice-planes (h, k, l). The scattered amplitude q by an atomic plane can be calculated [20] and from the values of primary extinction ε it is very easy to evaluate the size of the perfect domains in our material as the amount of atomic layers p in coherent perfect volume scattering in the diffracted direction and the spacing of the planes d_{hkl}.

The coefficient of secondary extinction g depends on the characteristic angular block distribution. For a Gaussian distribution of blocks, which can be used for many cases, the average angle u of block disorientation is [11]:

$$\langle u \rangle = \frac{1}{2g\sqrt{2\pi}}. \qquad (20)$$

The results of the system solution according to Eqs. (17), obtained values of corrected pole densities by the proposed method P_{cor} and corrected pole densities by the one [17] with the assumption that only secondary extinction is present P_{sec}, the coefficients of primary ε and secondary g extinctions and also the average sizes of coherent perfect domains L and the average angle of domains disorientation u in the selected direction are shown in Table II.

TABLE II. Calculated parameters for a partially cold rolled aluminum sample: P_{cor} - pole density corrected by proposed method, P_{sec} - pole density corrected by method [17], ε - coefficient of primary extinction, g - coefficient of secondary extinction, q - scattered amplitude by atomic plane, L - average sizes of subgrains, u - average angle of disorientation of subgrains.

hkl	P_{cor}	P_{sec}	ε	g	$q \cdot 10^4$	L μm	$u$min
111	5.72	5.82	0.7	367	1.53	1.82	1.9
200	8.03	8.05	0.93	556	1.07	0.95	1.2
220	10.8	11.3	0.85	663	0.42	3.41	1

As can be seen from Table II for all directions the extinction phenomena, primary as well as secondary, are present, but their values and influence on the determined correct pole density are different. This is connected with different size and disorientation of domains in different directions. The smallest primary extinction is observed for (200) reflection and smallest average size of domains is at $\langle 100 \rangle$ direction. Though the primary extinction for (111) reflection is the largest, the largest average size of domains is at $\langle 110 \rangle$ direction. The scattered amplitude q for a (220) atomic plane is three times less than for a (111) one. So, to obtain the same value of primary extinction for (220) reflection we need larger size of domains. The sizes of perfect coherent areas are smaller in the order of average grains size, which was observed by metallography. This fact indicates the presence of

defects, in particular, dislocations in these grains. We also obtained a different value of the secondary extinction parameter for direction $\langle 111 \rangle$, $\langle 100 \rangle$ and $\langle 110 \rangle$ that may be connected with inelastic deformation properties of aluminum at texturing.

5. Conclusions

A new X-ray diffraction approach for more accurate pole-density determination of textured materials has been described. We have shown that the coefficient of primary extinction for a given low-index reflection in the symmetric Bragg case is independent on X-ray radiation wavelength. According to such independence a new general procedure is proposed for the separation and calculation of unknown coefficients of primary and secondary extinctions for textured materials. A correction of pole-density measurements was made using the two-wavelength and second-order reflection technique.

In general, the pole density is a fundamental quantity for texture presentation and characterization [21]. The calculated parameters showed that the extinction phenomena, primary as well as secondary, might be present even in slightly textured materials. It is shown that application of the analytic solution for the extinction parameters is extremely useful in the refinement of pole densities since this correction eliminates the perspective systematic error. The magnitude of such error in some cases exceeds two times the directly calculated pole density (see Tables I and II). The elimination of the systematic error is a notable result.

The values of the determined parameters for primary and secondary extinctions can be used to obtain additional information about crystallite structure and to evaluate size and disorientation of coherent perfect domains in such kind of materials. The average size of coherent perfect domains was different in different directions and was largest at the $\langle 110 \rangle$ direction, which may be connected with inelastic deformation properties of aluminum. The value of secondary extinction was also somewhat different for different directions. That can be explained by the difference between textured and mosaic crystals, where this coefficient may be constant in any direction. In the case of accounting only secondary extinction, the corrected pole density is somewhat above.

The precision in any X-ray diffraction characterization of textured materials could be enhanced using improved extinction corrections. The proposed method and evaluation of crystallite morphology can be very useful at investigations of textured materials after thermal annealing and other treatments, when changes in diffracted beam from textured samples can be connected not only with reorganization of pole density but also with extinction phenomena depending also on structural perfection of grains.

Acknowledgements

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