# SOME OBSERVATIONS ON THE BACKGROUND SCATTERING IN X-RAY PATTERNS AND ITS UTILIZATION FOR LINE PROFILE CORRECTION

By

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#### 1. Characteristics and causes of scatter

The background as observed on X-ray patterns represents a diffuse level of intensity appearing along the whole pattern, against which narrow zones of characteristic interference lines of higher intensity emerge. This background may be attributed, so far as its causes are being investigated, to external causes (producing *extraneous scattering*), as well as, to causes closely related to, and characteristic of the material tested (producing *significant scattering*), the latter being a product of interaction between X-ray photons and atoms of the specimen.

The main cause of extraneous scattering may be found in spectral heterogenity of the incident X-rays, being itself a consequence of insufficient monochromatization. Components of continuous or "white" X-ray radiation as reflected from reflecting atomic planes produce diffuse scattering effects around each line peak. Background intensity is increased by random scattering at the diaphragm and other accessories (filters, screens, etc.) of the recording apparatus, as well as by the so-called air scattering, which is due to ionization of gas molecules along the path of the X-rays.

In case of film recording the fog effect depends on quality and storage conditions of the silver bromide emulsion, while in case of using G-M counter tubes, cosmic radiation effects and other radioactive contaminations within the test space will contribute to the increase of background intensity.

Significant scattering may also be the result placed on several effects. Itis known that X-ray photons interact with electrons placed on several electron shell of atoms. In this case the energy state of the atom is increased to a higher level, from the normal state into an excited one, after photons of the incident X-ray beam having been absorbed by the atom. A coherent X-ray scattering effect is obtained, if the photons are emitted on a frequency identical with that of the incident beam, the atom thus returning to its normal state of energy. As a result of this coherent scattering interference lines obeying to Bragg's law, characteristic X-ray patterns are obtained, and in material testing these are evaluated by well-known processes.

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Also significant of the material, while much less utilized for X-ray diffractometry, are the following effects :

1. Incoherent or Compton scattering, which is realized by the emission of a lower-frequency photon simultaneously emitted with an electron, its intensity being a function of the scattering angle.

2. Fluorescent scattering or rather fluorescent radiation, propagating in every direction in a strictly identical manner, and having a wavelength exceeding that of the primary exciting radiation. This is observed, if the material to be tested is exposed to an X-ray radiation having a wavelength equal to, or less than, the  $\lambda$  value for the absorption edge K, L, M. . . of the specimen material. The harder the primary X-ray radiation, the more intense the fluorescent scattering, while for a gamma-radiation of extremely short wavelength, almost the total scattering effect will be due to this type of scatter.

Let us finally mention 3. the Raman scattering and 4. the Auger effect, which will generally also increase the diffuse background intensity level of X-ray patterns, although they will only have a lower effect. Wave mechanical interpretations for these phenomena are dealt with by literature [1]. Even a short summary of these has to be omitted from this paper, as it would exceed its scope.

5. For the sake of comprehensiveness *thermal scattering* must also be mentioned. A diffuse scattering effect may also be due to the fact that atoms in crystallites are performing a continuous thermal motion, and always present therefore some displacement as against this their equilibrium position as defined by the crystal lattice points. This thermal vibration will broaden the interference lines, but at the same time intensify the continuous background level, proportionally to the increase in reflection angle, although not necessarily in a monotonic manner [2].

6. Similarly *imperfections of the crystal lattice* of the material to be tested will also engender a broadening of lines, resp. an increase in background intensity level. This, however, belongs to the range of problems associated with line profile evaluation in lattice strain analysis and therefore will be detailed in the next paragraph.

#### 2. Problems of line profile evaluation

Up-to-date evaluation of interference line distortions becomes a first-class problem especially during lattice deformation analysis or for determining crystalline grain size. According to data published in literature, accurate description of line profiles has been successfully tried by several authors, using Fourier coefficients [3, 4]. It has been proved that for observing and quantitatively representing lattice defects or density of dislocations, microstructural strain

processes, the intensity-density distribution of X-ray patterns, i. e. line profiles must be generally known to the utmost precision.

Actual distribution may be inferred to — after introducing different corrections — from diagrams directly obtained or through microdensitometric evaluation. The essence and the application of these corrections are dealt with elsewhere by the author [5]. According to literature sources a true reproduction of the actual line profile may be considered — after application of the necessary corrections — the more reliable, the more effectively the intensity level of background scattering can be reduced [6]. Especially during the estimation of background level at partially overlapping or coincident line patterns, errors may be committed resulting in over- or under-estimating the areas below the curve, and thus increasing the uncertainty of determining the integrated or Laue breadth of the line.

Thus, at first glance solution of this problem may depend upon the rate of how background effects can be eliminated in X-ray patterns.

## 3. Possibilities for reducing background scatter

a) A substantial part of extraneous scattering may be relatively easily eliminated. Insertion of a crystal monochromatizer, or of a beta=filter foil, will filter off the major part of continuous radiation. However, this procedure entails a significant decrease in overall intensity level, so that it may be only compensated at a rate of powerful increase in exposure times. This has a marked uneconomical effect.

The elimination of air scattering may be particularly essential when using soft X-ray radiations, as the scattering effect proportionally increases with the wavelength. It may seem advisable to evacuate the internal space of the recording cassette and to fill it with a gas of low scattering effect (e. g. H or He).

In a diffractometer using counter tubes, background intensity level due to continuous radiation is not critical [7], as particularly proportional counters in a special pulse-sorting circuit are usually sensibilized to a much narrower wavelength range, than film emulsions. Thus a direct counter will operate as a monochromatic receiver without any noticeable loss of intensity [8]. However, difficulties of another nature are observed in this case. Optimum choice of the time constant of a counting circuit may present a problem [9]. Increasing the time constant will result in a reduction of the random or statistical noise level, but it will also entail a shift in apparent line peak position. On the other hand, by accelerating the counting rate, lines of lower intensity will be smoothed into the background and their evaluation will become impossible.

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Atmospheric contaminations due to cosmic and other radioactive radiation sources will always be added to the background level by the counter tube. Their elimination in the form of a shielding-off of very hard rays has not yet been solved.

b) Elimination of significant scattering may also present serious difficulties. A rise in background level, due to fluorescent radiation may only be cancelled if the material for the X-ray tube target can be chosen so as to have an atomic number exceeded at least by 5, that of the constituent element prevalent in the specimen. Our most common target materials : Cr, Fe, Co or Cu are identical with, or very close to the metallic elements most frequently investigated in X-ray diffractometric tests. Thus the possibility of fluorescent scattering must always be taken into account, although its elimination can be solved in many cases [10]. Filtering effect of an aluminium foil placed between the object and the film may somewhat improve the conditions. Other kinds of significant scattering cannot be practically eliminated due to their intrinsic nature.

#### 4. Principles of experiments

The last sentence provides the key to the new procedure. Namely, if elimination of background scatter entails substantial difficulties, then it may be better to leave off from eliminating it, and to consider it as a *technical constant* characteristic of the specimen, the testing arrangement as well as recording conditions. As a working hypothesis for our experiments, we proposed to investigate whether the measured background scattering intensity can be used as a reference basis for evaluation, resp. for line profile corrections.

It is an absolute premise for the comparative evaluation of line profiles to strictly maintain constant — for the duration of a series of experiments — the setting of the testing arrangement, the recording technique and in case of films even the conditions of development. Otherwise any variation in line profiles may be a consequence, not only of a changed lattice strain rate, but also of a summarized or individual effect of several other parameters of recording technique, leading to obviously erroneous test results.

Let us see which parameters of the recording technique should be kept exactly constant.

I. The horizontal dimension a of the primary X-ray beam section, being itself a function of the following variables :

1. finite dimension of X-ray tube focus

2. size of collimating slit

3. divergence of the X-ray beam

4. distance between collimator and specimen

5. distance  $(T_a)$  between specimen surface and recording plane.

II. Angle of incidence  $\Psi_0$  of the primary X-ray beam against specimen surface.

III. Angle  $\alpha$  between the recording surface and the direction of primary radiation.

IV. Sensitivity of the film used for recording :

1. slope of the linear part of the characteristic curve

2. fog density.

V. Exposure, being a product of 1. X-ray tube current intensity and 2. exposure time.

VI. Development conditions : 1. activity and 2. temperature of the developer, 3. development time.

The first three parameters having geometrical character may be kept practically constant with relative ease, at least so far as their influence on line profile shape is concerned, because a precision in distances not higher than 0,1 mm and in angles not exceeding 1 degree is needed to achieve this. It may be solved by using templates, gauges and direct contact. Requirements concerning parameters IV. may not always be satisfied, as no assurance can be given that films used for recording belong to the same production batch and period. Criterion V. may be regarded even less as a constant, because voltage fluctuations could occur in an overloaded main network ; variations in intensity have an integrated effect during identical exposure times. Finally, the constancy of development conditions VI. is a question of laboratorium reliability.

Obviously parameters IV.---VI. are very difficult to control in a reliable manner. It appears to be much more favourable to work under less restrained recording conditions and reduce the recorded patterns to a common base using an appropriate method. This principle has been realized by our investigation.

#### 5. A mathematical approximation of the background scattering function

The influence of variables on the distribution of scattering level as a function of the running coordinate  $\varphi$  may be approximately described in a mathematical way by taking geometrical conditions into account, and also certain physical (X-ray optical) laws.

Relative intensity level of background scatter for a given angle  $\varphi$  will be directly proportional to F' (i. e. to the "working" projection of the reflecting surface layer F), to the cosine of the incidence angle  $\beta$  included by the reflected X-ray beam and the recording plane, as well as inversely proportional to the square of the path length h of the secondary beam.

This may be expressed by the formula

$$(I_s)_{\rm rel} = C \cdot \frac{F' \cdot \cos \beta}{h^2} \tag{1}$$

where the coefficient C also accounts for dependence on film sensitivity, exposure and development conditions.

It can be seen from fig. 1 that if the horizontal dimension of the primary X-ray beam is a, then the dimension of specimen surface exposed to X-ray radiation will be proportional to

$$F = \frac{a}{\sin \Psi_0} \,. \tag{2}$$



The useful projection of this surface in direction of the angle  $\varphi$ , i. e. the area influencing the recording plane is given by

$$F' = F \cdot \sin\left(\Psi_0 \pm \varphi\right). \tag{3}$$

Arrangements to the left, resp. to the right from the centre position can be accounted for by using fig. 2/a. and 2/b, resp. The angle of incidence  $\beta$  of the reflected beam is defined by fig. 1.

In case of a cylindrical camera arrangement according to Debye-Scherrer the path length h will be constant for any angle  $\varphi$  and equal to the camera radius simultaneously defining the distance  $T_a$ . In case of back-reflection patterns the path length h is continuously variable. To derive its expression for a general case the sine theorem may be used according to fig. 3:

$$T_a: h = \sin\left[180^\circ - (a + \varphi)\right]: \sin a$$

from where we have

$$h = \frac{T_a \cdot \sin a}{\sin (a + \varphi)} \,. \tag{4}$$

Here  $\alpha$  denotes — as given by the figure — the angle included by the primary beam and the recording plane. For a common back-reflection cassette we have  $\alpha = 90^{\circ}$  and the formula assumes the following simple form :

$$h = \frac{T_a}{\cos\varphi}.$$
 (4a)









For a combined conical camera [11] one has to substitute  $a = 45^{\circ}$ . For the latter case we have the following expression:

$$h = \frac{T_a \cdot \sin a}{\sin a \cdot \cos \varphi + \cos a \cdot \sin \varphi} = \frac{T_a}{\cos \varphi + \sin \varphi} .$$
 (4b)

Substituting all derived quantities into equation (1), the following general relation is obtained:

$$I_{s}(\varphi) = C \cdot \frac{a}{T_{a}^{2}} \cdot \frac{(\sin a \cdot \cos \varphi + \cos a \cdot \sin \varphi)^{2}}{\sin^{2} a} \cdot \frac{\sin \left(\Psi_{0} \pm \varphi\right)}{\sin \Psi_{0}} \cdot \cos \beta \quad (5)$$

For different types of recording cameras table I summarizes various expressions of the scattering function. Graphic interpretation of the calculated and measured level distribution  $(I_s)_{rel}$  can be seen from the sets of curves in fig. 4., 5. and 6.



Note. For a given test setting the quantities a and  $T_a$  may be regarded as constant. Thus the formulae for  $I_s$  do not contain them in explicit form, but they are included in the factor C.

#### 6. Experiments

Our views concerning the variation of background scatter level are supported by numerous experiments, carried out in recent times at the laboratory of the Institute for Mechanical Technology of Polytechnical University, Budapest. In order to clear the main problems, experiments have been grouped according to the following aims.

I. To measure the individual effect of the enumerated variables in recording technique :

1. on the slope of background intensity level,

2. on the shift of ordinates of line profiles.

II. To determine the eventual discrepancies between empirical and calculated background level distribution curves.

III. To investigate the relation between variations I/1. and I/2. due to the same cause; try to find an unequivocal relation between them.

Table I



Fig. 4



Fig. 5

IV. To check for a known loading rate i. e. for a known lattice deformation, whether X-ray patterns recorded under different conditions

1. actually show a technically constant slope in background intensity level, thus the summarized effect being of the variables investigated,

2. show no variables of a character excluding the possibility of simply superimposing scatter components,

3. do not indicate a disturbing influence by lattice deformation on background level variations and

4. yield full identity of interference lines by vertical correction — as based on characteristic technical constants — of the measured line profiles.



Fig. 6

Measurement's have been performed on X-ray diffraction units of the Seifert and VEM works. X-ray tubes with Co, Cr or Fe targets have been used, applying tube voltages of 30 to 40 kv and tube currents of 10 milliamps. Records have been made on Agfa-Laue X-ray no-screen films with emulsion layers on both sides. As recording equipment a cylindrical camera of 9 cm diameter (Unicam), a plane camera (Seifert) and a combined conical camera (own design) has been used. Evaluation has been performed on an MF—2 type non-recording microdensitometer, using a slit aperture of  $1 \times 18$  mm. The grey wedge as a rule has been adjusted so as to produce half density. The enlargement ratio between diagram and original record was 5 to 1.

The major part of the experiments was carried out on rolled plain carbon steel of 0,7 per cent C content, while the minor part which was concerned of extruded aluminium alloys of 0,58 per cent titanium content.

Results of several characteristic series of experiments are summarized in the diagrams shown on figs. 4 to 10.

#### 7. Discussion and conclusions

Comparison of the calculated curves of scatter with  $(I_s)_{r\in I}$  curves obtained from experimental measurements (cp. the diagrams plotted in *figs. 4, 5 and 6* with continuous, resp. dotted lines) permits to conclude an identical character of variation for both theoretical and empirical curves; the relatively satisfactory coincidence of these curves appears to prove, that description of phenomena along the theoretical considerations, already mentioned, yields a true first approximation.

Minor discrepancies between calculated and measured values are partly due to uncertainties in measurements (background level measurements through





film recording and densitometric analysis are less reliable, than those performed through direct counting of discharge pulses amplified electronically); on the other hand exposition and development could not be constantly maintained for the whole series of recordings, thus the factor C itself cannot be regarded as being of constant value.

Comparison of the curve sets shown also permits the conclusion that intensity level of background scatter is above all a function of variations in the path length h. This may be clearly established by the fact that in case of the Debye-Scherrer arrangement (fig. 4) no effect of h can be observed, while curve shapes are obviously modified in figs. 5 and 6 because of the latter effect (and also because of air scattering). Variations in the angles a,  $\Psi_0$  and  $\beta$  yield a qualitative influence on the curves which are close to the theoretically expected ones.

Using the diagrams of  $I_{rel}$  versus angle  $\varphi$  (vs.  $T_a$  tan  $\varphi$ ) as shown in *fig.* 7 it can be clearly established, that in case of identical geometrical arrangement and constant exposure the slope of increase in background scatter intensity

level and simultaneously the vertical dimensions of line profiles are proportionally varying to development time (but also similarly to developer temperature and activity). Influence of these three factors on the characteristic curve of the film (as seen in the scheme in the upper right corner of fig. 7) remains essentially identical. Any increase in the slope of the working section, characterized by  $\gamma$ , entails a proportional increase in background level and line profile.



Fig. 8

It may be seen from fig. 8 that slope angle of background scatter level  $(\omega)$  and line profile will also vary as a function of exposure under identical development conditions. Experience has shown that the relation is linear, insofar that the variation concerned falls outo the straight section of the density-exposure curve. Line profiles may be replotted proportionally between them to the angles  $\omega$ . This will be more detailedly referred to in the next paragraph. As a result of replotting, profile identities may be obtained within the tolerance limits of measurement uncertainty.

Curves shown in figs. 9/a and 9/b representing specimens under load, and after unloading, prove altogether that background intensity level remains practically constant, so that it may be regarded as a technical constant, sup-

posing that under unchanged circumstances exposure and development were conducted, and if a significant specimen load has not caused plastic deformation in the major part of crystallites. Microdensitograms taken from exposures



Fig. 9/a

under an angle of incidence  $\Psi_0 = 45^\circ$  show that curves on the " $\cdot$ /" side (fig. 9/a) are much more sensitive to line profile changes due to lattice strain, than curves



Fig. 9/b

on the "--/" side (fig. 9/b). This observation may be interpreted on the base of X-ray physics and is dealt with in detail elsewhere [5].

Experiments conducted with different kinds of radiation (fig. 10) resulted in establishing the fact that for corrective measures based on variations in scatter level the Co—K radiation may prove to be the most suitable in case of ferrous and aluminium alloys. Although the slope angle of the background level will be somewhat higher for Cr—K<sub>a</sub>, resp. Fe—K<sub>b</sub>, when taking the effect of the same recording parameter, this is largely counterbalanced by the non-negligible fact that any specific change in line profile due to a given lattice strain will be less, than for Co—K<sub>a</sub>. A loss in recording sensitivity may be accounted for by the fact that for the Cr-radiation the indicating array of atomic planes will give evaluable reflections, only at an angle value of  $\eta = 11^{\circ} 57'$  ( $\varphi \simeq 24^{\circ}$ ), while for the Fe—K<sub> $\beta$ </sub> radiation in the neighbourhood of  $\eta = 14^{\circ} 19'$  (i.e.  $\varphi \simeq 29^{\circ}$ ).



Using the above enumerated conclusions let us now see how line profiles may be corrected according to variations in background scattering.

#### 8. The zeta-correction procedure

The slope of a background scatter level belonging to a line profile to be corrected, can be computed by substituting the value of reflection angle  $2\eta$ as measured at the line peak in place of the angle  $\varphi$  in the expression for the first derivative of the corresponding  $I_s$ -function with respect to  $\varphi$ .

E. g. for a back-reflection camera adjusted to the perpendicular position, the derivative of the formula as taken from *table I* is given by :

$$\frac{d(I_s)_{\rm rel}}{d\varphi} = \tan \omega_1 = -C_1 \cdot \cos^3 \varphi \cdot \sin \varphi \tag{6}$$

For a Co—K<sub>a1</sub> radiation and steel testing the reflection angle corresponding to the indicating array of atomic planes (310) will be  $\varphi = 9^{\circ} 19' 30''$ . Thus the value to be substituted :  $\varphi = 2\eta = 18^{\circ} 39'$ .

Substituting into expression (6) we obtain tan  $\omega_1 = -C_1 \cdot 0.272$ 

The slope of curve b in fig. 8 most closely approximates this theoretical figure. As tan  $\omega_1$  equals — 0,23, we have for  $C_b$  a value of 0,846, the measured value of  $\omega_b$  being 13°.

This value is obviously negative, because decreasing scattering intensity  $I_s$  is obtained for increasing angles  $\varphi$  within the recorded range.

X-ray patterns taken under changed conditions (e. g. with a longer exposure) are referred to in this basic reference value. The slope as taken from the microdensitometric curve at the same angle  $\varphi$  (see curve *a*, fig. 8) equals  $\omega_a = 18^{\circ} 16'$ . The scales of both microdensitograms being identical, any variation in the slope angles may be only the result of a change in the value of *C* due to the different exposures.

Thus we have

- tan 
$$13^{\circ}$$
 = -  $C_b \cdot 0,272 = -0,846 \cdot 0,272 = -0,23$   
- tan  $18^{\circ} 16' = -C_a \cdot 0,272 = -1,210 \cdot 0,272 = -0,33$ 

The ratio of the C factors yields the correction factor  $\zeta$ , i. e.

$$\zeta = \frac{C_b}{C_a} \tag{7}$$

For our example  $\zeta = \frac{0.846}{1,210} = 0.7$ . Consequently all ordinates of the curve a must be multiplied by this factor to achieve full agreement between profiles b and a.

As the reference value  $\tan \omega_1 = \frac{d I_s}{d \varphi}$  may be considered as a technical constant by force of our experiments, any difference between actual  $\omega_i$  values of the individual X-ray patterns will only entail that setting, adjustment and development conditions have been unequal.

In order to achieve commensurability of line profiles all ordinate segments must be modified accordingly by the resulting correction factors  $\zeta_i$ . Thus a so-called *homologous set of curves* is obtained, where uncertainties due to random variations in recording conditions cannot disturb the evaluation of lattice strain as based on relative profile variations. In our case the quantity  $\omega_{ref}$  fulfils — using an analogy with operations on fractions — the role of the common denominator.

# 9. Application: a semi-quantitative X-ray method for measuring lattice strains

On fig. 11 an experimental homologous set of curves is shown. Under relatively constant recording conditions serial measurements have been taken from a steel plate specimen clamped to a bending fixture. By bending the specimen around known radii of curvature, step-wise increasing bending loads have been obtained. Load increase occurred in steps of approximately 10 kgs per sq. mm.



Fig. 11

After densitometric evaluation X-ray patterns have been subject to a zeta-correction procedure, the individual ordinate segments being shifted, parallelly to themselves, to the basic level. Thus comparison of the line profiles between themselves and rapid approximative information became possible about the magnitude of stresses. In microdensitograms pertaining to single stress steps, relative changes in profile are very marked; the line shape may be especially well-observed on the part between the  $K_{a1}$  and  $K_{a2}$  peaks.

It is possible therefore to gain approximative information with an accuracy of  $\pm 5$  kgs per sq. mm for steel and for a setting distance of  $T_a = 80$  to 90 mm, without having to perform the tedious work of precisely determining the location of a line peak or resolving the  $K_{a1a2}$  doublet. To give up the separation may be of direct advantage. It will not only mean an economy of time, but also enables the investigator to draw more reliable conclusions from the saddle portion on the  $I_{rel}(\varphi)$  curve between the line peaks  $K_{a1}$ ; and  $K_{a2}$ , as could have been obtained by analyzing the profile of any of the component curves. Both interference lines  $K_{a1}$  and  $K_{a2}$  and the general trend of the curve will be separately affected by a lattice strain. However, adjacent parts of the two modified curves will be superimposed one on the other : line profile changes are recorded with double sensitivity on the saddle portion of the resulting curve, illustrated clearly by fig. 11.

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#### Summary

For a quantitative analysis of lattice defects, density of dislocations, generally of any strain process in the microstructure of metals, intensity-density distributions of X-ray patterns, especially interference line profiles with the maximum of accuracy should be known. According to prevailing views up to the present, evaluation of line profiles may be considered the more reliable, the more the background intensity level on the X-ray patterns can be reduced. This background scattering effect may be due to several causes.

The introduction of the paper gives a schematic summary on the different kinds of scattering, and deals with the possibilities of their elimination, resp. reduction. It is stated that in case of film recording the presence of significant scatter is almost unavoidable.

Under these circumstances the concept has been evolved to utilize background scatter for the up-to-date evaluation of line profiles, instead of eliminating it. The author has arranged multiple experiments to observe the influence of different recording parameters on the slope of background scatter level, and also in conjunction therewith, the shift in ordinates of profiles. Under certain, rather easily reproducible recording conditions unequivocal relations among the variables investigated have been found. The influence of these variables has been mathematically described, taking geometrical and partly X-ray optical conditions into account. Computed curves of background scattering function show satisfactory agreement with empirical diagrams. The first derivative of the scattering function — after substituting numerical values characteristic of a given arrangement — may be considered as a technical constant, which may be used as a reference for the so-called zeta-correction procedure used for comparative evaluation of line profiles.

The paper is concluded by a demonstration of applying the correction method. The series of diagrams as given in the paper proves that approximative information on the stress distribution in a steel part can be obtained with an accuracy of  $\pm 5$  kgs per sq. mm in a relatively rapid and simple manner, without having to perform the tedious operations of determining line peak locations or separating the  $K_{\alpha_1\alpha_2}$  doublet. The correction procedure described, provided means for evolving a semi-quantitative X-ray method for measuring lattice strain.

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