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Generalised Least-squares Determination of Triaxial Stress States by X-ray Diffraction and the Associated Errors*

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Abstract

The determination of residual stresses via X-ray diffraction is briefly reviewed, with particular emphasis on the triaxial stress state. A new method is proposed for determining the general stress tensor, which considerably reduces the variances of the stresses due to counting statistics and gradients. The procedure involves a generalised least-squares solution of strains measured at various tilts of the X-ray beam to the sample, and a new set of tilts is recommended to minimise these errors.

1. Introduction

X-ray diffraction can be used to determine stresses by measuring the changes in interplanar spacing in a crystalline material. Classical stress determination using X-ray diffraction assumes a biaxial stress state where the stresses normal to the surface are zero. This assumption leads to equations from which the stress along the measurement direction σ_ϕ may be determined from the slope of a d versus $\sin^2\psi$ plot (Noyan and Cohen 1987). The angles ϕ and ψ are defined in Fig. 1 and d is the interplanar spacing perpendicular to the L_3 axis.

Triaxial stress states cause curvature in the d versus $\sin^2\psi$ plots (Noyan and Cohen 1984). This curvature has been observed in several experimental studies (Dölle and Cohen 1980; Ho *et al.* 1983). The presence of a stress normal to the surface, σ_{33}, will cause curvature in d versus $\sin^2\psi$ plots, while the shear stresses σ_{13} and σ_{23} produce different curves for negative and positive ψ tilts (ψ splitting). Dölle and Hauk have extended the classical stress analysis theory to include the determination of triaxial stress states (Dölle and Hauk 1976; Dölle 1979). This method uses the average strain for positive and negative ψ tilts versus $\sin^2\psi$ at ϕ equal to 0, 45° and 90° to determine the stresses σ_{11}, σ_{22}, σ_{33} and σ_{12}. The stresses σ_{13} and σ_{23} are determined from plots of the difference in strain for positive and negative ψ tilts versus $\sin 2\psi$.

In this paper a generalised least-squares method of determining triaxial residual stresses from diffraction data is presented. Being able to estimate the errors associated with a measurement is also quite important. Errors due to counting statistics and stress gradients with the Dölle–Hauk method have been investigated by Rudnik and Cohen (1986), Noyan (1983) and Noyan and Cohen (1984). These errors are

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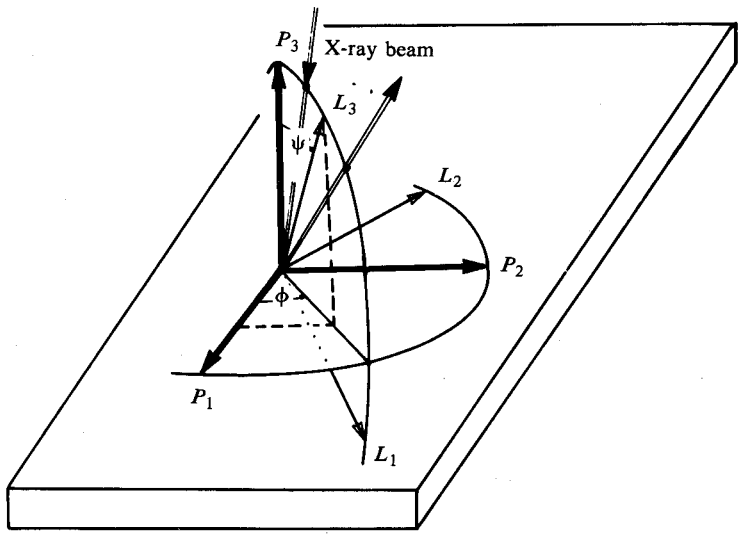

Fig. 1. The coordinate system.

investigated for the generalised least-squares method and it is shown that the method is less sensitive to errors than the Dölle–Hauk method.

2. Theory

Consider the two coordinate systems in Fig. 1. The P coordinate system is attached to the sample and is the coordinate system in which it is desired to measure the stresses. The L coordinate system is the laboratory system and is the system in which the diffraction measurements are made. The two coordinate systems are oriented with respect to each other by the angles ϕ and ψ. One may determine the interplanar spacing along planes perpendicular to the L_3 axis, $d_{\phi\psi}$, from the position of a diffraction peak. By knowing the unstressed planar spacing d_0 one may determine the strain along the L_3 axis:

$$e_{\phi\psi} = (d_{\phi\psi} - d_0)/d_0 . \tag{1}$$

Noyan (1985) discussed several methods of determining d_0. Using tensor transformations the strains in the P coordinate system may be related to the strain along the L_3 axis as

$$\begin{aligned} e_{\phi\psi} = {} & \epsilon_{11} \cos^2\phi \sin^2\psi + \epsilon_{22} \sin^2\phi \sin^2\psi + \epsilon_{33} \cos^2\psi \\ & + \epsilon_{12} \sin 2\phi \sin^2\psi + \epsilon_{13} \cos\phi \sin 2\psi + \epsilon_{23} \sin\phi \sin 2\psi . \end{aligned} \tag{2}$$

The ϵ_{ij} refer to strains in the sample coordinate system while $e_{\phi\psi}$ refer to strains measured in the laboratory coordinate system.

Equation (2) shows that the measured strains $e_{\phi\psi}$ are linear with respect to the strains in the sample coordinate system. Measuring strains in six independent directions is therefore sufficient to determine the strains ϵ_{ij} (Nye 1976). The accuracy may be improved by measuring more than six strains $e_{\phi\psi}$ and determining the strains ϵ_{ij} by a least-squares procedure (Imura *et al.* 1962).

To facilitate a matrix formulation of the least-squares procedure the following definitions are made:

$$\begin{aligned}
\epsilon_1 &= \epsilon_{11}, & f_1(\phi,\psi) &= \cos^2\phi \sin^2\psi,\\
\epsilon_2 &= \epsilon_{22}, & f_2(\phi,\psi) &= \sin^2\phi \sin^2\psi,\\
\epsilon_3 &= \epsilon_{33}, & f_3(\phi,\psi) &= \cos^2\psi,\\
\epsilon_4 &= \epsilon_{12}, & f_4(\phi,\psi) &= \sin 2\phi \sin^2\psi,\\
\epsilon_5 &= \epsilon_{13}, & f_5(\phi,\psi) &= \cos\phi \sin 2\psi,\\
\epsilon_6 &= \epsilon_{23}, & f_6(\phi,\psi) &= \sin\phi \sin 2\psi.
\end{aligned}$$

The residual between the measured and calculated strain along the L_3 axis is

$$r_i = \sum_{j=1}^{6} \epsilon_j f_j(\phi_i,\psi_i) - e_i. \tag{3}$$

The total weighted sum of the squared error R for n measurements of e is then

$$R = \sum_{i=1}^{n} \frac{1}{\mathrm{var}(e_i)} \left\{ \left(\sum_{j=1}^{6} \epsilon_j f_j(\phi_i,\psi_i) \right) - e_i \right\}^2. \tag{4}$$

Each error r_i is weighted by the inverse variance associated with the corresponding strain e_i. Equations exist for estimating these variances from the diffraction data (James and Cohen 1977), as discussed below. Thus, the most reliable measurements are weighted the most. Taking the partial derivatives with respect to each strain ϵ_j and setting them equal to zero to find the minimum results in the set of equations

$$\sum_{i=1}^{n} \left\{ \left(\sum_{k=1}^{6} \epsilon_k f_k(\phi_i,\psi_i) \right) - e_i \right\} \frac{f_j(\phi_i,\psi_i)}{\mathrm{var}(e_i)} = 0. \tag{5}$$

To formulate a matrix equation the **B** matrix and $\boldsymbol{E}$ vector are defined as

$$B_{jk} = \sum_{i=1}^{n} f_j(\phi_i,\psi_i) f_k(\phi_i,\psi_i)/\mathrm{var}(e_i), \tag{6}$$

$$E_j = \sum_{i=1}^{n} e_i f_j(\phi_i,\psi_i)/\mathrm{var}(e_i). \tag{7}$$

Provided that the strains are measured at ϕ and ψ angles that do not form a singular **B** matrix, the strains giving the least squared error are given by the solution of

$$\mathbf{B}\boldsymbol{\epsilon} = \boldsymbol{E} \quad \text{or} \quad \boldsymbol{\epsilon} = \mathbf{B}^{-1}\boldsymbol{E}. \tag{8, 9}$$

When the strains have been determined the stresses may be determined from the relations

$$\sigma_{ij} = \frac{1}{\frac{1}{2}S_2} \left(\epsilon_{ij} - \delta_{ij} \frac{S_1}{\frac{1}{2}S_2 + 3S_1} (\epsilon_{11} + \epsilon_{22} + \epsilon_{33}) \right), \tag{10}$$

where S_1 and $\frac{1}{2}S_2$ are the appropriate X-ray elastic constants (Marion and Cohen 1977; Perry *et al.* 1984) and δ_{ij} is the Kronecker delta function. The stresses may also be obtained directly by substituting σ for ϵ and the functions g_j for the functions

f_j in equations (6), (7) and (8), where

$$g_1(\phi,\psi) = \tfrac{1}{2}(\cos^2\phi\,\sin^2\psi)S_2 - S_1\,, \quad g_2(\phi,\psi) = \tfrac{1}{2}(\sin^2\phi\,\sin^2\psi)S_2 - S_1\,,$$
$$g_3(\phi,\psi) = \tfrac{1}{2}(\cos^2\psi)S_2 - S_1\,, \quad g_4(\phi,\psi) = \tfrac{1}{2}(\sin 2\phi\,\sin^2\psi)S_2\,,$$
$$g_5(\phi,\psi) = \tfrac{1}{2}(\cos\phi\,\sin 2\psi)S_2\,, \quad g_6(\phi,\psi) = \tfrac{1}{2}(\sin\phi\,\sin 2\psi)S_2\,. \tag{11}$$

3. Counting Statistical Errors

An estimate of the errors associated with a measurement is nearly as important as the measurement itself. In determining interplanar spacings by X-ray diffraction, intensities at different points along a diffraction peak are measured to determine the peak position 2θ. The interplanar spacing may then be determined from Bragg's law. The intensity measurements are subject to statistical counting error. James and Cohen (1977) gave formulae for determining the error in 2θ from the intensity measurements. Estimates of the errors in 2θ can also be determined from nonlinear least-squares fits of peaks to analytical functions.

The variance in e is computed from the variance in 2θ by (Rudnik and Cohen 1986)

$$\operatorname{var}(e) = \left(\frac{1}{d_0}\right)^2\left(\frac{\pi}{180}\right)^2\left(\frac{\lambda\cos\theta}{2\sin^2\theta}\right)^2\frac{\operatorname{var}(2\theta)}{2}\,, \tag{12}$$

where $\operatorname{var}(2\theta)$ is given by the peak location method. The variance in the strains ϵ_j may be calculated from the variance in each of the measured strains

$$\operatorname{var}(\epsilon_j) = \sum_{i=1}^{n}\left(\frac{\partial\epsilon_i}{\partial e_i}\right)^2\operatorname{var}(e_i)\,. \tag{13}$$

The variance in d_0 is not considered here but may also be included (Rudnik and Cohen 1986). The errors in the measured strains can be propagated through equation (9) by using (12) to determine the variance in each of the strains ϵ_j:

$$\operatorname{var}(\epsilon_j) = \sum_{i=1}^{n}\left(\sum_{j=1}^{6} B_{kj}^{-1}\frac{f_j(\phi_i,\psi_i)}{\operatorname{var}(e_i)}\right)^2\operatorname{var}(e_i)\,. \tag{14}$$

The variance in each strain value may be evaluated from equation (13) and used to propagate the error to the stresses using (10) and (12). The errors in the measured stresses may then be estimated from the standard deviations given by the square roots of the variances

$$\operatorname{var}(\sigma_i) = \left(\frac{1}{\frac{1}{2}S_2} - \frac{S_1}{\frac{1}{2}S_2(\frac{1}{2}S_2+3S_1)}\right)^2\operatorname{var}(\epsilon_i)$$
$$+\left(\frac{S_1}{\frac{1}{2}S_2(\frac{1}{2}S_2+3S_1)}\right)^2\{\operatorname{var}(\epsilon_j)+\operatorname{var}(\epsilon_k)\}\,; \quad i,j,k = 1,2,3\,, \tag{15a}$$

$$\operatorname{var}(\sigma_m) = \left(\frac{1}{\frac{1}{2}S_2}\right)^2\operatorname{var}(\epsilon_m)\,; \quad m = 4,5,6\,. \tag{15b}$$

Table 1. Comparison of the counting statistical errors in the strain and stress (MPa) tensors for (*a*) the Dölle–Hauk method and (*b*) a generalised least-squares method

The data are from a ground steel sample

(a) Dölle–Hauk

$$\epsilon = \begin{bmatrix} 1{\cdot}649 & -0{\cdot}139 & -0{\cdot}226 \\ -0{\cdot}139 & 1{\cdot}721 & 0{\cdot}013 \\ -0{\cdot}226 & 0{\cdot}013 & -1{\cdot}001 \end{bmatrix} \times 10^{-3} \pm \begin{bmatrix} 0{\cdot}088 & 0{\cdot}087 & 0{\cdot}026 \\ 0{\cdot}087 & 0{\cdot}080 & 0{\cdot}021 \\ 0{\cdot}026 & 0{\cdot}021 & 0{\cdot}064 \end{bmatrix} \times 10^{-3}$$

$$\sigma = \begin{bmatrix} 539{\cdot}74 & -24{\cdot}03 & -39{\cdot}15 \\ -24{\cdot}03 & 552{\cdot}16 & 2{\cdot}30 \\ -39{\cdot}15 & 2{\cdot}30 & 80{\cdot}41 \end{bmatrix} \pm \begin{bmatrix} 27{\cdot}08 & 15{\cdot}05 & 4{\cdot}58 \\ 15{\cdot}05 & 25{\cdot}30 & 3{\cdot}56 \\ 4{\cdot}58 & 3{\cdot}56 & 21{\cdot}97 \end{bmatrix}$$

(b) Generalised least-squares

$$\epsilon = \begin{bmatrix} 1{\cdot}515 & -0{\cdot}045 & -0{\cdot}234 \\ -0{\cdot}045 & 1{\cdot}888 & 0{\cdot}029 \\ -0{\cdot}234 & 0{\cdot}029 & -0{\cdot}936 \end{bmatrix} \times 10^{-3} \pm \begin{bmatrix} 0{\cdot}036 & 0{\cdot}043 & 0{\cdot}010 \\ 0{\cdot}043 & 0{\cdot}031 & 0{\cdot}009 \\ 0{\cdot}010 & 0{\cdot}009 & 0{\cdot}010 \end{bmatrix} \times 10^{-3}$$

$$\sigma = \begin{bmatrix} 527{\cdot}04 & -7{\cdot}90 & -40{\cdot}48 \\ -7{\cdot}90 & 591{\cdot}73 & 4{\cdot}99 \\ -40{\cdot}48 & 4{\cdot}99 & 49{\cdot}87 \end{bmatrix} \pm \begin{bmatrix} 10{\cdot}73 & 7{\cdot}39 & 1{\cdot}72 \\ 7{\cdot}39 & 9{\cdot}59 & 1{\cdot}53 \\ 1{\cdot}72 & 1{\cdot}53 & 5{\cdot}82 \end{bmatrix}$$

The counting statistical errors for the Dölle–Hauk method and the generalised least-squares method are compared in Table 1. The diffraction data are taken from sample C3 in Dölle and Cohen (1980). The sample is a normalised plain carbon steel ground along the P_1 direction. The results for the Dölle–Hauk method were calculated using the same program used in Rudnik and Cohen (1986). The value of d_0 was assumed to be known exactly. The standard deviations of the peak position 2θ were all of the order of 0·012°. These results show that the propagation of error through a generalised least-squares method results in improved counting statistical errors over the Dölle–Hauk method. In the generalised least-squares method each strain measurement e_i contributes to the determination of each strain ϵ_j to which it is not orthogonal in equation (2). This is a more efficient use of the available data than the Dölle–Hauk method and results in the improved counting statistical errors.

The counting statistical error in equation (15) depends only on the errors in e_i and not on the actual values. The tensor of counting statistical errors will thus be independent of the stress tensor and depend only upon the accuracies to which the interplanar spacings are measured.

It is possible to optimise ϕ_i and ψ_i to minimise the counting statistical errors so that the measurement time to achieve a given error may be minimised. Another consideration, however, is that it is still highly desirable to have a number of ψ tilts along a constant value of ϕ so that $d_{\phi\psi}$ versus $\sin^2\psi$ plots may be made. These plots provide a valuable visual check that the strains in the sample follow the theory and that the sample was properly aligned during the diffraction measurements (Noyan and Cohen 1987).

Table 2. Comparison of the counting statistical errors in the strain and stress (MPa) tensors for the use of (*a*) $\phi = 0$, 45° and 90° and (*b*) $\phi = 0$, 60° and 120°

The results are a computer simulation for a steel sample measured with Cr Kα radiation at $2\theta = 156°$. Each strain was assumed to have an error of 0·0001

(a) $\phi = 0$, *45° and 90°*

$$\epsilon = \begin{bmatrix} 1{\cdot}308 & 0{\cdot}000 & 0{\cdot}000 \\ 0{\cdot}000 & 1{\cdot}308 & 0{\cdot}000 \\ 0{\cdot}000 & 0{\cdot}000 & 1{\cdot}000 \end{bmatrix} \times 10^{-3} \pm \begin{bmatrix} 0{\cdot}117 & 0{\cdot}117 & 0{\cdot}031 \\ 0{\cdot}117 & 0{\cdot}117 & 0{\cdot}031 \\ 0{\cdot}031 & 0{\cdot}031 & 0{\cdot}039 \end{bmatrix} \times 10^{-3}$$

$$\sigma = \begin{bmatrix} -400{\cdot}00 & 0{\cdot}00 & 0{\cdot}00 \\ 0{\cdot}00 & -400{\cdot}00 & 0{\cdot}00 \\ 0{\cdot}00 & 0{\cdot}00 & 0{\cdot}00 \end{bmatrix} \pm \begin{bmatrix} 35{\cdot}43 & 20{\cdot}24 & 5{\cdot}44 \\ 20{\cdot}24 & 35{\cdot}43 & 5{\cdot}44 \\ 5{\cdot}44 & 5{\cdot}44 & 20{\cdot}91 \end{bmatrix}$$

(b) $\phi = 0$, *60° and 120°*

$$\epsilon = \begin{bmatrix} 1{\cdot}308 & 0{\cdot}000 & 0{\cdot}000 \\ 0{\cdot}000 & 1{\cdot}308 & 0{\cdot}000 \\ 0{\cdot}000 & 0{\cdot}000 & 1{\cdot}000 \end{bmatrix} \times 10^{-3} \pm \begin{bmatrix} 0{\cdot}117 & 0{\cdot}078 & 0{\cdot}030 \\ 0{\cdot}078 & 0{\cdot}117 & 0{\cdot}030 \\ 0{\cdot}030 & 0{\cdot}030 & 0{\cdot}039 \end{bmatrix} \times 10^{-3}$$

$$\sigma = \begin{bmatrix} -400{\cdot}00 & 0{\cdot}00 & 0{\cdot}00 \\ 0{\cdot}00 & -400{\cdot}00 & 0{\cdot}00 \\ 0{\cdot}00 & 0{\cdot}00 & 0{\cdot}00 \end{bmatrix} \pm \begin{bmatrix} 35{\cdot}43 & 13{\cdot}49 & 5{\cdot}13 \\ 13{\cdot}49 & 35{\cdot}43 & 5{\cdot}13 \\ 5{\cdot}13 & 5{\cdot}13 & 20{\cdot}91 \end{bmatrix}$$

Table 2 illustrates a comparison between use of the 'traditional' set of ϕ angles 0, 45° and 90° and the set 0, 60° and 120°. The data are a computer simulation for a steel sample with Cr Kα radiation. Each strain was assumed to have an error of 0·0001. While the normal stresses have the same errors, the errors in the shear stresses are reduced by using ϕ equal to 0, 60° and 120°. Thus greater precision may be obtained in the shear stresses by simply using 0, 60° and 120° for ϕ instead of 0, 45° and 90°.

4. Errors due to Gradients

Noyan (1983) and Noyan and Cohen (1984) have examined the effect of gradients in the stresses on the measurement of stresses by X-ray diffraction. Since the stresses normal to the surface must be zero at the surface, these stresses must exist as gradients in the sample. The stresses measured by X-ray diffraction are averages measured over the penetration depth of the X-ray beam. In making the measurements, however, the sample must be tilted at different angles to the X-ray beam which gives a different sampling depth for each tilt. Thus, the stresses form a different average for each tilt of the sample. Stress gradients, therefore, lead to curvature (but not ψ splitting) in d versus $\sin^2\psi$ plots and errors in the measured values of the stresses.

The average stress sampled by an X-ray beam is given by

$$\langle \sigma_{ij} \rangle = \int_0^\infty \sigma_{ij}(z) \exp(-z/\tau)\, dz \Big/ \int_0^\infty \sigma_{ij}(z)\, dz, \tag{16}$$

where z is the depth into the sample and τ is given by

$$\tau = (\sin^2\theta - \sin^2\psi)/2\mu \sin\theta \cos\psi \tag{17}$$

for ψ tilts around the θ-axis (ψ goniometry), and by

$$\tau = (\sin\theta \cos\psi)/2\mu \tag{18}$$

for ψ tilts around an axis parallel to the plane of the diffractometer (Ω goniometry).

If we assume, for instance, stress gradients of the form

$$\sigma_{ij}(z) = \sigma_{ij}(0) + a_{ij} z^{n_{ij}}, \tag{19}$$

the average stress becomes

$$\langle\sigma_{ij}\rangle = \sigma_{ij}(0) + K_{ij} \tau^{n_{ij}}, \tag{20}$$

where K_{ij} is a constant. This equation shows that the average stress sampled by the X-ray beam is a function of τ which is a function of the angle ψ.

When stress gradients are present, as given for example by equation (19), instead of constant stresses as assumed by the theory, the trigonometric dependence will not be as simple as given in the above equations for $f_j(\phi, \psi)$ or $g_j(\phi, \psi)$. There will be a systematic deviation from the theoretical dependence on ϕ and ψ due to τ, which is a function of ψ. The averaged stress is dependent on the unknown gradient. This systematic deviation will cause some error in the measured stress values when equation (2) is forced to fit the data by any procedure.

To determine the effect and magnitude of these gradient errors and to determine methods to minimise them, computer simulations of stress measurements on samples containing stress gradients were performed similar to those in Noyan (1983) and Noyan and Cohen (1984).

In the computer simulations the sample was assumed to be steel and the measurements done with Cr Kα radiation for the 211 diffraction peak at 156°2θ for several different stress gradients. The stresses in the plane of the sample surface σ_{11} and σ_{22} were assumed to be uniform. Gradients of the form (19) were used to calculate an average stress sampled by the X-rays for each ψ tilt. Equation (18) for Ω goniometry was used to determine the value of τ at each ψ tilt. A linear absorbtion coefficient of $\mu = 0{\cdot}09\ \mu\text{m}^{-1}$ was used. The measured strain values e_i were calculated and these were used to calculate the measured stresses by the generalised least-squares procedure. Different ψ ranges were tested with ϕ equal to 0, 60° and 120° in all cases.

Four groups of stress tensors with different gradients were examined to determine the effects of gradients in the different components of the stress tensors. The constants K_{ij} were selected to give a value of stress σ_{33} of about 100 MPa for a ψ range 0–60° except for the second group of stress tensors for which σ_{33} was zero.

(a) Group I

In group I tensors of the form

$$\sigma = \begin{bmatrix} -400 & 0 & 0 \\ 0 & -400 & 0 \\ 0 & 0 & K\tau^{n_{33}} \end{bmatrix}$$

Table 3. Computer simulation results for group I stress tensors

n_{33}	K_{33}	ψ range (degrees)	σ_{11} (MPa)	σ_{33} (MPa)	Error in σ_{11} (MPa)
1	22·6764	0–60·00	−422·6	100·0	22·6
1	22·6764	0–33·21	−476·0	79·4	76·0
1	22·6764	39·23–60·00	−407·5	88·3	7·5
2	5·0243	0–60·00	−442·8	100·0	42·8
2	5·0243	0–33·21	−564·2	52·6	164·2
2	5·0243	39·23–60·00	−411·5	76·5	11·5
3	1·0920	0–60·00	−460·7	100·0	60·7
3	1·0920	0–33·21	−661·6	20·8	261·6
3	1·0920	39·23–60·00	−413·2	65·3	13·2

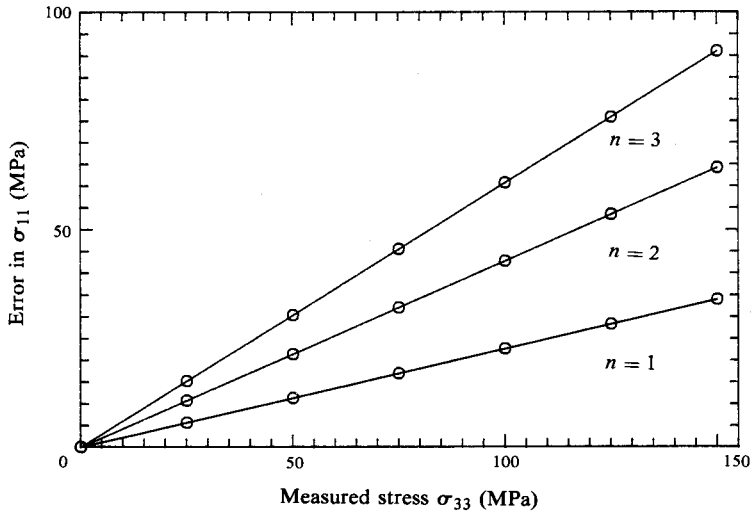

Fig. 2. Errors due to a gradient in σ_{33}.

were examined for n equal to 1, 2 and 3. Table 3 shows the results of the computer simulation with different ψ ranges for the measurement, while Fig. 2 shows the error in the stress σ_{11} as a function of the measured stress σ_{33} for different values of n for the ψ range 0–60°. These data show that the measured value of stress σ_{11} can be greatly affected by a gradient in σ_{33}, especially for low ψ ranges. This table also shows that this error can be minimised by using high ψ ranges.

(b) Group II

Group II tensors were of the form

$$\sigma = \begin{bmatrix} -400 + K\tau^{n_{11}} & 0 & 0 \\ 0 & -400 + K\tau^{n_{11}} & 0 \\ 0 & 0 & 0 \end{bmatrix}.$$

Table 4. Computer simulation results for group II tensors

n_{11}	K_{11}	ψ range (degrees)	σ_{11} (MPa)	σ_{33} (MPa)	Error in σ_{11} (MPa)
1	10·00	0–60·00	−366·2	−5·7	33·8
1	10·00	0–33·21	−337·8	5·2	62·2
1	10·00	39·23–60·00	−374·4	0·7	25·6
2	2·50	0–60·00	−367·7	−10·7	32·3
2	2·50	0–33·21	−307·5	12·8	92·5
2	2·50	39·23–60·00	−383·2	0·9	16·8
3	0·55	0–60·00	−369·8	−13·9	30·2
3	0·55	0–33·21	−282·2	20·8	127·8
3	0·55	39·23–60·00	−389·9	0·7	10·1

Table 5. Computer simulation results for group III tensors

n_{11}	K_{11}	n_{33}	K_{33}	ψ range (degrees)	σ_{11} (MPa)	σ_{33} (MPa)	Error in σ_{11} (MPa)
1	10·0	2	5·0	0–60·00	−408·7	93·8	8·7
1	10·0	2	5·0	0–33·21	−501·3	57·6	101·3
1	10·0	2	5·0	39·23–60·00	−385·9	76·9	14·1
2	2·0	2	5·0	0–60·00	−416·7	91·0	16·7
2	2·0	2	5·0	0–33·21	−489·4	62·6	89·4
2	2·0	2	5·0	39·23–60·00	−398·0	76·9	2·0
1	−10·0	2	5·0	0–60·00	−476·4	105·2	76·4
1	−10·0	2	5·0	0–33·21	−625·6	47·1	225·6
1	−10·0	2	5·0	39·23–60·00	−437·0	75·4	37·0
2	−2·0	2	5·0	0–60·00	−468·4	105·1	68·4
2	−2·0	2	5·0	0–33·21	−637·4	42·1	237·4
2	−2·0	2	5·0	39·23–60·00	−424·9	75·4	24·9

Here the gradient was in the stresses σ_{11} and σ_{22} and the stress σ_{33} was absent. Again (see Table 4) the high ψ range gives the least error in the normal stresses, while the low ψ range gives the greatest error. The nonzero values of σ_{33} are due to the forced fitting of equation (2) to the data.

(c) Group III

Combined gradients with tensors of the form

$$\sigma = \begin{bmatrix} -400 + K_{11}\tau^{n_{11}} & 0 & 0 \\ 0 & -400 + K_{11}\tau^{n_{11}} & 0 \\ 0 & 0 & K_{33}\tau^{n_{33}} \end{bmatrix}$$

were examined in group III. Both positive and negative K_{11} were used since values of K_{11} and K_{33} with the same sign have the opposite effect on the curvature in a d

versus $\sin^2\psi$ plot (Cohen *et al.* 1980). Table 5 shows the results from the group III tensors. When K_{11} and K_{33} have the same sign the error in σ_{11} nearly cancels out depending on the ψ range. The low ψ range again gives large errors. When K_{11} and K_{33} are of opposite sign and produce the same curvature in d versus $\sin^2\psi$, the high ψ range again gives the smallest error in σ_{11}.

Table 6. Computer simulation results for group IV tensors

n_{33}	K_{33}	ψ range (degrees)	σ_{11} (MPa)	σ_{33} (MPa)	σ_{13} (MPa)	Error in σ_{11} (MPa)
2	5·0	0–60·00	−442·6	99·5	76·6	42·6
2	5·0	0–33·21	−563·4	52·3	111·2	163·4
2	5·0	39·23–60·00	−411·5	76·1	59·2	11·5
2	5·0	0–26·57	—	—	121·3	—
2	5·0	33·21–45·00	—	—	80·6	—
2	5·0	50·77–60·00	—	—	44·2	—

(d) Group IV

To test the effect of combined gradients in σ_{33} and σ_{13}, group IV tensors were of the form

$$\sigma = \begin{bmatrix} -400 & 0 & K_{33}\tau^2 \\ 0 & -400 & 0 \\ K_{33}\tau^2 & 0 & K_{33}\tau^2 \end{bmatrix}.$$

The results of these tests are shown in Table 6. Once again, the smallest error in the normal stresses is with the high ψ range. The magnitude of σ_{13} decreases with increasing ψ range. This is to be expected as the penetration depth of the X-rays decreases with ψ and the high ψ ranges sample less of the gradient in σ_{13}. Noyan and Cohen (1984) found that the Dölle–Hauk method gave very sporadic results for σ_{13} depending on the ψ range, sometimes giving the wrong sign. The generalised least-squares method is far more consistent with respect to these shear stresses, and will give far more reasonable values for them independent of the ψ range.

5. Conclusions

(1) A generalised least-squares method of analysing diffraction data to determine triaxial stress states was presented along with equations to estimate the statistical counting error associated with the measurements. The calculations for a typical triaxial stress measurement such as the one in Table 1 take only a few seconds on a personal computer. Computation time is therefore insignificant in using this analysis method and it is well suited for use in an automated stress measurement system.

(2) The d versus $\sin^2\psi$ plots are a valuable visual check on the data and should be used and checked against the results for reasonableness.

(3) The use of ϕ equal to 0, 60° and 120° will give lower counting statistical error than the traditional values of 0, 45° and 90° for the shear stresses.

(4) High ψ ranges minimise the errors in the normal stresses due to stress gradients in the sample. The shear stresses σ_{13} and σ_{23} are adequately measured with any ψ range, lower ψ ranges sampling more of their gradient. The method will not give the wrong sign for these stresses as can happen with the Dölle–Hauk method.

(5) The generalised least-squares method gives lower statistical counting errors than the Dölle–Hauk method, and smaller errors due to the presence of stress gradients in the sample.

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