PERGAMON

Journal of Structural Geology 22 (2000) 1601–1607

JOURNAL OF STRUCTURAL GEOLOGY

www.elsevier.nl/locate/jstrugeo

On the strain-free lattice constants in residual stress evaluation by diffraction

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Received 17 November 1999; accepted 14 July 2000

Abstract

Any discussion of methodical problems in residual stress evaluation by diffraction (X-rays or neutrons) is based on the so-called 'general transformation equation'. The problem of the unknown strain-free lattice constants has to be treated by an appropriate use of the transformation equation. We outline several solutions known from the literature for this problem. In the most general case, we include the lattice constants and the strain/stress tensor components as unkowns into the evaluation the general transformation equation. This results in a non-linear equation system for the unknowns which can be solved numerically. © 2000 Elsevier Science Ltd. All rights reserved.

1. Introduction

The theory of residual stress evaluation (RSE) has been described in several review papers, for example by Dölle (1979); Noyan and Cohen (1987); Scholtes (1991); Hauk (1995); and Eigenmann and Macherauch (1995/1996). The core of the theory is the 'general transformation equation' (GTE) which relates the measured strain $\varepsilon_{33}\,(hkl,\Psi)'$ to the six independent components of either the strain tensor $\overset{2\leftrightarrow}{\varepsilon}$

$$\varepsilon_{33}(hkl,\ \Psi)' = (u_{32})^2\varepsilon_{11} + 2u_{31}u_{32}\varepsilon_{12} + 2u_{31}u_{33}\varepsilon_{13} + (u_{32})^2\varepsilon_{22} + 2u_{32}u_{33}\varepsilon_{23} + (u_{33})^2\varepsilon_{33} \quad (1)$$

or of the stress tensor $\overset{2\leftrightarrow}{\sigma}$

$$\begin{aligned}\varepsilon_{33}(hkl,\Psi)' &= \left(\tfrac{1}{2}S_2(hkl)(u_{31})^2 + S_1(hkl)((u_{11})^2 + (u_{21})^2 + (u_{31})^2)\right)\sigma_{11}\\ &+ \left(\tfrac{1}{2}S_2(hkl)(u_{32})^2 + S_1(hkl)((u_{12})^2 + (u_{22})^2 + (u_{32})^2)\right)\sigma_{22}\\ &+ \left(\tfrac{1}{2}S_2(hkl)(u_{33})^2 + S_1(hkl)((u_{13})^2 + (u_{23})^2 + (u_{33})^2)\right)\sigma_{33}\\ &+ 2\left(\tfrac{1}{2}S_2(hkl)u_{31}u_{32} + S_1(hkl)(u_{11}u_{12} + u_{21}u_{22} + u_{31}u_{32})\right)\sigma_{12}\\ &+ 2\left(\tfrac{1}{2}S_2(hkl)u_{31}u_{33} + S_1(hkl)(u_{11}u_{13} + u_{21}u_{23} + u_{31}u_{33})\right)\sigma_{13}\\ &+ 2\left(\tfrac{1}{2}S_2(hkl)u_{32}u_{33} + S_1(hkl)(u_{12}u_{13} + u_{22}u_{23} + u_{32}u_{33})\right)\sigma_{23}\end{aligned} \quad (2)$$

The measurement takes place in the laboratory coordinate system $\mathscr{L}$ along (by convention) the axis $\mathscr{L}_3$. Therefore, the measured strain $\varepsilon_{33}(hkl,\ \Psi)'$ is the component, ε_{33} of the strain tensor expressed[1] in $\mathscr{L}$ and has to be transformed into the strain tensor expressed in the sample system $\mathscr{P}$. The necessary transformation matrix U (with elements u_{ij}) from $\mathscr{P}$ to $\mathscr{L}$ depends on the diffractometer geometry, or more explicitly, on the orientation of the sample with respect to the diffraction plane. This orientation is expressed by a set of suitable orientation angles Ψ_1, Ψ_2, Ψ_3. In order to convert strains into stresses one needs elastic compliances. In RSE they are called X-ray elastic constants $S_1(hkl)$, $\frac{1}{2}S_2(hkl)$ and depend on the lattice plane (hkl), the texture within the sample, and particularly (if calculated) on the assumptions (Voigt and Reuss model, etc.) about the mechanical interaction of the crystallites in the polycrystalline sample, see Behnken and Hauk (1986).

The strain $\varepsilon(hkl,\Psi)$ is not truly measured, but calculated according to

$$\varepsilon(hkl,\Psi) = \frac{d(hkl,\Psi) - d_0(hkl)}{d_0(hkl)} \quad (3)$$

from the measured lattice plane distance $d(hkl,\ \Psi)$ which is different from the strain-free lattice spacing $d_0(hkl)$ in the presence of residual stresses. Eq. (3) poses the 'fundamental problem of RSE', because the strain-free lattice constants a_0, b_0, c_0, a_0, β_0, γ_0 of the lattice must be known in order to calculate $d_0(hkl)$. Hauk (1991) gives an overview of this problem. Since the sample contains residual stresses, one

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[1] The prime indicates a quantity given in $\mathscr{L}$.

0191-8141/00/$ - see front matter
PII: S0191-8141(00)00111-5

may doubt that it is possible to measure the strain-free lattice constants of the sample under investigation. In the following sections several procedures are outlined which actually provide the strain free lattice constants under certain restrictions. These restrictions concern the form of the GTE and the elasticity and crystal class of the material.

2. The $\sin^2 \Psi$-law

The so-called $\sin^2 \Psi$-law is nothing but a special form of Eq. (2). If follows from the assumption $\sigma_{13} = \sigma_{23} = \sigma_{33}$. For the following discussion we will change from $\varepsilon(hkl, \Psi)$' to $d(hkl, \psi)$ in the GTE. Using Eq. (2) and Eq. (3) (and assuming to be in the principal axis system of the stress tensor) one arrives at (with the usual denominations for the two orientation angles ϕ and ψ)

$$d(hkl, \phi, \Psi) = \Big(\tfrac{1}{2} S_2(hkl)(\sigma_{11}\cos^2\phi + \sigma_{22}\sin^2\phi)\sin^2\Psi + s_1(hkl)(\sigma_{11} + \sigma_{22})\Big) d_0(hkl) + d_0(hkl). \quad (4)$$

The unknown $d_0(hkl)$ enters into Eq. (4) as factor and as an additional term. One can carry out an error analysis and calculate the error $\Delta\sigma$ for σ_{11}, σ_{22} caused by an error $\sigma d_0(hkl)$ in $d_0(hkl)$. One finds that the relative error $\Delta\sigma/\sigma_{ii}$ is equal to the relative error $\Delta d_0(hkl)/d_0(hkl)$ which will amount to a few 0.1 percent under normal circumstances. Therefore it is practice to even arbitrarily set $d_0(hkl) = d(hkl, \phi = 0, \psi = 0)$. If the $\sin^2 \psi$-law *is* applicable and if high precision is not requested, then the d_0-problem is of minor importance.

3. Strain-free direction

From Eq. (3) we can define a direction $\vec{n}(\Psi^*)$along which the strain vanishes. For that purpose we consider two measurements for the two opposite sample orientations Ψ and -Ψ. Then the condition for $\vec{n}(\Psi^*)$ is

$$\varepsilon(hkl, \Psi^*) + \varepsilon(hkl, -\Psi^*) = 0 \quad (5)$$

from which follows $d(\Psi^*) = d(-\Psi^*) = d_0$. An explicit equation for Ψ^* follows from Eq. (5) after insertion of Eq. (2). For example, in the case of the usual $\phi\psi$-diffraktometer (sample tilt about the orientation angle ψ) and assuming $\phi_{33} = 0$ one obtains Hauk (1991)

$$\sin^2\psi_2^* = \frac{-S_1(hkl)}{\frac{1}{2}S_2(hkl)}\left(1 + \frac{\sigma_{22}}{\sigma_{11}}\right). \quad (6)$$

From Eq. (4) one sees that plotting the measured lattice spacings $d(hkl, \phi = 0, \psi)$ for constant azimuthal angle $\phi = 0°$ over ψ will give σ_{11} as the slope of the plotted line. Correspondingly one obtains σ_{22} from a plot for $\phi = 90°$. Inserting the resulting values into Eq. (6) will give the strain free direction ψ^* along which the strain free lattice spacing $d(hkl, \Psi^*) = d_0(hkl)$ can be measured.

If σ_{33} may not be assumed to be zero, then other procedures for finding Ψ^* must be applied. However, all known procedures rely on certain simplifying assumptions which may not be valid for the actual sample under investigation. If the material is elastically isotropic, then one may use the elastic modulus E and the Poisson ratio ν instead of X-ray elastic constants. If one further assumes that $v = 0.5$ (this assumption allows to lump together two terms containing σ_{33} in the $\sin^2 \varphi$-law, please refer to Hauk (1991)), then one gets (we do not cancel $\frac{\nu}{E}/\frac{1+\nu}{E}$ because of the subsequently necessary evaluation of Eqs. (8) and (9))

$$\sin^2\psi^* = \frac{\frac{\nu}{E}}{\frac{1+\nu}{E}} 1 + \left(\frac{\sigma_{22} - \sigma_{33}}{\sigma_{11} - \sigma_{33}}\right). \quad (7)$$

According to Eq. (4), for a plot of the measured lattice spacing $d(hkl, \phi, \psi)$ over $\sin^2 \psi$ one obtains

$$\frac{1+\nu}{E} d_0(\sigma_{11} - \sigma_{33}) \text{ for } \phi = 0^\circ, \quad (8)$$

$$\frac{1+\nu}{E} d_0(\sigma_{22} - \sigma_{33}) \text{ for } \phi = 90^\circ. \quad (9)$$

From these equations the quotient in Eq. (7) can be calculated and thus ψ^*. Further procedures for the determination of Ψ^* are described in the literature, e.g. the one from Torbaty et al. (1983), but they all depend on certain assumptions which restrict their applicability.

4. Special strain/stress states

If the sample is known to have a distinct stress state with some of the tensor elements $\sigma_{ij} = 0$, then the evaluation of the GTE may result in a procedure to determine the strain/stress tensor and the strain-free lattice constant simultaneously. For thin polycrystalline films on a bulk substrate corresponding procedures have been developed, see Witt and Vook (1968); Haase (1989); Segmüller and Murakami (1985); Perry et al., (1992); Wieder (1995a); Wieder (1995c). The main idea is due to Witt and Vook (1968). For a thin film (thickness of about 1 m) one may, assume that $s_{13} = s_{23} = s_{33} = 0$. Furthermore it is assumed that the residual stress in the film is caused by the mismatch between the thermal expansion coefficients a_f and a_s, of film and substrate, resulting in a thermal strain according to $\varepsilon_{th} = (a_f - a_s)\ (T_d - T_m)$ where T_d and T_m are the temperatures of the film during deposition and during measurement. Making the reasonable assumptions that $\varepsilon_{th} = \varepsilon_{11} = \varepsilon_{22}$ and $\varepsilon_{12} = 0$, one arrives from Eq. (1) at the following linear equation system Eqs. (10)–(12) for the determination of σ_{11}, σ_{22}, σ_{33}. In Eqs. (10)–(12), the coefficients S_{ijkl} are the elastic compliances of the single crystal transformed to the sample system $\mathscr{P}$.

$$\varepsilon_{th} = \varepsilon_{11} = S_{1111}\sigma_{11} + S_{1122}\sigma_{22} + (S_{1112} + S_{1121})\sigma_{12} \quad (10)$$

$$\varepsilon_{th} = \varepsilon_{22} = S_{2211}\sigma_{11} + S_{2222}\sigma_{22} + (S_{2212} + S_{2221})\sigma_{12} \quad (11)$$

$$0 = \varepsilon_{12} = S_{1211}\sigma_{11} + S_{1222}\sigma_{22} + (S_{1212} + S_{1221})\sigma_{12}. \quad (12)$$

Once σ_{11}, σ_{22}, σ_{33} have been found, the remaining unknown strain tensor components ε_{33}, ε_{23}, ε_{13} follow immediately from

$$\varepsilon_{33} = S_{3311}\sigma_{11} + S_{3322}\sigma_{22} + (S_{3312} + S_{3321})\sigma_{12} \quad (13)$$

$$\varepsilon_{23} = S_{2311}\sigma_{11} + S_{2322}\sigma_{22} + (S_{2312} + S_{2321})\sigma_{12} \quad (14)$$

$$\varepsilon_{13} = S_{1311}\sigma_{11} + S_{1322}\sigma_{22} + (S_{1312} + S_{1321})\sigma_{12}. \quad (15)$$

The connection to the fundamental problem of RSE is provided by the GTE again. For simplicity we restrict ourselves to cubic materials ($a_1 = a_2 = a_3 = a$) here, but the procedure is valid for any crystal class. The lattice constant $a = a(hkl,\ uvw)$ will depend on the measurement direction (given by the indices $u,\ v,\ w$ of the lattice plane normal $\vec{n}$ (uvw)) and on the lattice plane indices because of the strain $\varepsilon(hkl,\ uvw)$ acting on the lattice plane (hkl) in orientation $\vec{n}(uvw)$ within S. The explicit relation

$$a(hkl) = a_0\varepsilon(hkl, uvw) + a_0 \quad (16)$$

is nothing but the GTE in a particular form. One can use Eq. (16) to simultaneously determine $\overset{2\leftrightarrow}{\varepsilon}$ and a_0 from measured reflection positions $\Theta(hkl,\Psi)$ (where Ψ indicates the measurement direction within $\mathscr{P}$) by a least-square fit (Wieder, 1996b)

The above outlined procedure is just an example for procedures for the simultaneous determination of $\overset{2\leftrightarrow}{\varepsilon}$ or $\overset{2\leftrightarrow}{\sigma}$ and strain-free lattice constants a_0 b_0, c_0 adapted to the material under investigation. One can think of corresponding procedures for material classes other than thin films. The main advantage of these adapted procedures is to take into account only tensor components which will be different from zero and to neglect all other components which reduces the number of unknowns. However, in the general case all six tensor components may be different from zero and then correspondingly a general procedure must be applied.

5. General strain/stress state

If the full strain/stress tensor *and* the strain free lattice constants are to be determined simultaneously, then the GTE must be evaluated in its full form. We restrict the discussion to the GTE in Eq. (1), all arguments below apply equally well to the use of Eq. (2). Using Eq. (3) the GTE (1) takes on the form

$$d(hkl,\Psi) = ((u_{31})^2\varepsilon_{11} + 2u_{31}u_{32}\varepsilon_{12} + 2u_{31}u_{33}\varepsilon_{13} + (u_{32})^2\varepsilon_{22} + 2u_{32}u_{33}\varepsilon_{23} + (u_{33})^2\varepsilon_{33})d_0(hkl) + d_0(hkl). \quad (17)$$

In Eq. (17) the strain-free lattice spacing $d_0(hkl)$ appears explicitly as unknown on the right hand side. Since the tensor components ε_{ij} are also unknown and since products of the form $\varepsilon_{ij}d_0(hkl)$ arise, this equation is non-linear in the unknowns. Each single measurement of a reflection position $\Theta(hkl,\ \Psi)$ and thus $d(hkl,\Psi)$ will provide us with one single equation of the form Eq. (17). The idea of any general procedure is to collect a sufficient number of linearly independent equations of the form Eq. (17) to yield an equation system from which all unkowns can be calculated. Several procedures of this kind have been presented (Peiter and Wern, 1987; Wern, 1991, Wieder, 1996a; Wieder, 1995b), even depth-dependent (Genzel, 1999).

Before showing examples we want to discuss whether the equation system Eq. (17) is solvable. Recently Eigenmann and Macherauch (1995/1996) (see section 8.3 of the cited paper) stated that a simultaneous determination of the full stress tensor (with $s_{33} \neq 0$) and strain-free lattice constants is impossible. Eigenmann argues using a form of the GTE (eq. 133 in the cited paper) where he supresses the (hkl)-dependence of the GTE. Then his argument is right, one can not discern between changes in a hydrostatic stress state or corresponding changes in d_0 from the change in θ_0 for just a single (hkl). However, the GTE is (hkl)-dependent, we are allowed to take into account not just one (hkl)-reflection (as in traditional RSE), but any (hkl)-reflection which is available from the diffraction experiment.

However, even taking the (hkl)-dependence into account one may still doubt whether the equation system is solvable. The argument supporting the doubt is as follows: "Any change in $d_0(hkl)$ can be compensated by a corresponding opposite change in ε_{ij}." We give the following answer to this argument. If we inspect the dependency of $d(hkl,\Psi)$ on ε_{ij} and on $d_0(hkl)$ by forming the partial derivates, then we see that these dependencies are of different analytical form. Once we have a given set of equations which form a discrete restriction in the solution space, then it matters how a change in the variables changes the data. Speaking in a very loose manner, we have some sort of 'regularisation' by discretization which makes the equations system solvable.

Two main solution methods are available from mathematics for the problem in question, namely dedicated solution methods for nonlinear equation systems and the (more general) least-squares solution methods. If we intend to solve the equation system by one of the common iterative solution methods for non-linear equation systems, then we even can examine whether such an iterative method will converge. Banach's fixpoint theorem can be applied. From

Table 1
Simultaneous calculation of the strain tensor $\overset{2\leftrightarrow}{\varepsilon}$ or the stress tensor $\overset{2\leftrightarrow}{\sigma}$ and the strain free lattice constant a_0 for a simulated X-ray diffraction experiment on copper in grazing incidence diffraction. Simulated Bragg positions $\Theta(hkl, \Psi)^{\text{sim}}$ were calculated from an arbitrarily given tensor $\overset{2\leftrightarrow}{\varepsilon}$ or $\overset{2\leftrightarrow}{\sigma}$ (first column) and given a_0. From this set of simulated $\Theta(hkl, \Psi)^{\text{sim}}$ the tensor $\overset{2\leftrightarrow}{\varepsilon}$ or $\overset{2\leftrightarrow}{\sigma}$ was (re-)calculated (right column).

	input $\overset{2\leftrightarrow}{\epsilon}$, a_0	**output** $\overset{2\leftrightarrow}{\epsilon}$, a_0
ϵ_{11}	0.001	0.0011
ϵ_{12}	0.0	0.0
ϵ_{13}	-0.0005	-0.0005
ϵ_{22}	0.001	0.0011
ϵ_{23}	-0.0005	-0.0005
ϵ_{33}	-0.001	-0.0008
a_0	0.36150 nm	0.36147 nm
	input $\overset{2\leftrightarrow}{\sigma}$, a_0	**output** $\overset{2\leftrightarrow}{\sigma}$, a_0
σ_{11}	500 MPa	579 MPa
σ_{12}	75 MPa	75.8 MPa
σ_{13}	33 MPa	33.1 MPa
σ_{22}	450 MPa	529.0 MPa
σ_{23}	-90 MPa	-89.6 MPa
σ_{33}	-650 MPa	-375.9 MPa
a_0	0.36150 nm	0.36179 nm

this analysis (see Appendix A) we see that it is possible to solve the non-linear equation system Eq. (17) in principle. However, experimental data are always connected with experimental errors. The error propagation from the experimental data through the calculation into the results has to be analysed. We are not aware of such an exhaustive analysis which is therefore left for future work.

From the praxis of numerical data analysis one will tend to apply a least-squares method to solve the equation system Eq. (17). Least-squares routines are more easy to handle and have the advantage to average about (partially self-contradictory) experimental errors in the data. The drawback from the mathematical viewpoint is that no least-squares method is known which is guaranteed to end in the global minimum which corresponds to the solution of Eq. (17). However, one can perform numerical simulations to test the feasibility of the procedure actually implemented. In (Wieder, 1995b) a computer program (named SBGBBG) has been presented which performs such a least-squares analysis. Among others, SBGBBG allows to calculate reflection positions $\Theta(hkl, \Psi)^{\text{sim}}$ from a given tensor $\overset{2\leftrightarrow}{\varepsilon}$ or $\overset{2\leftrightarrow}{\sigma}$ and given strain-free lattice constants. Then one can use SBGBBG to calculate

Table 2
$\overset{2\leftrightarrow}{\varepsilon}$ for an ion-plated aluminium thin film, calculated without (left) and with (right) fit of a_0

a_0 **taken from literature**	**with fitting** a_0 **from data**
$\begin{pmatrix} -0.0010 & 0.0 & -0.0001 \\ & -0.0008 & -0.0002 \\ & & 0.0023 \end{pmatrix}$	$\begin{pmatrix} -0.0016 & 0.0 & -0.0001 \\ & -0.0012 & -0.0004 \\ & & 0.0030 \end{pmatrix}$
$a_0^{lit} = 0.40496$ nm	$a_0^{fit} = 0.40487$ nm

$\overset{2\leftrightarrow}{\varepsilon}$ or $\overset{2\leftrightarrow}{\sigma}$, and the lattice constants from the set of simulated $\Theta(hkl, \Psi)^{\text{sim}}$. We carried out several simulations for strain and stress tensors of arbitrary components. These simulations show that it is possible to determine simultaneously $\overset{2\leftrightarrow}{\varepsilon}$ or $\overset{2\leftrightarrow}{\sigma}$, and the strain-free lattice constants also in the triaxial case. Table 1 gives some results of simulations.

The comparison between given and calculated quantities gives an impression of the *numerical* accuracy achievable by a least squares fit. Up to now, an exhaustive error analysis has not been made for this least squares routine implemented in SBGBBG. Table 1 is meant as a first example only. Depending on the requested accuracy the present accuracy may be sufficient of insufficient. If one intends to do stress analysis with an accuracy of $\pm$ 1 MPa as seems to be necessary for geological applications, then the procedure in question is not good enough. However, in other cases one might be glad to have a reasonable estimate of the stresses which can be obtained by this procedure.

As an example for real data analysed by SBGBBG, the following measurement on a thin film of ion-plated aluminium on a silicon wafer may be mentioned. The measurement was done in grazing incidence diffraction with parallel beam geometry. Details on residual stress evaluation by grazing incidence diffraction can be found elsewhere (Wieder, 1995c). Nine different lattice planes (*hkl*) (from (111) to (224)) were used. From Table 2 one gets an impression about the changes in $\overset{2\leftrightarrow}{\varepsilon}$ calculated with a_0 set to be equal to the literature value for bulk Al and with a_0 simultaneously fitted to the data. For all practical purposes in residual stress determinations on thin films, these differences are negligible.

As a more difficult example, where two unknown lattice constants a_0 and c_0 are to be fitted, the results for a thin film of galvanic zinc are provided in Table 3.

6. Rietveld Analysis

Sections 2–5 concerned topics from RSE by diffraction in

Table 3
$\overset{2\leftrightarrow}{\varepsilon}$, $\overset{2\leftrightarrow}{\sigma}$ or for a thin film of galvanic zinc, calculated without (left) and with (right) fit of a_0, c_0

tensor	a_0, c_0 taken from literature	with fitting a_0, c_0 from data
$\overset{2\leftrightarrow}{\epsilon}$	$\begin{pmatrix} -0.0010 & 0.0 & 0.0 \\ & -0.0009 & 0.0 \\ & & 0.0007 \end{pmatrix}$	$\begin{pmatrix} -0.0009 & 0,0 & 0.0 \\ & -0.0007 & 0.0 \\ & & 0.0006 \end{pmatrix}$
$\overset{2\leftrightarrow}{\sigma}$	$\begin{pmatrix} -198\,\text{MPa} & 0.0 & 0.0 \\ & -191\,\text{MPa} & 0.0 \\ & & 4\,\text{MPa} \end{pmatrix}$	$\begin{pmatrix} -110\,\text{MPa} & 0.0 & 0.0 \\ & -97\,\text{MPa} & 0.0 \\ & & 9\,\text{MPa} \end{pmatrix}$
	$a_0^{lit} = 0.26648$ nm $c_0^{lit} = 0.49470$ nm	$a_0^{fit} = 0.26642$ nm $a_0^{fit} = 0.49424$ nm

the traditional manner which means RSE by analysing reflection positions $\Theta(hkl,\Psi)$. An X-ray or neutron diffractogram of a polycrystalline sample contains far more information then just the reflection positions. For polycrystalline materials, one can perform a Rietveld analysis of the entire scattered intensity $I(\Theta)$. One forms a theoretical model of the structure and fits the theoretical intensity $I(\Theta)^{\text{theo}}$ calculated for the model structure to the measured intensity $I(\Theta)$. The structure parameters like lattice constants a_0 b_0, c_0 are parameters of the fit. The first Rietveld analyses were done to refine known structure models. Today, complete structure analyses from powder data are feasible. As progress continues, in the last years the description of the samples 'real structure', i.e. crystallite size, texture, strain and stress, has become a major application of the Rietveld analysis (Le Bail, 1992; Ferrari and Lutterotti, 1994; Scardi et al., 1993). New Rietveld programs emerge which explicitly take into account residual stresses (Lutterotti, 1999).

We do not intend to describe these Rietveld-based procedures for RSE. We just stress that these Rietveld procedures (per se) simultaneously determine strain-free lattice constants and strain/stress tensors simultaneously. As in traditional RSE, measurements for several different sample orientations (within $\mathscr{L}$) have to be done in order to get enough information to fit a strain or stress tensor to the data. We expect that these procedures will become standard for the 'real structure analysis' of materials, including RSE.[2]

7. Discussion

The acceptance of a method depends on the requirements to be fulfilled by the method. All the above outlined procedures for evaluating the strain-free lattice constants are of a certain accuracy and one will accept a certain method if its accuracy is considered to be sufficient. This sufficiency will depend on the samples under investigation. For bulk shot-peened steel surfaces often one is interested just in the sign (+ or −) of the stresses (tensile or compressive). An error of ±50 MPa is considered as acceptable in common RSE. For thin films (from physical vapour deposition) one is content with even less accuracy. In the opinion of the present authors, the available procedures for the fundamental problem of RSE are of sufficient accuracy if compared to the usually requested accuracy in RSE. The situation may be different for geological applications, where an error of maximal ±1 MPa has been claimed (Pintschovius, 1999). However, such a small error limit poses a problem to RSE

[2] The developers of Rietveld procedures do not care about the deep-rooted doubts of traditional RSA concerning the simultaneous determination of $\overset{2\leftrightarrow}{\varepsilon}$ and a_0.

even if the strain-free lattice constants may be assumed to be known.

Acknowledgements

Many thanks to Prof. Dr. Jürgen Schreiber, Fraunhofer-Institut fur zerstörungsfreie Werk stoffprüfung, Dresden, for valuable discussions. Also many thanks to Dr. Klaus Ullemeyer for his work as editor.

Appendix A

Without mathematical strictness we outline how one can prove the solvability of the non-linear equation system (17) by use of Banach's fix point theorem:Consider a non-linear equation system $F_1, ..., F_n \equiv \mathbf{F}$ of n equations F_i which can be brought into the form $\mathbf{F}(\mathbf{x}) = \mathbf{x}$ which then can be solved in the rectangular, n-dimensional interval $A \subset \Re^n$ by an iteration ν, $\nu + 1$,…

$$\mathbf{F} = (\mathbf{x}^{\nu}) = \mathbf{x}^{\nu+\mathbf{1}} \tag{A1}$$

if Eq. (A1) has a fix point $\mathbf{x}^*$ on A (Bronstein et al., 1998). A sufficient condition for Eq. (A1) to be fulfilled can be found by means of the Jacobi matrix (Graf von Finkenstein, 1977)

$$\mathbf{J}(x_1, ..., x_n) = \begin{pmatrix} \frac{\delta F_1}{\delta x_1} & \cdots & \frac{\delta F_1}{\delta x_n} \\ \frac{\delta F_1}{\delta x_1} & \cdots & \frac{\delta F_1}{\delta x_n} \end{pmatrix} \tag{A2}$$

If for any matrix norm $\| \mathbf{J} \| \leq L < 1$ for all $\mathbf{x} \in A$, then a fix point exists in A. The partial derivations of the GTE (17) with respect to the unknowns ε_{ij} and a_0 (cubic case) are (with Einstein's summation convention in place)

$$\frac{\delta d}{\delta \varepsilon_{ij}} = u_{3i} u_{3j} \frac{1}{\sqrt{h^2 + k^2 + l^2}} a_0 \tag{A3}$$

$$\frac{\delta d}{\delta a_0} = u_{3i} u_{3j} \varepsilon_{ij} \frac{1}{\sqrt{h^2 + k^2 + l^2}} a_0 + \frac{1}{\sqrt{h^2 + k^2 + l^2}} \tag{A4}$$

Since the $\| u_{ij} \|$ as elements of a rotation matrix are always smaller than 1 and the $\| \varepsilon_{ij} \|$ are smaller than ≈ 0.05 one can conclude that Eq. (A4) is always smaller than 1. For Eq. (A3) all terms are smaller than 1, except the term a_0 which may be >1. Using a corresponding scaling of Eq. (17) will allow to keep $a_0 < 1$. We conclude that for every element j_{ij} of $\mathbf{J}$ one has $j_{ij} < 1$. Then any matrix norm like the row norm $\sqrt{\sum_j j^2_{i=\text{const},\, j}}$ or the column norm will be less than $L < 1$ on A (which follows from the scaling of a_0 and from $\varepsilon_{ij} < 0.05$).

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