A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
Volume 53 2008 Issue 1

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CORRECTIONS FOR RESIDUAL STRESS IN X-RAY GRAZING INCIDENCE TECHNIQUE

O POPRAWKACH PRZY WYZNACZANIU NAPRĘŻEŃ WŁASNYCH RENTGENOWSKĄ METODĄ STAŁEGO KĄTA PADANIA

Grazing incidence technique (called also GID-$\sin^2\psi$ method) can be used to study samples with important stress gradients. Using this method, it is possible to perform a non-destructive analysis of the heterogeneous stress field for different (and well defined) volumes below the surface of the sample. Moreover, the stress can be measured at very small depths of the order of a few μm. Asymmetric geometry is used in this technique. The penetration depth of radiation is almost constant in a wide 2θ range for a given incidence angle α. It can be easily changed by an appropriate selection of α angle (or also by using a different type of radiation). This enables the investigation of stress variation with depth below the sample surface.

There are, however, some factors which have to be corrected in this technique. The most important is the refraction of X-ray wave (with refraction coefficient smaller than one). It changes the wave length and direction of the beam inside a sample. These two effects cause some shift of a pick position, which should be taken into account in data treatment. For small incidence angles ($\alpha \leqslant 10°$) the correction is significant and it can modify the measured stress even of 70 MPa. The refraction correction decreases, however, with increasing the incidence angle. Other corrections (absorption, atomic factor, Lorentz-polarization factor) are less important for the final results.

The studied corrections were tested on ferrite powder samples and on a sample of AISI316L stainless steel.

Keywords: X-ray diffraction, internal stresses, stress determination, grazing incidence method, AISI316L stainless steel

Metoda stałego kąta padania (zwana także metodą GID-$\sin^2\psi$) może być zastosowana do badania materiałów o dużym gradiencie naprężeń własnych. Przy jej pomocy można dokonać nieniszczącej analizy niejednorodnych naprężeń w różnych (dobrze zdefiniowanych) częściach próbki poniżej jej powierzchni. Naprężenia mogą być wyznaczone na bardzo małych głębokościach, rzędu kilku μm. W metodzie używa się geometrii asymetrycznej. Głębokość wnikania jest prawie stała w szerokim przedziale kąta 2θ przy ustalonej wartości kąta padania α. Głębokość tą można łatwo zmieniać dobierając odpowiednio wartość kąta α (oraz także używając różnego typu promieniowania). Wszystko to pozwala wyznaczyć zmienność naprężeń w głąb próbki.

Omawiana technika wymaga jednak uwzględnienia kilku czynników korygujących. Najważniejszym z nich jest załamanie fali promieniowania rentgenowskiego (współczynnik załamania mniejszy od jedności). Zmienia ono zarówno długość jak i kierunek propagacji fali wewnątrz próbki. Oba te efekty powodują delikatne przesunięcie piku dyfrakcyjnego, które powinno być uwzględnione przy analizie wyników pomiaru. Dla małych kątów padania ($\alpha \leqslant 10°$) poprawka jest znacząca i może ona zmienić wartość wyznaczanych naprężeń nawet o 70 MPa. Poprawka wynikająca z załamania fali rentgenowskiej maleje jednak ze wzrostem kąta padania. Inne poprawki (absorpcja, czynnik atomowy, czynnik Lorentza-polaryzacji) są mniej istotne dla dokładności pomiarów.

Powyższe poprawki zostały przetestowane na próbkach proszkowych żelaza oraz na próbce stali nierdzewnej AISI316L.

1. Introduction

Classical $\sin^2\psi$ method is one of basic methods for measuring the residual stresses and elastic properties of polycrystalline materials. Main disadvantage of this method is a variable penetration depth, which depends on angle. For this reason the classical $\sin^2\psi$ method cannot be used to study materials with a high stress gradient. The geometry based on the grazing angle incidence X-ray diffraction (so-called grazing incident diffraction method, GID-$\sin^2\psi$) is discussed and applied for stress measurement. Using this method, it is possible to perform a non-destructive analysis of the heterogeneous stress for different (and well defined) volumes below the surface of the sample. Moreover, the stress can be measured at very small depths, of the order of a few

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micrometers. The incidence angle is small and consequently it is necessary to take into account additional factors which are not significant in classical geometry.

2. Methods of stress determination

The standard $\sin^2\Psi$ method of stress determining is based on the measurement of interplanar spacing for various directions of the scattering vector. These directions are defined by φ and ψ angles (Fig. 1). Using diffraction, the mean interplanar spacing $< d(\phi, \psi) >_{\{hkl\}}$ averaged only for reflecting grains which possess the scattering vector normal to the $\{hkl\}$ crystallographic planes is measured [11]. In the standard $\sin^2\Psi$ method the positions of diffraction peak corresponding to a single reflection *hkl* are analysed. In the case of X-ray diffraction the measurement is performed for the near surface volume, which is limited by radiation absorption [1]. The penetration depth z, defined as the distance from the sample surface for which $1 - \frac{1}{e}$ of total intensity of the incident beam is absorbed, can be calculated from the formula:

$$z = \frac{-\ln(1 - G_z)}{2\mu} \sin\theta \cos\psi, \tag{1}$$

where: μ is the linear absorption coefficient, $G_z = 1 - \frac{1}{e} \cong 0.63$, θ is the Bragg angle and ψ angle defines the inclination of the scattering vector Q from the normal to the sample surface (Fig. 1). Because the penetration depth strongly depends on θ and ψ angles, this method cannot be used when a large macro-stress gradient appears.

The grazing incidence diffraction geometry, so-called GID-$\sin^2\psi$ method (Fig. 2), is characterized by a small and constant incidence angle α and by different orientations of the scattering vector (changing 2θ angle for a constant wavelength). The parallel beam geometry is used to minimise errors connected with sample misalignment [2]. Only detector moves in this geometry and ψ_{hkl} angle is expressed by equation:

$$\psi_{hkl} = \theta_{hkl} - \alpha. \tag{2}$$

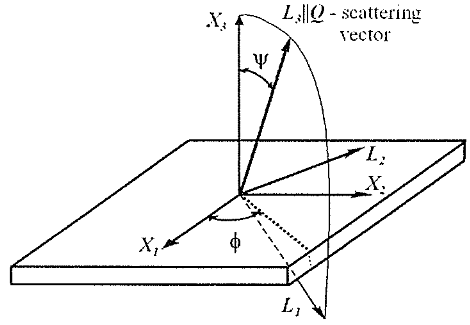

Fig. 1. Orientation of the scattering vector with respect to the sample system X. The ψ and ϕ angles define the orientation of the L system (L_2 axis lies in the plane of the sample surface). The laboratory system, L, defines the measurement of the interplanar spacings $< d(\phi, \psi) >_{\{hkl\}}$ along the L_3 axis

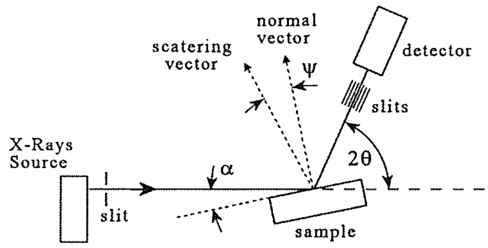

Fig. 2. Geometry of GID-$\sin^2\psi$ method

The ψ_{hkl} angle depends on incidence angle (α) and type of reflection $\{hkl\}$. The possible values of $\psi_{\{hkl\}}$ angles are limited to the number of *hkl* reflections used in the experiment.

For this geometry the penetration depth t is described by [3]:

$$z = \frac{-\ln(1 - G_z)}{\mu\left[\frac{1}{\sin\alpha} + \frac{1}{\sin(2\theta-\alpha)}\right]}, \tag{3}$$

where α is incident beam angle.

The main advantage of GID-$\sin^2\psi$ method is a constant or almost constant penetration depth for a fixed α value and for a radiation of given type. However, the penetration depth can be changed by selection of the incidence angle. This gives a possibility to investigate materials with a stress gradient. Choosing appropriate α values and type of radiation it is possible to measure stresses from different volumes below the surface.

The penetration depths vs. $\sin^2\psi$ for classical and GID geometry according Eq. 1 and Eq. 3 are presented in Fig. 3.

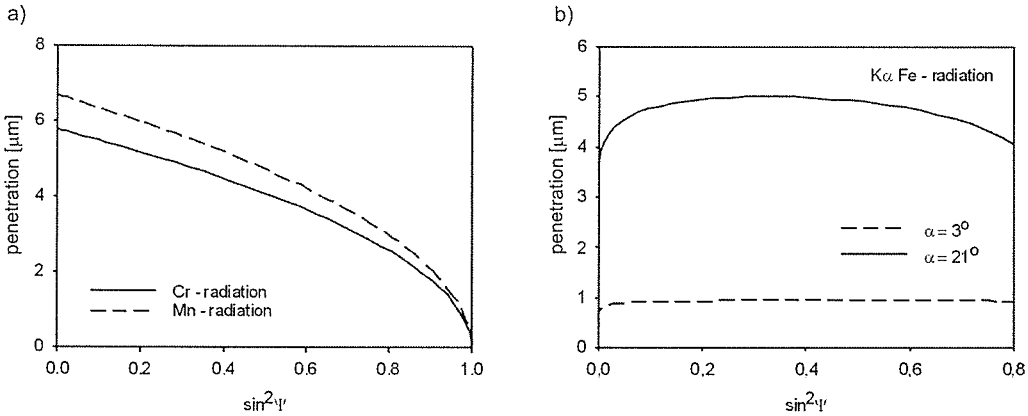

Fig. 3. The penetration depth for {211} reflection in steel vs. $\sin^2\psi$: a) for classical geometry using different wavelengths, b) for GID method for two different incidence angles

3. Grazing incidence geometry applied for stress determination

In GID-$\sin^2\psi$ method, the $< d(\phi,\psi) >_{\{hkl\}}$ interplanar spacing is measured in directions defined by the ϕ and ψ angles for different hkl reflections. These experimental data can be easily analysed by the multi-reflection procedure and residual stresses can be determined for every incidence angle α [4, 5, 6]. The interplanar spacing measured in the L_3 direction (Fig. 1) is given by the well known relation, which can be rewritten for equivalent lattice parameters a_{hkl}:

$$< a(\phi,\psi) >_{\{hkl\}} = \Big[s_1(hkl)(\sigma^M_{11}+\sigma^M_{22}+\sigma^M_{33}) + \frac{1}{2}s_2(hkl) (\sigma^M_{11}\cos^2\phi + \sigma^M_{22}\sin^2\phi + \sigma^M_{12}\sin 2\phi)\sin^2\psi_{\{hkl\}} + \frac{1}{2}s_2(hkl)\,\sigma^M_{33}\cos^2\psi_{\{hkl\}} + \frac{1}{2}s_2(hkl)(\sigma^M_{13}\cos\phi + \sigma^M_{23}\sin\phi)\sin 2\psi_{\{hkl\}}\Big] a^o + a^o, \qquad (4)$$

where: $a_{hkl} = d_{hkl}/\sqrt{h^2+k^2+l^2}$ are equivalent lattice parameters, σ^M_{ij} is the average macrostress for the penetration depth t corresponding to a given incidence angle, while $s_1\{hkl\}$ and $\frac{1}{2}s_2\{hkl\}$ are the diffraction elastic constants for the studied quasi-isotropic sample, calculated for different hkl reflections related to $\psi_{\{hkl\}}$ angles by Eq. 2.

The $< a(\phi,\psi) >_{\{hkl\}}$ parameters can be fitted applying the least square procedure and, consequently, the values of a^0 and the macrostress σ^M_{ij} can be found. The $< a(\phi,\psi) >_{\{hkl\}}$ versus $\sin^2\psi_{\{hkl\}}$ plot is linear in the case of quasi-isotropic sample.

4. Corrections in grazing incidence diffraction geometry

Similarly as in symmetrical Bragg-Brentano geometry, to estimate stresses it is necessary to consider all factors which influence the final result and to apply accurate corrections. A diffraction peak for a given diffraction pattern depends on several parameters [7, 8]:

- multiplicity
- temperature factor
- absorption factor
- Lorentz-polarization factor
- structure factor
- refraction factor.

Residual stress measurement is based on the peak position analysis. The first two factors (multiplicity and temperature factor) do not change the peak position but they modify intensity of the peak and FWHM (Full Width at Half Maximum). For these reasons they can be neglected in elastic stress analysis. The next factors from the list above are significant in stress analysis and they should be considered in diffraction data analysis. The value of absorption, Lorentz-polarization and structure factors are well known and are widely discussed in the literature. In the present paper only the refraction factor will be discussed.

Refraction index for X-ray radiation in metals is slightly smaller than unity [9, 10]. For this reason the phase velocity of electro-magnetic wave in material is higher than the velocity outside material (in the air). If anomalous dispersion is ignored, the refraction index factor for X-ray range is given by:

$$n = 1 - A\lambda^2\frac{\rho Z}{M}, \qquad (5)$$

where:

$$A = \frac{N_A e^2}{2\pi m_e c^2} = 2.7019 \cdot 10^{10} \ \left[\mathrm{cm}/_{\mathrm{mol}}\right] \qquad (6)$$

and: N_A – Avogadro's number; e – electron charge, m_e – electron mass, c – velocity of light, M – molecular weight, ρ – density, Z – atomic number and λ wavelength in cm^{-1}.

The index of refraction n of X-rays is slightly smaller than one. For wavelengths below 2Å, δ is of the order of 10^{-4} to 10^{-5}, depending on the density of the material.

Propagation direction of electro-magnetic wave changes during passing by the boundary of two media. This change depends on refraction index of a material and is described by Snell's law. Refraction causes a change in 2θ angle and a shift of peak position. For this reason a correction has to be introduced to the Bragg's law. The total correction consists of two factors:

- the first one takes into account a different wavelength in a material (change of so-called optical path),
- the second one takes into account a change of propagation direction on the boundary between two media.

Using simple geometric relations, one finds the resulting shift of the peak position:

$$\Delta 2\theta = \delta \left[ctg\alpha + ctg(2\theta - \alpha) + 2tg\theta \right]. \qquad (7)$$

The above correction depends on the incident beam angleα, the Bragg angle 2θ and on the material constant (δ). The variation of the total correction for refraction versus incidence and Bragg angles are shown in Fig. 4.

The correction for refraction strongly depends on the incidence angle α. For small α the shift can easily exceed 0.01° and then with growing α angle the shift decreases. What concerns the Bragg angle, the correction is important for low (below 20°) and for high (above 160°) values of 2θ angle.

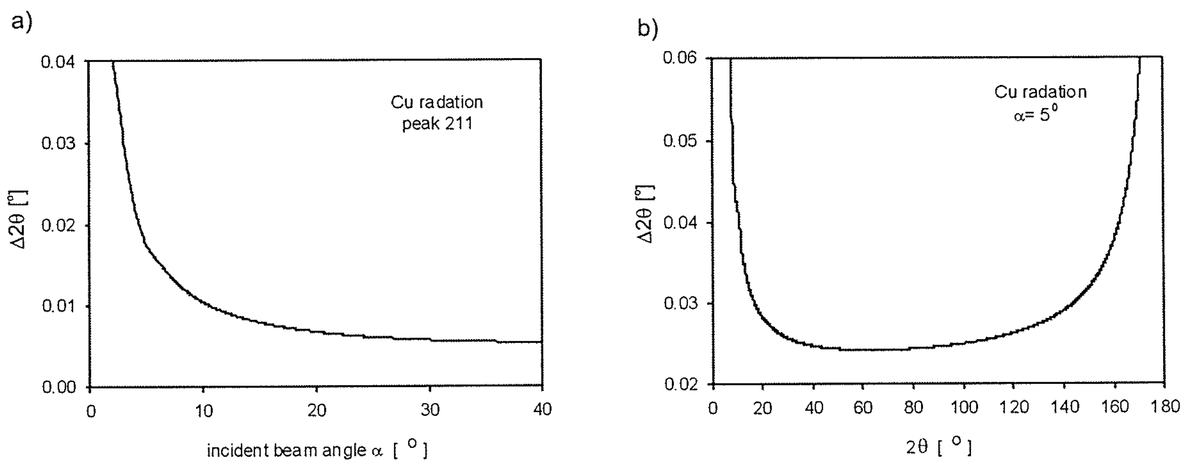

Fig. 4. a) Correction $\Delta 2\theta$ versus α for 211 peak in steel, b) Correction $\Delta 2\theta$ versus 2θ for the constant incidence angle $\alpha = 5°$. The graphs were calculated for Cu radiation

5. Experimental verification

The reference ferrite powder sample was prepared. The grazing incidence diffraction measurements were performed with X-pert Philips and Seifert X-ray diffractometer using Cu and Fe radiations, respectively. The interplanar spacings were determined for different reflections *hkl* (see Eq. 4.) and analysed using multi-reflection method. The experiment was repeated for various incidence angles α corresponding to different penetration depths t (Table 1).

The alignment of experimental set-up for grazing incidence diffraction geometry was first checked on a powder sample of ferrite iron. In this case, zero stress should be obtained for each incidence angle α, i.e., for different penetration depths t. Application of the corrections leads to a better agreement between two series of measurements (with Cu-radiation and Fe-radiations) – Fig. 5. A relatively low value of the measured pseudo-stress in the powder sample ($\sigma_{11}^M = \sigma_{22}^M \approx -25$ MPa) was found independently of the α angle. (Fig. 5). This value should be treated as a possible systematic error for other measurements.

The variation of the lattice parameter a_0 obtained by the multi-reflection analysis was studied as a function of the penetration depth t (or α), with and without corrections (Fig. 5). As expected, after correction the a_0 value does not depend on the depth and it is equal to 2.8663±0.0002Å. Like in the case of residual stress, corrections improve the agreement between results obtained with different radiations.

TABLE 1

Penetration depth of X-ray radiation in steel

Radiation /absorption coeff. (cm^{-1})/	Incidence angleα[°] and penetration depth t [μm] – grazing incidence diffraction method:						
	3°	6°	9°	12°	15°	18°	21°
Cu/μ_l = 2395/	0.21	0.39	0.55	0.69	0.82	0.93	1.02
Fe/μ_l = 554/	0.89	1.67	2.36	2.97	3.53	4.01	4.42
Average penetration depth for standard $\sin^2\psi$ method [μm]:							
Mn/μ_l = 700/	6.13						

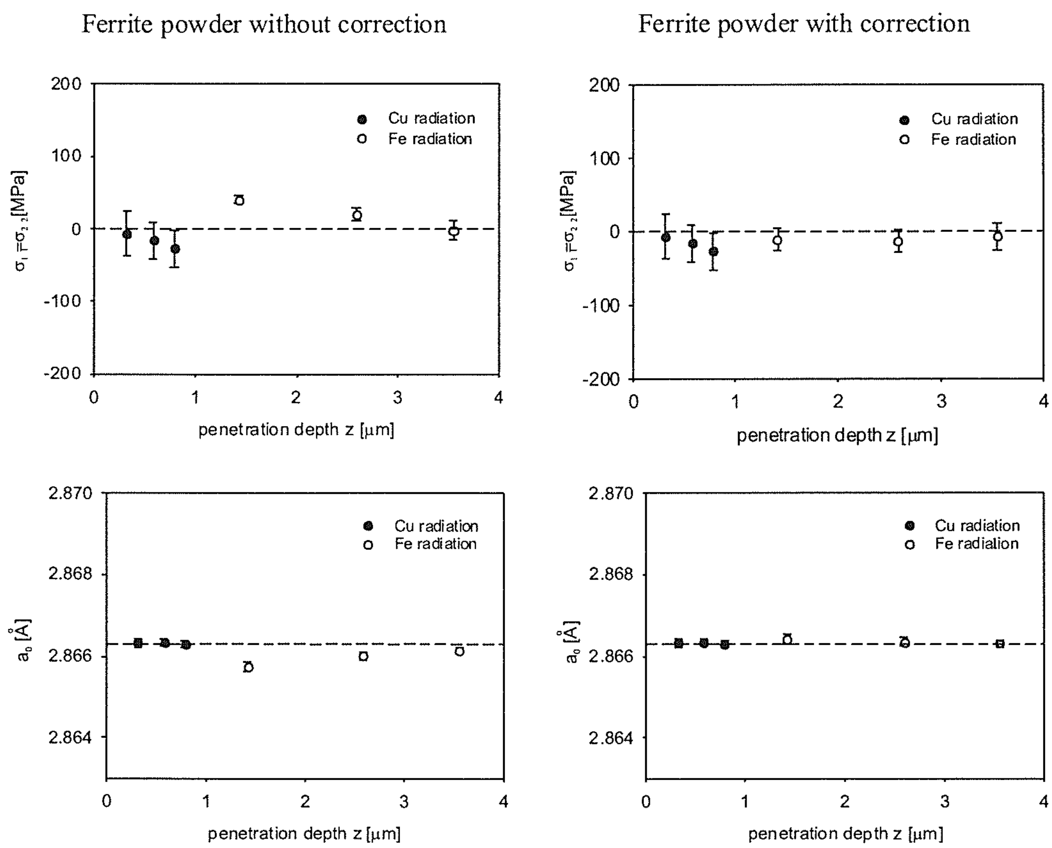

Fig. 5. $\sigma_{11}^M = \sigma_{22}^M$ and a_0 versus penetration depth z for the ferrite powder sample. Cu and Fe radiations and multi-reflection method were used. On the left – results without corrections, on the right- results with corrections are shown

TABLE 2

Chemical composition of the studied steel (mass %)

	C	Si	Mn	P	S	Cu	Ni	Cr	Mo
316L	0.02	0.56	1.67	0.041	0.041	0.35	11.14	17.24	1.96

TABLE 3

Mechanical properties of the as received materials

Specimen	0.2% proof stress (MPa)	Ultimate tensile strength (MPa)	E (GPa) Young modulus
316L	200	535	196

The discussed corrections were applied next to austenite steel samples after grinding. Chemical composition and mechanical properties of the material used for sample preparation are listed in Table 2 and Table 3. The surface of the sample, produced from the 316L stainless steel, was ground at the work piece speed of v_w = 4 m/min and the depth of cut equal to d_c = 4 μm was applied.

For the 316L stainless steel samples studied in the present work two independent diffraction elastic constants ($s_1(hkl)$ and $\frac{1}{2}s_2(hkl)$) were calculated using Voigt, Reuss and the self-consistent models for the sample surface and interior [11]. The calculations were performed using single crystal elastic constants. The surface of the 316L stainless steel was subjected to grinding treatment in one direction. Consequently, the asymmetry of planar stresses (i.e., $\sigma_{11}^M \neq \sigma_{22}^M$) is expected. The GID-sin$^2\psi$ method was applied using Cu and Fe radiations. In order to calculate the diffraction elastic constants, the self-consistent model was used [11]. The values of σ_{11}^M and σ_{22}^M stress components in function of penetration depth are shown for 316L sample in Fig. 6.

A very good quality of fitting and small uncertainty of the determined stress suggest that the self-consistent approach for sample surface gives the best estimation of residual stresses (quite similar values were obtained with the Reuss model). It should be noted that good continuity of the measured stresses versus depth was obtained using the grazing incidence diffraction with Cu and Fe radiations. The values determined by GID-sin$^2\psi$ method approach those measured by the standard diffraction method (using Mn radiation and 311 reflection). As for the previously studied samples, the stress- free parameter a_0 is almost constant versus penetration depth (a_0 = 3.5951 ± 0.0009 Å, see Fig. 6).

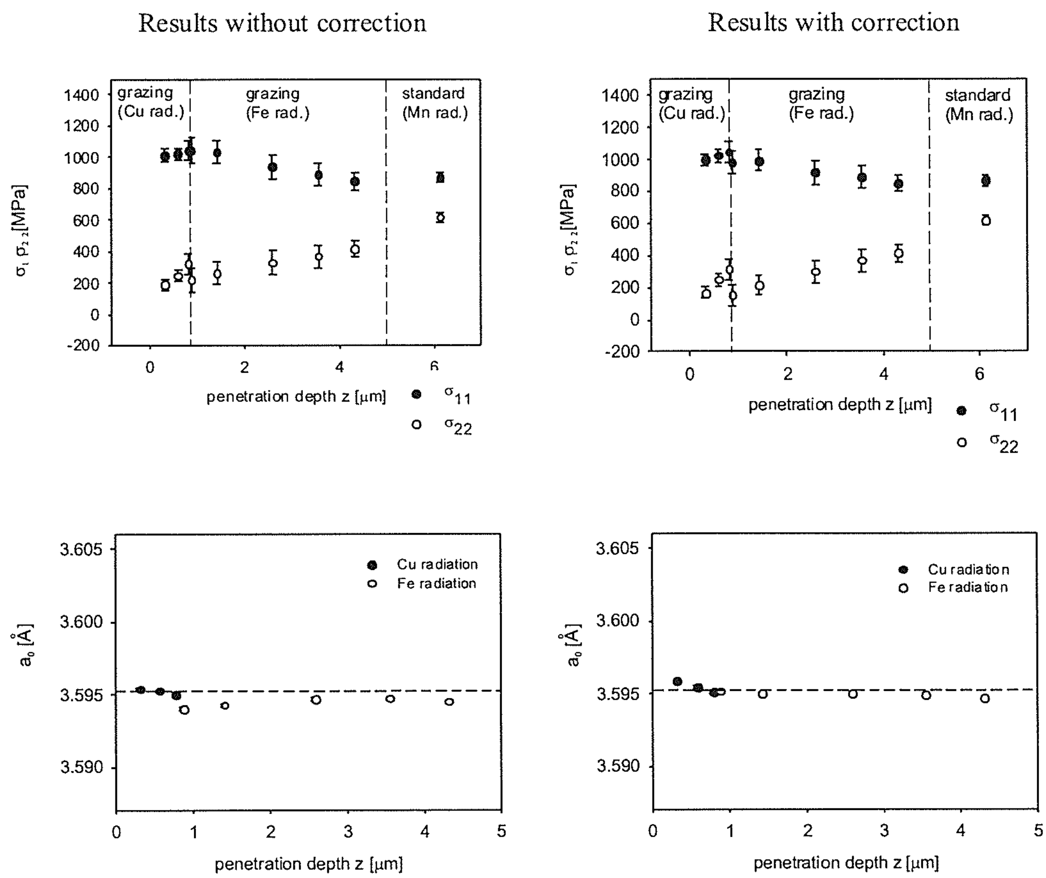

Fig. 6. Stress components (σ_{11}^M and σ_{22}^M) and stress-free equivalent lattice parameter a_0 versus penetration depth z for the 316L ground sample. The self-consistent model for free surface was used to calculate the diffraction elastic constants. On the left – results without corrections, on the right – with corrections

6. Conclusions

Asymmetric geometry is applied in the grazing incidence diffraction method. Penetration depth of radiation is almost constant during experiment and it can be easily changed by an appropriate selection of incidence angle or by using different type of radiation. Variation of penetration depth enables investigation of materials with stress gradient. Classical $\sin^2\psi$ method cannot be applied for this purpose, because penetration depth strongly vary during experiment.

Refraction of electro-magnetic wave (with refraction coefficient smaller than one) causes two effects: it changes the wavelength and the direction of the beam inside a sample. The two effects change the 2θ angle and shift the pick position. This shift has to be considered in data treatment. For small incidence angles ($\alpha 10°$) the corrections are significant and can modify the resulting stress even of 70 MPa. The refraction correction decreases with growing incidence angle. Other corrections (absorption, atomic factor, Lorentz-polarization factor) are less important for final stress values.

Acknowledgements

The work was supported by the Polish Ministry of Science and Higher Education (MNiSW) and by EU Network of Excellence "Complex Metallic Alloys".

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Received: 3 December 2007.