Managing alignment errors is the key to success in obtaining reliable XRD stress data. For the classical $\sin \gamma$ residual stress analysis using relative peak positions only two alignment errors are relevant: specimen displacement and incident beam misalignment [1,2]. For advanced residual stress analysis methods using absolute peak positions two more alignment errors become relevant: zero beam shift and cradle axis misalignment [2]. Hence, for triaxial stress analysis and multiple $\{hkl\}$ stress analysis all four of these alignment errors must be taken into consideration.

When employing focusing optics all the alignment errors must be determined and corrected for. The corrections can be done by tuning the hardware (for large errors) and/or by means of software (for the remaining errors). The full procedure to determine all errors will be discussed in this paper. When employing parallel beam optics, a simplified procedure can be followed since only the zero beam shift needs to be determined. The relevance of each error for focusing optics and parallel beam optics is indicated in Table I.

The complete procedure to obtain accurate absolute peak positions involves measuring peak positions of a calibration/reference powder sample over the full 20 and the full $\psi$ range. The peak shifts with respect to the theoretical peak positions are analyzed with a multivariate linear least squares (multiple linear regression) fitting procedure. This method enables the possibility to also include other sources of peak shift, like: error in stress-free lattice parameter, thermal expansion, error in wavelength, transparency, beam divergence, et cetera. The relevance of these errors will be discussed. The fit parameters obtained -describing the remaining alignment errors- are used for a software correction of the measured peak positions.

The first results obtained with the procedure show an absolute peak position accuracy of $\pm 0.005^\circ$ 20 over the full 20 and the full $\psi$ range with only 3 hours measurement time. The optimization of the measurement strategy and the improvement of the statistical peak position accuracy will be investigated.

Table I: Relevance of various alignment errors for different XRD stress analysis methods for focusing optics (F) and parallel beam optics (P). The classical $\sin \gamma$ methods, omega-stress and psi-stress, are stress methods using relative peak positions. Tri-axial stress analysis and multiple $\{hkl\}$ stress analysis are methods using absolute peak positions.

<table>
<thead>
<tr>
<th>Alignment error</th>
<th>Relative methods</th>
<th>Absolute methods</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Omega</td>
<td>Psi</td>
</tr>
<tr>
<td>Zero beam shift</td>
<td>- , -</td>
<td>$\gamma$, -</td>
</tr>
<tr>
<td>Specimen displacement</td>
<td>F, -</td>
<td>F, -</td>
</tr>
<tr>
<td>Equatorial beam misalignment</td>
<td>F, -</td>
<td>$\gamma$, -</td>
</tr>
<tr>
<td>Axial beam misalignment</td>
<td>- , -</td>
<td>F, -</td>
</tr>
<tr>
<td>Cradle axis misalignment</td>
<td>- , -</td>
<td>$\gamma$, -</td>
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</table>
DIFRACTION ANALYSIS OF IRON MATERIALS AFTER SURFACE MACHINING

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The aim of the contribution is to present the possibilities of X-ray diffraction analysis for characterisation of surface layers after machining by using electro discharge machining (EDM) and electro chemical cutting (ECM), in comparison with "classical" methods like milling and grinding. The intention of the paper is to exemplify X-ray diffraction capability of simultaneous phase, and both the macro- and micro-stress analysis. The research is being carried out on two sets of steel specimens:

- a low-alloy steel (Czech grade 14 220 – 0.15% C, 1.0% Cr, 1.4% Mn) – standard for studying the effect of heat and structure phenomena on the shaped surfaces,
- a tool steel (Czech grade 19 436 – 2.0% C, 12.0% Cr, 0.4% Mn) – typical material for dies, casting moulds, shearing and working tools.

This work was supported by the Grant Agency of the Czech Republic (grant No. 106/03/1039).

IN SITU NEUTRON DIFFRACTION STUDY OF THE LOW CYCLE FATIGUE OF THE \(\alpha-\gamma\) DUPLEX STAINLESS STEEL

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The mechanism of the low cycle fatigue in \(\alpha-\gamma\) duplex stainless steel was examined by the neutron diffraction technique performed in situ upon mechanical exposure consisting of the tension-compression cycles with the strain amplitude of 0.8%. Deformation response of both constituent components, i.e. ferrite and austenite was studied in detail during the first and the last applied cycle. Furthermore, the evolution of the residual strains in both phases after tensile and compressive unloading was monitored during cycling. Information on the evolution of lattice strains and microstrains has been obtained from the diffraction profiles collected during the fatigue experiment. The results show different deformation behavior and hardening of austenite and ferrite. The evolution of internal stresses and residual stresses detected in both phases during individual cycles is strongly affected by initial stresses present in both phases. These thermal stresses are introduced into the individual phases of the duplex steel during cooling from high (homogenization) temperature due to different thermal expansion coefficients of the constituent phases.

NEUTRON DIFFRACTION MEASUREMENTS OF STRAIN/STRESS STATE INDUCED BY A WELD DEPOSITED PASS

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It is well known that in the case of components exposed to a thermomechanical load much attention is paid to investigations of strain/stress state of a basis material in the vicinity of welds where a nuclei of microcracks can arise. Neutron diffraction is a non-destructive method for determination of residual stresses in crystalline materials [1-3]. It can also be used for determination of applied stresses. The corresponding procedure can be employed within the interior of materials and adjacent to surfaces. In fact, neutron diffraction provides the values of elastic strain components paral-
So, the strain in the (hkl) set of planes is given as

\[ \varepsilon_{hkl} = \frac{d_i - d_o}{d_o} \approx -\Delta \theta \cot \theta_o \]

The direction in which strain is measured is along the scattering vector \( Q \) and is perpendicular to the diffracting planes. Stress and elastic strain in solids are 2nd rank tensors and are linked through the solid’s elastic constants. As neutron diffraction can measure the elastic strain within a defined gauge volume in a crystalline solid, it is possible to calculate the stress in that volume provided the materials elastic constants are known. As they are 2nd rank tensors, the full three dimensional elastic strain variation must be known if the stress within a given gauge volume is to be completely described. To measure the full 3D strain tensor requires measurement of the elastic strain along a number of directions. If the principal strain directions within the body are known then a minimum of three orthogonal directions must be determined. In our contribution we present the results of strain measurements two samples of steel plates of 15Ch2MFA. This steel is used for construction of reactor vessels with different weld deposited passes of Inconel 52. The thickness of the plate was 7 mm and the weld passes of the high of 1 mm or 3.5 mm and the width of 5 mm or 10 mm, respectively, were used. The irradiated gauge volume of 2x2x3 mm³ was situated in the middle of the plate, i.e. in the depth of 3.5 mm. Fig. 2 shows the photographs of the individual samples and Fig. 3 shows the coordinate system and corresponding orientation of the plates during the measurements. Figs. 4 and 5 display the results of the experimental measurement of the strain components. The measurements were carried out on the dedicated strain diffractometer SPN-100 [4,5] installed at the
research reactor LVR-15 in Řež [6] when using the neutron wavelength of 1.65 Å. As the neutron diffraction strain measurements need a sufficiently high monochromatic neutron current, it requires test pieces to be transported to the neutron source.

Acknowledgement
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T3 - P55

RESIDUAL STRESS MAPPING IN THE ELECTROLYTE LAYER OF A HIGH-TEMPERATURE SOLID OXIDE FUEL CELL

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A single cell of a high-temperature solid oxide fuel cell stack consists in the case of the flat package concept of five layers stacked to form a sandwich. Gas tightness of the central layer, the electrolyte membrane, dividing the cathode from the anode space of the cell is crucial for the electrochemical function. Gas tightness is influenced by the residual stress state of this 10 µm thin membrane made from yttria-stabilized zirconia. The residual stress distribution of such a membrane has been determined at four processing steps of a cell to study its development during the manufacturing process.

A plate-like half-cell of 5.5 x 5.5 cm² was used as sample. This half-cell consisted of the supporting NiO/ZrO₂-Y₂O₃ anode with 1.8 mm thickness, the anodic functional layer of about 10 µm and the electrolyte, which is in this study the topmost layer (Fig. 1). The sin²ψ method was applied for strain measurement at room temperature. The sample area illuminated through a polycapillary half-lens and a cross-slit collimator with Cu-Ka radiation was about 1 mm in diameter. A pattern of 10x10 measuring points 5 mm apart from each other were located across the cell.

Fig. 1: Manufacturing steps of a solid oxide fuel cell. Processing steps 3-5 (step 4 twice sintered and flattened) were used for stress mapping.
Dis tri butions of re sid ual stresses in the elec trolyte layer are shown in Fig. 2 at four dif fer ent man u fac tur ing steps. In all graphics the width of the col our code is set to 200 MPa; its cen tre is lo cated at the cal cu lated av er age stress. This al lows a di rect com par i son of colours in the graphics. Fig. 2a con firms the zero stress lev el in the as-de po sited state of the only d ried elec trolyte layer. Af ter sin tering at 1400°C/5h/air for achiev ing a tight elec trolyte the re sid ual stress de creases to a level o f -560 MPa. S ince the cell slightly bends dur ing sin tering it must be flat tened by a n ad di tional heat ing step at 1360°C for 1h un der an ex ter nal load of 1 kg. Dur ing that flat ten ing the stress level re mains un changed. Re duc tion of the an ode (re duc tion of NiO t o Ni) leads to a de crease of the com pressive stress within the elec trolyte from -560 MPa to about -518 MPa.

Both stress levels de ter mined at room tem per a ture are suf fi cient to en sure that the thin elec trolyte layer re mains even at the ser vice tem per a ture 800°C of a cell un der com pression. This is im por tant to avoid stress crack for ma tion dur ing ser vice. The ex per i men tal ob ser va tion was con firmed by thermo-elastical mod el ling.

Distributions of residual stresses in the electrolyte layer are shown in Fig. 2 at four different manufacturing steps. In all graphics the width of the colour code is set to 200 MPa; its centre is located at the calculated average stress. This allows a direct comparison of colours in the graphics. Fig. 2a confirms the zero stress level in the as-deposited state of the only dried electrolyte layer. After sintering at 1400°C/5h/air for achieving a tight electrolyte the residual stress decreases to a level of -560 MPa. Since the cell slightly bends during sintering it must be flattened by an additional heating step at 1360°C for 1h under an external load of 1 kg. During that flattening the stress level remains unchanged. Reduction of the anode (reduction of NiO to Ni) leads to a decrease of the compressive stress within the electrolyte from -560 MPa to about -518 MPa.

Both stress levels determined at room temperature are sufficient to ensure that the thin electrolyte layer remains even at the service temperature 800°C of a cell under compression. This is important to avoid stress crack formation during service. The experimental observation was confirmed by thermo-elasitical modelling.
Determination of Lattice Parameters in Real Metal Materials, Characterized by Substructure Nonuniformity and Anisotropy

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Real metal materials show significant substructure inhomogeneity both because of the statistical nature of structure formation processes and due to regular differences in the lattice condition depending on grain orientations. In this connection, determination of lattice parameters by the standard X-ray method involves essential indefiniteness, which needs to be analyzed for the sake of adequate description of material. It is of importance also to consider the question concerning relationship between the technically attainable precision of some separate measurement and the physically reasonable characterization of the sample with inevitably inhomogeneous lattice condition.

Since any metal material in consequence of technological treatment acquires a texture, characterized by definite distributions of crystallographic axes, it was proposed to modify the procedure of diffractometric determination of lattice parameters in such a manner, that measurements of X-ray line profile and texture are to be combined. As a result, instead of a single value of interplanar spacing \(d_{hkl}\) along the normal to the surface, we obtain a multitude of values \(d_{hkl}(\psi, \phi)\), corresponding to all possible orientations \((\psi, \phi)\) of axes \(<hkl>\). These axes belong to grains with different orientations, whereas in each grain there are several axes of the same type, whose number depends on the multiplicity factor \(p(hkl)\).

A set of interplanar spacings \(d_{hkl}\) along axes \(<hkl>\) of grains with the same orientation gives a tensor description of lattice condition in these grains. Since in any massive polycrystal some anisotropic elastic microstrains always are acting, its lattice parameters can not be correctly determined on the basis of a single measurement, characterizing the interplanar spacing of the sample only by an unreal non-physical supposition of the isotropic homogeneous lattice condition. When constructing the distribution of interplanar spacing \(d_{hkl}\) in the stereographic projection of the sample, we are passing to its multi-dimensional description. A practical procedure to construct these distributions was elaborated, including correction of measured values for defocalization effects and calculation of the physical profile of X-ray line [1].

Elastic microstresses, acting in several neighbouring grains of the polycrystal, by definition are mutually equilibrated, so that within the region, irradiated by X-ray diffractometric study, there are grains, subgrains or blocks, showing microstrains of equal absolute values by opposite signs. As a result, the distribution of measured interplanar spacings \(d_{hkl}\) versus integral intensity of the X-ray line \((hkl)\), registered as reflection from crystallographic planes \(<hkl>\) with corresponding orientations, proves to be symmetric about some average level [2]. This level gives the interplanar spacing for the case of absence of elastic microstrains, whereas other factors, influencing lattice parameters, correspond to the most probable condition.

The above approach is illustrated by measurements of the lattice parameter “c” for \(\alpha\)-Zr phase of the Zr-2.5%Nb alloy. In Fig.1 diagrams of Peak Position (0004) versus Pole Density in PF(0001), proportional to Integral Intensity of the X-ray line (0004), are presented both for cold-rolled and recrystallized sheets. Each point in diagrams corresponds to the definite direction \((\psi, \phi)\) of the basal axis of HCP lattice in the stereographic projection of the sample. It is seen, that annealing of the rolled sheets results in an increase of the lattice parameter “c” by additional scattering of the distribution, which at the same time becomes more symmetric and regular. In Fig.2 for the same samples distributions of volume fractions of grains with different values of the lattice parameter “c” are shown. Distributions were calculated using Pole Figure of Peak Position (0004) and Texture Pole Figure (0001), so that the lattice condition in the sample is described in the fullest manner.


NEUTRON DIFFRACTION RESIDUAL STRESS ANALYSIS OF COMPOSITE TUBE FROM AUSTENITIC STAINLESS STEEL WITH WELDED FERRITIC STEEL CLADDING

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Compared to carbon and alloy steels, all corrosion resistant alloys are expensive. In many cases, corrosion resistance is required only on the surface of the material and carbon or alloy steel can be clad with a more corrosion resistant alloy. Cladding can save up to 80% of the cost of using solid alloy. Cladding of carbon or low alloy steel can be accomplished in several ways including roll bonding, explosive bonding, weld overlaying and “wallpapering”. Clad materials are widely used in the chemical process, offshore oil production, oil refining and electric power generation industries. Weld overlaying is commonly used to clad the surfaces of fabricated steel structures.

Welded ferritic steel cladding on austenitic stainless steel tube is used also to suppress tensile stresses or to build compressive stresses on its outer surface and as a result, to reduce stress corrosion. An analysis of residual stresses through the ferritic cladding into the austenitic parent material can be helpful for the optimization of the corresponding welding technique.

We have used neutron diffraction for 3D-residual stress mapping of such composite tube in which 22 mm thick multilayer spiral weld overlay from ferritic steel was clad on 13 mm thick austenitic stainless steel pipe. For a small (11 mm thick along the hoop direction and 30 mm thick along the axial direction) specimen cut from the composite tube, strain measurements have been made at three European neutron sources: 1) TKSN-400 triple-axis and 2) High Resolution Fourier Diffractometer (IBR-2 pulsed reactor in Dubna), and 3) ENGIN instrument (ISIS pulsed source in RAL). To compare as-delivered austenite and ferrite steels with the same after weld clad process the auxiliary in-situ stress-rig neutron diffraction experiment was carried out on SPN-100 to study mechanical characterizations of samples cut from the inner (austenite) and outer (ferrite) layers of the composite tube, respectively.

The results of stress calculations from the experimental data are compared with the data obtained by the X-ray diffraction method, the destructive turning out technique (TOT) and the theoretical predictions using the finite element method (FEM). Stress differences in two mutually perpendicular directions were in qualitative accordance for all measurements, while the absolute stress values obtained from neutron data differed strongly from the TOT and FEM results. At least, two reasons one can attract for explanation: uncontrollable influence of 2nd kind microstresses on the result of neutron measurements; incorrect determination of $a_0$ (or $d_0$) values for both layers of the tube. We plan to resolve this disagreement by performing neutron diffraction experiments with anew prepared samples together with a series of strain-free reference samples prepared by means of the special comb-technology.

RESIDUAL STRESS MAPPING ON THE LASER MODIFIED SURFACE OF STEEL BY X-RAY DIFFRACTION

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X-ray diffraction stress analysis has been applied to two types of steel (to a carbon steel and a corrosion-proof steel) heat treated by a laser beam. Strain of the interplanar distances of the lattice family of (220) produced in the surface layers of the investigated materials by radiation of a continuous CO\textsubscript{2} – laser has been determined using the AXS Bruker diffractometer D8 equipped with the great area position sensitive detector HI-STAR. From this lattice strain, the distribution of the residual macrostress both on the modified surface and bellow the surface has been calculated. The information obtained in this way can be to advantage used e.g. by optimizing parameters of the laser heating to increase locally the microhardness (to decrease abradability) of the surface of machine products.
Parameters of the treatment applied

A continuous 2500 W CO₂ laser has been used with a 6 mm diameter spot of the defocused beam on the surface of treated material. Samples of two marks of steel (ČSN 12050 and ČSN 17027) were processed, being moved with the velocity of 700 mm/min with respect to the beam. The surface of the samples has been heat-treated in this way along one track, two parallel tracks or three partially overlapped tracks (Fig. 1, 2).

The results obtained are further illustrated by the stress distribution on the surface of the ČSN 12050 carbon steel; the samples were moved with the velocity of 700 mm/min with respect to the laser beam.

a) Measurement in the surface heat treated by one passage of the laser beam

Compressive stresses appear in the laser hardened region while virtually no stresses or small tensile stresses are found outside the laser track.

b) Measurement in the surface heat treated by two passages of the laser beam

From the residual stress distribution on the surface hardened by two passages of the laser beam it is obvious that the stress along the first (left-side) laser track relaxed (diminished by about 100 MPa) due to the second passage of the laser beam (along the right-side track).

c) Measurement in the surface heat-treated by three passages of the laser beam
The effect of the relaxation of the macrostresses is even more distinct on the surface hardened by three partially overlapped laser beam passages than in the case of two laser beam passages.

From the depth profile of the stress it is evident that the applied laser treatment influenced the material of the samples up to the distance of some 0.8 mm from the surface. The difference of the stress distribution in the two marks of steel (ČSN 12050 and ČSN 17027) is only quantitative, being a consequence of the different real structure of these two steels.

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In the case of very precious and rare art manufactures, usually having a complex geometry, the conventional X-Ray Diffraction technique can be very difficult or even impossible to be applied. However by means of microbeam, small area can be analysed and the microstructure of differently treated surfaces can be discussed.

Recently, two-dimensional (2D) detectors have been developed for laboratory X-Ray diffractometers. Thus, nowadays it is possible to collect with high speed and high quality diffraction data of a significant part of the diffraction cones and the structure and microstructure of small surface area can be evaluated. Moreover, by the analysis of the diffraction cones distortion, the residual and/or applied stresses can be calculated [1].

In the present work the structure, microstructure, and residual stress present on gold artfacts due to the manufacturing are discussed mainly on the basis of bidimensional

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**RESIDUAL STRESS MEASUREMENT OF GOLD ARTFACTS BY DeBYE RING ANALYSIS**

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²SPIN Srl S. Casciano V.P. (Fi), Italy

In the case of very precious and rare art manufactures, usually having a complex geometry, the conventional X-Ray Diffraction technique can be very difficult or even impossible to be applied. However by means of microbeam, small area can be analysed and the microstructure of differently treated surfaces can be discussed.

Recently, two-dimensional (2D) detectors have been developed for laboratory X-Ray diffractometers. Thus, nowadays it is possible to collect with high speed and high quality diffraction data of a significant part of the diffraction cones and the structure and microstructure of small surface area can be evaluated. Moreover, by the analysis of the diffraction cones distortion, the residual and/or applied stresses can be calculated [1].

In the present work the structure, microstructure, and residual stress present on gold artfacts due to the manufacturing are discussed mainly on the basis of bidimensional
X-Ray microdiffraction (μXRD). In particular, by the analysis of the Debye rings deformation, the map of the residual stress field of sample was calculated and discussed in terms of manufacturing.

Other techniques (optical and electron microscopies, EDX, microXRF and SIMS) were applied to characterize the samples and used in the discussion.

The potentialities of microbeam techniques for jewellery applications will also be shown.


SIMULTANEOUS EVALUATION OF ELASTIC AND THERMAL STRAINS IN THERMALLY-CYCLEDR THIN FILMS

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Residual strains/stresses in thin films or in sublayers of sandwich structures represent an important physical parameter decisively influencing the structural integrity and the performance of devices. When studying the mechanical behaviour of the thin films, one of the main difficulties is the identification and quantification of specific phenomena contributing to the strain/stress state. In the case of thermal stresses, the actual magnitude is usually calculated on the basis of thermal expansion coefficients (TECs) obtained from the literature [1-4].

The main goal of this contribution is to discuss a new methodology for the simultaneous evaluation of in-plane elastic and thermal strains in thin films from elevated-temperature X-ray diffraction measurements.

The diffraction data are routinely used to evaluate in-plane elastic strains and subsequently also residual stresses in thin films. Using an appropriate experimental procedure and data treatment, it is possible to evaluate also TECs of the film and the substrate. Thus the actual magnitude of the in-plane thermal strains can be calculated using the experimental TECs for every measurement temperature. In the case of structures with residual stress-temperature hysteresis, the unstressed lattice parameter of the film can be refined without the knowledge of x-ray elastic constants [5].

The proposed methodology is demonstrated on various thin film/substrate systems (e.g. Al/Si(100), Cu/Si(100), Cu/SiO₂(110), Mg-implanted GaN/Al₂O₃(0001)).

X-RAY DIFFRACTION RESEARCHES OF INTERNAL STRESSES AT THE NI-BASED ALLOY AT THE THERMAL CYCLING

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The high specific heatproof of Ni-based alloy (1,3% W, 3,2% Mo, 5,6% Al, 4,5% Ti, 12-15% Co) is conditioned by the promoted maintenance of the intermetallic \( \gamma' \)-phase \( \text{Ni}_3(\text{Al,Ti}) \). Small accordance of parameters of crystalline lattices of \( \gamma \) and \( \gamma' \)-phases creates pre-conditions for the homogeneous origin of excretions with low surface energy and low level of coherent stresses. High concentration of Co at minimum maintenance of W (1,2%) is promotes in creation at the alloy of the intermetallic \( \gamma' \)-phase \( \text{Ni}_3(\text{Al,Ti}) \), permeable for dislocations.

The alloy after casting contains a big amount of the macro and micropores, that in the turn accelerates development of microcracks, suddenly reduces plasticity and heatproof of the alloy at the high temperature loading. In [G.Ya.Bazelyuk, B.Ya.Vinokurov, Ye.A.Kalmykov. Sposib termichnoji obrobky vyrobit iz zarostijkh splaviv. Patent No. 20563A Ukraine. (Russian)] it is shown that thermal cycling in the soft mode results in “healing” (narrowing) of casting mykropores under action of the internal stresses arising up because of different coefficients of thermal expansion of \( \gamma \) and \( \gamma' \)-phases. There was of interest the research of level of internal stresses arising up after thermal cycles and their influence on the processes of extraction of \( \gamma' \)-phase.

It is shown that the level of thermal squeezing stresses at the room temperature grows with the increase of number of thermal cycles in \( \gamma \) and \( \gamma' \)-phases is increases.

X-ray diffraction researches of the samples of our alloy was found the primary orientation of the structure after thermal cycling treatment, that was confirmed by means of electron microskopy. The lengthening of the coherent excretions in direction of \( \{100\} \) can be explained by influence of internal stresses on the excretion of \( \gamma' \)-phase during the process of thermal cycle (\( \tau = 2-4 \) hours, at \( T=1173 \) K).

ACCOUNTING FOR SECONDARY EXTINCTION IN THICKNESS MEASUREMENT OF THIN FILMS BY X-RAY ABSORPTION

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Due to the texture of either the polycrystalline film or the substrate underlying it, the measurement of film-thickness by x-ray diffraction or x-ray absorption could be compromised if measured intensities are used for methods based on kinematical theory. This theory assumes that there is no mutual shadowing between crystallites reflecting in each sample direction and the attenuation of incident intensity is caused by the ordinary absorption only. Due to parallelism of crystallites in given direction(s) of a textured sample, the incident intensity is additionally attenuated by x-ray diffraction. As a result of diffraction the crystallites lying beneath receive less incident intensity compared to that they would receive in absence of any shadowing and thus contribute to the diffracted intensity less that causes secondary extinction (SE). It should be noted that SE enhances accordingly the effective absorption in the range of reflection. Since the SE is inherent diffraction property of textures, considering it is the only way to correctly solve the problem for film-thickness measurement. The aim of the present investigation is to develop an innovative method for thickness measurement based on absorption by the film of x-rays diffracted by a textured substrate under-lying it. After the problem is defined, the way will be schematically described how the measured intensity affected by extinction should be converted into intensity corresponding to kinematical approximation.

Consider a film of phase \( \beta \) mounted on a textured flat-plate substrate of phase \( \alpha \) appearing infinitely thick to x-rays. Especially, the \( \alpha \)-phase may be amorphous, polycrystalline or single-crystalline. To measure the film thickness one needs kinematical intensities \( I_{\text{kin},\beta} \) and \( I_{\text{kin},\alpha} \), corresponding, respectively, to a reflection from a clean substrate, and from the same substrate with a film of thickness \( t \) mounted on it, i.e.

\[
I_{\text{kin},\beta} = I_{\text{kin},\alpha} \exp(-2\mu_a t / \sin \theta_\beta )
\]

where \( \mu_a \) is the linear absorption coefficient of the \( \alpha \)-phase and \( \theta_\beta \) is the Bragg angle of the respective substrate reflection.

Accounting for SE, the kinematical intensity is defined by

\[
I_n = y I_{\text{kin}}
\]
where \( I_0 \) is the measured intensity affected by extinction, and \( y \) is the extinction factor which is always less than unity (cf. Sabine [1]). If the extinction factor is equal to unity, which corresponds to extinction-free conditions, the measured intensity is then equal to the kinematical one, i.e. \( I_0 = I_{\text{kin}} \). For the case of pure SE Chandrasekhar [2] gave for \( y \):

\[
y = \frac{\mu}{\mu_s} \quad (3)
\]

where \( \mu \) is an effective absorption coefficient. In the symmetrical Bragg geometry, as a first order approximation for the SE correction, the effective absorption coefficient should be used [3]:

\[
\mu_e = \mu + gQ(p_2 / p_1^2) = \mu + \varepsilon \quad (4)
\]

where \( g \) is the SE coefficient related to the texture and microstructure of the sample, \( Q \) is the reflectivity per unit crystal volume, and \( \varepsilon \) is the SE itself. The symbol \( p_2 \) denotes polarization factors for monochromatized x-ray beam

\[
p_s = \left[ 1 + \cos^2(2\theta_0) \right] \left[ 1 + \cos^2(2\theta_0) \right] \quad (5)
\]

where \( n=1,2,\ldots \), and \( \theta_0 \) is the Bragg angle of the monochromator.

The kinematical intensity defined above can be calculated by a method developed earlier for measuring the parameters of the texture and secondary extinction [4-6]. Taking advantage of a reflection pair characterized with the same pole density, this method is responsive to account simultaneously for the anisotropy effects of both the texture and the secondary extinction corresponding to the same crystal direction.

The model samples of this investigation are textured Ni foils of a few microns thickness mounted on textured Ag substrate representing o-preferred orientation. For the purpose of pole density and SE determination, the XRD measurements were carried out with conventional diffractometer using Bragg-Brentano focussing geometry, while the XRD measurements in another investigation were carried out with diffractometer using asymmetric diffraction geometry, which accounts for the texture effect only [7].


**T3 - P63**

**INFLUENCE OF EXTINCTION PHENOMENON ON DETERMINATION OF THE ORIENTATION DISTRIBUTION FUNCTION**

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As has already been recognized [1,2,3], pole figures obtained from textured materials by X-ray diffraction are affected by the extinction phenomenon. In the papers [2,3] concerning textured films, only secondary extinction has been taken into account on the basis that the films are of fine lamellar structure. The phenomenon of the primary extinction depends on the extinction length, which is about 1 \( \mu \)m for the Bragg case geometry of diffraction for most materials. Since a coherent crystal domain of the size of one third of the extinction length already shows the primary extinction, a grain of such size, from a crystal could exhibit the primary extinction and not only the secondary extinction takes place. Especially, for the recovered and recrystallized materials, where large perfect crystallites exist, also strong primary extinction is present. Taking advantage that the primary extinction affects strongly the low index reflections, and that the secondary extinction can be considered as the additional term \( gQ\varepsilon \varepsilon C_2/C_1^2 \) in the coefficient of absorption [1,4], where \( g \) is the secondary extinction coefficient, related to the crystallites angular deviation from the mean direction, \( \varepsilon \) is the primary extinction coefficient, \( Q \) is the kinematical integrated reflectivity per unit volume, and \( C_1 \) and \( C_2 \) are the polarization factors, the necessity of pole density correction was demonstrated for the measurements with low index reflections, as done commonly [1].

In the present work, the appropriate conditions were found using two X-ray wavelengths (Cu and Mo radiations and both, low and high index reflections) to differentiate the primary and secondary extinction contributions and to correct pole densities. An aluminum sample after rolling and annealed at 360 °C was used as example to investigate the influence of the extinction phenomenon on the orientation distribution function (ODF).
Applying measurements with Cu radiation to determine the ODF for low index pole figures for the reflections 111, 200 and 220 on the one hand, and high index pole figures for the reflections 222, 400 and 420 on the other, using the PopLA package, the resulting ODFs were evaluated and compared (see Fig. 1 and Fig. 2, respectively).

As can be seen, the ODF obtained with low index pole figures shows systematically lower pole density values than that obtained with high index ones as expected due to the primary extinction contribution. ODFs obtained from low as well as from high index pole figures are also affected by the secondary extinction. Extinction is also found to be anisotropic due to the primary extinction coming from recrystallized non equiaxial crystallites.

As can be expected, the corrections of pole figures due to the extinction phenomenon [1] will give new changes in the ODF and the values of pole densities will increase more essentially as observed in Fig. 2, where mainly the secondary extinction is present.


Fig. 1 ODF obtained with low index pole figures.

Fig. 2. ODF obtained with high index pole figures.

PRELIMINARY RESULTS ON A SIMPLE TECHNIQUE TO CORRECT THE XRD INTENSITIES FROM THE PREFERRED ORIENTATION EFFECTS

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Introduction. X-ray powder diffraction is a non-destructive technique for characterising crystalline materials. The method has been widely adopted for qualitative and quantitative phase analysis, where precise and reproducible intensity measurements are required. Successful application of quantitative methods requires careful, proper sample preparation in order to obtain a specimen that presents a very large number of randomly oriented, uniformly sized crystallites to the x-ray beam. See the excellent and concise article by Bish and Reynolds in Bish and Post [1] for a summary of most of what you need to know for all-purpose XRD sample preparation. Unfortunately, absolute random particle orientation can only exist if the shape of the particles is spherical. In real samples random grain morphologies are rarely the norm. Instead, almost all materials exhibit some degree of preferred orientation, and the measured diffraction intensities will therefore be incorrect [2]. Many methods have been proposed to overcome this thorny problem; some via theoretical mathematical approaches, others by randomising the samples [3], but no method has yet to resolve the problem. Moreover, methods calling for prolonged comminution cannot be considered valid alternatives. In fact, several authors have shown that prolonged grinding causes many materials and minerals to undergo radical changes in their physical and chemical characteristics [4]. It is in this perspective that the present paper describes a method which enables minimising preferred orientation effects in diffraction intensity measurements on minerals with easy cleavage fracture by
using a conventional diffractometer with a modified sample-holder.

**Experimental trials.-Theory** In a conventional X-ray diffractometer system with Bragg-Brentano parafocusing geometry, Bragg reflection occurs mostly from particles whose crystallographic planes are approximately parallel to the sample-holder’s external surface. For this reason, a homogeneous, randomly oriented powder must be used. If we rotate the sample-holder by a few degrees around the diffractometer’s horizontal axis, as shown in Fig. 1b (such rotation was obtained by modifying the sample-holder as illustrated in figure 1a), the geometry of the apparatus is consequently modified, together with the focusing conditions. Thus, Bragg reflection is broadened and the intensity decreased, but the contribution to Bragg reflection originates from planes that are not parallel to the sample’s external surface. The intensity measurements were made at various positions, rotating the sample holder in a counter-clockwise direction until the ratio between full width at half maximum (°2θ) (FWHM) and the integral breadth of each intensity measurement approximated unity. In order to check the effectiveness of the proposed method in reducing the effects of preferred orientation, we carried out a series of tests on calcite and quartz samples. The easy cleavage of the calcite phase can cause preferred orientation in powder diffraction specimens when the powder is compacted during X-ray test sample preparation. On the other hand, quartz has a minimal degree of preferred orientation, so it can be used for comparisons between a mineral with hard effect (calcite) and a free mineral (quartz) by orientation. The intensity measurements were performed with a Philips commercial diffractometer, the selected peak (104) for calcite and (101) for quartz were scanned by the 0/20 method with a step of 0.005°, measuring for 2 seconds per step. A precise determination of the net area (°counts), FWHM (°2Theta) and integral breadth, was made by PC APD 3.6 software for automated powder diffraction analysis.

**Results and Discussion.-** Five independent calcite and quartz samples were prepared for testing (the powder was packed into the sample-holder each time). Figure 2a-b summarise and show the results obtained from the tests performed. The various angular positions of the sample-holder during the X-ray measurements are indicated as a, a’, etc., starting at the “a” position, where the plane of the powder was tangent to the focusing circle, up to the end point when the ratio FWHM / Integral breadth approached unity. The angular positions a’, a”, ... were not at

![Figure 1a](image1a.png)

![Figure 1b](image1b.png)

![Figure 2a-b](image2a-b.png)
the same positions in each of the five tests of the calcite and quartz samples examined; only the $a$ position remained constant. Figures 2a and 2b report the regression lines obtained for the calcite and quartz phases, respectively, by plotting the net-area values versus the FWHM / Integral breadth ratios. Only the 3 samples are included for the sake of clarity, though the trend is the same for all the samples. The plots in figure 2a (calcite) and 2b (quartz) show a different trend: while the calcite lines converge in a area centring about an approximate value of 800 (Net area (°counts)) and a FWHM/Integral breadth ratio of 1.00 (corresponding to a calcite diffraction intensity free of preferred orientation effects), the quartz lines are parallel, revealing “no immediate convergence”. The standard deviation is about 19% for the initial calcite measurements and 2.2% for the final ones, while for initial quartz position is 2.85%. Therefore, the proposed method appears to effectively correct the diffraction intensity for preferred orientation effects. It is up to the analyst to decide whether he should also utilise alternative techniques at the same time.

CORRELATION BETWEEN TEXTURE AND TABLETTING PROPERTIES OF SOME PHARMACEUTICAL TABLETS

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Pharmaceutical tablets are usually made by compressing crystalline powder. The resulted tablet is a polycrystalline object and its properties may depend on the orientation of the crystallites. In the present study an x-ray diffraction method was used to characterize the texture of various pharmaceutical one-component tablets. The results of the texture measurements were then linked to the tabletting properties of the materials from which the studied tablets were compressed.

The studied materials were lactose, mannitol, sodium chloride, ibuprofen, dicalcium phosphate and aspirin. The tablets were compressed using a tabletting simulator or a hydraulic press. Three different pressures were used in order to study the effect of the compression force to the texture. The used tablet moulds were flat-faced with a diameter of 8 or 13 millimeters. The texture measurements were done with X'Pert Pro MPD diffractometer equipped with ATC-3 texture goniometer.

Most of the studied materials texturized in compression. The extent of texture appeared to be dependent on the compaction behavior of the tablet material. If the compaction behavior was elastic no clear texture or no texture at all was observed. The plastic and brittle materials had clear texture but the clearest and strongest texture was observed from materials whose compaction behavior were proper combination of plastic and brittle.

The compaction pressure had no effect on the direction of texture. Furthermore, the extent of texture was changed only a little when the pressure was increased. The particle size was found to have major effect on the formation and quality of texture.

It has been suggested that the texture of pharmaceutical tablets may affect the dissolution properties, breaking strengths and hardness of tablets, for example. Therefore, texture analysis of pharmaceutical substances could give valuable information about their properties. Based on this study, the texture analysis seems to offer new and powerful nondestructive research tool for pharmaceutical physics.

CRYSTALLOGRAPHIC TEXTURES IN NACRE OF SOME RECENT AND FOSSIL MOLLUSCS

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In many molluscs, nacre is the innermost lining of their shells, often displaying a characteristic play of colors. We have examined nacre from a number of recent and fossil shells using the technique of oblique-texture X-ray diffraction [1], aiming at identifying and/or quantifying preferred orientation of aragonite platelets. Among the fossil shells (as old as Mesozoic), we included only those in which aragonite has not recrystallized to calcite. The following techniques were used: X-ray diffraction, SEM, TEM.

The chief types of aragonite textures had been established earlier [2]; the purpose of this research was to make use of crystallographic textures and microstructures of molluscan shells for application in phylogenies. However, here we are merely pointing out the diffraction effects, whereas the systematic and phylogenetic importance of this study is being dealt with elsewhere [3].

In the material studied, we encountered two basic patterns, (i) preferred orientation of aragonite with [001]* as axis of the texture and directions [100]* and [010]* random and (ii) parallel orientation of all crystallographic axes of aragonite.

The first type was found in the gastropods (living and fossil members of Vetigastropoda) and fossil cephalopods (Mesozoic members of Ammonoidea). The texture differs in the degree of its perfection and in the orientation the axis of the texture with respect to the surface of the shell. There are shells whose axis of texture is perpendicular to the surface, but in most of them the platelets are inclined in a roof-tile fashion, with the axis of texture deviating by about 4 degrees from the normal to the surface. There is evidence that this crystallographic pattern has developed at least 200 Ma ago.

The second type was found in recent cephalopods (Nautilus). When mounted on a precession camera, this nacre gives quasi-single-crystal X-ray patterns.
The SEM photos indicate a visually appealing parallel alignment of the platelets, but do not betray whether the material belongs to one type of texture or the other. In order to examine whether the platelets themselves are twinned (aragonite is well known for its twinning on (110)), we used the TEM technique on one specimen. So far, only untwinned single-crystal platelets have been observed.

The project is in progress, it will include more mollusc species and shells of different geological age.

**T3 - P68**

**THE EFFECT OF DOPING ON THE STRUCTURAL MICROSTRUCTURAL AND THERMAL EXPANSION OF GALIUM NITRIDE (GaN)**


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The crystal structure, microstructure and thermal expansion of pure, Al and Gd doped Gallium Nitride were carried out, in order to locate the atomic positions of the doping atoms, to see the effect of the dopant elements on the crystallite size, strain and thermal expansion of GaN lattice. The solid state reaction method was used for the doping process. PHILIPS X’pert MPD X-ray diffractometer was used with CuKα radiation and 0.02° 20 step for data collection. The pure and Al doped GaN diffraction data were collected at (-200, -100, 25, 100 and 200°C). Rietveld and the two step method were used for structure analysis. The two step method was adequate in analyzing the present GaN compound; this is due mainly to the inherited high degree of lattice defects in this sample. The location of the Al atoms were found to be accommodated in positions different from those of the Gd atoms in the unit cell. The obtained crystallite size and microstrains were different in the three samples (pure Al and Gd doped GaN). They were also different along the a and c-axis of the hexagonal cell.

The crystallite size and microstrain results are anisotropic and different in value among the three different sample (pure, Al and Gd doped GaN). The thermal expansion for the different ranges of temperature were also anisotropic and different along the different axes, a and c of the GaN cell. In general the thermal expansion coefficient were found to be very small over the temperature range (-200 to 200°C) which will allow these materials to be used in optoelectronic devices very safely in this range of temperature.

In this work the crystallite, microstrains and lattice thermal expansion are correlated with the structural changes produced by doping.

**T3 - P69**

**ORIENTATION DISTRIBUTION FUNCTION ODF DRAW INTO THE STANDARD PROJECTION (001)**

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A new program ODFSP2.EXE has been set up to draw the Orientation Distribution Function (ODF) into the Standard projection (001).

Preferred orientation of crystallite in the polycrystalline samples is marked as its texture. It can be described by means of the ODF expressing the crystallite quantity in the volume unit of the sample with a specific orientation of crystallographic axes towards the axes defined for the sample.

The orientation of the crystallographic axes of crystallite in the sample towards the axes of the sample is defined by three Euler’s angles F2, F, F1. To define the crystallite orientation better we introduce the concept of so called Ideal Orientations of the crystallite: (HKL)/uvw/.

(HKL) : crystallographic planes parallel with the plane of the rolling, /uvw/ : crystallographic direction in this plane and parallel with the direction of rolling.

Three definite Euler’s Angles F2,F,F1 correspond to any ideal orientation where the plane (HKL) is given by the angles F2, F and the direction /uvw/ is given by the third angle F1 (for given F2, F).

Any crystallite orientation is in the sample given by three Euler’s angles: F1,F,F2.

The ratio of this orientation as present in the sample is given by the ODF value that shows the ODF as a function of Euler’s Angles.

$$\text{ODF} = f(F2,F,F1) = f'(HKL)/uvw/$$

This function can be visualized in so called Euler’s Space as defined by three rectangular axes F2, F, F1.
The texture can be shown as a diagram (and printed on paper) in two following ways:

1) By a Section in the Euler’s space namely for constant angles F2 or F1. The positions of the Ideal Orientations can be drawn on the respective angles F2, F1, F into the section. The rate of ODF contents in the sample can be read out from the position of the Ideal Orientation and the corresponding ODF values.

By drawing the ODF into the Standard Projection (001).

Any point in the Euler’s space with F2, F, F1 coordinates defines the (HKL) and /uvw/ poles in Standard Projection. The relative ODF value related to these poles can be visualised using a colour scale. The ODF is drawn into the Standard Projection by the program: ODFSpx.EXE that was compile on the Faculty of Nucl. Sci. and Physical Engineering CTU in Prague.

The poles of the plane (HKL) are drawn in the IVth quadrant in rhombic form, the poles of of the direction /uvw/ are drawn in the IInd and IIIrd quadrants in square form.

The drawn poles (HKL) and /uvw/ are set on the plane of the standard projection basic circle specific domains to which it is possible to coordinate numerical values of the index (HKL) or /uvw/ respectively, with a network of standard projection pole.

The ODFSPx.EXE program allows also drawing the poles of the discrete ideal orientations (HKL)/uvw/ to the polar domain of the specimen.

The calculation of ODF values is made by the program’s files POPLA. [1]

http://www.lanl.gov/orgs/mst/cms/poplalapp.html


Fig. 1 [2]

Fig. 2b

Texture of an axial planar section throught a bar. pressed from alloy: AA 6262 (AlMgSiPb). Systems of two fibrous textures are visible:

a) (HKL)[111] where the crystallographic direction [111] are parallel with the axis of the bar; the crystallographic planes (HKL) are the planes of the zone with zonal axis [111].

b) (010)[001] where the crystallographic directions [001] are parallel with the axis of the bar and the planes (010) are zonal planes with the axis [001].
T3 - P70

RESIDUAL STRESSES IN ROLLED STEEL SHEETS

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Residual stresses of annealed and cold rolled sheets were determined by the \( \sin \Psi \) method, using the Mo, Co and Cr X-ray tubes. The anisotropy of the steel sheets was determined by textural data measurements and analyses. Pole figures (on the crystallography planes \((732), (103)\) and \((112)\)) were measured to determine the optimal measuring range of the residual stresses. The residual stresses were measured in three basic directions in the rolling, \( 45^\circ \) degree, and transversal direction. Rayflex program calculated the tensor of the residual stresses. The results of the estimated residual stresses in materials rolled on 5 stand mill, after continuous annealing and after second reduction on the 2 stand mill are presented in this contribution and will be used for optimization of the production process and for products quality improvement.

T3 - P71

DEFORMATION AND RECRYSTALLISATION TEXTURE OF TIN PLATES

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Double reduction is the final stage in tin steel sheets production and introduces the deformation texture into material. Textural parameters are very important for material further treatment. The annealing mode, in batch furnaces or continuous and the subsequent double reduction parameters are the main factors influencing the crystallographic texture. The X-ray textural goniometer and Mo and Co X-ray tubes were used for textural measurements. The texture of rolled steel sheets is characterized by \((\alpha, \gamma, \eta)\) textural fibers.

The orientation distribution function - ODF was calculated from pole figures data measured on \((110), (200), (112)\) and \((103)\) planes, using the WIMV method. The coefficient of normal anisotropy \( r \) was calculated by means of popLA program. Real values measured on samples from production are presented in this contribution. Tin sheets annealing mode can be easily distinguished from the character of \( \alpha \) and \( \gamma \) fibers. Influence of the annealing mode and the effect of double reduction rate on the texture of tin plates were found. The optimal and limited double reduction rates on deformationaly advantageous crystallography texture of tin plates were estimated.

T3 - P72

RIETVELD REFINEMENT OF WHOLE \( \Omega-2\Theta \) PATTERN FOR TEXTURED AND STRESSED THIN FILMS

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Thin films structure and microstructure parameters predetermine their material properties (e.g. microhardness, chemical resistance, etc.). Due to the dependence of surface energy upon the crystallographic orientation of the surface planes, thin films tend to grow with more or less strong preferred orientation (texture). The texture complicates the determination of microstructural parameters by means of x-ray diffraction due to a limited extent of grain orientations and, thus, a limited angular range, in which a particular reflection \( hkl \) is observed.

Due to the symmetry of the deposition process (the only principal axis is the growth direction, i.e. the surface normal), we may anticipate the axial symmetry of both structural and mechanical properties of thin films deposited on polycrystalline or amorphous substrates. The so-called fiber texture is introduced [1].

In Rietveld refinement, all the structural parameters must be expressed in terms of either theoretical or empirical functions of a limited number of parameters. The same holds for experimental conditions (instrumental broadening and aperture function). We use the following functional expressions for particular effects on the diffraction:

- **Profile function**: The Pearson VII profile is expected. The width in \( q \)-space is expressed as \( (\Delta q)^2 = G^2 + (M |q|)^2 \),
Figure 1: Measured and simulated omega-20 maps of diffracted pattern of TiB$_2$ deposited on steel substrate. The texture is very close to 001 normal to surface and angular FWHM is about 2.5 degrees. The scale of grey is logarithmic. Upper windows show detailed maps of several reflections.

according to the anticipation that the broadening is caused by the limited grain size and microstrain. Both $G$ and $M$ are angular dependent $G = G_{\theta_0} \cos^2 \omega + G_{\theta_1} \sin^2 \omega$ (similarly for $M$) to account for a columnar structure and different microstrain in normal and lateral direction.

Texture function: We expect the general HKL crystallographic direction to have maximum in normal direction and to have simple $\omega$ dependence defined by two width/shape parameters $X$ and $n$:

$$ P_{\text{hk}} = A \exp[-(1-\cos \omega)^n / X^n] $$

$A$ being the normalization constant. Any other hkl reflection pole function can then be derived from (1) as follows:

$$ P_{\text{dl}} = A \int \exp[-(1-\cos \delta)^n / X^n] d\delta $$

$$ \cos \delta = \sin \cos \Omega_{\text{dl}} \cos \phi + \cos \cos \Omega_{\text{dl}} $$

Residual stress: Both the extreme models of elastic behavior of polycrystalline materials can be evaluated. The Voigt model, assuming the unique strain, leads to the stiffness tensor averaging, the result of which is the texture dependent ‘Poisson ratio’ (more exactly the ratio of lateral to normal component of the uniaxial strain tensor) and, also, the mean lateral stress can be obtained numerically. The Reuss model, assuming the unique deformation, requests the averaging of compliance tensor for a particular orientation of every hkl reflection to obtain its strain tensor. The dependence of deformation upon $\sin^2 \psi = \sin^2 \omega$ is no longer linear within the Reuss model accounting for the preferred orientation. The complete strain tensor (either constant or hkl and $\omega$ dependent) is then used to calculate the $\omega$ and 20 position corrections.

Absorption, irradiated area: The absorption correction is derived straightforwardly from the kinematical diffraction theory. The linear absorption coefficient of each phase is calculated as:

$$ \alpha = \frac{4\pi}{\lambda} |n_{\text{magn}}| = \frac{2\pi}{\lambda} \sum f_i^n $$

where $f_i^n$ is the imaginary correction for atomic scattering factor of each atom $i$ in the elementary cell of volume $V$; $\lambda$ is radiation wavelength and $r_i$ classical electron radius. If necessary, atomic occupancies are used as weighting coefficients. The intensity of diffraction of the substrate is attenuated by the film above and exponentially to the infinity, according to the path lengths of incoming and outgoing beam.

In order to account for a non-diffracting material in the layer, the correction factor of the total layer’s absorption coefficient is also refined, which allows to properly simulate both the substrate and film reflection intensities together. The factor is always found greater than one.

Total Intensity: The total intensity is the product of both the $\omega$ (texture) and 20 (line broadening) profile, absorption factor, Lorentz and polarization factor. The instrumental broadening is accounted for only by simulating the diffraction pattern using two wavelenghts of CuK$\alpha$ doublet, as the angular broadening of the parallel beam geometry is small with respect to the physical broadening of the samples. The background is accounted for as a floating polynomial fitting the experimental data in symmetrical scan with manually extracted peaks. The $\omega$ profile of the background is refined as a layer-to-substrate ratio of contribution to the background in symmetrical scan and using the same absorption correction as for the diffraction.

An example of measured and simulated omega-20 maps are shown in Figure 1 below.