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SUMMARY

In the presented work, the problems related to the X-ray diffraction profiles and their physical interpretations are discussed. The thesis tries to keep global view of possibilities and characteristics of X-ray diffraction in material research and related areas. The main contribution of the presented work lies in three areas: theoretical research and comments to theories concerning the used X-ray diffraction method, developments of computational algorithms, and particular applications. In-house written programs were developed for reading the data, profile fitting and physical interpretations. The presented particular applications cover macroscopic and microscopic stress analysis, crystallite size determination as well as phase and texture analysis of various steel samples, the study of preferred orientation in thin zeolite layers and Rietveld refinement of non-stoichiometric $SrHfO_3$ phase.

RESUMÉ

V prezentované práci jsou diskutovány problematiky vztahující se k rentgenovým difrakčním profilům a jejich fyzikální interpretaci. Disertační práce se snaží o širší pohled na možnosti a charakter rentgenové difrakce v materiálovém výzkumu a přidružených oblastí. Hlavní přínos této práce leží ve třech oblastech: teoretický výzkum a komentář k teoriím rentgenových difrakčních metod, vývoj vlastních výpočetních algoritmů a konkrétní aplikace. Byli napsány vlastní programy pro čtení dat, fitování a fyzikální interpretaci difrakčních profilů. Prezentované applikace pokrývají: analýzu napětí, stanovení velikosti krystalitů, studium textury a fázovou analýzu u ocelových vzorků; studium přednostní orientace zeolitických tenkých vrstev; Rietveldovu analýzu nestechiometrické fáze $SrHfO_3$.

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1. State of the art

The field of material science and engineering has been particularly active in exploiting diffraction as an analytical tool. This is because there exists a mutual relation between the materials structure and their physical and chemical properties. Since diffraction methods provide information about crystal structures, they can be used to characterize new structures, as well as the presence and the amount of known structures – qualitative and quantitative phase analysis. Materials scientists are also interested in deviations from the perfect crystal structure or the so-called real structure, which encompasses a variety of parameters, such as texture, ordering, interstitial impurities, residual stresses, or grain sizes, and their effects on material behavior.
Diffraction analysis is perhaps the most powerful technique for investigating the microstructure by exploiting, especially, its sensitivity for the atomic arrangement and also the element specificity of the scattering power of an atom. The X-ray diffraction is important for its accessibility and for fast measurements.

The schematic algorithm of X-ray diffraction data processing is shown in Fig. 1. On one side is the diffraction pattern that represents input from the measurement. On the other side is "reality" – the real state of subject. The today's most often used approximation of reality is based on atomic model. For today's computers is not possible to compute diffraction pattern of macroscopic sample from position and state of every particular atom. Consequently, some models consisting of any larger object are used. Many different approaches were suggested as larger object model. They are divided into two main categories: i) the models based on some phenomenological ideas [Klug 1974, Warren 1969] and ii) the models rising from some physical-based conceptions [Krivoglaz 1996]. The phenomenological models are based on modified mosaic structure consisting of coherently diffracting domains of various size and deformations, alternatively including also stacking faults. The microstructure models are based on spatial distribution of various kinds of individual lattice defects, their concentration and correlations [Kuzel 2003]. For example, the first group of model interprets the crystal defects mainly as microstrains, the second one speaks about dislocations. The advantage of the first approach is universality, but the second one is closer to reality. However, the theoretical descriptions are not yet complete for all possible cases.

In general, two basic concepts of diffraction pattern evaluation are used. The first goes from data to description of microstructure; the second one starts from an idea of microstructure or structure and goes to diffraction pattern.

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The first usually describes the peaks in diffraction pattern by some analytical functions [Keijser 1983]. However, other parameterizations of profiles can be used. The first concept can be represented for example by Williamson-Hall, $\sin^2\psi$, or Cohen-Wagner plot, where the diffraction profiles are usually described by one of the analytical functions, whose parameters are further plotted on the graph. These graphs are designed so that it allows fitting of the straight line across the point. Parameters of these straight lines are further interpreted as crystallite size, microstrains, and components of macroscopic residual stress tensor or lattice parameters. Since the measured diffraction pattern is influenced by measuring instrument, appropriate correction for instrumental effects should be executed. This concept is represented by two most upper arrows in Fig. 1.

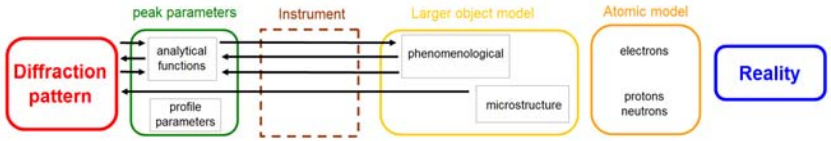

Figure 1. The schematic of X-ray diffraction pattern processing. The arrows show the most often used ways of processing of diffraction patterns.

The second concept is represented for example by Rietveld method [Rietveld 1969] (the second arrows from the top in Fig. 1) or by Whole Powder Pattern Modeling method [Scardi 2002] (the bottom arrow in Fig 1). Here, the diffraction pattern calculation is based on an idea of structure, microstructure, and a detailed knowledge of the used diffraction device. On the base of difference between measured and computed diffraction patterns, the initial model can be modified. The comparison and improvement of the model goes until sufficiently small difference is reached.

The advantage of the first basic concept is that it can be easily applied, using essentially only pen and paper. Its disadvantage is that it can suitably describe only comparatively straightforward models – whenever a more detailed structure characteristic is required or some statistical errors arrive, this approach can lead to wrong or unrealistic interpretations. As an example, the Williamson-Hall plot for large crystallites can be taken: the statistical errors involved can be responsible for negative values of the crystallite size as determined by this method. The second basic principle proceeds from physically plausible values of the evaluated parameters. Its disadvantage may lie in computational difficulties that call for sophisticated computer programs and, in case of comprehensive models, require very advanced and efficient computers. Another problem may arise with initial values of structure/microstructure parameters, their correlations and the stability of

refinement. Some method of difference minimization can be found in [Comba 2009].
Besides these two extreme cases, also a combination of both is possible and used (the third arrows from the top in Fig. 1). It starts from both ends and both ways meet in profile parameters. Thus, it looks like the second extreme way, but instead of the diffraction pattern only the parameters of diffraction peaks are compared. This concept can be represented for example by general lest-squares determination of residual stresses [Winholtz 1988] or by several home made programs presented in the thesis. The advantage of this combined approach is its easy implementation and the possibility of quick refinement. The number of data point is considerably reduced in comparison to whole powder pattern modeling.

2. Aim and motivation of the thesis

The main goal of the thesis was to get experiences with X-ray diffraction profile analysis. Instead of focusing to one topic, several material characteristics were studied. The main attention is devoted to crystallite size, microstrain and macroscopic stress determination. However, quantitative phase composition and texture or preferred orientation have been studied as well.

At the beginning of my doctoral studies in 2004, both X-ray laboratories, where I worked (Department of Solid State Engineering at FNSPE of CTU and Department of Metals of Institute of Physics of AS CR), cooperated on research project dealing with analysis of stress state induced by surface machining of ferrous materials. The project envisaged processing of large numbers diffraction data. Unfortunately, at that time, both laboratories suffered from a lack of suitable software. Hence the first specific aim was to create in house made software for X-ray diffraction data processing, consisting of reading the data from file generated by the diffractometers, describing the diffraction profile (analytical functions were chosen), and physical interpretation of single line profile parameters in the sense of crystallite size and microstrain determination. Another research project was aimed at investigation of depth gradients; therefore, the attention was directed at creating a program for macroscopic stress analysis. The main motivation was to make a computation algorithm for macroscopic stress analysis that would follow the last concept discussed in previous chapter and directly compare calculated and fitted peaks position instead of following the classical approach of fitting straight lines through several modified data points.

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The arrival of modern diffractometers with X’Pert HighScore Plus and TOPAS evaluation software, respectively, significantly broadened the scope of possible diffraction analyses. Characterization of materials by means of quantitative phase composition, texture, precise value of lattice parameters pointed to suitability of non-traditional approaches, which were sometimes beyond the boundaries of common powder diffraction.
One of the aims was to use Rietveld refinement method as a tool for more complex sample characteristic. A certain part of the thesis is devoted to samples, which represent a boundary between powder and single crystal diffraction and correspondingly effects of wavelength spectral distribution and instrumental settings on non-broadened diffractions profiles were analyzed.
As the experimental experiences grew, it was found that the precise sample and instrument alignments play in particular cases very important role in correct diffraction pattern interpretation. Consequently, partial attention was paid also to this problematic.

Eventually, the exerted effort should in the future point to solving crystal structure from powder diffraction data and quantum-mechanics structure modeling.

The main goals of the thesis are summarized in the following list:

1. Provide information about size, strain, and stress in shot peened steel samples.
2. Present the program for non-uniform macroscopical stress state determination on real samples.
3. Perform phase and crystal size analysis on textured, plastically deformed steel AISI 301.
4. Present X-ray diffraction study of zeolite layers.
5. Test the effective intensity of diffraction profiles from the point of view of R factor.
6. Rietveld refinement of non-stoichiometric $SrHfO_3$ phase.

3. Results

The own contribution of the author of the thesis is divided to three parts. The first part deals with own theoretical research and comments to theories concerning used method of X-ray diffraction. The second one describes the development of own computational algorithms for reading the data, profile fitting and physical interpretations of x-ray diffraction profiles. The third one present particular applications that cover: macroscopic and microscopic stress analysis, crystallite size determination as well as phase and texture analysis on various steel samples, the study of preferred orientation in thin zeolite layers and Rietveld refinement of non-stoichiometric SrHfO3 phase.

3.1 Comments to theory and theoretical research

This part discussed fourteen subsections containing comments to, and own experience with, method of physical interpretations of diffraction profiles, own theoretical research and experimental works that proof and deepen the used theory of the used X-ray diffraction method. The most extensive themes discussed spectral wavelength components, effect of sample inclination to broadening and shifting of diffraction peaks and the study of efficiency of intensity of diffraction profile.

Spectral wavelength components. The particular spectral distributions could be easily measured using in-house diffractometer by diffraction from a single crystal sample. Fig. 2 shows a part of diffraction pattern of Al_2O_3 single crystal, the diffracting plane (0 0 12) (the tungsten L-lines on the right-hand-side come from diffraction (0 0 18)) was parallel to sample surface. The measurement was performed using a diffractometer with θ-θ Brag-Brentano geometry. The tube's anode was made from Co. Two lines for two different measurements, one with β-filter (Fe) and the other without, are plotted.

The Kα-satelites component was identified, described and incorporated to Rietveld refinement.

Sample inclination. In practice, diffraction pattern under various tilt conditions is required for the study of oriented treated or prepared samples. The attention is devoted only to the case when sample is inclined in χ axis by angle ψ.

The sample inclination can cause additional sample displacement in cases when sample was already displaced from goniometer axis or when the primary beam does not point to center of rotation, see equation (1).

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List of the author's publications relating to the thesis

The works are divided two parts. The first part contain article in journals with impact factors and the second part consist of reviewed article in other journals, book of abstracts or posters.

Publication with Impact factor:

1. L. Straka, O. Heczko, H. Seiner, N. Lanska, J. Drahokoupil, A. Soroka, S. Fähler, H. Hänninen, A. Sozinov: „*Highly mobile twinned interface in 10 M modulated Ni–Mn–Ga martensite: Analysis beyond the tetragonal approximation of lattice*“, Acta Materialia **59** (2011), pp. 7450–7463 doi:10.1016/j.actamat.2011.09.020

2. F. Laufek, A. Vymazalová, D. A. Chareev, A. V. Kristavchuk, Q. Lin, J. Drahokopil, T. M. Vasilchikova: „*Crystal and Electronic structure study of $AgPd_3Se$*”, Journal of Solid State Chemistry, **184**, (2011), pp. 2794-2798. doi 10.1016/j.jssc.2011.08.019

3. G. Suchaneck, R. Labitzke, B. Adolphi, L. Jastrabik ,P. Adamek, J. Drahokoupil, Z. Hubicka, D.A.Kiselev, A.L.Kholkin, G.Gerlach, A.Dejneka: „*Deposition of PZT thin film onto copper-coated polymer films by mean of pulsed-DC and RF-reactive sputtering*“, Surface & Coatings Technology **205** (2011), pp. S241-S244. doi:10.1016/j.surfcoat.2011.02.050

4. A. Michalcová, D. Vojtěch, J. Čížek, I. Procházka, J. Drahokoupil, P. Novák: “*Microstructure characterization of rapidly solidified Al-Fe-Cr-Ce alloy by positron annihilation spectroscopy*“ Journal of alloys and Compounds, **509**, (2011), pp. 3211-3218 doi:10.1016/j.jallcom.2010.12.049

5. J. Kopeček, P. Haušild, K. Jurek, M. Jarošová, J. Drahokoupil, P. Novák, V. Šíma: „*Precipitation in the Fe-38 at.% Al-1 at.% C alloy*“, Intermetallics, **18**, (2010), Pages 1327-1331 doi:10.1016/j.intermet.2010.03.027

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$$\Delta\theta = \frac{(s + x\tan\psi)\cos\theta}{R\cos\psi}, \qquad (1)$$

where x is displacement from center of rotation, ψ is inclination angle, R is radius of goniometer, s is specimen displacement and $\Delta\theta$ is shift of diffraction angle θ.

The effect of broadening of diffraction peaks due to sample inclination was described by additional convolution to computed instrumental function with TOP-Hat function with cos θ dependence.

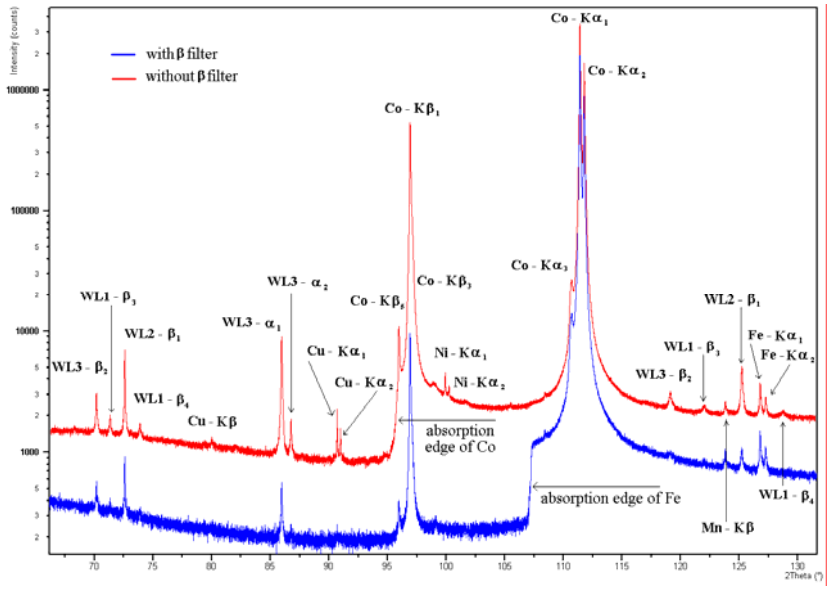

Figure 2. The part of the diffraction pattern of Al_2O_3 single crystal. All observed lines originate from diffraction 0 0 12, except the L lines of tungsten on the right-hand side that originated from 0 0 18. The red line corresponds to the measurement without β-filter and the blue one for the measurement with β-filter.

The efficacious intensity of a profile. This part tries to answer how precisely the profiles should be measured. It was found that R_{wp} factor reach minimum when the maximal intensity of diffraction profile is about 10 000 counts, then errors due to insufficient model prevail the statistics benefits. Additionally was

found that including Kα-satelites component can improve the description of diffraction profiles, see Fig. 3.

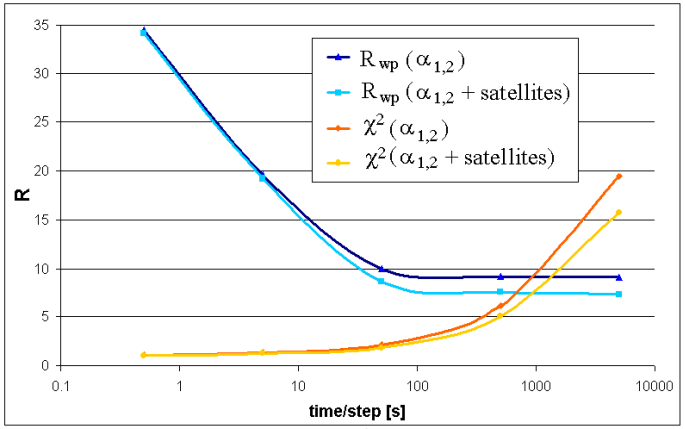

Figure 3. A comparison of the R_{wp} and χ^2 factor for model including (lighter) and not including (darker) Kα-satellites components.

3.2 Computational algorithms

The majority of programs developed during my doctoral studies were coded in Microsoft Excel program. As such, they should be readily transportable to any computer on which Excel is installed and can be readily modified by a user to meet their individual needs. The Excel supports VBA (Visual Basic for Application) computer language in which all macros were written.

First group of programs presents software for reading the data from various files generated by several diffractometers. The second group shows program that used Pearson VII function to describe profiles. The software is not only limited to describing classical 2θ profiles but also the ψ- and ω-profiles can be fitted. The third group deals with description of three programs for crystallite size, microstrain and macrostress determinations. The first of them interprets the peak profile parameters to crystallite size and microstrain using Single line Voigt function method and macroscopic stress by $\sin^2\psi$ method. The second one serves to macroscopic stress determination using general least square refinement. This program is suited for description of general stress state with gradients. The thickness of the layer and several corrections (for temperature, sample displacement, etc.) are included. The third one replaces the classic Williamson-Hall plot by least squares analysis of observed and measured diffraction peaks' breadths. Compared to classic Williamson-Hall plot the data measured with various wavelengths can be used and refined at once.

crystallite size and microstrains. The correlation between refined parameters is discussed using correlation matrix. The simultaneous Rietveld refinement of several measurements is performed with bounding some parameters. This process lowers the correlations between refined parameters. Important parameters were thus given with higher accuracy.

From the whole number of findings, the following three take my biggest interest. Their originality lies in the fact that they were not expected:

a) The possibility of measuring of the relatively precise spectral wavelength distribution by diffraction on perfect single crystal.

b) The possibility of determination of sample displacement by observing peak position during its inclination.

c) The relation between macro and micro stresses observed by parametric plot that was independent on intensity of shot peening.

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is comparable to errors in peak position determination. An interesting matrix was designed to observe difference between measured and calculated peak position. This matrix is useful during process of refinement and shows area where the agreement is worse than the average and thus suggest next step of refinement.

iii. The third application shows diffraction study of plastically induced phase transformation in an austenitic steel. Main question was the volume fraction of plastically induced martensite. Unfortunately, the samples were textured and phase analysis is in this case problematic. Thus, the problem was solved by measuring the texture and subsequently by creating homemade software for phase analysis that enables correction for texture. The texture was described by commercial programs X'Pert Texture. However, the improved corrections (background, defocusing) for pole figures were carry out in another homemade program, which was create especially for this kind of problem. The second task was evaluation of crystallite size. Since required diffractions for size-stress analysis were observed only for inclined sample, the description of inclination broadening were proposed and involved to program TOPAS.

iv. The characterization of preferentially oriented zeolite layer was the theme of fourth parts. Although the Crystallographic Preferred Orientation (CPO) indexes were commonly used by zeolite community, the usability of particular diffraction for CPO indexes was discussed only very roughly. Thus, several diffraction combinations for CPO indexes calculation are now properly discussed. Moreover, the diffractions 10 0 0 and 0 10 0, which were not before considered, were suggested as a best choice for resolution between *a*-, *b*-preferred orientations for a strongly oriented layer. A special version of homemade peak fitting software had to be designed to describe also Kβ component. For a strongly oriented layer the CPO indexes were not able to distinguish between several different layers. Consequently, the measurement of ψ-scans was suggested. In order to get more appropriate distribution of orientation, an instrumental ψ-broadening was measured on the single crystal. The deconvolution was then applied to measured ψ-scans and the OSD (Orientation spread distribution) curves were subsequently obtained. The comparison of the OSD curves for 10 0 0 and 0 10 0 diffractions shows a strong relation between these two orientations supporting the theory about *a*-twin growing from *b*-oriented crystals.

v. The Rietveld refinement of the non-stoichiometric sample of Sr_{1-x} Hf_{1+x} O_{3+x} phases was presented. The sample was sensitive on airy CO_2 and H_2O; therefore, the measurements have to be performed quickly without any sophisticated preparation. The time evaluation of phase contents was determined together with occupancy of Hf and O atoms, lattice parameters,

Corrections for non-ideal standard and elastic anisotropy are also included. Fourth part present software for phase analysis of strongly textured steel samples. The fifth section shows program that enables reading of .xrdml pole figure files, their correction and rewriting back to .xrdml file. The last section present small utility that computes peak position for various wavelengths (several Tungsten lines is prescribed) from a given unit cell. This utility enables calculation of the angle between two crystal directions.

3.3 Specific applications

The theoretical and software parts were enclosed by presenting concrete experimental work of five different problematic. The first presents possible relations between microstructure parameters in steel samples after shot peening. The second one shows the usefulness of least-square analysis in macroscopic residual stress determination. The third part is devoted phase analysis in case of strong texture. The fourth part deals with zeolite layers with very strong preferred orientation. The last subchapter presents the possibilities of Rietveld refinement.

Shot-peened steel sample. The shot peening caused symmetric depth distribution of both macroscopic and microscopic residual stress. Macroscopic stresses are compressive and reach the maximum surface value between - 400 and - 550 MPa. The depth profiles of particular types of stresses for all five investigated steels are similar. The macroscopic stresses show larger differences in the course, the microscopic stresses in the surface value, depending on both: material and intensity of blasting. The relationship between macroscopic and microscopic stresses is independent of the intensity of blasting for a particular material. Hence, knowledge of this dependence for a given material would enable us to evaluate macro stresses from micro stresses and vice versa.

Parameters of the shot peening process have only little effect on the magnitude of the induced compressive macrostress, which is primarily a function of the mechanical properties of the material. Subsurface range of this stress depends on intensity of the process; broadly speaking, the range is approximately 0.2 mm for peening intensity of 0.2 mmA and 0.4 mm for peening intensity of 0.4 mmA.

The surface region of shot peened steels is affected by severe plastic deformation. It is interesting that from the point of view of crystallite size and macroscopic stress the surface region is saturated and more intensive shot penning did not decrease these two parameters. However, from the point of view microstresses, the surface region is not saturated and yet more intensive shot peenig will results in higher microscopic stresses.

Surface hardening by shot peening was observed namely for Mn-Cr steel (B), low-alloyed tool Mn-Cr-V steel (D), and high-speed heavy-duty Mo-W-Cr steel (E); on the average by about 0.8 GPa. Weak hardening was found in the remaining two steels: carbon steel (A) and refractory Cr-Mo-V steel (C). Similar linear dependence was found between macrostresses and hardness in the subsurface region.
The indentation is a less sensitive method than the X-ray diffraction. Surface roughness influences the measurements for small indents. Deviations of the results of indentation are also greater because of a large influence of the surroundings of the indentation spot (various grains, grain boundaries, precipitates). On the other hand, namely the micro hardness in a larger depth indicates that the investigated steels differ considerably. This is not clearly manifested by the residual stresses estimated by X-ray diffraction. The intensity of the shot peening has weaker influence on the micro hardness than on the results of X-ray diffraction measurements.

It was observed that more intensively shot-peened samples differ from the samples blasted with lower particle intensity mainly in the width of the affected zone, which was approx. 0.4 mm and 0.2 mm respectively. Significant correlation was observed between the depth profiles of macroscopic residual stress and crystallite size. The biggest correlations were observed between microhardness and microstresses (close to one), but not for all materials. So the correlation between microhardness and microstresses caused by shot peening depends on material. A little lower correlations were observed between microhardness and macrostresses with the same material dependence as in previous case. No change in the phase content due to surface treatment was found.

The correlation coefficient is constructed for identification of linear relations between two variables. However, the construction of the parametric plot is more time consuming than calculation of correlation coefficient, the non-linear relations can be found and described. Moreover, the parametric plots can be used for identification of errors during processing of large amount of data.

Macroscopic stress gradients and grinded surface of steel. The use of least square analysis in macroscopic stress determination is very useful tool for non-specific stress state. The ψ-splitting or stress gradients can be easily set down, see Tab. 1. The very good agreement between measured and calculated data was find already including only linear gradient of stress components. The average difference (0.006°) was lesser than the maximal error in peak position determination (±0.01°). The minor improvement was reached by including quadratic gradient of stress components; the average difference was then equal

description of profiles were created. These versions include fitting of classic doublet of $K\alpha_{1,2}$ as well as incorporation of Kα-satellites or/and Kβ spectral components. Specific version was designed to fit the ω-profiles or ψ-profiles. Three programs for physical interpretation of diffraction profiles were build:

i. The program for interpretation of profiles parameters by Single line Voigt function method to crystallite size and microstrain and $\sin^2\psi$ method to macroscopic residual stress.
ii. The second interpretation software uses general least squares analysis to description of non-uniform stress state with gradient to surface normal.
iii. The last interpretation software is inspired by the Williamson-Hall method. However, instead of fitting some straight line across measured breadth, the breadths are calculated from initial values of crystallite size and microstrain and the initial values are further varied to get the smallest difference between measured and calculated integral breadths. This technique enables to use simultaneously the breadths given by measurements with different wavelengths. Moreover, correction for elastic anisotropy and not-ideal standard are also covered.

The last chapter presents five particular applications of X-ray diffraction profiles interpretations:

i. The crystallite size, micro- and macro- stress depth gradients were observed in the set of steel after various conditions of shot peening. Besides the surface values of microstructure parameters and the thickness of affected layer by shot peening, also very important relations between microstructure parameters were observed. First by correlation coefficient, that shows the biggest relation between macroscopic residual stress and crystallite size. The second one by parametric plot between macro and micro stresses showing that this plot can distinguish between materials. It also indicates that intensity of shot peening does not play an important role in macro – micro stress characteristic. Moreover, microhardness measurements show that shot peening increases hardness only for some materials, while the rest are not affected. The calculations of correlation coefficients show very big correlation between microhardness and microstresses for the materials, whose microhardness were affected by shot peening.
ii. The homemade program for macroscopic stress analysis was presented. The surface layers of ground steel samples were chosen as subject of study, because the grinding produces a non-uniform stress state with big depth gradients in diffraction volume. This program enables successful fit of observed peak position. The average difference, for six refined sample, between calculated and measured peak position was ca. 0.004° [2θ], which

4. Conclusions

Several topics that broadened or proofed the known theoretical concepts of X-ray diffraction were discussed:

i. The first main topic was devoted to spectral wavelength distribution of a particular laboratory X-ray source. It was found that besides the main spectral components Kα and Kβ, the spectrum also contains several tungsten L lines and several Kα lines from minor element impurities (Cu, Fe, Ni, Mn) of Co anode matrix. Although, these subsidiary spectral wavelength components have no or very low importance to common applications of powder diffraction, their contribution can be significant for study of strongly oriented samples, polycrystalline samples on single crystal support or single crystal itself. Short program was written to help with this problem by computing 2θ position for several Tungsten L lines and by calculating mutual angle between two crystallographic directions. Usually it is considered that the main Kα spectral components consist from only Kα1 and Kα2, however the detailed view shows also presence of several minor Kα-satellite components. These components were described by several analytical functions and included to the Rietveld refinement in the program TOPAS. It was also shown that including these Kα-satellite components can decreases the R-factor during refinement of a profile. This is especially important in structure refinement procedure or in microstructure interpretation of subtly broadened peaks.

ii. It was discovered that the shift of diffraction angle during inclination can determine the sample displacement. The effect of broadening of diffraction peaks was described and also included to the Rietveld refinement in the program TOPAS. This description was used in crystallite size determination of textured steal samples.

iii. A more detailed view of penetration depth for radiation of three different wavelengths (Cr, Co, Cu) into a steel material is presented for various diffraction peaks and sample inclinations. The theory of gradients was integrated to own made software for macroscopic residual stress determination.

Several homemade software related to the primary topic of theses were developed. Besides several macros for reading the data from different diffractometer files, various programs using Pearson VII function for

to 0.004°. The very low improvement was reached after including refinement of non-stressed peak position and correction of temperature coefficient. The convergence of simultaneously fitted samples is lower than in one sample case, so increasing of the number of iterations or another appropriate step have to be done. The process of refinement can end in local minimum; therefore, the sequential fitting of parameters as was shown above is suggested.

Table 1. The final component of stress tensor. The surface values, linear part and quadratic part of components σ_{11}, σ_{22}, σ_{13}, σ_{23}, σ_{33} [MPa] of stress tensor, coefficient of temperature expansion and lattice parameter of non-stressed state were refined.

	surface value						linear component						qudratic component					
	σ_{11}	σ_{22}	σ_{33}	σ_{12}	σ_{13}	σ_{23}	σ_{11}	σ_{22}	σ_{33}	σ_{12}	σ_{13}	σ_{23}	σ_{11}	σ_{22}	σ_{33}	σ_{12}	σ_{13}	σ_{23}
1A	-169	-434	0	0	67	-1	11.7	62.6	-0.5	0.0	-2.1	0.2	0.3	-2.1	0.0	0.0	0.1	0.0
3A	-512	-703	0	0	35	0	65.3	127.2	-2.8	0.0	0.8	-0.1	-1.5	-5.5	0.2	0.0	0.0	0.0
5A	-92	-419	0	0	111	-8	4.9	74.0	2.4	0.0	-18.2	0.8	1.7	-2.2	0.0	0.0	0.7	0.1
8A	-196	-448	0	0	78	-1	8.1	47.7	-1.3	0.0	-7.3	0.0	0.8	-1.3	0.0	0.0	0.3	0.0
9A	-223	-449	0	0	84	-3	16.0	56.3	0.2	0.0	-9.3	0.9	0.9	-1.6	0.0	0.0	0.3	0.0
10A	-245	-497	0	0	68	-8	-14.6	75.7	-2.2	0.0	-3.8	2.2	3.1	-3.6	0.0	0.0	0.0	0.0

Phase transformation induced by plastic deformation. In low-nickel content austenitic steel AISI 301, deformation-induced martensitic transformation starts only after low amount of plastic deformation (less than 2%). During straining, the volume fraction of α'-martensite rapidly prevails over the volume fraction of ε-martensite, see Fig. 4.

Since the texture maximum of austenite {111} roughly corresponds to martensite {110} it is possible to easily make the crystallite size analysis from crystallite with the same orientation to sample coordination system. The deformation process decrease the crystallite size of austenite, but to contrast the crystallite size of martensite are lightly increasing. As for phase analysis so for texture analysis give all three diffraction technique similar results. Since the x-ray diffraction take information from surface, the EBSD from local part in sample center and neutron diffraction from several samples it can be state that deformation process is relatively homogeneous and surface values do no differ from bulk values.

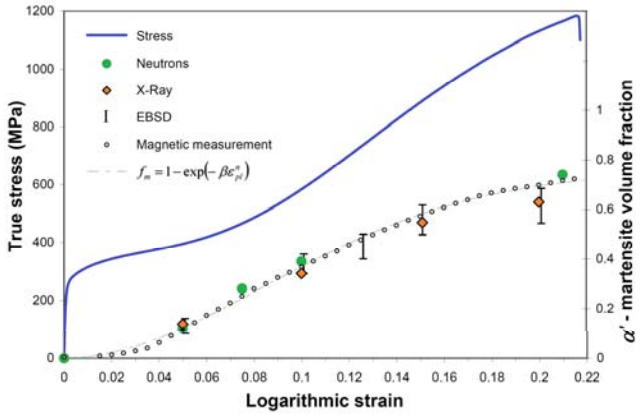

Figure 5. Evolution of α' martensite volume fraction after tensile deformation.

Zeolite membranes. The X-ray diffraction pattern measured in symmetrical Brag-Brentano geometry is excellent tool for the first visual comparison of crystallinity and preferred orientation of crystallites. Detailed information about orientation can be given by appropriate crystallographic preferred orientation (CPO) indexes. The precise information about spreading of *a*-, *b*-orientations provides orientation spread distribution (OSD) curves calculated from ψ-scans, see Fig. 6. The highest preferred *a*-, *b*-orientation was achieved for silicalite-1 layer prepared on the silicon wafer - A.

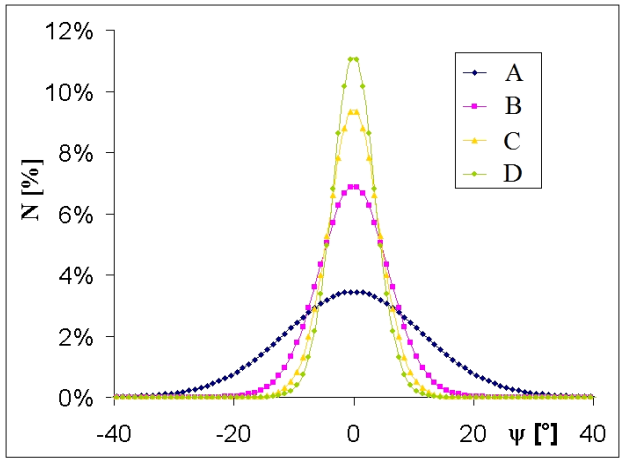

Figure 6. The OSD curves from diffraction 400+040 for studied Silicalite-1 layers. Each point corresponds to ψ-interval of 1°.

Rietveld refinement of $Sr_{1-x}Hf_{1+x}O_{3+x}$ phases. The Rietveld method is very strong tool that is able to work with diffraction pattern consisting of many diffraction peaks of several phases that are moreover particularly or considerably overlapped. Many of structure or microstructure parameters can be calculated by this method. Last but not least, important parameters from this method are: phase content, lattice parameters, crystallite size, microstrain, occupancy factors. The X-ray diffraction did not give the precise answer for occupancy factors. However, the lattice parameters or occupancy factor can be further compared with other non-diffraction experiments or ab initio calculations. Time evolution of particular phases contents in weight percents are shown in Fig. 7.

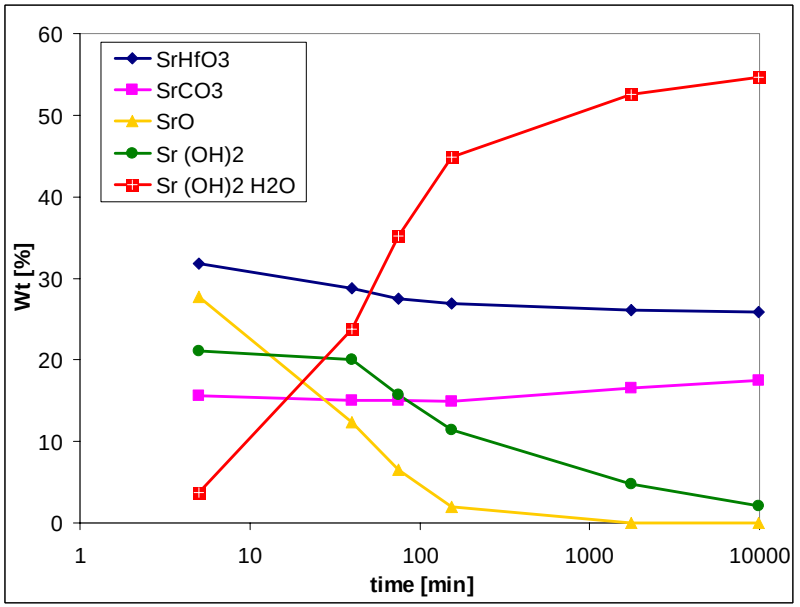

Figure 7. The weight percent time evolution of particular phase content in SHO sample.