Experimentální Analýza Napětí 2011
Experimental Stress Analysis

Recent Application Capabilities of X-ray Diffraction Technique for Residual Stress Measurements in Materials Science and Industry

Nikolaj Ganev[1]

Abstract: The goal of the contribution is to outline the basic principles and characteristics of X-ray stress measurement and at the same time to sketch some possibilities of applying this experimental technique in materials science and mechanical engineering. The paper contains examples presenting the recent experience of the X-ray diffraction laboratory of the Czech Technical University in Prague with studying residual stress fields in surface layers of metals and alloys.

Keywords: X-ray diffraction, Residual stresses, Shot peening, Up-cut and down-cut grinding

1. Introduction

Residual stresses on the surface of polycrystalline materials and beneath it belong among the most important parameters of surface quality. Under elastic-plastic deformation, individual crystallites are differently deformed and this gives rise to microscopic internal stresses, which are accompanied by macroscopic stresses [1, 2]. It has been shown [3] that, in general, compressive residual stresses in the material can favourably improve the dynamic strength by about 50%; on the other hand, tensile RSs could deteriorate it by about 30%.

Lately we have been witnessing a growth of interest in the surface qualities of solids. However, this fact is not surprising, when we become aware of the fact that any interaction with material is being realized over its free surface. Surface layers can influence in a decisive way the employment on the whole volume of material. Surface layers are primarily important in processes of brittle and fatigue fracture and the like.

Various surface engineering procedures, as well as many conventional technologies, introduce stresses on particular engineering products either intentionally or involuntarily. These stresses are confined to shallow surface layers only several micrometres thick. In this way, considerable stress gradients may be created which influence significantly the different characteristics of the products, sometimes favourably, sometimes detrimentally. There is no analytical technique which allows us to evaluate such non-uniform stress fields in a nondestructive way as efficiently as X-ray diffraction.

Stress measurement, especially that of residual stresses, represents one of the most wide-spread and technically important applications of X-ray diffraction. In Czech Countries this area of experimental physics has already reached a 70 year old tradition. From the point of view of national engineering industry traditionally much attention has been paid to the X-ray

[1] *Prof. Ing. Nikolaj Ganev, CSc.; Department of Solid State Engineering, Faculty of Nuclear Sciences and Physical Engineering, CTU in Prague; Trojanova 13, 120 00 Prague 2, Czech Republic; nikolaj.ganev@fjfi.cvut.cz*

stress analysis, namely for its capability of reliable and effective checking of metal and alloy surface treatment technologies.

2. Residual stress classification

Mechanical stress within an isolated body of which all parts are of the same temperature is called residual stress (RS). The internal forces and moments in a body with RS are in a mechanical equilibrium. The most generally recognised RS classification is based on the extent of acting stress [1, 2].

Macroscopic (first-order) residual stresses are approximately homogeneous in a macroscopic volume (many crystallites) of the material. Its linear dimensions are at least of the order of millimetres.

Microscopic (second-order) RS are approximately homogeneous in volumes of a dimension comparable to the grain size. The force and moment equilibrium is supposed even in a great number of crystallites.

Submicroscopic (third-order) RS are inhomogeneous even in volumes comparable to interatomic distances.

State of RS is always a superposition of all the three kinds defined above. Macroscopic RS are believed to be the most important from the point of view of industrial applications.

3. Basic principles and characteristics of X-ray stress measurement

X-ray stress analysis exploits the fact that X-rays are diffracted by crystal lattices. The rays being diffracted at adjacent lattice planes interfere with each other and produce an intensity maximum if the difference between the path length of the two rays is an integer multiple of the employed X-ray wavelength λ. This yields Bragg's condition for the angular position θ of the interference peak:

$$n\lambda = 2d\sin\theta, \quad (1)$$

where d is the distance between the reflecting lattice planes of the type *{hkl}*. If the crystal is under mechanical stress, the lattice plane spacing d is modified with respect to the unstressed state. That causes angular displacement of the interference maximum. Differentiation of Eq. (1) yields the correlation

$$\varepsilon = \frac{d - d_0}{d_0} = -\mathrm{cotg}\,\theta_0(\theta - \theta_0) \quad (2)$$

between the lattice strain ε occurring in the deformed crystal and the angular displacement of the interference line $(\theta - \theta_0)$. Here d_0 and θ_0 stand for the corresponding values of the stress free crystal.

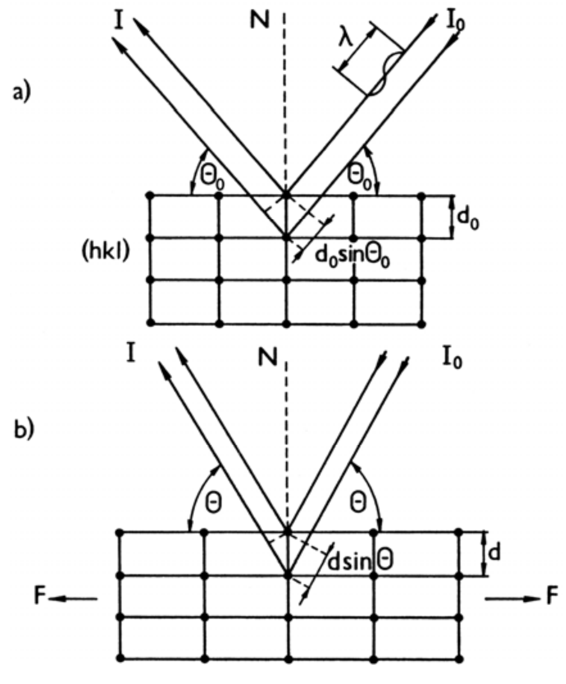

Fig. 1. Deformation of interplanar spacing and shift of the diffraction maximum.

Values of RS are subsequently obtained using relations of elasticity theory with proper elastic constants.

X-ray diffraction has some main attributes different from the other experimental techniques for stress determination:

- The investigated specimens have to be of crystalline or partly crystalline structure. When multiphase systems are investigated, it is possible to analyse lattice strains of each phase component.
- Diffraction measurements in a thin surface layer are completely nondestructive. The destructive X-ray residual stress analysis can be performed by sequentially removing surface layers usually by electro-chemical polishing. A correction with respect to the occurred stress relaxation should be involved in the evaluation procedure.
- As mentioned above, macroscopic residual stress determination is based on angular shift of diffraction lines. Homogeneous microscopic stresses can result in a symmetrical broadening of diffraction lines. X-ray diffraction method enables to study these two kinds of residual stresses separately.
- Stress interpretation of diffraction diagrams exploits evaluation of diffraction line (profile) parameters. The method, therefore, can be used only for materials whose diffraction peaks are not too diffuse. Accuracy and reliability of the method decrease in large grain size objects and textured materials.

Although the principle of stress measurement, “what the elongation is like, such is the force” was formulated by Hooke’s law in 1678, its diffraction interpretation was realized only 250 years later. The oldest information about the diffraction research on changes induced in solid state substance structure by exterior forces came from Sankt Petersburg, where shortly after World War 1 (1913 - 1924) Joffe and Kirpicheva used Laue’s method for studying elastic constants [4] and for assessing the anisotropy of temperature dependence on the elastic limit in monocrystals of NaCl, CaSO, and in some natural minerals [5]. In polycrystalline materials the primacy in this area of experimental physics is shared between two Americans, H.H.Lester and R.H Aborn, who in 1925 measured the lattice strain of a-Fe crystallites in an elastically deformed steel sample using the Debye-Scherrer method: the interplanar distances change linearly with the stress [6].

In Czechoslovakia, the first measurements of residual stress by means of X-rays were probably performed by A. Kochanovská in 1936. She investigated the origin of cracking in the cover of shells. This research was of military character and therefore its results could not be published. Between 1937 – 1939 the Czech physicist P. Skulari paid attention to X-ray stress analysis of forged aluminium, to head treated iron and steel, and to residual stress non-homogeneities near welded seams. Kochanovská and Skulari are credited with introducing diffraction stress analysis in X-ray laboratories of Czechoslovak universities as well as research institutes.

At present X-ray diffraction for residual stress investigation is used in several Czech laboratories. However, the most systematic development in this field of experimental stress analysis is concentrated at the Faculty of Nuclear Sciences and Physical Engineering of the Czech Technical University in Prague. Following examples of solved problems illustrate the recent experience of the laboratory team with applications of X-ray stress measurements in materials science and mechanical engineering.

4. Residual stresses in surface layers of shot-peened steels

Shot peening consists of the controlled bombardment of the metal surface by spherical shot including steel shot, steel and stainless steel pieces of wire, ceramic or glass beads [7]. The shots may be driven by a high velocity stream of air or liquid or by mechanical device in which the shots are fed into a rotating wheel and thrown at the desired velocity. The treatment causes plastic flow of the surface layers, thereby inducing surface compressive stresses, change of microstructure and may cause phase transformation in the surface layers.

Although shot peening have been used widely in machined industry for a long time, it is still in the focus of interest of scientists and technologists [7].

When the beneficial effect of imposed compressive residual stresses by shot peening is evaluated, usually only depth distribution of macroscopic (first kind) ones is taken into account. The unique ability of X-ray diffraction methods to determine both the macroscopic residual stress and mean value of micro-strain and crystalline size in the irradiated volume is not commonly applied for complete characterization of degree of severe plastic deformation imposed into the surface layer affected by the shots' stream.

4.1. Samples and methods

The set of analysed samples (50 x 50 x 5 mm^3) was prepared from five steels: mild carbon steel C45 (*A*), low carbon Mn-Cr steel 16MnCr5 (*B*), corrosion-resistant steel M300 (*C*), tool low-alloyed Mn-Cr-V steel for the cold working 90MnCrV8 (*D*), and high speed heavy duty steel M41 (*E*). The samples were shot-peened by using two different intensities of blasting specified by using Almen test [7] as 0.2 mmA (samples signed by number 11) and 0.4 mmA (samples signed by number 13). In order to analyse the stress gradients beneath the samples surface the layers of material was gradually removed by electrolytic polishing. Prior to shot peening the samples were annealed (stress-relieved) in *Ar* at 550 ºC for 2 hours.

The measurement was performed on an ω-goniometer Siemens with *Cr-Kα* radiation. The line *{211}* of *α-Fe* phase was measured. Nine different tilts angles (ψ) from 0° to 63° were used. The $sin^2\psi$ method was used for determination of macroscopic residual stress [2]. The X-ray elastic constants $\frac{1}{2}\, s_2 = 5.95\cdot10^{-6}$ MPa^{-1}, $-\, s_1 = 1.325\cdot10^{-6}$ MPa^{-1} were used in stress calculations. The single line Voigt function method was applied for corrections of instrumental broadening and determination of microstrains and crystallite size. The microstresses σ^{micro} was calculated from microstrains *e* using Hooke's law ($\sigma = e\,E$) with the Young modulus $E^{211} = 216$ GPa to be comparable with macroscopic residual stress σ^{macro}.

4.2. Results and discussions

The samples were measured in two perpendicular directions. For reasons of clarity the average values for these perpendicular directions are used there. Ascertainment that shot peening is symmetrical treatment [7], give us the competence for the mentioned averaging. The single line Voigt function method [1] offer values of particle size *D* and microstrain *e* (or σ^{micro}) for each of the tilt angles ψ, i.e. for $sin^2\psi = 0, 0.1, 0.2, \ldots 0.8$. In order to compare these data with macroscopic residual stress, the values for all tilts and also for two perpendicular directions were averaged.

Fig. 2 shows the depth profiles of macro and micro stresses for the samples from steels *B* and *C*. The shot peening caused symmetrical both macroscopic and microscopic residual stresses. The macroscopic stresses are compressive. The depth profile of microscopic stresses has nearly the same hyperbolic decreasing tendencies. The surface values differentiate both among steels and intensities of blasting. All microscopic stresses are decreasing to zero with increasing depth aside the M300 steel (*C*) which reaches the lowest value approx. 100 MPa.

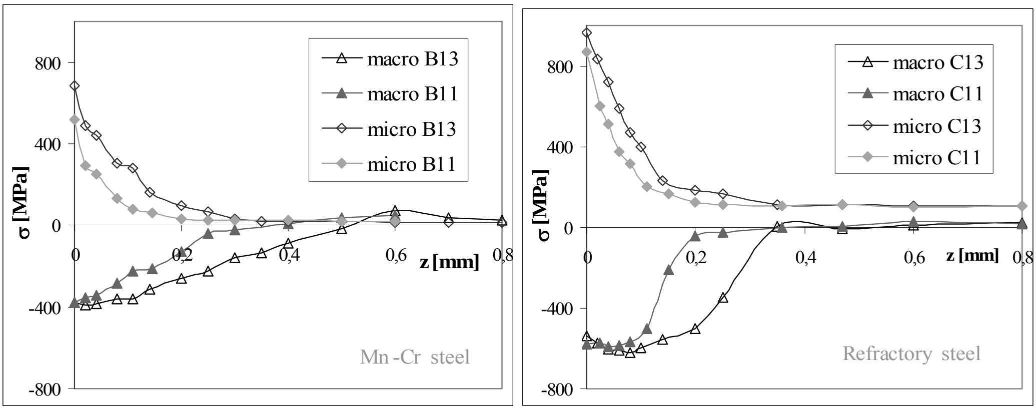

Fig. 2. Depth profiles of macro and micro stresses for two intensity of blasting (11-low, 13-high) for all materials *B* and *C*. The standard deviations are approximately ± 30 MPa

The Fig. 3 is parametrical plot between macro and micro stresses where on the axis *x* are plotted microscopic residual stresses σ^{micro} and on the axis *y* are plotted macroscopic residual stresses σ^{macro}. Since dependences for both intensities of blasting are for all materials very similar (whereas the curves for both intensities do not differ much from one another for the same material) and on the curves for different materials are shifted each other, it can be stated that the relations between macro and micro stress depend primarily on the material characteristics independently of intensity of peening. Thus, dependencies for only two materials (*B*, *C*) are plotted as limiting curves for the remaining materials (*A*, *D*, *E*).

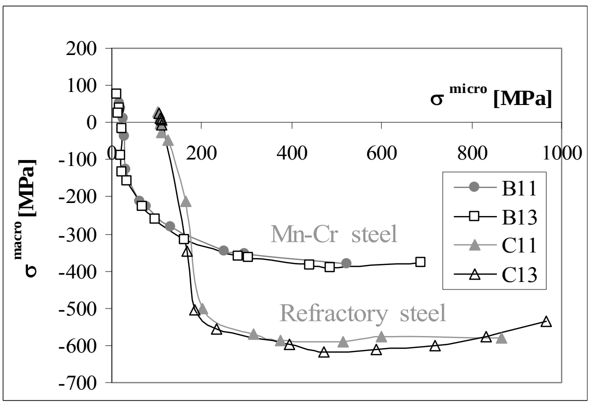

Fig. 3. Parametric plot of macrostresses vs. microstresses for materials *B* and *C*

These experiments imply that relationships between macroscopic and microscopic stresses are independent on intensity of blasting for the particular material. Hence, knowledge of this dependence for a given material would enable to evaluate macro stresses from micro stresses and vice versa.

The shot peening caused symmetrical both macroscopic and microscopic residual stress depth distributions. The macroscopic residual stresses are compressive and they reach the maximum surface value between – 400 and – 550 MPa. Depth profiles of particular types of stresses for all five investigated steels are similar. Parameters of the shot peening process have only a little effect on the magnitude of the compressive macrostress induced which is primarily a function of the mechanical properties of the material. Subsurface range of this stress depends on intensity of the process.

5. Influence of various cooling environments during grinding on residual stresses

The experiments within this investigation were designed with the aim to study the influence of cooling media used for conventional face grinding on the resulting state of macroscopic residual stress. Primarily, the influence of three cooling environments, namely ambient air, emulsion of water, and synthetic fluid, and cooled air of –28°C from vortex tube, was assessed on the surface and approximately 400 µm beneath. The grinding together with milling have a common distinctive feature lying in the fact that both can be performed in two modes, i.e. up-cut or down-cut. During conventional face grinding, these modes are alternating and it is not known which mode was the final one; therefore, secondly, a machining experiment consisting of just up-cut or down-cut face grinding was carried out.

All samples were squared plates 5.5 mm thick and 50 mm in dimensions. In these samples, texture was not significant and sizes of coherent scattering domains were convenient for residual stress determination using diffraction measurements.

5.1. Assessment of three cooling environments during conventional face grinding [8]

The samples for this study were made from corrosion-resistant steel M300 and low carbon Mn-Cr steel 16MnCr5. The measurements were carried out in the so-called Bragg-Brentano parafocusing geometry of a ω-goniometer with *CrKα* radiation. Components of macroscopic stress tensor in the grinding direction (σ_{11}), in the direction perpendicular to grinding (σ_{22}), shear stresses (σ_{13}, σ_{23}, σ_{12}) and normal component (σ_{33}) were evaluated by using the method of Dölle and Hauk [1]. Depth distributions of macroscopic residual stresses were obtained by diffraction measurements in combination with stepwise electro-chemical layer removal.

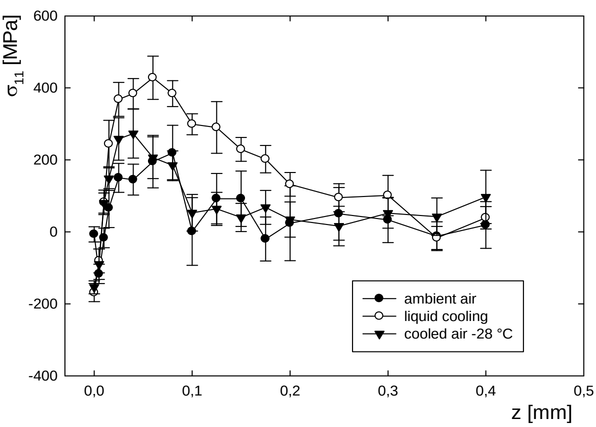

Fig. 4. Depth distribution of macroscopic residual stress σ_{11} in the corrosion-resistant M300 steel

Detailed consideration of the results leads to following conclusions:

- Measurements of residual strains on the surface of ground samples proved that grinding causes anisotropic triaxial state of residual stress.
- Inclination of the axes of principal stresses in respect to the samples surface is decreasing with the increasing depth.
- Absolute values of surface residual stresses in the direction perpendicular to grinding $|\sigma_{22}|$ are larger than those in the grinding direction $|\sigma_{11}|$.
- Cooling using liquid is characterized by higher compressive stresses on the surface in comparison with cooled air from vortex tube and ambient air. The liquid conducts heat away from the surface more effectively which suppresses tensile thermal stresses and thus the effect of mechanical deformation causing compressive stresses dominates.
- Maximum of the depth distribution is reached for both materials in the area of 50 to 80 µm beneath the surface. Maximal stress values σ_{11} and σ_{22} in Mn-Cr steel are approximately the same (250 ± 25 MPa) for all samples, but the corrosion-resistant steel

M300 behaves differently in case of liquid cooling. This sample has maximum stress value approximately 150 MPa larger maximal σ_{11} (430 ± 60 MPa) and approximately 50 MPa larger maximal σ_{22} when compared with the other ways of cooling.

- Normal stresses σ_{11} and σ_{22} in Mn-Cr steel are equal, with respect to the experimental inaccuracy, to each other from the depth of 20 μm onwards. The levelling off the normal stresses σ_{11} and σ_{22} in M300 steel happens in the depth of 150 μm.

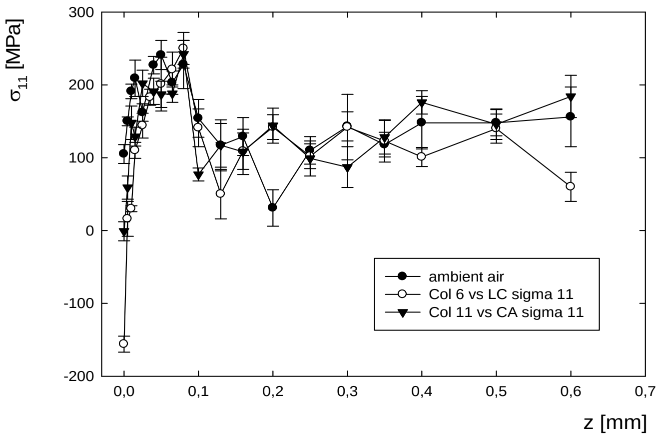

Fig. 5. Depth distribution of macroscopic residual stress σ_{11} in the low carbon Mn-Cr steel 16MnCr5

- In all the studied distributions, the shear stress σ_{31} decreases with increasing depth and is negligible from the depth of 50 μm onwards. Values of shear stresses are not influenced by cooling method.
- Surface value of normal stress σ_{33} reaches approximately MPa, local extremes are observed in the depth of 50 μm. This behaviour is common for both steels and does not show any variation with the cooling method.

5.2. Comparison of up-cut and down-cut grinding modes [9]

There is a pronounced distinction in the fashion of material removal between up-cut and down-cut grinding which could lead to unequal states of surface residual stress. By means of X-ray diffraction analysis, ground plates made from three types of steel, mild carbon steel C45, corrosion-resistant steel M300 and high speed steel M41, were investigated in order to evaluate, and compare macroscopic residual stresses. With respect to the main sources of residual stress generation, i.e. plastic and thermal deformation, machining process was carried out in two types of cooling environment, ambient air and emulsion of water and synthetic fluid.

In general, there exist two possibilities for grinding direction assignment, in the direction of material removal progress and in the opposite direction to it. Having the information about the geometry of grinding for each sample, the diffraction measurements were performed for both options, i.e. in two coordinate systems mutually rotated by 180°. In order to obtain full stress tensor, the diffraction line {211} of *α-Fe* phase was measured in both positive and negative tilts in three azimuths 0°, 45°, 90° on an θ/θ Bragg-Brentano ω-goniometer X'Pert PRO with *CrKα* radiation. For all samples, the azimuth 0° was chosen in the direction of material removal progress and in the opposite one.

In particular, the ground surfaces were assessed in accordance with (i) differences between up-cut and down-cut ground plates, (ii) impact of cooling environment, (iii) ambiguity of choice of azimuth φ = 0° as the grinding direction. The conclusions from the performed analysis are following:

- Way of cooling has an appreciable impact on normal residual stresses σ_{11} and σ_{22} in respect to the grinding modes. Liquid cooling results in approximately the same values of residual stresses in both grinding modes, whereas for down-cut dry grinding higher compressive normal RS were recorded in comparison to the up-cut mode.
- Shear stress σ_{31} calculated from psi splitting in grinding direction changes its sign when the reference frame is rotated by 180°. Moreover, the negative σ_{31} always occurs when the primary X-ray beam impinges the surface in the opposite orientation with regard to the assumed direction of grinding wheel rotation. The sign of shear stress σ_{31} can be, hence, used for determination of grinding wheel rotation direction.
- Negative shear stresses σ_{31} are systematically lower, in an absolute value, in comparison with positive shear stresses.

6. Conclusions

On the base of a long-term experience it should be notice, that diffraction technique for residual stress measurement provides a very efficient wide applicable tool, convenient both for fundamental research in material sciences, and for solving the everyday problems of manufacture. X-ray diffraction determination of stresses is based on a transformation of crystal lattice deformations into mechanical stresses using equations of the linear theory of elasticity. While the technique of lattice deformation measurement does not cause any substantial problems, the evaluation of stresses should be carried out very deliberately. Since any solved problem requires an individual access, the assertion, that X-ray method for residual stress measurements can be apply commonly in manufacture does not seem to be legitimate.

Experimental equipment with both, the diffractometer devices and classical X-ray cameras exploiting two dimensional detection of diffracted radiation is necessary when practical tensometric problems are to be solved.

Acknowledgements

The research was supported by the Project No 101/09/0702 of the Czech Science Foundation and by the Project MSM 6840770021 of the Ministry of Education, Youth and Sports of the Czech Republic.

References

[1] Hauk, V., Structural and Residual Stress Analysis by Nondestructive Methods, Elsevier, 1997.

[2] Kraus, I., Ganev, N., “Residual Stress and Stress Gradients”, in Industrial Applications of X-ray Diffraction, Marcel Dekker, New York, 2000, pp. 793–811.

[3] Youtsos, A., G., Residual Stress and Its Effect on Fracture and Fatigue, Springer, 2006.

[4] Joffe A.F.and Kirpicheva M.V.: Phil. Mag. 43, 1922, 204.

[5] Joffe A.F. and Kirpicheva M.V. and Levitski M., Nature 113, 1924, 424.

[6] Lester H.H. and Aborn R.H., Army Ordnance 6, 1925/26, 120, 200, 283, 364.

[7] Drahokoupil, J., Ganev, N., Čerňanský, M., Boháč P., Stranyánek M., and Čtvrtlík R., “Stress and Hardness in Surface Layers of Shot-peened Steels”. Z. Kristallogr. Supl. 27 (2008) pp. 89 – 96, ISSN 0930-486-X.

[8] Pala Z. and Ganev N., “Impact of various cooling environments on distribution of macroscopic residual stresses in near-surface layers of ground steels”, Materials Science and Engineering A 497 (2008) pp. 200 – 205, ISSN: 0921-5093.

[9] Pala Z., Ganev N., and Drahokoupil J., ”X-ray diffraction study of anisotropic state of residual stress after down-cut and up-cut face grinding”, Pow. Dif. 24 (2009) 99-101.

[10] Kraus I. and Ganev N., Technické aplikace difrakční analýzy, ČVUT, Praha, 2004. ISBN 80-01-03099-7.