

About the Capability of X-ray Diffraction Technique for Diagnostics of Technological Residual Stresses

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Abstract: The contribution is focused on the selected recent experience of the X-ray diffraction laboratory of the Czech technical university in Prague with industrial applications of X-ray diffraction residual stress determination on cold-worked surfaces, for assessing effect of working conditions and parameters of conventional and unconventional methods of machining. Since any interaction with work-piece during machining is realized over its free surface, the state of residual stresses in surface layers could have a predominant impact on the reliability and service life of machine components. As any process of machining accompanied by inhomogeneous plastic deformation leads to formation of residual stresses, their volume distribution is of great importance. Recently that is the reason of the growing interest for complementing traditional materials' characteristics like strength, toughness, and wear resistance, with information about field of residual stresses.

Keywords: X-ray diffraction, Residual stress, Milling, Shot-peening, Electro discharge machining

1. Introduction

Interaction with material during its machining involves its free surface and hence the surface state and its properties have significant impact on utility properties of the whole component. One of the crucial factors to be considered is both surface and depth distribution of residual stresses. Creation of residual stresses accompanies all technological process with inhomogeneous plastic deformation and inhomogeneous thermal fields.

Residual stresses (RS) present in surface layers, which have been subjected to various surface treatments ranging from both traditional and progressive machining to shot peening, result from combined influence of mechanical and thermal effects. In some cases even phase transformation can cause substantial change in residual stresses' distribution. If only mechanical load is present, thin surface layer is being

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plastically deformed and the rest forming the elastic core is deformed elastically. During and after elastic unloading, the elastically deformed volume strives to return to its original state and thus it interacts with the plastically deformed surface volume. The surface is at the same time exposed to sources of heat emerging e.g. from the friction between the tool and work-piece. The volume closer to the heat source yields to thermal expansion which is, however, hindered by colder deeper volumes. Overall, the resulting state of residual stress and both its surface and depth distribution depend on which of the thermal or plastic effects will dominate. However, the process of stress relaxation should not be neglected. The character and magnitude of residual stresses in surface layers is always a function of the treated material, type of treatment and its working conditions. An array of parameters of surface integrity determines the utility properties and residual stresses do belong among them. Compressive stresses generally increase dynamic strength and vitally increase surface resistance to corrosion and crack initiation and propagation. On the other hand, tensile stresses are considered detrimental since they facilitate erosion of surface layers during contacts with outer environments and other objects.

Therefore, residual stresses play important role in the process of surface treatments and machining optimization from the quality point of view.

2. Selected results of X-ray diffraction industrial application

X-ray diffraction (XRD) is a tool which is being increasingly used in industrial research and development and X-ray tensometry is no laggard. Introduction of modified or new production technologies is more frequently accompanied by X-ray tensometry analyses. Selected results of cooperation between X-ray diffraction laboratory of Czech Technical University in Prague and its industrial partners during last four years are presented in the contribution.

2.1. Residual stress gradients of ground and roller burnished axle seats

XRD analysis was done on two samples of axle seats marked A1N and MA1N. The aim was to measure the ground sample A1N at the surface and in the depths of 0.1, 0.2, and 0.3 mm; the roller burnished sample MA1N was analysed up to the depth of 2 mm.

2.1.1. X-ray diffraction technique

XRD "one-tilt" method was applied to study biaxial state of RS [1]. The incident Xray CrK α beam directed by a cylindrical collimator of 1.7 mm in diameter reached the sample surface at an angle of $\psi_0 = 45^\circ$ in the axial and transversal direction, in which the surface components of stress σ_A and σ_T , respectively, were analyzed. The record of the {211} α -Fe diffraction line intensity curve was obtained from a position sensitive detector (imaging plate). The experimental inaccuracy does not exceed 40 MPa.

Due to the limitations of X-ray penetration depth, the X-ray diffraction technique can be used only for surface layers of few micrometers in thickness. In the

case of conventional X-ray diffraction device, investigation of stress depth profiles is performed in combination with electrochemical polishing.

2.1.2. Selected results

Values of axial (σ_A) and transversal (σ_T) residual stresses obtained in the middle of the ground sample A1N and roller burnished sample MA1N are shown in Figure 1.



Fig. 1. Depth profiles of axial (σ_A) and transversal (σ_T) residual stresses; (a) ground sample A1N, (b) roller burnished sample MA1N.

2.1.3. Conclusions

During this diffraction investigation of residual stresses depth distribution ground sample A1N exhibits subsequent decay of continuous diffraction pattern onto discrete diffraction spots (Fig. 2b) corresponding to diffraction of individual crystallites. This observed material behaviour, which substantially limits reliability of diffraction tensometry, is caused by larger crystallite sizes in the bulk.



Fig. 2. Diffraction patterns of α -Fe {211} lines obtained at the surface (a) of the ground sample A1N and after removal of 0.4 mm thick layer (b).

On the contrary to the ground sample, the real structure of the roller burnished sample enabled to reliably determine residual stress depth distribution up to the requested threshold of 2 mm (Fig. 1b). Owing to the continuous character of diffraction patterns (Fig. 3), the values' inaccuracies do not exceed 40 MPa.



Fig. 3. Diffraction patterns of α -Fe {211} lines obtained at the surface (a) of the roller burnished sample MA1N and after removal of 2 mm thick layer (b).

The depth distribution of axial (σ_A) and transversal (σ_T) residual stresses measured in the middle of the roller burnished sample MA1N (Fig. 1b) have analogous character. Compressions observed in axial direction are systematically higher than in transversal direction.

2.2. Gradient of residual stresses of shot-peened helical spring SNCF 1 KAT

The object of this research was to establish the depth profile of RS from the segment of shot-peened helical spring *SNCF 1 KAT*. Outer side was shot-peened by intensity corresponding to Almen A = 0.54 and inner side by A = 0.43.

Tensometric measurement was carried out by XRD "one-tilt" method in combination with electrochemical etching. Three areas were analysed, namely on inner (1), outer (2) and lower (3) surface always in the direction perpendicular to the inner axis of the spring (Fig. 4).



Fig. 4. Scheme of the measured areas on the sample.

2.2.1. Results

Residual stresses measured in areas 1, 2, and 3 in the above mentioned direction on the surface and in the depths of 0.1, 0.2, 0.3, 0.4, and 0.5 mm are schematically shown in Figure 5a.



Fig. 5. Depth distributions $\sigma(z)$ of shot-peened helical spring *SNCF 1 KAT* (a) and parabolic spring of *Fiat Ducato* (b).

2.2.2. Conclusions

Distributions of residual stresses obtained on all analysed samples are qualitatively and quantitatively similar.

The biggest compressive residual stresses (-647 MPa, -631 MPa, -639 MPa, -678 MPa) from the depth 0.1 - 0.2 mm under the surface from areas 1, 2, and 3 are identical with respect to experimental error.

The applied shot-peening mode leads to creation of considerable compressive residual stresses in the depth of 0.3 mm beneath the surface which exceed -450 MPa in all measured areas.

2.3. Residual stresses of shot-peened parabolic springs for Fiat Ducato

Samples used in this research were taken from shot-peened parabolic springs used in Fiat Ducato. One spring plate (sample 1) was shot-peened with intensity A = 0.64 and the other (sample 2) with intensity A = 0.78. The industrial partner provided us the information that these parabolic springs exceed the minimal required lifetime of one hundred thousand cycles.

The experimental method applied was the same as in the case of helical spring (chapter 2.1.1.) and results in the form of RS depth distributions are shown in Fig. 5b.

2.3.1. Conclusions

RS depth distributions of both samples resemble qualitatively. Higher shot-peening intensity (sample 2) leads to lower values of surface compressive RS in comparison with sample 1. The maximal compressive RS –1020 MPa and –1067 MPa were measured in the depth of 0.3 mm in sample 1 and sample 2 respectively. Applied shot-peening mode cause considerate RS even in the depth of 0.5 mm, where higher shot-peeing intensity leads to larger absolute values of compressive RS.

2.4. Surface residual stresses of steam generators

Following diffraction analyses were done in order to verify usage of new nickel based alloy Inconel 600 which are to replace widely used austenitic steel in steam generators and nuclear reactors. The surfaces of the studied specimens were treated by either water jet peening (WJP) or fiber laser peeing (FLP) in order to create a prestrained surface layer. XRD measurements were performed before and after the specimens were exposed to environment with similar corrosive and radioactive parameters as in real circumstances.

2.4.1. X-ray diffraction technique

XRD technique " $\sin^2\psi$ " [2] was carried out by θ - θ goniometer X'Per PRO PANalytical with CrK α radiation. The diffraction line {220} of Ni was analysed and RS were evaluated presuming that the state of residual stresses was biaxial. The dependences of $2\theta^{220}$ versus $\sin^2\psi$ were measured in two mutually perpendicular azimuths $\sigma_{\rm T}$ and $\sigma_{\rm L}$.

2.4.2. Results

Results of XRD analysis of two selected specimens before and after their exposure in environment simulating real environment are shown in Table 1.

Used surface treatment	State of the specimen	σ_{T} , MPa	σ _L , MPa
Water jet peening	Before	-442 ± 41	-501 ± 32
	After	-293 ± 24	-320 ± 25
Fiber laser peening	Before	-640 ± 57	-334 ± 37
	After	-436 ± 37	-166 ± 48

Table 1. Macroscopic residual stresses σ_T , σ_L before and after testing of chosen samples

2.4.3. Conclusions

XRD tensometry is a suitable tool for new technologies verification, especially in environments extremely sensitive to creation and propagation of cracks and to effects of radioactivity and corrosion on the material strength such as in the case of steam generators, nuclear reactors or aircraft components.

Both the applied surface treatments, i.e. water jet peening and fibre laser peening, are capable of creation appreciable compressive RS on the surface of nickel based Inconel 600 alloy. Moreover, although the compressive RS do diminish after sample testing, they character remains preserved.

2.5. Verification of new technology in aircraft diffuser production

Hitherto, aircraft diffusers have been manufactured by conventional milling which constitute substantial part of production costs. Progress in machining technologies and gradual sophistication of electro discharge machining (EDM) enabled to use this technique for machining of nickel alloy Inconel 718 which is frequently used in

diffusers production. The application of EDM would lead to considerable cost savings, but several properties of the final product has to be checked. One of the controlled parameters are RS that were investigated by XRD.

2.5.1. Results

The state of RS in the studied samples was determined by " $\sin^2 \psi$ " method and in addition to RS values of lattice parameter were evaluated. Results are to be seen in Table 2.

Used technology	σ _T , MPa	σ _L , MPa	a _e , nm
EDM graphite electrode -finishing	483 ± 64	433 ± 47	0,35973
EDM Cu electrode -finishing	0 ± 15	-1 ± 62	0,36034
EDM graphite electrode - stocking	388 ± 59	292 ± 64	0,35936
EDM Cu electrode - stocking	217 ± 34	321 ± 77	0,35845
Side parallel milling	-798 ± 57	-343 ± 24	0,36167
Side up cut milling	-546 ± 47	$-\!405\pm67$	0,36170

Table 2. Macroscopic residual stresses σ_T , σ_L and values of lattice parameter a_e , determined from
diffraction line {220} of Ni

2.5.2. Conclusions

The biaxial isotropic state of macroscopic stresses was identified by X-ray diffraction analysis in all surfaces cut by electro discharge machining (Table 2).

Significantly higher values of tensile stress obtained by means of EDM in the case of finishing by graphite electrode correspond to the higher thermal load used in process of machining.

Anisotropic state of residual stresses found on the surfaces of milled samples reflects the character of mechanical interaction between the tool and the workpiece.

The results of lattice parameters in surface layers (Table 2) show that EDM leads to distinctively lower values in comparison with milling. It can be assumed that diffusion of atoms of the electrode (graphite, copper) into the surface layers of machined sample is responsible for the difference in lattice parameters.

3. Conclusions

The content of this contribution reveals that the main objective is to give a representative review of XRD tensometry potential for application in industrial research and development. The process of residual stresses generation itself can give useful information about the nature of various processes which take place during material processing. Reliable monitoring of RS during manufacturing can hence minimize future pitfalls. As the XRD belongs to surface experimental techniques, it is most suitable for investigations of surface treatments such as grinding, shotpeening, roller burnishing, milling, etc. In combination with force-free layer removal procedures such as electrochemical polishing, XRD is a powerful tool for study of

real structure gradients as well. Diffraction techniques are not limited by shape and mechanical properties which is a common hindrance for other tensometric methods. In addition to information about the state of RS, XRD is used for determination of qualitative and quantitative phase composition, texture, grain size and other parameters of real structure of polycrystalline materials.

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References

- Kraus I. and Ganev N., "Residual Stress and Stress Gradients," in *Industrial Applications of X-Ray Diffraction*, Chung F.H., Smith D.K., eds. (Marcel Dekker, New York, 2000), pp. 793-811. ISBN 0-8247-1992-1.
- [2] Hauk V. et al., Structural and Residual Stress Analysis by Nondestructive Methods (Elsevier Science B. V., 1997). ISBN 978-0-444-82476-9.