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X-ray diffraction techniques for semi-destructive and non-destructive determination of residual stress depth distributions

Zdenek Pala[1], Nikolaj Ganev[2] & Kamil Kolařík[3]

Abstract: Determination and study of residual stress depth distributions is an important step in the progress of structure description of materials. The depth profiling is often done by combining the chosen X-ray diffraction technique on a conventional laboratory diffractometers in reflection mode and successive layer removal. Semi-destructive layer removal should be done with minimal impact to the structure of the remaining layers; the most widely used technique is electro-chemical polishing. Another possibility for depth distribution investigation is to employ synchrotron radiation in non-destructive transmission mode. In the contribution, a comparison between conventional XRD laboratory and synchrotron experiments is offered.

Keywords: X-ray diffraction; residual stress; synchrotron source; transmission and reflection geometries

1. Introduction

Various surface treatments and surface enhancement processes lead not only to a complex microstructure, but also to a specific state of residual stress. The fact that residual stresses (RS) superimpose with load stresses was the chief reason for their incorporation into the set of parameters branded as *surface integrity*. RS are of special importance in dynamically loaded components where they significantly reduce or enhance the service life. Therefore, the knowledge of RS spatial distribution is needed not only in machine tool production, but in automotive and aerospace industries as well.

A complete determination of RS spatial distribution in a real object is a challenging assignment for either simulation or experimental approach. When the experimental one is pursued, steel objects of up to 30 mm can be measured by neutron diffraction techniques, the most common one is called “strain scanning” [1] and takes advantage of comparatively low absorption of neutrons. However, a fairly large gauge volume of several mm^3 has to be used in order to make this experiment viable. When the component is even larger, either cutting into smaller specimens

[1] *Ing. Zdenek Pala; Fac. of Nuc. Sci. and Phys. Engineering, Czech Technical University in Prague; Trojanova 13, 120 00 Prague, Czech Republic; zdenek.pala@fjfi.cvut.cz*
[2] *prof. Ing. Nikolaj Ganev, CSc.; Fac. of Nuc. Sci. and Phys. Engineering, Czech Technical University in Prague; Trojanova 13, 120 00 Prague, Czech Republic; zdenek.pala@fjfi.cvut.cz*
[3] *Ing. Kamil Kolařík, PhD.; Fac. of Nuc. Sci. and Phys. Engineering, Czech Technical University in Prague; Trojanova 13, 120 00 Prague, Czech Republic; zdenek.pala@fjfi.cvut.cz*

accompanied by strain redistribution and/or relaxation is performed in combination with diffraction techniques or the inherently destructive contour [2] or deep hole drilling [3] methods have to be employed.

Due to appreciable developments of X-ray diffraction techniques during the last decade, they are a suitable tool for analyses of RS spatial distribution in the cases when surface areas of several hundreds of micrometres are of interest. The depth profiling is most frequently done by combining the chosen X-ray diffraction technique on a conventional laboratory diffractometers configured in reflection mode and successive layer removal. Semi-destructive layer removal should be done with minimal impact to the structure of the remaining layers; the most widely used technique is electro-chemical polishing. Another possibility for depth distribution investigation is to employ synchrotron radiation. Energies of X-ray photons from a synchrotron can be chosen in a wide spectrum with maximum exceeding 100 keV, thus, facilitating RS measurements in non-destructive transmission mode in which the beam penetrates through the whole sample. In the most advanced synchrotron sources, the cross-section of incoming X-ray beam can be focused down to several square μm which is especially useful when extremely steep gradients are anticipated. In this contribution, a comparison will be done between RS depth distribution in steels measured in reflection geometry by X-ray diffraction technique combined with polishing and in transmission geometry obtained at synchrotron source.

2. Effective penetration depth of X-rays

The gauge or irradiated volume during diffraction measurements is given by the irradiated surface area, which is directly set by diffractometer slit systems, and by the so called penetration depth. Traditionally and most frequently, this quantity is described by the *effective penetration depth* T_{ef} [4] that determines the thickness of a layer providing 63.2 % out of the entire diffracted intensity. T_{ef} is given by the absorption μ of the given diffracting volume for the impinging X-ray beam wavelength, the Bragg angle θ and by the geometric alignment of the goniometer. The structural information gained from classical Bragg-Brentano goniometer changes with the changing 2θ angle and is, therefore, influenced by possible steep structural gradients. To illustrate this, we present in Fig. 1 detailed analysis of penetration depth for the most commonly performed residual stress determination in ferritic steels. For the detailed description of the experiment geometry, especially in respect to the sample tilt ψ and sample azimuth φ, see e.g. [5].

In the transmission geometry, a vital role is played by the sample's transmittance, or the ratio of photons which are not absorbed by the sample of given thickness. Transmittance is a function of μ and the thickness of the sample in the direction of the beam path. Since the absorption is wavelength dependent and synchrotron sources have different fluxes for different photon energies, the final choice of wavelength and maximal thickness has to be decided according to the detector efficiency. The general tendency is to ensure the maximal thickness in order to avoid cutting of the sample or at least to limit the influence of the sample volume impacted by the sectioning.

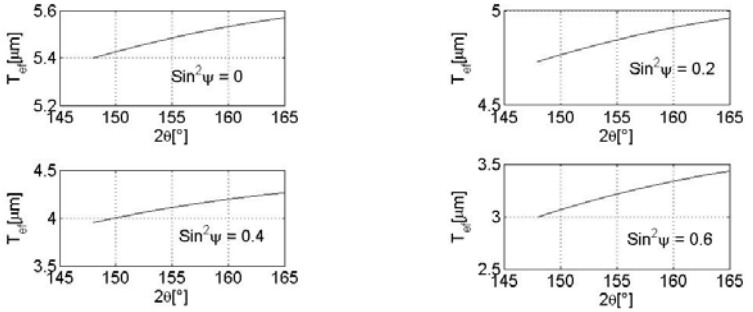

Fig. 1. Penetration depths T_{ef} as functions of sample's tilt ψ and 2θ. The calculations are done for {211} α-Fe diffraction line measured in Bragg-Brentano semifocusation by CrKα radiation. We considered the so called ω goniometer [7] when the interplanar lattice spacing is measured for various orientation of {211} planes in respect to the sample's surface given by the tilt ψ while the tilt and Braggs' angles are in the same plane. Value of absorption coefficient μ = 890.1 cm^{-1} was calculated from table in Appendix 8 of [4].

3. Sample and experimental techniques

Macroscopic residual stresses depth distribution was analysed in a samples made from high carbon martensitic chromium steel M300 (X36CrMo17). Both bases of rectangular plates with dimensions 50×50×5.5 mm^3 were subjected to finish surface grinding in order to introduce triaxial state of residual stress with appreciable shear stresses in the subsurface layers. Transmission ratio for 50mm thick iron plates and photons with energy of 130 keV is of the order of 10^{-5} which would represent a beamstop. Hence, the samples measured with synchrotron radiation had to be cut to cuboids with dimensions 12×12×5.5 mm^3; in this case the transmission ratio is approximately 0.092.

In the X-ray diffraction laboratory, the sample was investigated by analysing {211} diffractions of α-Fe with CrKα radiation (λ = 2.29106 Å) and ω-diffractometer equipped with a scintillation detector. Dölle and Hauk method was implemented for calculation of the strain tensor [6] and the calculation of stress tensor was done from the strain tensor by using the generalized Hooke's law [5] with X-ray elastic constants calculated according to the Eshelby-Kröner method [7] for the measured α-Fe {211} diffraction planes. For the surface layer removal, an apparatus for automatic, micro-processor controlled electrolytic polishing and etching of metallographic specimens, was used. In the synchrotron laboratory, the diffraction patterns in the form of Debye rings [8] were detected by large detector High Energy Detector Array (HYDRA) consisting of an array of 4 amorphous silicon detectors which provide a much larger size and corresponding maximum radius than using a single detector. In order to gain sufficient intensities of Debye rings, photons with energy of 130 keV (λ = 0.09537 Å) were scattering in volume defined by setting the primary slits to 10 µm × 200 µm. Measuring time in each depth was 4 s and the step of depth profiling was set to 10 µm.

4. Results

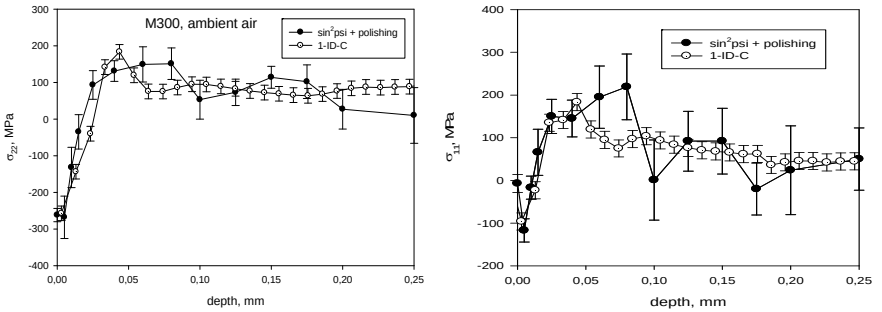

Fig. 2. Macroscopic residual stress components σ_{11} (in the grinding direction) σ_{22} (the transverse direction to grinding) of *M300* steel sample; comparison of synchrotron measurements and an approach combining modified $sin^2\psi$ method with successive electro-chemical polishing.

5. Conclusions

Depth distribution of residual stresses as an important surface integrity parameter can be determined either semi-destructively by combining laboratory X-ray diffractometer with polishing or non-destructively when synchrotron beam is available. Measuring of ground steel sample by both techniques yields similar depth distributions and there is no pronounced difference between the method involving polishing and the method for which the sample had to be sectined. Both experimental approaches are useful for evaluation of surface enhancement treatments with affected zone up to 1 mm.

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