CHARACTERIZATION OF SHOT PEENED COMPONENTS BY X-RAY DIFFRACTION: A METHOD ON ITS WAY FROM THE LABORATORY INTO INDUSTRIAL PRODUCT DEVELOPMENT

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ABSTRACT
A main beneficial effect of shot peening is the creation of compressive residual stresses. Thus, reliable and accurate methods for determining residual stresses are needed to allow controlling the process, verifying models and assessing strength mechanisms. Since a long time X-ray diffraction (XRD) has been the choice method for basic residual stress investigations. Due to missing a norm or practice guides being internationally accepted, this method has been hindered to be also the first choice in case of acceptance tests.

This gap is now going to be closed. A European standard for residual stress determination using X-ray diffraction has been evaluated by an expert group and will be available shortly. This paper summarizes the basic principles of the method, sketches the range of applications covered by the norm and explains some of the limiting cases defined by the norm. Special emphasis is given to the relevance of the norm for residual stress investigations on shot peened materials.

SUBJECT INDEX
X-ray, diffraction, residual stress, standard, quality control.

INTRODUCTION
Since the first papers about using X-ray diffraction as a tool for determining stresses where published in the early 20th century, this technique has become one of the most popular methods currently used for the analysis of residual stresses in crystalline materials. In addition, it is assumed to be the most precise method compared to other popular methods like the hole drilling technique (Lord, 2002). Within the last years several groups in, e.g., France, Germany and Great Britain developed national practice guides for the X-ray stress measurement technique. The commonly agreed bodies of these practice guides are the basis for a new European standard. The discussions in the working group (Technical Committee CEN/TC 138 "Non-destructive testing", WG10) clearly showed that XRD in general is not the push-of-a-button method and therefore the standard procedures as well as the limiting cases have to be defined carefully. Nevertheless, in addition to the standardized measurement and evaluation procedure – the possibility for a verification of economic measurement procedures had to be included to allow industry to widely apply the method for product development and quality control. The resulting draft for the new European standard (WI 00138097) outlines this presentation. The basic principles of the method and the limitations are summarized.
Special emphasis is given to practical aspects of testing of real components and to strategies for economic measurements.

**BASIC PRINCIPLES OF X-RAY STRESS ANALYSIS**

The basic principles of the X-ray residual stress analysis are well known and described in detail elsewhere (Hauk, 1997). The technique is based on the measurement of the angular shift of diffraction lines being caused by stresses. The principle of the experimental set-up and diffraction is sketched in Fig. 1. Bragg's law gives the relationship between the spacing $d$ of lattice planes of the crystallites of a polycrystalline material and the angular position $\theta$ of diffraction lines using monochromatic X-rays of wavelength $\lambda$ (n= whole-numbered multiple):

$$2d \sin \theta = n \cdot \lambda$$

(1)

The orientation of the examined lattice planes with respect to the specimens coordinate system is defined by the angles $\varphi$ and $\psi$ of the normal of the lattice planes with respect to the orthogonal sample system $S_1$, $S_2$ and $S_3$ ($S_3$ is the normal of the specimen).

![Fig. 1: Sketch of the (iso-inclination) measurement set-up for stress determination by X-ray diffraction.](image)

Stresses change the lattice spacing $d$ und thus also the diffraction angle $\theta$. The dependency between the relative change of the lattice spacing ($=\text{lattice strain } \varepsilon_{\varphi\psi}$) and the peak shift is given by:

$$\varepsilon_{\varphi\psi}^{(M)} = \ln \left[ \frac{d_{\varphi\psi}}{d_0} \right] = \ln \left[ \frac{\sin \theta_{\varphi\psi}}{\sin \theta_0} \right] = \ln[\sin \theta_{\varphi\psi}] - \ln[\sin \theta_0]$$

(2)

On the basic theory of elasticity, for a macroscopically isotropic crystalline material the formula to express the strain in the direction defined by the angles $\phi$ and $\psi$ is:
where $S_{ij}^{(hkl)}$ and $\frac{1}{2}S_{ij}^{(hkl)}$ are the X-ray elasticity constants for the family of lattice planes $\{hkl\}, \sigma_{11}, \sigma_{22}, \sigma_{33}$ are normal stress components in the directions $S_1, S_2$ and $S_3, \tau_{12}$ is the shear stress within the plane defined by $S_1$ and $S_2, \tau_{13}$ is the shear stress within the plane defined by $S_1$ and $S_3$ and $\tau_{23}$ is the shear stress within the plane defined by $S_2$ and $S_3$.

The determination of the stress components is performed through the measurement of a sufficient number of strain components at different angles $\phi$ and $\psi$ followed by a fit of the strain distribution using the stress components as parameters to be optimized.

In case of shot peened materials generally the shear stresses $\tau_{12}$ and $\tau_{23}$ are negligible and a plot of the strain components $\varepsilon_{\psi\psi}$ versus $\sin^2\psi$ ($\phi = \text{constant}$) can be fitted by a straight line (see Fig. 2, (a)). This is the well known $\sin^2\psi$ - method (Müller, 1961).

**LIMITING CASES FOR STATE OF THE ART STRESS EVALUATIONS**

The new European standard defines the following limitations for state of the art measurements:

1. Stress gradients within the diffracting volume;
2. Lattice constants gradients within the diffracting volume;
3. Coarse grain materials;
4. Highly textured materials;
5. Multiphase materials;
6. Surface roughness $R_s$ should be lower than the minimum average information depth;
7. Non-flat surfaces: The irradiated area should be smaller than 0.4 times the radius of curvature of the analysed surface in the direction of the stress component to be determined;
8. Overlapping diffraction lines;
9. Broad diffraction lines.

The limiting cases No. 1 – 4 can easily be detected by inspection of the $\varepsilon_{\psi\psi} - \sin^2\psi$ distribution (see Fig. 2).

(1) In case of shot peened materials, the effect of stress gradients are in general less important due to the small penetration depth of X-rays. Nevertheless, different attempts to handling such situations have been presented (Hauk, 1997).

(2) Lattice constant gradients are present if, e.g., shot peening is performed on materials with a gradient of composition. Methods to correct such effects are available (Prußener, 1983).

(3) Coarse grains in general do not affect stress measurements of shot peened materials as the introduced plastic deformation dramatically reduces the domain size. But, approaching the non-deformed substrate, an increasing effect of large domain sizes
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may be obtained. In most cases this problem can be solved by increasing the number of diffraction crystallites through translations and/or angular oscillations of the specimen, by using lattice plains with higher multiplicity factor or by using X-rays with a higher penetration depth.

(4) Highly textured materials comprise the more complicated problems in X-ray stress analysis. The available attempts to treat severe textures are based on the additional determination of the orientation distribution function (ODF) and applying assumptions concerning the elastic interaction between the crystallites, see, e.g. (Hauk, 1997). In many cases of shot peening induced textures, the effects are less dominant and can be simply reduced by optimizing the experimental set up (using a larger range of \( \psi \) - angles and/or using lattice planes with a high multiplicity factor).

(5) The uncertainties resulting from multiphase materials are often underestimated and the reason behind diverging stress results derived from, e.g., XRD and hole drilling measurements. XRD - measurements are performed on specific lattice plains belonging to one single phase. In case of multiphase materials, different stress states may exist in different phases which partially compensate. Thus, the evaluation of the mean macroscopic stress state needs the determination of stress components of all significant phases and the calculation of the volume-weighted average of all phase-specific stresses. Remember that one of the most important materials, hardened steel, is such a multiphase material consisting of - among other things - ferrite, cementite, carbides and retained austenite.

(6) - (9) The limiting cases No. 6 through 9 are not explained in detail here as the problems resulting from those conditions generally can be solved by experienced experimenters.

**Fig. 2:** Regular and “limited case” \( \sin^2 \psi \) distributions: (a) regular distribution with (dashed line) and without shear stress components (solid line), (b) stress and/or lattice constant gradients, (c) texture, (d) coarse grains. Mixtures are possible.

REPRODUCIBILITY AND ACCURACY

Reproducibility

The reproducibility of X-ray residual stress measurements has been stated through a number of national and international round robin tests. Astonishingly the reproducibility has not significantly been improved through the last 30 years. A German round robin test (12 participants) on hardened ground steel in 1982 resulted in a reproducibility of ± 40 MPa for stress states amounting 460 up to 670 MPa (FA Spannungsmessbeechnik, 1985). In 2002 a European round robin test (12 participants) under comparable conditions on hardened and shot peened steel resulted in a reproducibility of ± 79 MPa.
for a stress level of 480 MPa (Gibmeier, 2002). The latter and other round robin tests (François, 2000, Ferreira, 2002, Fry, 2002) indicated that, besides the influence of the operator, the use of different positioning methods contributes most to the scatter of the results. Performing a recalculation of the round robin raw data using a uniform peak treatment reduced the scatter by 50%. Anyhow, the new European norm does not give a distinct recommendation concerning the use of appropriate peak treatment methods. The reason behind may be that, recommending specific peak treatment methods, may improve the reproducibility of stress measurements performed at different laboratories but does not answer the question what the accurate stress value may be. Under certain circumstances the dependency of the stress results on the peak treatment may be related to the material characteristics and the stress state (Pfeiffer, 1994). Thus, it is advantageous to have software available, which, by using different peak treatment methods, points out a potentially existing influence of the data treatment on the result. Then it is helpful, if assistance for the interpretation of the dependencies and the selection of appropriate treatment parameters is provided.

**Accuracy**
The accuracy of the stress evaluation is affected by different factors. A certain stress value may be reproduced by different labs with a high reproducibility but this value still may differ from the true stress amount. It is obvious that the so-called X-ray elastic constants (XEC) directly determine the stress value calculated from the measured lattice strains. The XEC may be calculated on basis of the single crystal constants of elasticity or can be measured using loading experiments. In general, the effect of the phase composition, the texture and of fabrication procedures on the XEC is more significant compared to the effect on the macroscopic constants of elasticity (Hauk, 1997). In order to evaluate accurate stress amounts by XRD, reference specimens with well known XECs are needed and the residual stress state has to be verified by other methods. Round robin tests providing such specimens are on the way, see e.g. current activities in VAMAS TWA20. The principle procedure for creating such reference samples is fixed in the new standard.

**ECONOMY**
The advantage of a reliable stress measurement method will be exploited by industry only if the measurements can be performed in a satisfying timeframe. The “Normal Procedure” described by the new standard will not generally fit these needs. In order to allow less time consuming measurements under the framework of the norm a so called “Dedicated Procedure” has been defined. The Dedicated Stress Measurement Procedure can be applied to series of very similar specimens (routine analyses). Specimens are regarded as “very similar” if the differences between their stress states (not the stress values), their chemical and phase compositions, their texture, their microstructure are expected to be insignificant for the stress values to be determined. For a series of such specimens a laboratory can define and describe a Dedicated Stress Measurement Procedure. In order to conform to the norm such a Dedicated Stress Measurement Procedure has to be validated through the execution of the Normal Procedure.
CONCLUSION
X-ray diffraction is a valuable tool for the determination of residual stresses. The technique is widely accepted for a broad range of applications in materials science. The acceptance of the method for quality control purpose and design in mechanical engineering had been derogated through the lack of a transnational accepted procedure. This gap has been now closed by the evaluation of a European norm. This norm clearly separates the commonly accepted state of the art of XRD-stress measurements methods from the limiting cases, for which the existence of uncertainties are well known but not yet eliminated. For state of the art measurements a normal and a dedicated procedure are defined which should allow determining stress states with high accuracy and reliability while fulfilling the economic needs of industrial research and quality control. Thus, an important and reliable tool for quantitative residual stress engineering will be available shortly.

ACKNOWLEDGMENTS
The author would like to thank all members of WG10 who contributed to the WI 00138097 standard for the very procreative discussions and cooperative work.

REFERENCES
VAMAS TWA20, proposed project No. 3, contact jerry.lord@npl.co.uk.