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Energy Resolved Residual Stress Analysis with Laboratory X-Ray Sources

Eigenspannungsanalysen unter Verwendung energieauflösender Detektoren und Laborröntgenquellen

Kurzfassung/Abstract

It is well known that existing residual stress fields play an important role for strength and lifetime of components. Consequently there is a great interest in the availability of fast, reliable and possibly nondestructive methods for their determination. In this context, X-ray diffraction methods play an important role in technical practice as well as in scientific research. They are based on the determination of lattice strains from which residual stresses are determined applying Hooke's law with appropriate elastic constants. In this paper – after a short survey of the basic principles – characteristic features of energy resolved methods for laboratory applications compared with angle resolved methods are outlined. A corresponding measuring device is presented and characteristic examples are given to demonstrate the possibilities and limitations of the method. ■

Keywords: Residual stress, energy resolved analysis, nondestructive testing, surface treatment, gradients

Aufgrund der Bedeutung, die Eigenspannungen für die Zuverlässigkeit und Beanspruchbarkeit von Komponenten besitzen, besteht ein großes Interesse an der Verfügbarkeit schneller, zuverlässiger und möglichst zerstörungsfreier Messverfahren. In diesem Zusammenhang kommt heute röntgenographischen Verfahren eine besondere Bedeutung in der Praxis zu. Sie basieren auf der Messung von Gitterdeformationen, aus denen unter Verwendung elastischer Konstanten Spannungen berechnet werden. In der vorliegenden Arbeit wird – nach einer kurzen Einführung in die Grundlagen – gezeigt, welche Besonderheiten bei energieauflösenden Verfahren im Vergleich zu den etablierten winkelauflösenden Verfahren bei der Anwendung im Labor bzw. einem industriellen Umfeld existieren. Ein entsprechendes Gerät mit seinen Möglichkeiten wird vorgestellt und anhand kennzeichnender Beispiele werden die zurzeit bestehenden Möglichkeiten und Grenzen energieauflösender Eigenspannungsanalysen aufgezeigt ■

Schlüsselwörter: Eigenspannungen, energiedispersive Analyse, zerstörungsfreie Prüfung, Oberflächenbehandlung, Gradient

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1 Introduction

In the field of industrial engineering, a well-known fundamental principle is that manufacturing induced residual stress states have a considerable influence on strength and lifetime of components [1–3]. There is a great and still increasing demand to include existing residual stress distributions into design rules. Consequently for the full exploitation of the potential of highly and complexly loaded components, e. g. for light weight constructions, the exact and reliable determination and assessment of existing residual stress distributions is crucial. The correct consideration of residual stress states already in the design phase of components is an important step towards short production cycles, to increase the degree of safety and reliability of relevant parts as well as for the economical use of materials. Hence, the availability of fast, reliable and efficient methods for residual stress analysis in the future will be a key factor for the successful manufacturing of advanced materials and structures and for the reduction of production costs [4–6].

Different types of methods for the analysis of residual stress states have been developed in the past, based on the physical consequences of residual stresses in materials and components. Typical methods, which are well accepted and widely used for industrial applications are diffraction techniques (X-ray diffraction, neutron diffraction), mechanical methods (sectioning techniques, hole drilling etc.), but also ultrasonic techniques as well as magnetic methods have been developed and applied with success [3, 7, 8]. For the X-ray diffraction technique as well as for the hole drilling method, national or European standards of good measurement

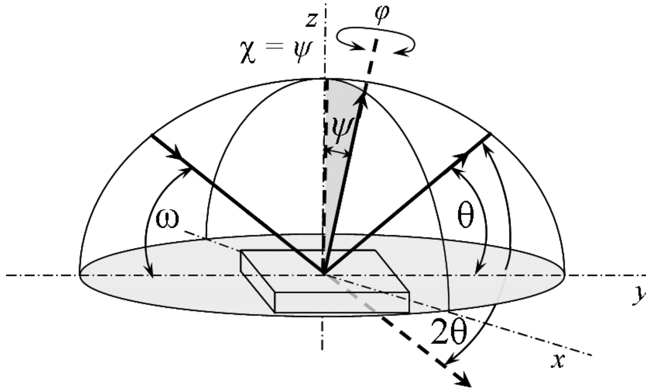

Fig. 1. Coordinate system and labeling of angles

Bild 1. Verwendetes Koordinatensystem und Winkelbezeichnungen

practice have been worked out. They describe the measurement procedure and possible errors of both techniques, however, only for relatively simple standard cases [9–12]. In many practical cases, e. g. for mechanically surface treated or machined components or after surface hardening, not only surface values but complete depth distributions of residual stresses in the processed surface layers are of interest. Consequently, methods allowing depth resolved residual stress analyses are of particular interest.

X-ray diffraction, which is established as a standard process for residual stress analysis, is based on the determination of lattice strains of polycrystalline materials in well defined orientations with respect to the specimen system of coordinates [13, 14]. Lattice strains $\varepsilon_{\varphi,\Psi}^{hk\ell}$ measured at {hkl}-planes can be calculated using

$$\varepsilon_{\varphi,\psi}^{hk\ell} = \frac{d_{\varphi,\psi}^{hk\ell} - d_0^{hk\ell}}{d_0^{hk\ell}} \tag{1}$$

$d_{\varphi,\Psi}^{hk\ell}$ is the lattice distance in a direction given by φ, Ψ and $d_0^{hk\ell}$ is the lattice distance of the stress-free state. The measuring geometry and the significance of the angles φ, Ψ is explained in Figure 1.

Then from the measured lattice strain distributions residual stresses are determined using theory of elasticity with appropriate diffraction elastic constants [15]. Generally, two possibilities exist to measure lattice strains by X-ray diffraction which, both, are based on Bragg's law: angle resolved X-ray scattering with quasi-monochromatic X-rays (characteristic radiation of X-ray tubes with appropriate anode elements and, if appropriate, complemented by monochromator systems), which leads to the expression

$$\varepsilon_{\varphi,\psi}^{hk\ell} = -\Delta\theta_{\varphi,\psi}^{hk\ell} \cdot \cot\theta_0^{hk\ell} \tag{2}$$

$\theta_{\varphi,\Psi}^{hk\ell}$ and $\theta_0^{hk\ell}$ are the directions of the diffracted beams of the material under investigation with residual stresses and in the stress-free condition, respectively. For the case of energy resolved diffraction

$$\varepsilon_{\varphi,\psi}^{hk\ell} = \frac{E_0^{hk\ell}}{E_{\varphi,\psi}^{hk\ell}} - 1 \tag{3}$$

is valid. E is the energy of the diffracted beam depending on the wavelength of the X-rays. Here X-ray synchrotron radiation or bremsstrahlung of X-ray tubes with e. g. W-anodes can be used. Both methods are schematically sketched in Figure 2. In each case appropriate procedures to determine the interference line positions have to be applied. For reliable residual stress analyses a strain resolution of at least $\Delta d^{hk\ell} / d_0^{hk\ell} = 10^{-4}$ has to be achieved. This implies that for angle resolved X-ray scattering an angular resolution of $\Delta\Theta = 0.005°$ and for energy resolved scattering an energy resolution of $\Delta E = 1$ eV is required [16, 17]. For the complete analysis of the full stress tensor, exact values of the lattice parameter $d_0^{hk\ell}$ of the stress–free state are crucial.

Due to the well-known Lambert-Beer law the wavelength of the used X-rays, the material investigated and the path of the incoming and diffracted beam within the material investigated determine the information depth of the respective measurements. In principle, the information measured can be confined to small volumes by appropriate mask systems [14, 18, 19]. In all cases, how-

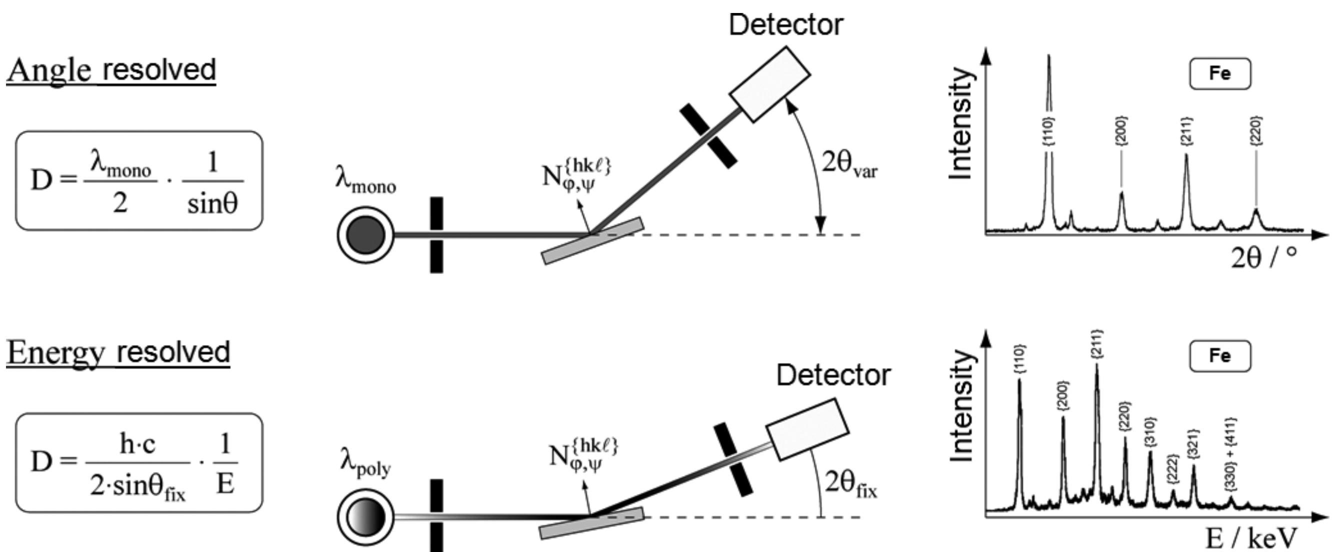

Fig. 2. Schematic representation of angle resolved and energy resolved diffraction experiments

Bild 2. Schematische Darstellung winkelauflösender bzw. energieauflösender Verfahren zur Eigenspannungsanalyse

ever, the lattice strains measured are weighted averages of the real lattice strain distributions within the probed materials volume. This is of paramount importance for the case of very steep near surface gradients as e. g. for thin layers or ground surfaces.

For the analysis of residual stress depth distributions different strategies exist. In industrial practice, successive electrolytic layer removal and measurements using the conventional $\sin^2\Psi$-method at the respective new surfaces, and thus destructive methods, are applied. This is a time consuming procedure and, in addition, residual stress relaxation processes have to be taken into consideration. Nondestructive methods can be divided in real-space and Laplace-methods. In the first case, the measured volume is restricted by appropriate aperture systems to gain information from the volume of interest only [14, 18, 19]. In the second case, methods are based on the fact that, due to the exponential attenuation of the applied radiation, in all cases weighted averages of the lattice strains within the penetration depth of the X-rays used are gained. Consequently differences exist between the measured depth distributions $\sigma_{ij}(\tau)$ and the desired distributions $\sigma_{ij}(z)$ (z is the distance from surface and τ is the mean penetration depth of the X-rays used). The transfer of the measured distributions $\sigma_{ij}(\tau)$ to the required values $\sigma_{ij}(z)$ is achieved by an inverse Laplace-transformation, which, however, not always provides stable and reasonable solutions [20]. Different approaches are described in literature. The variation of the information depth of the measurement is realized by different wavelengths of the X-rays used or by appropriate geometrical arrangements yielding different beam paths of the X-rays within the material investigated. In all cases data treatment following the universal plot method proposed in [21] is useful. Comprehensive surveys about the different strategies available to calculate residual stress fields from measured lattice strain distributions can be found in e. g. [14, 22–24].

2 Key Factors of Energy Resolved Residual Stress Analysis under Laboratory Conditions

Although both methods are basically equivalent, in practice by far most of the residual stress analyses today are carried out on the basis of angle resolved instruments and energy resolved X-ray diffraction is less common. Originally this was due to the fact that the angular resolution of available detectors was much better than the unsatisfactory energy resolution of energy resolved detectors. First successful attempts to determine lattice distances by energy resolved diffraction experiments are described in [25, 26]. Most of the work published in this field since then deals with the use of white high energy synchrotron radiation. In this context, groundbreaking strategies outlined in [27–29] have to be mentioned applying the universal-plot method to analyze stress-depth distributions. Further systematic studies with regard to measuring and evaluation strategies can be found in e. g. [30–32]. In this context also special technical aspects of energy resolved detectors [33] were investigated.

A fundamental advantage of energy resolved diffraction is that measurements are carried out under fixed 2Θ-directions and, hence, no rotation around the corresponding diffractometer axes during measurement is necessary. In this way one single measurement allows the analysis of numerous {hkl}-planes and their interference lines. This large amount of information is especially useful in case of structural, texture or line profile analyses. In addition, measurements under complex geometrical restrictions can be realized due to the lack of angular rotations. For residual stress analyses, simultaneous measurements at different {hkl}-planes allow the realization of different information depths during one single experiment. Hence, the possibility exists for a completely non-destructive qualitative assessment or even a quantitative determination of residual stress depth distributions in near surface layers. This opens up interesting prospects for quality control in industrial processes, when reliable information about near surface residual stress fields is mandatory, as e. g. in the case of machined or heat treated parts [34].

Energy resolved diffraction experiments are commonly carried out at synchrotron facilities. Due to the restricted access to the corresponding beamlines, however, there is an increasing interest in energy resolved residual stress analysis under laboratory conditions or even on the factory floor. Compared with synchrotron radiation experiments laboratory energy resolved residual stress analyses are subjected to quite different boundary conditions due to significantly reduced X-ray intensities as well as increased beam divergence. This requires special measures to reduce measuring times. In Figure 3 a technical drawing of a laboratory diffractometer for energy resolved X-ray diffraction is shown. The device, which is assembled in the laboratory of Institute of Materials Engineering at University of Kassel is based on a four-circle-diffractometer with a closed eulerian cradle and a φ-circle. The X-ray tube is horizontally arranged. In the center, there is a sample holder allowing controlled x-y-z-displacements of the samples. The eulerian cradle together with the sample holder can additionally be rotated around a vertical axis to allow a complete rotation of the measurement configuration around the scattering vector. In this way, all angular positions necessary for the application of the different measurement strategies for residual stress analysis mentioned above can be realized. It is interesting to note that two detector systems, which can be horizontally inclined, are assembled direct-

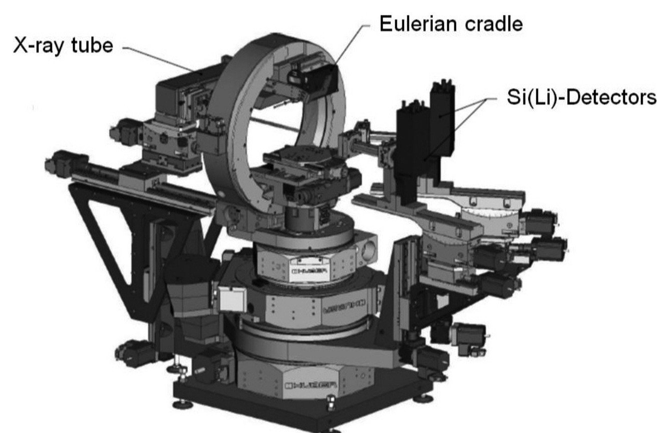

Fig. 3. Technical drawing of a laboratory diffractometer for energy resolved X-ray diffraction (technical realization: Huber Diffraktionstechnik Gmbh & Co. KG, Rimsting)

Bild 3. Technische Zeichnung eines Labordiffraktometers für die energieauflösende Röntgenbeugung (Herstellung: Huber Diffraktionstechnik Gmbh & Co. KG, Rimsting)

ed to the single X-ray source. This allows measuring strategies with minimized total measuring times. If all appropriate angles are adjusted, the sample only has to be tilted during a measuring procedure. For energy resolved residual stress analyses usually small 2Θ-ranges are used. Hence, in the present case, the maximum achievable 2Θ-value is 32°. Due to constant 2Θ-values during the measurement, the required angle resolution of the diffraction device is low. A calibration measurement carried out at a norm powder is enough to determine the exact diffraction conditions. Nevertheless the low diffraction angles require flat and smooth specimen surfaces. On the other hand, as mentioned above, the analysis of energy values requires a high energy resolution of the detector system, which decreases with higher X-ray energies because of absorption characteristics of the analyzing crystal. Eventually, with the device shown in Figure 3, also angle resolved measurements can be carried out. There is enough space and flexibility in the primary and secondary beam path to implement additional optical components. Finally a special camera system together with a laser triangulation device is used for exact sample positioning [35].

The quality of the X-ray sources and detectors used are crucial for fast and reliable energy resolved residual stress analysis [24, 33]. State of the art is the use of X-ray tubes with W-anodes providing a bremsspectrum of high intensity and a broad energy range. Less common, but by far more powerful and providing a high beam intensity are metaljet X-ray sources [36]. In the past, complex detector systems with limited energy resolution were the most detrimental bottleneck for energy resolved residual stress analyses in laboratories or in an industrial environment. In addition, energy resolved X-ray detectors were heavy and big and had to be continuously cooled with e. g. liquid nitrogen. Thus the necessary geometric arrangements and angle settings for strain measurements in real components were difficult to realize. Nowadays, however, new detector concepts are available, which overcome these restrictions and have a sufficient energy resolution. A survey about different types and principles of detector systems can be found in [37]. In [38] a compact Si(Li)-semiconductor detector is presented with a Peltier-cooling system covering X-ray energies up to 60 keV. It has, however, to be pointed out that commercially available detector systems cannot be applied without special consideration of their e. g. temperature and spectral sensitivity or dead time behavior (see e. g. [24, 33]).

3 Examples of Energy Resolved Residual Stress Analyses under Laboratory Conditions

In case of energy resolved diffraction, X-ray intensities over the whole wavelength or energy range of the detector system are recorded simultaneously and hence, all diffraction peaks within this range are available for evaluation with one measurement only. A characteristic example is given in Figure 4. The diagrams were measured at Fe-powder using the radiation of a W-anode and a Si(Li)-detector of the type X-ray Si(Li) Detection Unit SXRD-05150 with Peltier cooling system (Baltic Scientific Instruments). In addition to the different diffraction peaks also characteristic X-ray fluorescence peaks are visible. Measurements were carried out under two different 2Θ-directions, which leads to a characteristic energy shift of the diffraction peaks, while the positions of the fluorescence peaks remain unaffected. For the probed depth both the geometry of the beam path as well as the wavelength or energy of the diffraction peak is of interest and can thus be adjusted by the 2Θ-directions applied. In this way an optimized 2Θ-direction can be chosen which leads to a spectrum with many, however, not overlapping diffraction peaks.

In Figure 5 measurements of angle resolved diffraction (right) are compared with results of an energy resolved experiment (left). The object of the experiment was the running surface of a used roller bearing made of quenched and tempered steel 100Cr6 and measurements were carried out in running direction. For energy resolved diffraction at 2Θ = 20° the energy positions of {200}-peaks vs. $\sin^2\Psi$ are plotted while for angle resolved diffraction using Crkα-radiation the peak positions 2Θ of {211}-planes are plotted as a function of $\sin^2\Psi$. These lattice plains were chosen because of their similar penetration depths of about 4.91 µm (energy resolved diffraction) and 4.61 µm (angle resolved diffraction). For both methods, as expected, linear distributions are determined and from their slope the corresponding residual stress

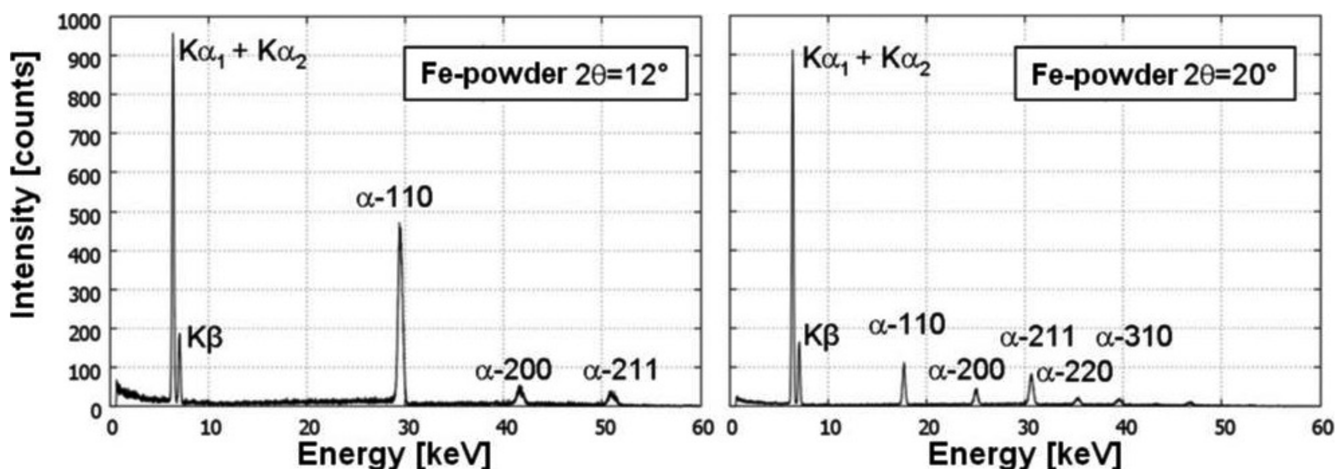

Fig. 4. Diffraction peak positions of Fe-powder for 2Θ = 12° (left) and 2Θ = 20° (right). Measurements were carried out using a W-anode (60 kv, 45 mA) and a Si(Li)-detector. Fluorescence peaks are also visible.

Bild 4. Interferenzliniendiagramm von Fe-Pulver für 2Θ = 12° (links) und 2Θ = 20° (rechts). Die Messungen wurden mit einer Röntgenröhre mit W-Anode (60 kV, 45 mA) und einem Si(Li)-Detektor durchgeführt. Fluoreszenz-Peaks sind ebenfalls gezeigt.

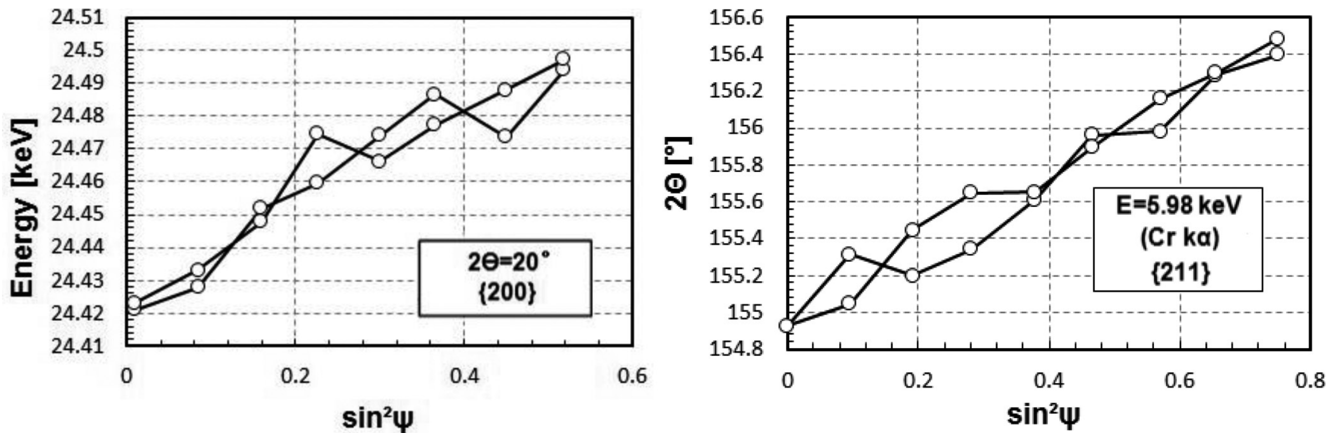

Fig. 5. Diffraction peak positions as a function of sin²Ψ determined in rolling direction at the running surface of a used roller bearing made of quenched and tempered steel 100Cr6 for energy resolved diffraction (left) and angle resolved diffraction (right)

Bild 5. Interferenzlinienlagen als Funktion von sin²Ψ ermittelt in Überrollungsrichtung eines Wälzlagers aus martensitisch gehärtetem und angelassenem 100Cr6 für energieauflösende (links) und winkelauflösende Röntgenbeugung (rechts)

values can be calculated. The diffraction elastic constants $s_1(200) = -2.03 \times 10^{-6}$ MPa^{-1}, $\frac{1}{2}$ $s_2(200) = 8.09 \times 10^{-6}$ MPa^{-1}, $s_1(211) = -1.27 \times 10^{-6}$ MPa^{-1} and $\frac{1}{2}$ $s_2(211) = 5.81 \times 10^{-6}$ MPa^{-1} were used. In case of energy resolved measurement, −698 ± 7 MPa is determined while for the case of angle resolved measurement −656 ± 4 MPa is found. The discrepancies between both values can possibly be attributed to the fact that different volumes are probed in both cases caused by different diffraction conditions and, hence, due to the expected steep residual stress depth gradient, different mean values of the near surface residual stresses in the measured volumes are determined. If, however, all the different diffraction peaks measured by an energy resolved diffraction experiment are included in the analysis, as mentioned above, a depth-sensitive information can be gained. Here different approaches are possible. Below, results of the multi-wavelength method (in Ψ-mode) and the Ω-χ-φ-method are compared. Applying the multi-wavelength method the different diffraction peaks of a spectrum are evaluated by the conventional sin²Ψ-method [24]. For each diffraction lattice plane one (mean) residual stress value can be determined at the corresponding energy dependent mean penetration depth. Applying the Ω-χ-φ-method, using appropriate combinations of the respective instrument angles (see Figure 1), a constant information depth during the whole measuring process is realized because the length of the X-ray beam path within the probed material is kept constant [39, 40]. Also in this case, the residual stresses are determined according to the sin²Ψ-method. To reach constant information depths between 0.5 µm and 2 µm of the first diffraction peak {110}, ω is varied between 0.891° and 8.938°, χ is varied in the range of 0° and −60° and φ is chosen between 1.476° to 206.075° based on the relation

$$\frac{\tau \cdot \mu(E)}{\cos(\chi)} = \frac{\sin(2\theta - \omega) \cdot \sin(\omega)}{\sin(\omega) + \sin(2\theta - \omega)} \quad (4)$$

The near surface depth distributions of residual stresses nondestructively determined in rolling and in transverse direction

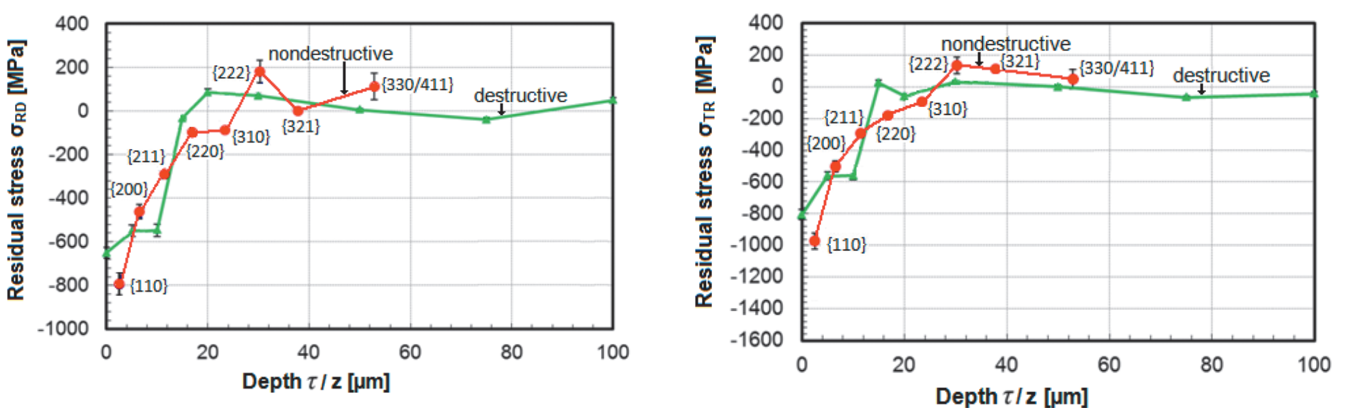

Fig. 6. Near surface depth distributions of residual stresses nondestructively determined in rolling (RD, left) and in transverse direction (TR, right) of the sample investigated in Fig. 5 applying the multi-wavelength method. The results are compared with data of experiments using the conventional destructive sin²Ψ-method combining electrolytic polishing and step by step measurements.

Bild 6. Zerstörungsfrei unter Anwendung der Multiwellenlängen-Methode ermittelte oberflächennahe Eigenspannungstiefenverteilungen in Überrollungsrichtung (RD, links) und senkrecht dazu (TR, rechts) des in Bild 5 beschriebenen Bauteils. Die Ergebnisse werden verglichen mit zerstörend unter Anwendung elektrolytischer Abtragschritte nach der sin²Ψ-Methode ermittelten Werten

applying the multi-wavelength method for the same component as investigated in Figure 5 is shown in Figure 6. They are compared with results of experiments using the conventional destructive $\sin^2\Psi$-method which combines electrolytic polishing and step by step measurements. Obviously, there is a good qualitative correlation between the results of both methods showing maximum compressive residual stresses near the surface, which continuously decline up to a surface distance of approximately 30 µm. Directly at the surface, nondestructive energy resolved analyses yield higher compressive residual stress values than the conventional angle resolved method. It should be noted that residual stress values measured conventionally by electrolytic polishing are attributed to the respective removed depths while in the case of energy dispersive diffraction, they are assigned to the mean penetration depths corresponding to the {hkl}-planes measured.

In Figure 7 results of the Ω-χ-φ-method are compared with those of the multi-wavelength method for measurements in rolling direction. Again a satisfactory agreement is observed. The determined residual stresses of both methods show the same gradient and the Ω-χ-φ-method provides more data points especially at low information depths. However, due to very small incident angles the scattering of the residual stress values is larger and the measurement effort is higher compared to the multi-wavelength method.

Nevertheless, the examples presented in Figures 6 and 7 clearly show the potential of energy resolved methods for the nondestructive analysis of residual stress depth distributions in e. g. machined or plastically deformed near surface layers. As a consequence destruction or damage of components can be avoided and, moreover, as compared with existing procedures, analysis of residual stress depth distributions is considerably faster.

4 Conclusion

Energy resolved X-ray residual stress analysis offers specific benefits compared with conventional angle resolved diffraction. For laboratory applications suitable diffractometer configurations have to be used which, due to low intensity and beam quality, differ from conventional diffractometer types. Appropriate detector types exist, which, at least to a reasonable extent, fulfill the necessary requirements. Characteristic examples of measurements are presented demonstrating the basics and the potential of energy resolved residual stress analysis.

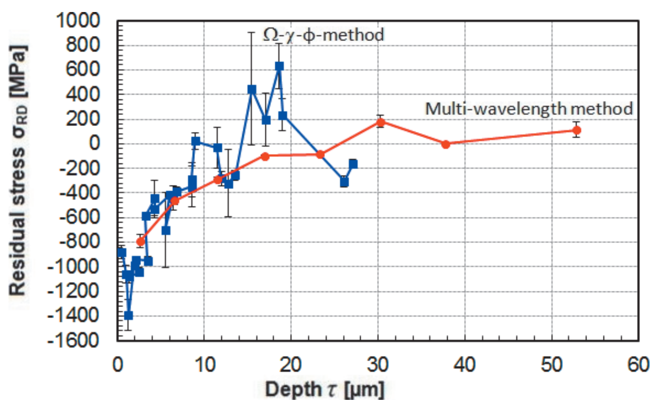

Fig. 7. Comparsion of residual stress depth distributions nondestructively determined in rolling direction of the sample investigated in Fig. 5 applying the multi-wavelength and the Ω-χ-φ-method. For the Ω-χ-φ-method 2Θ = 20° was chosen and measured planes were {110}, {200}, {211}, {220}, {310}, {222}, {321} and {330}.

Bild 7. Vergleich der in Überrollungsrichtung zerstörungfrei ermittelten Eigenspannungstiefenverläufe aus Multiwellenlängen- und Ω-χ-φ-Methode für das in Bild 5 beschriebene Bauteil. Die Messungen nach der Ω-χ-φ-Methode erfolgten bei 2Θ = 20° an den Ebenen {110}, {200}, {211}, {220}, {310}, {222}, {321} and {330}.

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