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Sebastian Send, Dominik Dapprich, Mikko Palosaari. Fast residual stress determination by means of two-dimensional x-ray diffraction using the $\sin 2 \psi$ -method. ICRS 11 - The 11th International Conference of Residual Stresses, SF2M; IJL, Mar 2022, Nancy, France. hal-04015041

HAL Id: hal-04015041

<https://hal.science/hal-04015041v1>

Submitted on 7 Mar 2023

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FAST RESIDUAL STRESS DETERMINATION BY MEANS OF TWO-DIMENSIONAL X-RAY DIFFRACTION USING THE $\sin^2\Psi$ -METHOD

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ABSTRACT

The conventional $\sin^2\Psi$ -method in accordance with EN 15305 is an established tool for non-destructive residual stress analysis by means of X-ray diffraction. However, depending on the geometrical and structural properties of the investigated sample, its applicability on laboratory X-ray diffractometers can be limited. The arising difficulties are associated with the dimensioning of the used X-ray detector system, which may lead to insufficient counting statistics or impractically long measurement times. Two-dimensional X-ray diffraction offers new possibilities for residual stress analyses exploiting the full 2D position resolution of an X-ray area detector. This work introduces a new approach based on the direct integration of 2D Debye-Scherrer ring sections to 1D intensity spectra for strain determination. In a first step, its limitations are identified by means of simulations considering data acquisition by an Xstress DR45 diffractometer in modified- χ geometry. In a second step, the performance of the Xstress DR45 is tested for an operation mode with discrete tilt steps (standard mode) as per EN 15305 and an alternative mode with continuous tilt movement (sweep mode). For that purpose, experimental studies were conducted for various materials measured under different geometrical conditions. The results of the data analyses show that the sweep mode of the Xstress DR45 is a reliable alternative to the standard mode allowing for fast residual stress measurements in applications of the $\sin^2\Psi$ -method.

Keywords: residual stress analysis; two-dimensional X-ray diffraction; X-ray diffractometer; X-ray area detector

1. Introduction

For decades X-ray diffraction has been used as a non-destructive method for residual stress determination in polycrystalline materials [1,2]. The procedure is based on a measurement of the direction dependent lattice strain and requires knowledge of the X-ray elastic constants of the sample. In industrial applications a variety of materials are investigated including, among others, steels with martensitic, austenitic or two-phase microstructure, Ni-based alloys, Ti alloys and carbides. The best known and most established X-ray diffraction technique for residual stress analysis is the $\sin^2\Psi$ -method standardized in EN 15305 [3]. In practice measurements are carried out in the most suitable operation mode of the used X-ray diffractometer taking into account the penetration depth of the incident X-ray beam as well as geometrical aspects like sample dimensions and accessibility of the measurement position.

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From an economic point of view residual stress analyses must be enabled within the shortest possible measurement time and without loss of precision. This highly demanding requirement motivates the development and continuous improvement of laboratory X-ray diffractometers. The performance of an X-ray diffractometer in a particular experiment manifests in the time needed to detect the Bragg peak of the sample with sufficient statistics, which depends on the power of the X-ray tube, absorption losses in air, the scattering power of the material and the size and sensitivity of the detector system. Nowadays, semiconductor radiation detectors with a quantum efficiency above 90% for 5-10 keV X-rays are frequently used in modern X-ray diffractometers. In recent years linear MYTHEN detectors [4] have successfully been applied for laboratory residual stress analyses. Exploiting the capability of single-photon counting the measurement time of flat ferritic steel samples could be reduced to about 1 min on an Xstress G2R diffractometer. Moreover, the energy threshold enables fluorescence suppression giving rise to improved signal-to-noise ratios when analyzing Ti alloys with Cu $K\alpha$ radiation [5].

A further speed-up of residual stress measurements can be achieved by replacing the linear MYTHEN detectors by Si-based X-ray area detectors with comparable properties in terms of sensitivity and resolution. In this case, two-dimensional X-ray diffraction [6] serves to record sections of Debye-Scherrer rings, from which residual stresses can be calculated with higher statistics and accuracy [7]. Another advantage of using area detectors is the availability of large virtual oscillation angles reducing the need for mechanical oscillations to study residual stresses in coarse-grained and textured materials [8].

The newly developed Xstress DR45 diffractometer is equipped with two area detectors with 256x256 square pixels and a pixel size of $55 \times 55 \mu\text{m}^2$ covering an angular window of 18° ($\Delta 2\theta$) at a nominal detector distance of 45 mm. In this work, the application of an Xstress DR45 for residual stress analyses based on the $\sin^2\psi$ -method in accordance with EN 15305 after direct integration of the 2D data volume is examined. For that purpose, the standard operation mode using discrete tilt steps and the sweep mode with continuous tilt movement are compared.

2. Experimental

Figure 1 illustrates the data acquisition of the Xstress DR45 operated in standard mode and in sweep mode. Residual stress measurements are carried out in modified- χ geometry by means of two area detectors with tilt angles between -45° and $+45^\circ$. In this configuration the tilt axis is perpendicular to the measurement direction. Moreover, the diffractometer allows for a full ϕ -rotation around the vertical axis between -180° and $+180^\circ$. In the unrotated state ($\phi=0^\circ$) the Xstress DR45 provides the normal stress σ_{11} and the shear stress τ_{13} assuming that the stress component σ_{33} in the direction of the surface normal is negligible. The residual stresses are calculated by averaging the measured strains of both detectors between $\sin^2\chi=0$ and $\sin^2\chi=0.5$ and applying multiple linear regression with the variables $\sin^2\chi$ and $\sin 2\chi$ as per EN 15305.

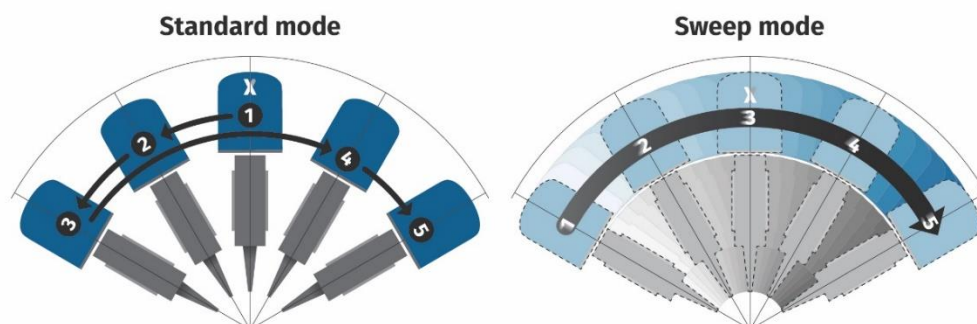


Fig. 1. Operation modes of the Xstress DR45 (schematical image). The numbering of the positions 1-5 indicates the sequence of the tilt movement steps and does not correspond to the real number of tilts in the measurements.

In standard mode, discrete tilt angles are defined between $\chi=-45^\circ$ and $\chi=+45^\circ$ with equidistant $\sin^2\chi$ -values starting with an exposure at normal incidence $\chi=0^\circ$ (position 1) and proceeding via $\chi=-45^\circ$ (position 3) to $\chi=+45^\circ$ (position 5). The necessary number of tilts ensuring a sufficiently high fit accuracy depends on the presence of shear stresses. EN 15305 requires at least seven tilts and recommends more than nine tilts to quantify ψ -splitting. Experience has shown that a setting with nine tilt angles is well-suited to measure normal stresses and shear stresses in fine-grained, untextured materials without the need for χ -oscillations. In this sense, nine tilt angles were used to evaluate the performance of the Xstress DR45 in standard mode, which is limited by the shortest possible exposure time of 1 s per tilt angle.

In sweep mode, a continuous tilt movement is performed over the complete tilt angle range between the starting point $\chi=-45^\circ$ (position 1) and the end point $\chi=+45^\circ$ (position 5) acquiring data in constant time intervals. Under this condition, data points are generated in non-equidistant $\sin^2\chi$ -steps and with a non-uniform density, which decreases from low $\sin^2\chi$ -values to high $\sin^2\chi$ -values. Experimental strain data were attributed to 18 discrete and equidistant tilt angles in order to obtain enough statistics for residual stress determination. In particular cases (such as coarseness or texture) a larger number of tilt angles may be required. A measurement in sweep mode physically corresponds to a measurement in standard mode with additional tilt oscillation, twice as many tilt angles and reduced exposure time. The complete tilt movement cannot be carried out faster than the shortest possible sweep time of 5 s (exposure time 0.3 s per tilt angle) due to electromechanical constraints.

The 2D data volume provided by the area detectors is transferred from pixel space to angular space for strain determination. Following theoretical conventions, the recorded section of the Debye-Scherrer ring can be described by an intensity profile $I(\gamma, 2\theta)$ with the circumferential angle γ and the diffraction angle 2θ . In modified- χ geometry the detector midpoints are located at $\gamma=0^\circ$ (detector A) and $\gamma=180^\circ$ (detector B), respectively. In the configuration of the Xstress DR45 system the tilt of the detectors with respect to the sample surface results in an elliptical deformation of the Debye-Scherrer ring. Further data processing involves a direct integration of the distribution $I(\gamma, 2\theta)$ over a user-defined interval $\Delta\gamma$ (Figure 2) and strain calculation based on the integrated spectrum $I_{\Delta\gamma}(2\theta)$ for each tilt angle (Figure 3). In the last step of data treatment, the stress components σ_φ and τ_φ are determined as per EN 15305. For residual stress analyses of ferritic materials with Cr K α radiation generating the 211 reflection of α -Fe at $2\theta_0=156.4^\circ$ the recorded section of the Debye-Scherrer ring extends over a range of about $\Delta\gamma=45^\circ$. In this work, the associated 2D data were integrated over a sector with an opening angle of $\Delta\gamma=40^\circ$. The performance of the Xstress DR45 in standard mode and sweep mode was studied within systematic test measurements of various materials. Depending on the experimental conditions (material, radiation, X-ray beam size) standard mode and sweep mode were compared with each other in terms of the shortest possible measurement time derived from the recorded data.

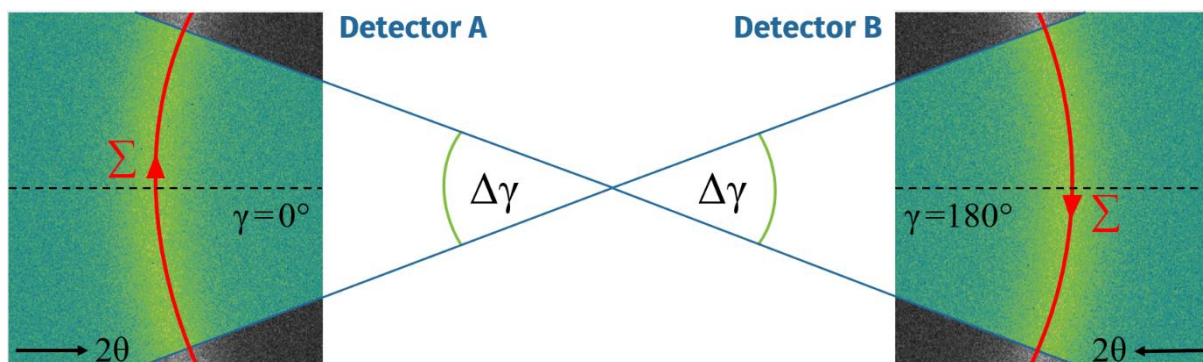


Fig. 2. Integration of 2D diffraction data acquired by the Xstress DR45 over sectors of the Debye-Scherrer ring with opening angle $\Delta\gamma$. In the displayed situation a ferritic steel sample was irradiated at normal incidence $\chi=0^\circ$.

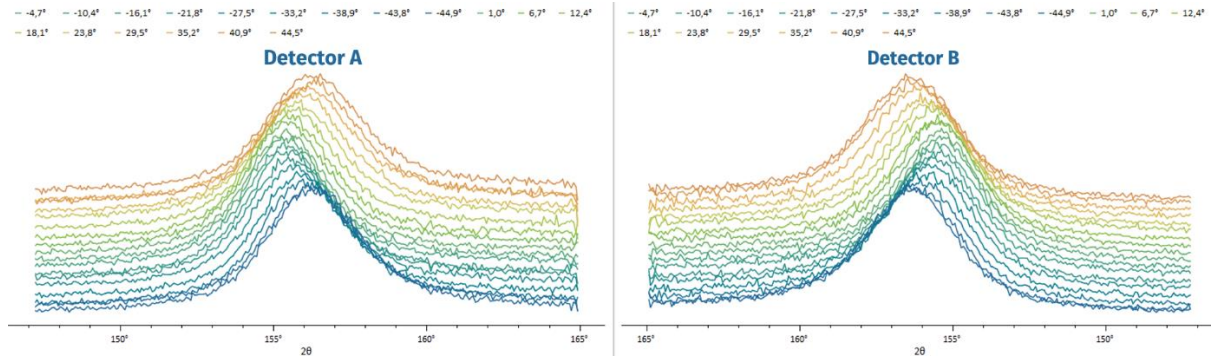


Fig. 3. Integrated data of a ferritic steel sample with a compressive normal stress of -593 MPa obtained for 18 tilt angles in sweep mode. For the graphical representation the measured intensity spectra were rescaled.

3. Results

Prior to experimental investigations of stressed materials, the applicability of the integration method for 2D data was verified by means of simulations. For that purpose, the geometrical conditions of the Xstress DR45 operated in standard mode were used to quantify the effect of the opening angle $\Delta\gamma$ on the calculated residual stresses and their uncertainties. Including α -Fe, Al and WC with different X-ray elastic constants and diffraction angles for Cr $K\alpha$ radiation in the simulations, a wide range of practically relevant situations could be covered. The results also apply to residual stress measurements of Ni or Ti alloys with Mn $K\alpha$ and Cu $K\alpha$ radiation, respectively, taking the related diffraction angles into account.

Considering various triaxial stress states it could be verified that in the unrotated case ($\varphi=0^\circ$), only the shear stress component τ_{13} has an impact on the accuracy of the measurement results. Consequently, simulations were confined to stress states with varying measurands σ_{11} and τ_{13} . In a first step, the profile $2\theta(\gamma)$ of the deformed Debye-Scherrer ring as a result of stress was determined for modified- χ geometry and nine tilt angles with equidistant $\sin^2\chi$ -values up to $\sin^2\chi=0.5$ from basic principles [9]. In a second step, averaging the distribution $2\theta(\gamma)$ for both detectors over the integration ranges between $\Delta\gamma=0^\circ$ and $\Delta\gamma=\Delta\gamma_{\max}$ around $\gamma=0^\circ$ and $\gamma=180^\circ$ for each tilt angle ($\Delta\gamma_{\max}=45^\circ$ for α -Fe 211, $\Delta\gamma_{\max}=25^\circ$ for WC 102), the experimental strain $\varepsilon(\sin^2\chi)$ was derived from the data. In a third step, the associated residual stress values $\sigma_{11,\text{exp}}$ and $\tau_{13,\text{exp}}$ with the standard deviations $s(\sigma_{11,\text{exp}})$ and $s(\tau_{13,\text{exp}})$ were obtained by means of least-squares fits of the strain data. Figure 4(a) shows the simulated deviation of the fitted stresses from the pre-defined values σ_{11} and τ_{13} for the α -Fe 211 ($2\theta_0=156.4^\circ$) and the WC 102 reflection ($2\theta_0=135.8^\circ$) generated by Cr $K\alpha$ radiation. The relative deviation increases parabolically as a function of $\Delta\gamma$ independent of σ_{11} and τ_{13} . No discrepancies occur in the limit of an infinitely small integration range ($\Delta\gamma=0^\circ$), whereas the largest possible integration range leads to relative deviations from the nominal stress values of about 0.22% (α -Fe 211) and 0.25% (WC 102), respectively, which are negligible for practical applications.

The standard deviations $s(\sigma_{11,\text{exp}})$ and $s(\tau_{13,\text{exp}})$ of the fitted stress values do not depend on the normal stress σ_{11} and follow linear relationships to the magnitude $|\tau_{13}|$ of the shear stress and to the integration range $\Delta\gamma$ according to

$$s(\sigma_{11,\text{exp}}) = c_\sigma |\tau_{13}| \Delta\gamma \quad (1)$$

$$s(\tau_{13,\text{exp}}) = c_\tau |\tau_{13}| \Delta\gamma \quad (2)$$

In equations (1) and (2) c_σ and c_τ are fit parameters depending on the diffraction angle $2\theta_0$ and $\Delta\gamma$ is in degrees. The corresponding simulation results for α -Fe 211 are depicted in Figure 4(b).

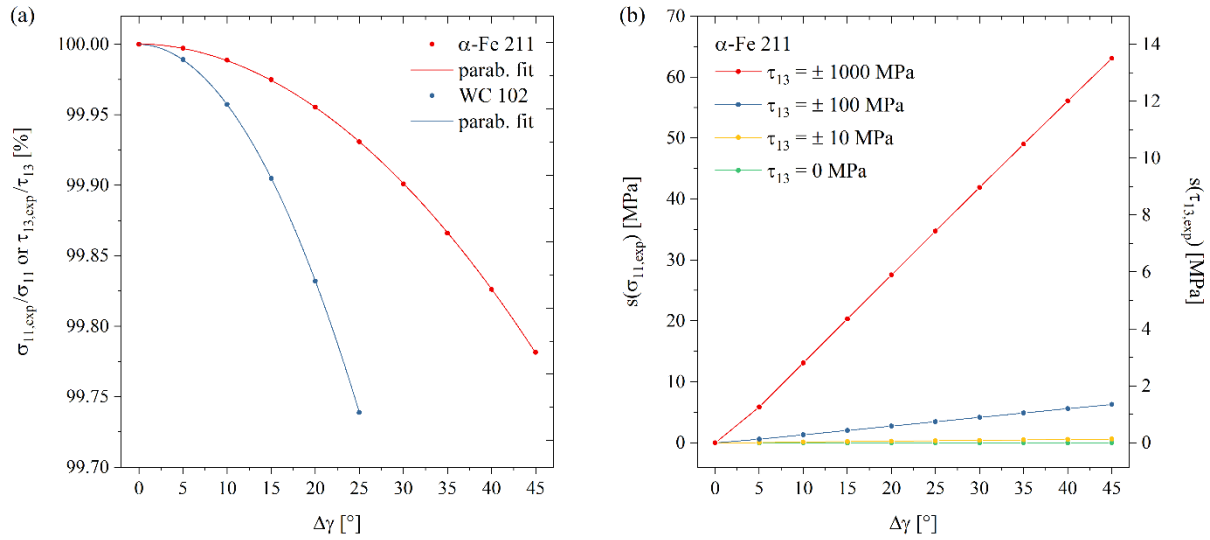


Fig. 4. Simulation results of the $\Delta\gamma$ -effect on the measured residual stresses $\sigma_{11,exp}$ and $\tau_{13,exp}$ and their accuracies using Cr $K\alpha$ radiation. (a) Discrepancy between the experimental stress values and the nominal stress values for the α -Fe 211 and the WC 102 reflections up to the limit $\Delta\gamma_{max}$ of the integration range. (b) Standard deviations of the calculated residual stresses for the α -Fe 211 reflection and increasing shear stress magnitude.

Table 1 gives an overview of the numerical simulation results for the considered situations with Cr $K\alpha$ radiation. In addition to α -Fe 211 and WC 102 the Al 222 reflection ($2\theta_0=156.7^\circ$) and the Al 311 reflection ($2\theta_0=139.3^\circ$) were incorporated in the simulations. The X-ray elastic constants $\frac{1}{2}S_2$ were derived from single-crystal data using the Kröner model [10]. The results show that the elastic properties of the material do not have a significant effect on the calculated residual stresses and their accuracies. Enhanced relative deviations of the stress values from the pre-defined values are observed when analyzing materials with smaller diffraction angles $2\theta_0$ compared to α -Fe 211. In those cases, the maximum standard deviation s_{max} of the normal stress and the shear stress increase by about 10%, not exceeding values of 6.9% and 1.5% of the shear stress magnitude, respectively. It can be concluded that the effect of the integration range $\Delta\gamma$ on the measurement precision requires attention only when a high shear stress component τ_{13} is present. In such a situation, a larger exposure time can compensate for statistical losses caused by a reduced $\Delta\gamma$ -range if more accurate residual stress values are needed.

Table 1. Numerical simulation results of the $\Delta\gamma$ -effect for various experimental conditions with Cr $K\alpha$ radiation.

Material	α -Fe	Al	Al	WC
hkl	211	222	311	102
$2\theta_0$	156.4°	156.7°	139.3°	135.8°
$\frac{1}{2}S_2$ [10^{-6} MPa $^{-1}$]	5.78	18.51	19.46	1.64
$\Delta\gamma_{max}$	45°	45°	27°	25°
Max. relative deviation	0.22%	0.21%	0.25%	0.26%
c_σ [10^{-3}]	1.395	1.376	2.515	2.775
c_τ [10^{-3}]	0.299	0.295	0.540	0.596
$s_{max}(\sigma_{11,exp})$	$0.063 \tau_{13} $	$0.062 \tau_{13} $	$0.068 \tau_{13} $	$0.069 \tau_{13} $
$s_{max}(\tau_{11,exp})$	$0.013 \tau_{13} $	$0.013 \tau_{13} $	$0.015 \tau_{13} $	$0.015 \tau_{13} $

In order to compare the performance of the Xstress DR45 in standard mode and sweep mode, an inter-laboratory qualified (ILQ) reference sample from ferritic steel with an isotropic normal stress state and negligible shear stresses was studied in a first test experiment. Residual stress measurements were carried out with Cr K α radiation from a 270 W X-ray tube and different collimators with diameters between 3 mm and 0.3 mm allowing for an intensity variation over two orders of magnitude. In standard mode, using nine tilt angles from $\chi=-45^\circ$ to $\chi=+45^\circ$, an exposure time of 1 s per tilt angle provides enough statistics for residual stress calculation with sufficient accuracy over the available intensity range. In sweep mode, the duration of the complete continuous tilt movement between $\chi=-45^\circ$ and $\chi=+45^\circ$ (sweep time) was successively increased starting with the lowest possible value of 5 s until the normal stress σ and its standard deviation $s(\sigma)$ stabilized. Figure 5 summarizes the experimental results for the situations with the 3 mm collimator and the 0.3 mm collimator as obtained with 18 tilt angles.

In the case of the highest incident intensity (3 mm collimator) the measured normal stresses vary within a 10 MPa wide band in accordance with the certified reference value $\sigma_{\text{ref}}=-593$ MPa of the sample taking into account a critical difference of 15 MPa. The relative standard deviation $|s(\sigma)/\sigma|$ is about 2% for a sweep time of 5 s, about 1% for a sweep time of 10 s and about 0.5% for a sweep time of 15 s and above, which can also be achieved in standard mode. Furthermore, repeatability tests were conducted for the ILQ reference sample giving a relative repeatability standard deviation of 0.5% for both operation modes. In the situation with the lowest incident intensity (0.3 mm collimator) strong variations of the normal stress values occur for sweep times between 5 s and 15 s, whereas the data points reach a stable level at sweep times between 20 s and 55 s. It should be noted that most of the measured normal stresses are out of the acceptance band, which is uncritical as the sample is not perfectly homogeneous and its certified reference value, repeatability and reproducibility apply only to the 3 mm collimator.

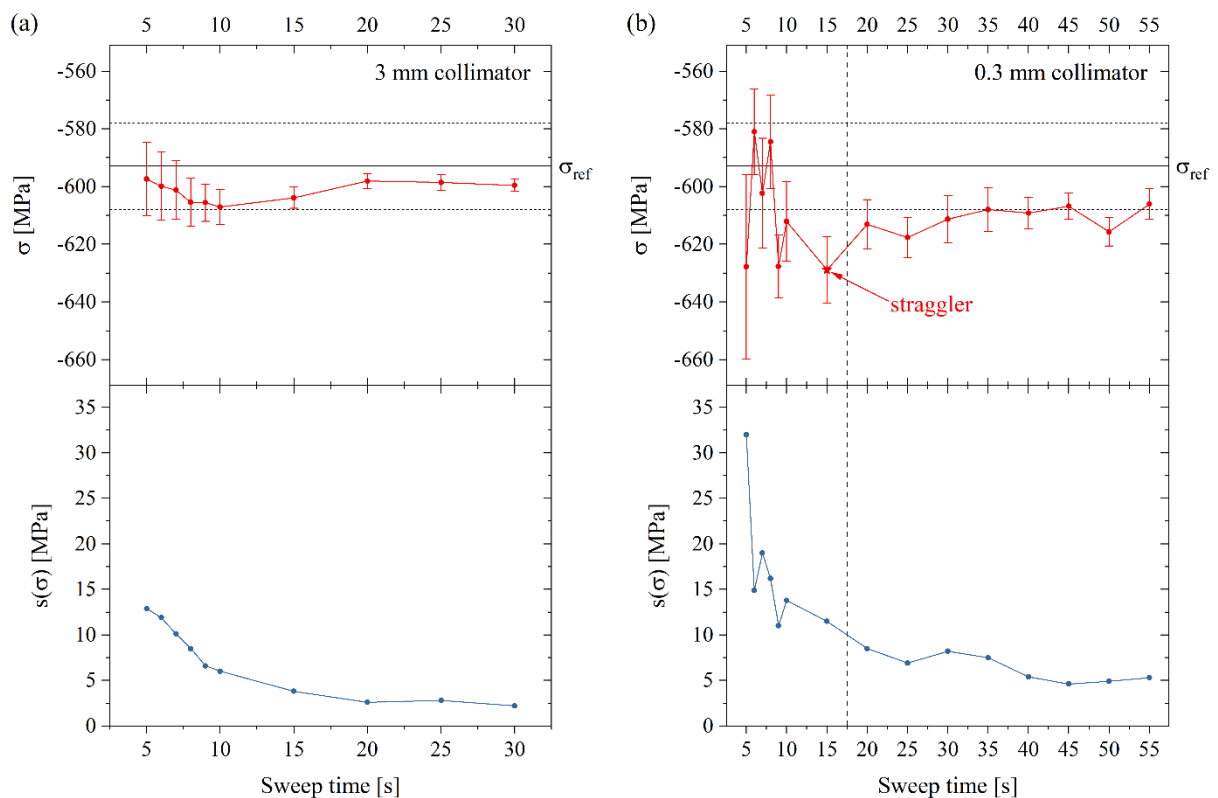


Fig. 5. Experimental results of the sweep mode tests for an ILQ reference sample from ferritic steel with a 3 mm collimator (a) and a 0.3 mm collimator (b), respectively. In the latter case, the dashed vertical line separates the region with stable data points from the region with strongly varying data points including the identified straggler.

The stability of the data points was quantitatively investigated by means of Grubbs' tests for one outlier starting with the three normal stress values at sweep times of 55 s, 50 s and 45 s and adding gradually one data point recorded at the next lower sweep time. In accordance with the classification of ISO 5725-2 [11] the residual stress values measured for sweep times between 20 s and 55 s are considered as correct. Including the data point at a sweep time of 15 s in the Grubbs' test gives rise to a straggler at the 5%-level, which indicates an instability in the data. The relative standard deviation $|s(\sigma)/\sigma|$ is about 5% for a sweep time of 5 s, below 2% for sweep times above 15 s and below 1% for sweep times between 40 s and 55 s. A relative fit accuracy of 2% is usually sufficient for many practical applications, so that reliable residual stress data can be obtained for ferritic materials with sweep times of 5 s for a 3 mm collimator and 20 s for a 0.3 mm collimator, respectively.

Analogue experimental studies were carried out for Ni alloys (INCONEL[®]) under different statistical conditions by means of Mn K α and Cr K β radiation generating the 311 reflection at diffraction angles $2\theta_0$ of about 150° (Mn K α) and 147° (Cr K β). Table 2 compares the applicable sweep times with the exposure times per tilt angle and the total measurement times in standard mode for ferritic steel and INCONEL. For sufficiently high statistics provided by K α radiation and collimator diameters of at least 1 mm the measurement time in sweep mode is shortened by a factor of 7 compared to standard mode, reducing to about 2 for small beam sizes. When using Cr K β radiation the residual stress measurements are accelerated by an average factor of 3.5, which seems independent of the beam cross section. The difference between Cr K α and Cr K β radiation with respect to the gain in measurement time is caused by the different signal-to-noise ratios of the detected Bragg peaks. Similar observations were made for two-phase steel samples analyzing simultaneously the α -Fe 211 reflection of the ferritic phase with Cr K α radiation and the γ -Fe 311 reflection of the austenitic phase with Cr K β radiation ($2\theta_0=148.9^\circ$).

For the situation with the highest possible statistics (3 mm collimator) equivalent results were obtained for carbides (WC-Co) and various Ti alloys (Ti6242, Ti64 and Ti6246) investigated with Mn K α and Cu K α radiation, respectively. In the first case, favored by the large scattering power of the WC phase, a sweep time of 5 s is sufficient for reliable residual stress determination based on the WC 201 reflection ($2\theta_0=132.3^\circ$) compared to a total measurement time of 34 s in standard mode (exposure time per tilt angle 1 s). In the second case, both the α -Ti 213 reflection ($2\theta_0=139.6^\circ$) and the α -Ti 302 reflection ($2\theta_0=148.7^\circ$) were used to quantify the residual stresses in the α -Ti phase. Due to the significantly lower signal-to-noise ratio an enhanced sweep time of 15 s is required to achieve acceptable accuracy in residual stress analyses of α -Ti compared to a total measurement time of 115 s in standard mode (exposure time per tilt angle 10 s).

Table 2. Comparison of sweep mode and standard mode for various measurement conditions in terms of sweep time (t_{sweep}), exposure time (t_{exp}) and total measurement time (t_{meas}). The results refer to INCONEL alloy 718.

Material (radiation)	Mode	Time	Collimator diameter				
			3 mm	2 mm	1 mm	0.5 mm	0.3 mm
Ferritic steel (Cr K α)	sweep	t_{sweep}	5 s	5 s	5 s	10 s	20 s
	standard	t_{exp}	1 s	1 s	1 s	1 s	1 s
INCONEL (Mn K α)	standard	t_{meas}	34 s	34 s	34 s	34 s	34 s
	sweep	t_{sweep}	10 s	10 s	10 s	20 s	40 s
INCONEL (Cr K β)	standard	t_{exp}	1 s	1 s	2 s	5 s	10 s
	standard	t_{meas}	34 s	34 s	43 s	70 s	115 s

4. Conclusions

In this work the performance of an Xstress DR45 diffractometer for laboratory residual stress measurements by means of the conventional $\sin^2\psi$ -method in accordance with EN 15305 was evaluated. The two-dimensional position resolution of two X-ray area detectors allows for a direct integration of the recorded Debye-Scherrer ring sections to one-dimensional intensity spectra for strain determination. Simulations revealed that the calculated residual stress values are practically unaffected by the integration procedure and that the associated fit accuracies are slightly deteriorated only in case of large shear stresses. Experimental studies were conducted for selected materials analyzed under different conditions in modified- χ geometry and with the Xstress DR45 operated in standard mode with discrete tilt angles as well as in sweep mode with continuous tilt movement. If the signal-to-noise ratio of the recorded Bragg peak is sufficiently high, residual stress measurements in standard mode can be carried out with an exposure time of 1 s per tilt angle resulting in a total measurement time of 34 s for nine tilt angles. In contrast, twice the number of tilts can be used in sweep mode providing residual stresses with 2% relative fit accuracy for a sweep time of 5 s. A significant reduction of the measurement time by a factor of about 2 can also be achieved in sweep mode if a small X-ray beam size is required due to the geometrical properties of the sample. The results of this work demonstrate the statistical benefit from the replacement of linear X-ray detectors by 2D X-ray detectors, making the Xstress DR45 a well-suited diffractometer for fast residual stress measurements in various applications.

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