



Non-destructive Neutron Surface Residual Stress Analysis

J. Rebelo Kornmeier¹ · M. Hofmann¹ · W. M. Gan² · J. Gibmeier³ · J. Saroun⁴

Received: 19 November 2018 / Accepted: 5 August 2019 / Published online: 13 August 2019
© Springer Science+Business Media, LLC, part of Springer Nature 2019

Abstract

Diffraction is a powerful tool for investigation of residual as well as applied stresses in engineering materials and components. Both X-ray and neutron diffraction can be used for this purpose. The interest in neutron stress analysis stems from the high penetrating power of neutrons when compared to laboratory X-ray sources, i.e. several cm instead of a few tens of μm in metallic materials. This contribution gives an overview on current instrumental and methodical developments for non-destructive through surface strain measurements, which bridges the gap between X-rays and neutrons analysis.

Keywords STRESS-SPEC · Surface residual stresses · Neutron diffraction · Spurious strains · Double radial collimators

1 Introduction

Neutron diffraction opens up the possibility to analyze residual stresses in the interior of technical components rather than just at the surface. On the other hand with the advent of second and third generation synchrotron sources, hard X-

rays have become a powerful competitor for stress analysis in bulk solids. Due to the very high brilliance of synchrotron sources one could think that neutrons would have been driven out of business. However, neutrons have the huge advantage of enabling a constant spatial resolution due to the possibility to define cubic gauge volumes in all required measuring directions. Consequently, new neutron instruments dedicated to stress or texture analysis, have been commissioned or are under construction until today, e.g. the Andes diffractometer currently being designed at LAHN in Argentine. The neutron diffractometer STRESS-SPEC located at Germany's neutron source "Forschungs-Neutronenquelle Heinz Maier-Leibnitz" (FRM II) in Garching is an instrument optimised for different material experimental analysis, namely strain [1], texture [2] and phase analysis [3]. Due to the still increasing user demand it is continuously developed and extended for new engineering and materials science applications.

This contribution focuses on an overview on current instrumental and methodical developments for through surface strain measurements, which bridge the gap in penetration depth between X-ray and neutron stress analysis. Measurements to cover the complete strain profile from the surface into the bulk of a component using neutron diffraction have seen an increased demand in recent years (see for example measurements on [4–7]). These type of measurements close to the surface are possible in two different ways: (1) reducing the gauge volume size to the desired spatial resolution or (2) keeping a gauge volume size as big as reasonable and only partially submerge it into the surface to achieve the required spatial resolution. The first measurement strategy is often impossible to realize, due to time restric-

✉ J. Rebelo Kornmeier
joana.kornmeier@frm2.tum.de
<http://www.frm2.tum.de>;
<http://www.mlz-garching.de/stress-spec>

M. Hofmann
Michael.hofmann@frm2.tum.de
<http://www.frm2.tum.de>;
<http://www.mlz-garching.de/stress-spec>

W. M. Gan
weimin.gan@hzg.de
<http://www.mlz-garching.de/stress-spec>

J. Gibmeier
jens.gibmeier@kit.edu
<http://www.iam.kit.edu/wk/index.php>

J. Saroun
saroun@ujf.cas.cz
<http://neutron.ujf.cas.cz/~saroun/index.html>

¹ Heinz Maier-Leibnitz Zentrum (MLZ) FRM II, Technische Universität München, Munich, Germany

² German Engineering Materials Centre at MLZ, Helmholtz-Zentrum Geesthacht, Geesthacht, Germany

³ Karlsruhe Institute of Technology, Institute for Applied Materials, Kaiserstr. 12, 76131 Karlsruhe, Germany

⁴ Nuclear Physics Institute of the CAS, v.v.i., 250 68 Rez, Czech Republic

tions for the measurements, due to characteristics like coarse grain structure of the material and due to sample dimensions or geometry. In case of the second approach, the measured strain must be corrected for spurious strains. These are mainly due to divergence and wavelength spread across the partially immersed gauge volume and can be of the same magnitude as the strains usually measured in engineering components [8–10]. For correction of these spurious strains an analytical model was developed for STRESS-SPEC [11] that takes into account for the first time a horizontal bending monochromator and is since then a routine tool for corrections of strain measurements close to surfaces and at interfaces [12, 13]. Further developments of the analytical method for treatment of spurious strains include smearing effects of measured strain distributions due to the finite gauge volume and have been demonstrated for a four-point bending test [14].

Another approach for non-destructive measurements with neutron diffraction close to surfaces or interfaces is reducing spurious strains in course of the measurements as much as possible. The divergence and wavelength spread in the primary beam are the main sources of the spurious strains in monochromatic neutron beams. They can be minimized when collimators in the neutron beam are used [15]. At STRESS-SPEC the primary neutron beam (incoming beam on the sample) is monochromatic after diffraction using a crystal monochromator in the white neutron beam from the reactor. In this study, a double focusing radial collimator to be used in the primary beam has been simulated and optimised by Monte Carlo simulations, using the SIMRES [16] code. The final double radial collimator has been designed and constructed according to the simulation results and tested experimentally.

2 Simulation of a Double Radial Collimator

A double radial collimator for the primary beam was first simulated and optimized to the STRESS-SPEC instrument with the objective to produce a gauge volume with a field of view (FOV) of 1 mm in the horizontal dimension. In addition, a large focus distance together with the constraints of construction, were further important requisites. The simulations resulted in a design compromise of a collimator delivering a gauge volume of 1 mm width and 3 mm height at a focus position of 70 mm. The schematic representation of the double collimator is shown in Fig. 1 where the collimator defining the vertical dimension has 20 slits and the one defining the horizontal dimension has 6 slits, respectively. In Fig. 2 simulations of the intensity distributions in horizontal and vertical directions are compared for the double collimator as designed (Fig. 2a), and an equivalent traditional slit in Fig. 2b. The traditional slit has the same dimensions of 1 mm (width) \times 3 mm

(height) and the slit to sample distance 70 mm the same as the focus distance of the collimator.

The distinct form of the neutron beam (at the same distance) in horizontal and vertical directions is shown in Fig. 2a for radial collimator and slit (Fig. 2b) in the plane perpendicular to the incident beam. The double collimator shows a typical triangular shape in both directions. The slit changes the form of the profile, from uniform distributions immediately after the slit exit, to a trapezoidal form at 70 mm distance. These changes are due to the angular divergence of the beam and therefore comparisons of both profiles are only possible by the Gaussian full width at half maximum (FWHM), which accounts for beam profiles differences. At the focus position for the primary double collimators, these results in 0.928 mm and 2.883 mm for the width and height of the sampling volume and 1.52 mm and 2.34 mm for the slit system, respectively. The horizontal dimension is larger after the slit system when compared to the radial collimator at the focus distance, most likely due to the use of the bent Si monochromator. Although the sampling volume defined by the radial collimators, 2.48 mm³, is smaller than by the slit, 3.29 mm³, the intensity per volume experiences only a slight decrease, less than 5%. This means that although the integral intensity when using the double radial collimators is smaller, this loss is compensated by the smaller beam divergence and consequently much smaller sampling volume.

The spurious strains simulations of a stress-free steel sample surface scanned out of the gauge volume, defined by the double radial collimator before the sample and a radial collimator with a FWHM of 1 mm in the diffracted beam, are shown in Fig. 3a. In the same figure, the Monte Carlo simulations in reflection and transmission mode are compared to the respective calculations using the analytical model [11]. The corresponding simulation results, for the traditional slit system, are pictured in Fig. 3b. In the graphics in Fig. 3 the position zero, at the x-axis, corresponds to the gauge volume half immersed in the material. Negative values indicated that the gauge volume is less than half immersed in the material and on contrary positive values means that the gauge volume is more than half immersed. In both figures reflection corresponds to the scattering vector being normal to the sample surface where transmission represents that the sample surface is parallel to the scattering vector.

Both, Monte Carlo simulations and the analytical calculations show similar agreement for both scattering modes. The simulations show that the double collimators reduce considerably the spurious strain effect, not only in transmission mode but also in reflection mode where spurious strains are almost negligible. Besides that, using collimators, has the additional advantage of easing sample positioning, due to the relatively large focusing distance enabling collision free movement of bigger samples.

Fig. 1 Schematic representation of the double monochromator simulated for STRESS-SPEC instrument by the SIMRES [16] code. The double radial collimator is shown in front of the steel sample while the scattered beam is detected through a secondary radial collimator

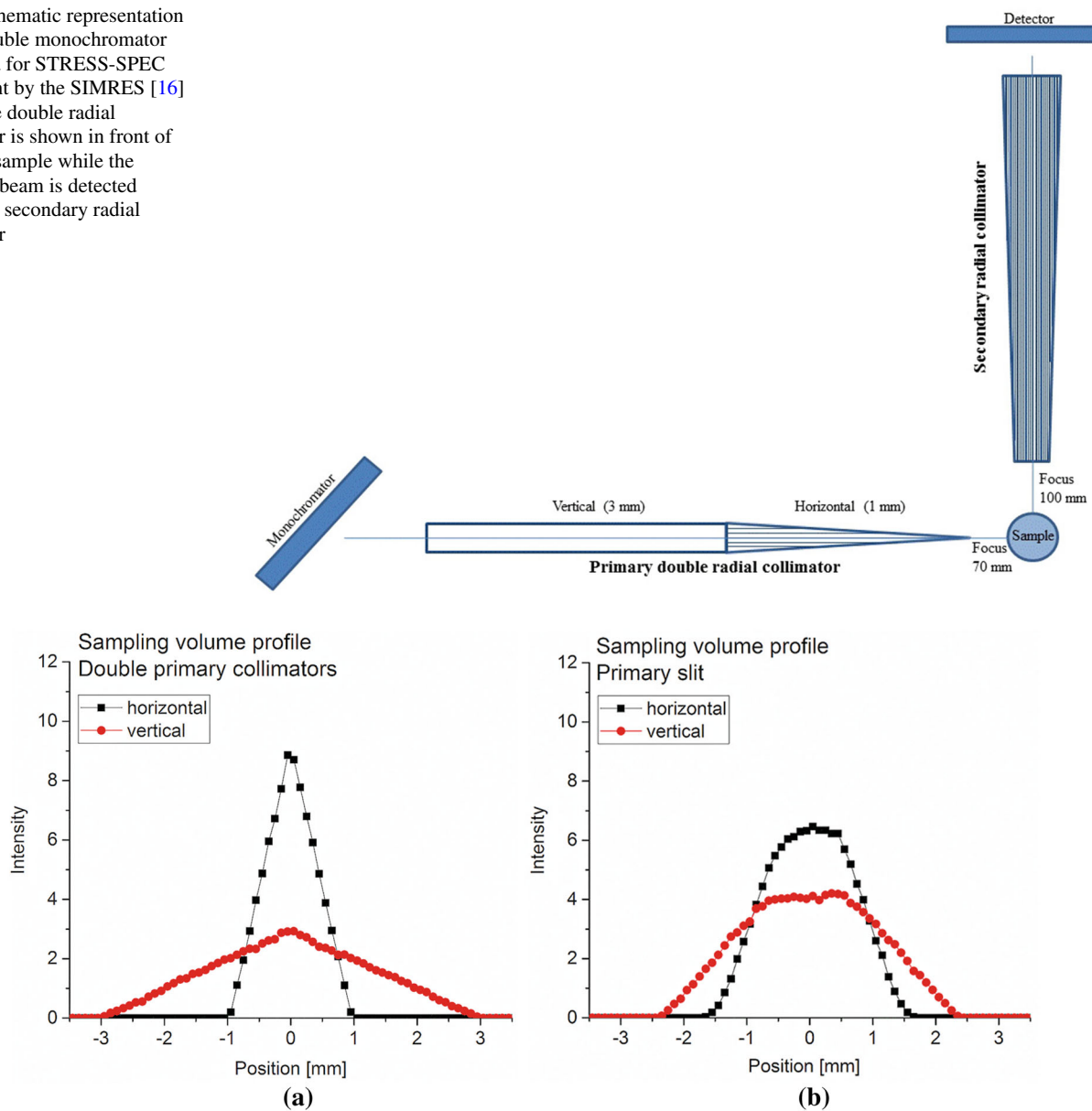


Fig. 2 Sampling horizontal and vertical simulations at a distance of 70 mm after the exit of the **a** double primary radial collimators and **b** traditional primary slit

3 Design and Construction of the Double Focussing Radial Collimator

The design of the double focussing collimator was to ensure that the dimensions at its exit close to the sample to be as small as possible. Figure 4 shows the individual collimators as well as the assembled double collimator in detail (Fig. 4a).

The collimator which defines the vertical dimension is further away from the sample whereas the horizontal dimension is defined by the collimator closest to the sample. The collimator which defines the vertical dimension is tapered towards the sample, with dimensions of 30 (w) \times 60 (h) mm² at the

neutron beam entrance and 30 (w) \times 30 (h) mm² at the exit. Twenty channels with overall length of 404 mm enable a 3 mm beam height definition after the collimator at the focal length of 424 mm, see Fig. 4b. The horizontally focussing collimator defining the width of the beam impinging on the sample has the dimensions of 30 (w) \times 30 (h) mm² at its entrance and 5 (w) \times 15 (h) mm² at the respective exit. Six channels with total length of 354 mm enable the definition of the gauge width of 1 mm at the collimator focal length of 70 mm (Fig. 4c).

The assembly of the complete double collimator permits future variations, by the possibility to exchange the horizontal

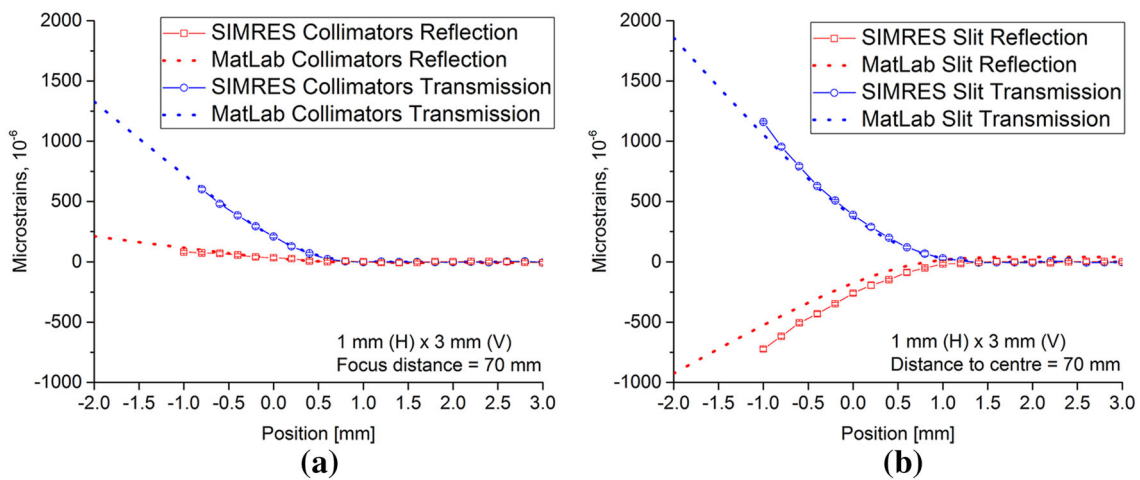


Fig. 3 Comparison of spurious strains calculated through Monte Carlo simulations and from the analytical model [11] when using either the double radial collimator (a) or a traditional slit setup (b) with the same nominal gauge volume dimensions and distance to the goniometer center



Fig. 4 a The constructed and assembled radial collimators, b side, c top view of the collimator defining the vertical and horizontal dimensions respectively, and d STRESS-SPEC setup for a test measurement where the adjustable mounting system of the primary optics (double radial collimator) is visible

focusing collimator by one with a wider horizontal gauge dimension and larger focal distance in case larger samples or sample environments are used.

4 First Measurements with Neutrons

After optical alignment of the double collimator an image plate camera with $130 \mu\text{m} \times 130 \mu\text{m}$ pixel resolution [17] was used to visualize the beam profile at the sample position. The image as well as the corresponding analysis are shown in Fig. 5. The image was collected with 5 s exposure time. A Si monochromator at a take-off angle of approximately $2\theta_M = 76.4^\circ$ was used for producing a wavelength of around 1.68 \AA .

Good agreement of the shape of the simulated and measured intensity distribution is found, although the measured width is larger in horizontal as well as in vertical direction. The horizontal widths derived from the simulated and measured intensity distribution curves are 0.99 and 1.31 mm, respectively. While the simulated and measured heights of the gauge volume expressed as FWHM of the corresponding vertical intensity distributions are 3.05 and 3.36 mm. The larger widths/heights derived experimentally can be attributed to the simulations not accounting for any imperfections nor for possible transmission through the foils.

A first test measurement on a stainless steel (γ -Fe) sample, which has undergone a mechanical surface treatment, was performed using the double focusing radial collimator in the primary beam and a radial collimator with $\text{FOV} = 1 \text{ mm}$ in the diffracted beam before the detector (see also the

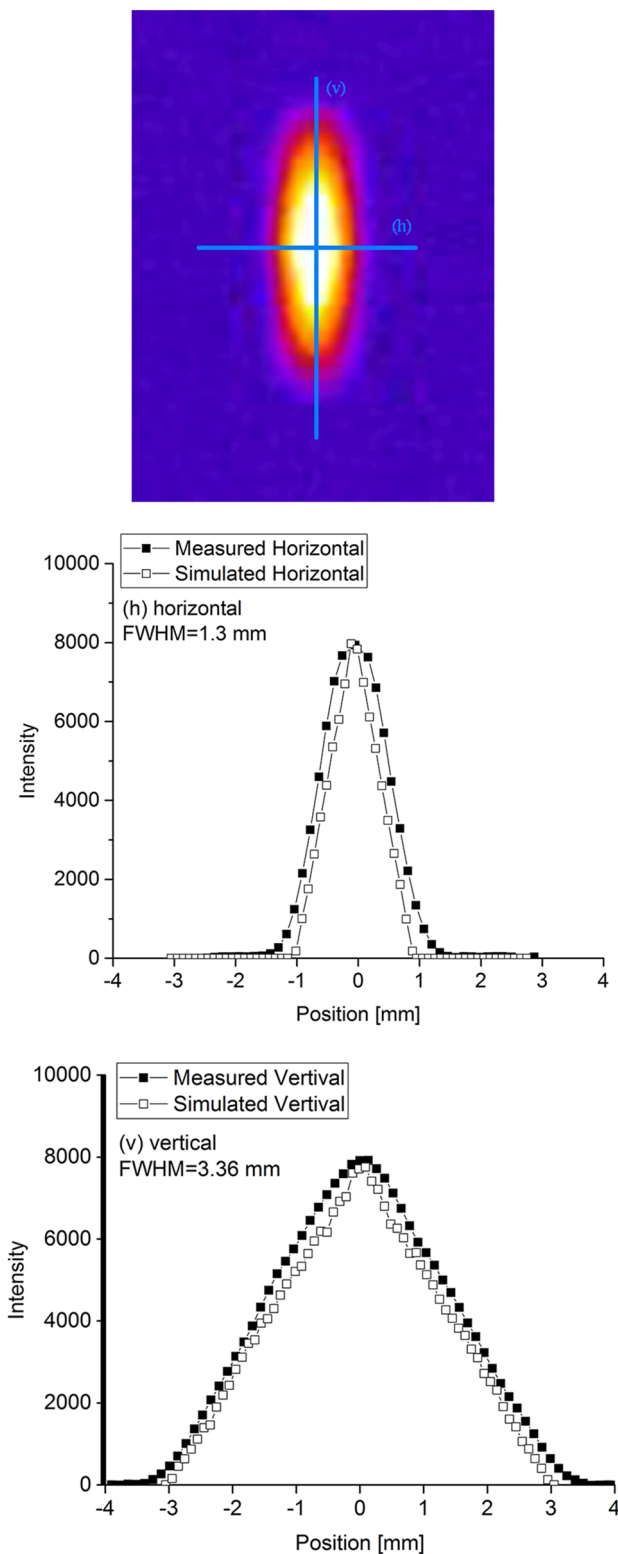


Fig. 5 The image of the beam at the focus position of the double focusing radial collimator is shown on the left. The corresponding horizontal and vertical intensity distributions at the positions indicated in the image are depicted on the right. For comparison the results of the Monte Carlo simulations are also shown in the figure

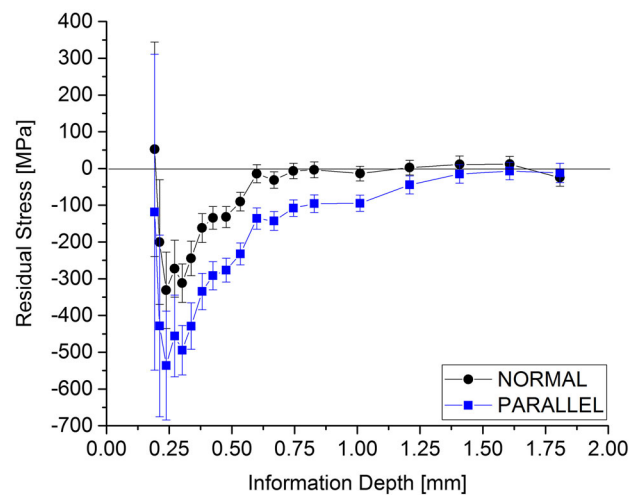


Fig. 6 Residual stress profiles for the normal and parallel directions to the sample surface determined from the neutron measurements. Please note: the normal strains are usually measured in reflection geometry while the parallel strains are measured in transmission mode

setup depicted in Fig. 4d). Measurements were carried out using the $\{220\}$ γ -Fe diffraction line at a scattering angle of $2\theta_S \approx 84.3^\circ$. Residual stresses were calculated [8] assuming that the principal stress directions are parallel and normal to the sample surface and that the in-plane residual stresses are axisymmetric. The diffraction elastic constants used to calculate the residual stress were $E_{220} = 207$ GPa and $\nu_{220} = 0.28$ [18]. No corrections for spurious strains were carried out for the measurement points close to the surface (Fig. 6).

The measurement results show that experimental residual stress profiles can be determined non-destructively by neutron diffraction from a position close to the surface into the bulk of a component. Close to the surface, the stress in the normal direction is almost zero as required from mechanical boundary conditions, implying the correctness of the measured strain distribution. Moreover, the results also show that this setup allows enough spatial resolution to distinguish a minimum of compressive residual stress at around $250 \mu\text{m}$.

5 Conclusions

Using focusing collimators instead of gauge volume defining slits in the primary neutron beam is an approach for bridging the gap of non-destructive strain measurements with neutron diffraction from the surface until the bulk. This is due to the considerable reduction of the spurious strains effect in measurements close to surfaces or interfaces. In this study, Monte Carlo simulations on an optimal double radial collimator for STRESS-SPEC specifications were carried out. They show that the sampling volume is smaller than using traditional

slits of the same dimensions compensating the intensity loss at the focus distance. The intensity per volume experiences only a slight decrease, around 5%, when the double radial collimator is used. Moreover, the simulations also show that spurious strains at the focus distance of the collimator are negligible in reflection (normal) mode and strongly reduced in transmission (parallel) mode.

Measurement of the intensity distribution within the gauge volume defined by the double radial collimator show a good agreement with the simulations. In future, additional radial collimators will be built to satisfy distinct sample environment needs required by different STRESS-SPEC setups. These will increase distances to goniometer center enabling larger translation space for manipulation of bulky samples. First test experiments on a steel sample which has undergone a mechanical surface treatment indicate that the spatial resolution is adequate to resolve details close to surfaces up to about 150 μm .

Acknowledgements The authors gratefully acknowledge the funding by the German Research Foundation (DFG) within the Project HO 3322/2-1, HO3322/2-2 and Czech Science Foundation Project No. 16-08803J, and by the German Research Foundation (DFG) within the Project HO 3322/4-1 and GI 376/11-1.

References

- Hofmann, M., Schneider, R., Seidl, G.A., Rebelo Kornmeier, J., Wimpory, R., Garbe, U., Brokmeier, H.G.: The new materials science diffractometer STRESS-SPEC at FRM-II. *Physica B* **385–386**, 1035–1037 (2006)
- Brokmeier, H.G., Gan, W.M., Randau, C., Völler, M., Rebelo Kornmeier, J., Hofmann, M.: Texture analysis at neutron diffractometer STRESS-SPEC. *Nucl. Instrum. Methods Phys. Res. A* **642**, 87–92 (2011)
- Zinth, V., von Lüders, C., Hofmann, M., Hattendorff, J., Buchberger, I., Erhard, S., Rebelo Kornmeier, J., Jossen, A., Gilles, R.: Lithium plating in lithium-ion batteries at sub-ambient temperatures investigated by in situ neutron diffraction. *J. Power Sources* **271**, 152–159 (2014)
- Cavaliere, P. (ed.): Chapter: 16. In: *Cold-Spray Coatings. Recent Trends and Future Perspectives*, 1st edn. Springer, Cham (2018)
- Vladimir, L., Andrew, V., Valarezo, A., Sanjay, S.: Neutron through-thickness stress measurements in coatings with high spatial resolution. *Mater. Sci. Forum* **905**, 165–173 (2017)
- Ramjaun, T.I., Stone, H.J., Karlsson, L., Gargouhri, M.A., Dalaei, K., Moat, R.J., Bhadeshia, H.K.D.H.: Surface residual stresses in multipass welds produced using low transformation temperature filler alloys. *Sci. Technol. Weld. Join.* **19**(7), 623–630 (2014)
- Gibmeier, J., Back, H.C., Mutter, M., Vollert, F., Vaßen, R., Rebelo Kornmeier, J., Mücke, R., Vaßen, R.: Study of stability of microstructure and residual strain after thermal loading of plasma sprayed YSZ by through surface neutron scanning. *Physica B* **551**, 69–78 (2018)
- Hutchings, M.T., Withers, P.J., Holden, T.M., Lorentzen, T.: *Introduction to the Characterization of Residual Stress by Neutron Diffraction*. Taylor and Francis, London (2005)
- Webster, P.J., Mills, G., Wang, X.D., Kang, W.P., Holden, T.M.: Impediments to efficient through-surface strain scanning. *J. Neutron Res.* **3**, 223–240 (1995)
- Rebelo Kornmeier, J., Gibmeier, J., Hofmann, M.: Minimization of spurious strains by using a Si bent-perfect-crystal monochromator: neutron surface strain scanning of a shot-peened sample. *Meas. Sci. Technol.* **22**, 065705 (2011)
- Šaroun, J., Rebelo Kornmeier, J., Hofmann, M., Mikula, P., Vrana, M.: Analytical model for neutron diffraction peak shifts due to the surface effect. *J. Appl. Crystallogr.* **46**, 628–638 (2013)
- Köhler, H., Rajput, R., Kahzan, P., Rebelo Kornmeier, J.: On the influence of laser cladding and post-processing strategies on residual stresses in steel specimens. *Phys. Procedia* **56**, 250–261 (2014)
- Coppola, R., Crescenzi, F., Gan, W., Hofmann, M., Lie, M., Visca, E., You, J.-H.: Neutron diffraction measurement of residual stresses in an ITER-like tungsten-monoblock type plasma-facing component. *Fusion Eng. Des.* (2019). <https://doi.org/10.1016/j.fusengdes.2019.01.059>
- Šaroun, J., Rebelo Kornmeier, J., Gibmeier, J., Hofmann, M.: Treatment of spatial resolution effects in neutron residual strain scanning. *Physica B* (2018). <https://doi.org/10.1016/j.physb.2018.01.013>
- Pirling, T.: Neutron strain scanning at interfaces: an optimised beam optics to reduce the surface effect. *Mater. Sci. Forum* **347–349**, 107–112 (2000)
- Šaroun, J., Kulda, J.: Raytrace of neutron optical systems with RESTRAX. In: *Modern Developments in X-Ray and Neutron Optics*. Springer Series in Optical Sciences, vol. 137, pp. 57–68. Springer, Berlin (2008)
- Defendi, I., Egerland, S., Kastenmüller, A., Mühlbauer, M., Panradl, M., Schöffel, T., Zeitelhack, K.: FRM II Annual Report (2005)
- Eigenmann, B., Macherauch, E.: Röntgenographische Untersuchung von Spannungszuständen in Werkstoffen. *Mater.-wiss. Werkst.* **27**, 426–437 (1996)

Publisher's Note Springer Nature remains neutral with regard to jurisdictional claims in published maps and institutional affiliations.