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New Developments of the Materials Science Diffractometer STRESS-SPEC

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Abstract. The high flux neutron diffractometer STRESS-SPEC at FRM II, Garching Germany, offers a flexible instrument setup suitable for fast and surface residual strain measurements. Likewise bulk, local or gradient texture analyses are feasible. Here improvements of the hardware (detector, slits) of the instrument as well as developments on methods for residual stress analysis are presented. A new detector system developed in-house was recently installed and successfully commissioned. Compared to the original delay line detector the new detector provides much higher resolution and allows event mode type measurements. Results of the commissioning measurements show a performance increase of nearly a factor of 2 compared to the former detector. Moreover the new analytical model, recently developed for surface spurious strain corrections, was successfully applied at a welded austenitic steel sample. Thus non-destructive measurements from the (200 μm) into the bulk (several millimeters) are possible without any extra time consuming experiments for spurious strains corrections.

Introduction

Residual stresses and texture influence significantly the mechanical properties of technical parts in service. Non-destructive analysis of both by neutron diffraction is of increasing importance and is nowadays taken into account in engineering applications. The high flux at the diffractometer STRESS-SPEC allows fast and surface residual stress mappings as well as global and local texture analysis at different and challenging new materials.

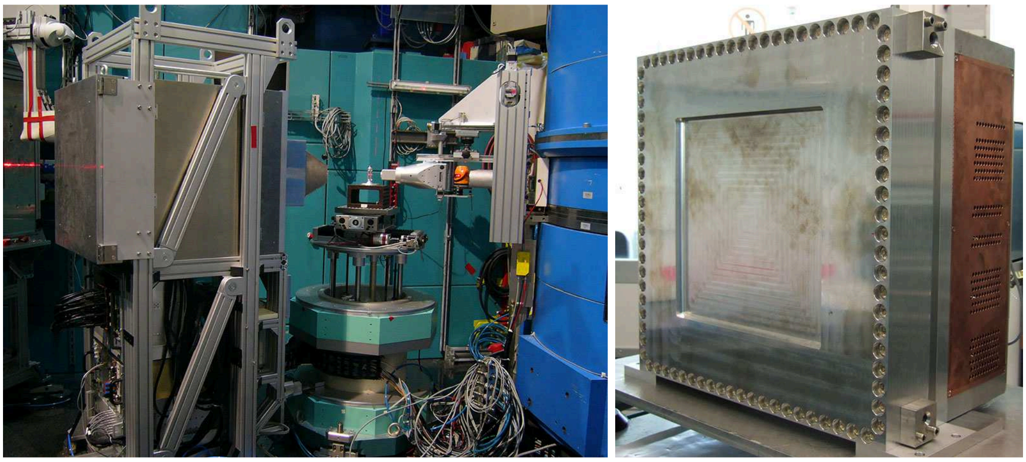
The materials science diffractometer STRESS-SPEC is operated by the Technische Universität München, the Technische Universität Clausthal and the German Engineering Materials Science Centre (GEMS), Helmholtz-Zentrum Geesthacht, at Forschungsneutronenquelle Heinz Maier-Leibnitz (FRM II). This diffractometer can be optimised either for texture analysis and/or strain measurements [1,2]. Its flexible instrument setup can be easily varied by using three different monochromators: Ge (511), bent silicon Si (400) and pyrolitic graphite PG (002). This selection of monochromators and the possibility to change the take-off angles from $2\Theta_M = 35^\circ$ to 110° allows finding a good compromise between resolution and intensity for each measurement problem [1].

The gauge volume defining optical system of primary and secondary slits is designed with regard to reproducibility of geometrical alignment and sturdiness. Both slit systems are linked to the sample table and detector in such a way that the center of the beam remains the same under all conditions. Instead of the secondary slit a radial collimator can be used in front of the detector. Samples can be aligned using theodolites and a camera system. A further STRESS-SPEC feature is the positioning system consisting of a Stäubli-6-axes robotic arm for texture and strain measurements (workload up to 30 kg) which can be mounted instead of the standard sample table. It enables more degrees of freedom for movements than an Eulerian cradle and can be also used as automatic sample changer for texture measurements [3].

To further improve the options at STRESS-SPEC a new detector was recently installed and successfully commissioned. In addition the original incoming beam slit system was upgraded for automatic gauge volume definition. During detector commissioning, measurements from the surface (200 µm) into the bulk, of a welded austenitic steel sample from a round robin exercise within the framework of the Network on Neutron Techniques Standardization for Structural Integrity (Net [4]) were successfully carried out.

New Hardware at STRESS-SPEC

The detector was developed with the design based on a concept of a Multi Wire Proportional Chamber (MWPC) developed within the scope of “MILAND” Joint Research Activity [5] of the NMI3 consortium (program 6 framework of the European Commission) [6]. It has an active area of 250 × 250 mm^2 and is filled with a gas mixture of 4 bar ^{3}He + 2 bar CF_4 resulting in a detection efficiency of almost 70% for thermal neutrons. Prior to the installation at STRESS-SPEC, the detector was thoroughly tested. The results of those experiments showed that a very high position resolution of Δx,y < 1.3 mm (FWHM) in both directions can be reached. Fig. 1 shows the detector in its new detector housing, together with the ancillary electronics and data acquisition system which is fully integrated into the instrument control software using the QMesyDAQ interface [7].



(a) (b)

Fig. 1 – (a) The MWPC detector including electronics in its housing. (b) The front view of the detector with electronics mounted at the rear side of the detector.

The first commissioning experiments at STRESS-SPEC clearly showed the resolution gains compared to the previous delay line detector. Fig. 2 shows images of the monochromatic (λ=1.66Å) transmitted neutrons through a BN-mask which was mounted directly on the front of the corresponding detectors. The BN-mask has the dimension of 255 × 255 × 5 mm^3 where 44 holes having a diameter of 2 mm and spaced vertically and horizontally by 39 mm were machined. Moreover the BN-mask is constituted at the centre by stripes of 1 × 20 mm^2 spaced by 3 mm as well as a fine raster of 9 holes of 0.5 mm diameter spaced by 5 mm. While features like the 1 mm vertical and horizontal stripes in the centre of the mask are blurred or even not visible (the small holes raster) using the old detector they can be easily discerned using the new MWPC detector. Full analysis of the resulting images confirms the results of the previously position resolution measurements. In addition, the detection efficiency is around 40% higher than with the old system. Combining this with much narrower peak profile, Fig. 3(a), results in an efficiency increase in case of residual strain measurements of almost a factor of 2 (Note: for rough comparison in residual strain measurements a figure of merit of FOM ≈ I/FWHM^2 can be utilized [8]).

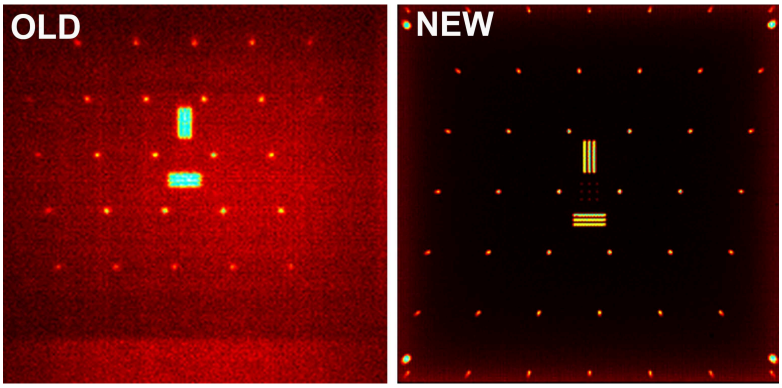
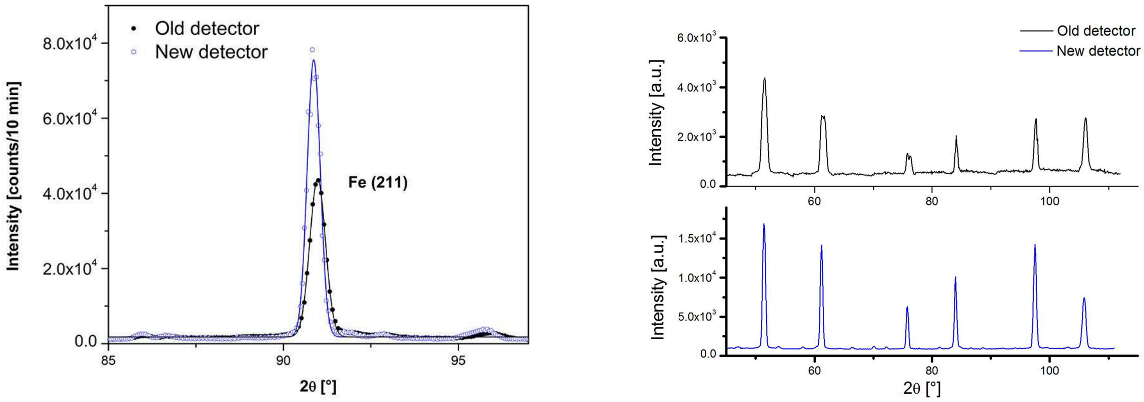


Fig. 2 – BN mask seen from the old delay line detector (left) and the new MWPC detector (right).



(a) (b)

Fig. 3 – (a) {211} α-Fe diffraction peak from a 1 mm cylinder of ferritic steel, FWHM = 0.53° with the old detector and FWHM = 0.44° and with the new one (b) Diffraction patterns of Si standard powder with the old detector (top) and the new MWPC detector (bottom).

In addition, Fig. 3 (b) shows a comparison of powder diffraction patterns of Si measured with both detector systems, showing besides the narrower peak profiles a significantly improved and almost flat background of the new MWPC detector. This together with the increased detection efficiency is beneficial for full pattern diffraction studies and the texture analysis program of the instrument. The detector is now in full user operation and it is foreseen to be further developed by facilitating its event mode data acquisition capabilities.

Besides the new detector, a new slit system defining the incoming, monochromatic beam just before the sample was installed. The new primary slit is fully motorised and allows defining gauge dimensions continuously from 0.1 × 0.1 mm^2 up to 7 × 17 mm^2, see Fig. 4. With the new system it is also possible to adjust the position of the slit with respect to the beam axis and sample centre in a more efficient and reproducible way. In addition due to the use of a precision safety clutch the slit can be repositioned without further realignment in case of a collision with the sample.

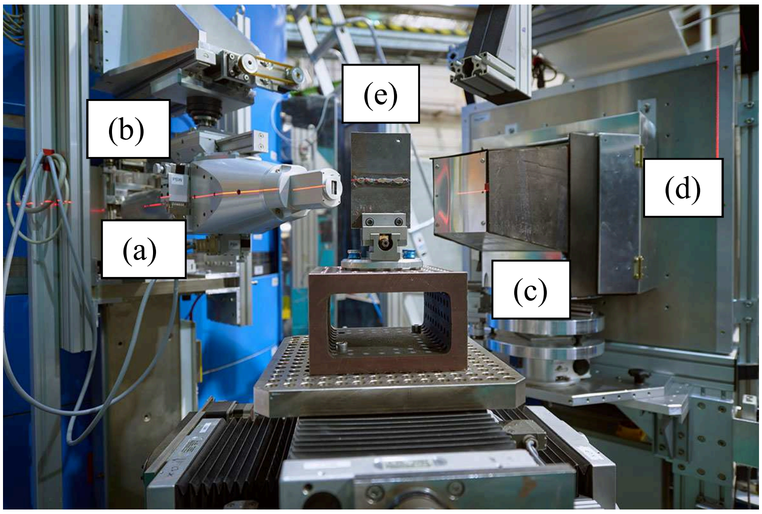
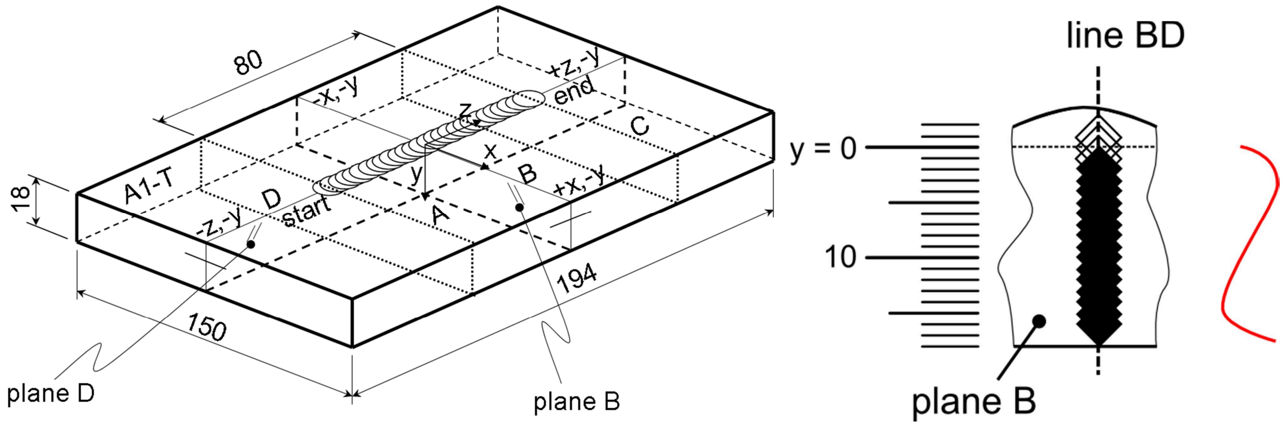


Fig. 4 – STRESS-SPEC: (a) Primary slit system; (b) precision safety clutch; (c) radial collimator; (d) detector; (e) sample

Method Developments – Spurious Strain Corrections

Thanks to the high flux reactor FRM II and the latest STRESS-SPEC improvements, measurements from the surface into the bulk, has seen an increase demand. A new developed analytical model for STRESS-SPEC [9] is now a routine tool for corrections of strain measurements close to the surface. Whenever the gauge volume is not totally immersed in the sample the measured strain must be corrected for the respective spurious strain [10]. As an example, a welded sample from the, NeT-Task Group 4 (TG4), a three-pass slot weld specimen in austenitic stainless steel [11], will be presented.

All specimens are made from an AISI type 316 austenitic stainless steel sample which is also used as a material for the fusion reactor ITER. The plates are 18 mm thick, 194 mm long and 150 mm wide, Fig. 5. The slot for the weld is about 10 mm wide and 80 mm long at the top face and 6 mm wide at its bottom. The edges of the plates are machined to obtain rectangular and parallel edges for easier and more reproducible specimen mounting for the strain measurements.



(a) (b)

Fig. 5 – (a) TG4 sketch specimen and coordinate system. The origin of the right handed coordinate system is on the top surface in the centre of the plate, y pointing perpendicularly through thickness, z pointing in direction of torch traverse, and x perpendicularly to the weld line. Start and end refer to the centre of the weld torch at z -40 and +40 respectively. The neutron measurements were done along the line BD, Fig. 5 (b), the intersection between planes B and D [12].

Neutron measurements were carried out on two different samples: after welding, Fig.6 (a) and after welding and subsequent machining, Fig. 6 (b) for the three main directions: longitudinal, transverse and normal to the weld direction. For the welded sample a set of cuboids were produced for reference measurements, d_0. Material of three different cuboids was extracted from three different locations: One from the parent material, a second one from the upper part of the weld metal and a third one from the lower part of the weld metal, see detail at [12]. For the welded and machined sample, since no strain free cuboids were available in time, the strains were calculated considering the assumption that the residual stress for the normal direction from the surface to the parent material is zero.

(a) (b)

Fig. 6 – STRESS-SPEC measuring setup for (a) AISI 316 sample after welding and (b) AISI 316 sample after welding and machining

The bent silicon Si (400) monochromator was set at a take-off angle of $2\theta_M = 76.5°$, which provides a wavelength around 1.68 Å. The measurements were performed around a scattering angle of $2\theta_S \approx 101^o$ for the austenite, {311} γ-Fe diffraction line. A gauge volume of $2 \times 10 \times 2$ mm^3 was defined by the primary slit and secondary radial collimator for the measurements of the transversal and normal directions for the welded and machined sample. A $2 \times 2 \times 2$ mm^3 gauge volume was used for the measurements of the longitudinal direction. For the weld sample in all three main directions a gauge volume of $3 \times 2 \times 3$ mm^3 was selected. Elasticity constants of E_{311} = 175 GPa, ν_{311} = 0.31 for the {311} γ Fe line, were used for residual stresses calculation [13].
Measurements were done along the samples cross sections (along y) starting in the middle of the weld line, x=z=0, Fig. 5. For the welded sample from a depth of around 1 mm underneath the surface until 16 mm with step size in y-direction of Δy=1 mm. For the welded and subsequent machined sample, the measurements were carried out from a depth of 200 µm from the surface into the bulk, 16 mm. Primarily with a step width of 0.2 mm, until 1 mm depth, and then with steps of 1 mm. Therefore, the measured strains for the positions where the gauge volume were not totally immersed at the material must be corrected for the respective spurious strains. The spurious strains were calculated using the analytical model developed for the STRESS-SPEC diffractometer according to the chosen instrument setup. In Fig. 7 the residual stresses profiles for the welded and welded and machined sample after spurious strains corrections are plotted for the longitudinal, transverse and normal directions. For the residual stress profile of the welded and machined sample, the positions depth (whenever the gauge volume was not totally immersed in the material) are corrected for absorption and represents the gravity centre of the partially immersed gauge volume.
The influence of the welding plus machining is minimal from 10 mm depth upwards. However, there is a significant influence of the machining on the welded residual stress profile from the surface until the depth of around 4 mm. The profiles in longitudinal and transverse directions show that the stress values decreases abruptly reaching compressive values near the surface. The effect of machining was fulfilled and verified by the measurements: reduce the tensile residual stress to even compression at the surface. Finite element simulations which are undergoing will be compared with these experimental results and published elsewhere.

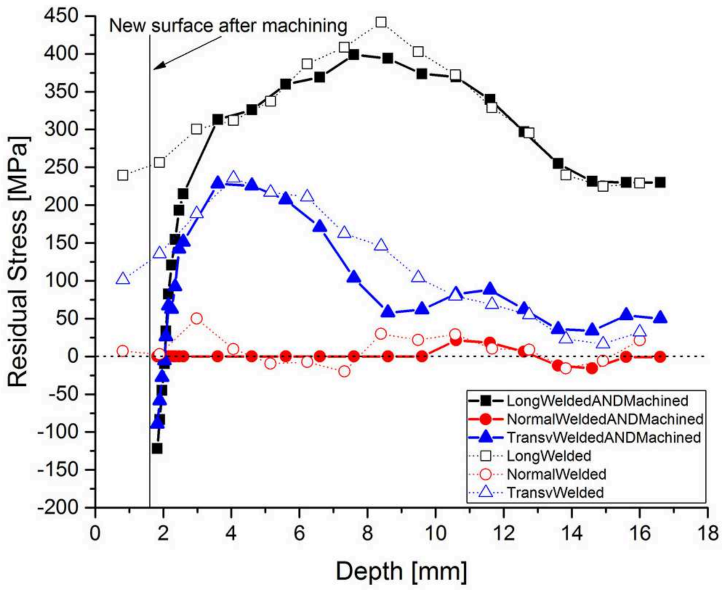


Fig. 7 – Residual stress profile across the AISI316 sample cross section, from the surface (200 µm) to the bulk, 16 mm depth. Comparison of residual stresses of the samples: welded (open symbols) and welded and machined (closed symbols). Comparisons are shown for the three main directions: longitudinal, transversal and normal to the weld direction. Errors are not plotted for the sake of clarity but they are maximal 30 MPa for the surface measurements and much smaller for the other ones.

Conclusions

In this work the main and latest improvements of the material diffractometer STRESS-SPEC were presented. Due to these improvements, relevant increase in speed and precision of strain and texture measurements could be achieved. Consequently, for instance, time consuming measurements from the surface into the bulk can now be routinely carried out. A new analytical model for spurious strains calculations for any possible instrument setup and measured material permits corrections of the measured strains at the surface, eliminating extra time consuming measurements for spurious strains corrections.

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