Session IX - Welding, Fatigue and Fracture 1

S9 - 1

IMPROVEMENT OF SURFACE RESIDUAL STRESS IN THIN WELD METAL MATERIALS BY LOW-ENERGY SHORT-PULSE LASER PEENING

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Laser peening (LP) is a post-processing method to improve fatigue strength, residual stress, and stress corrosion resistance of structural welds and mechanical parts. LP can induce compressive residual stress on the surface of a material by using the impact force of high-pressure plasma, which reaches several GPa, generated by irradiating a high-intensity laser beam on a metal surface covered with water [1,2]. This residual compressive stress on the material surface is one of the main factors that improve the strength of the material.

In conventional LP, the laser pulse energy is large (0.1-10 J) and the pulse width is long (several ns to several tens of ns). Therefore, deviation from the optimal LP application conditions may cause macro melting on the metal surface due to the large thermal effect of laser irradiation. This would put the metal surface in a state of tensile stress after laser irradiation, resulting in the starting point of embrittlement and cracking of the material. On the other hand, the microchip laser [3] used in this study has a pulse width and pulse energy that are approximately 1/10 or less than those of conventional LP lasers, and has the advantage of minimizing the thermal effects on the metal surface and minimizing the adverse effects described above. By taking advantage of the above features, we have developed a technology to introduce shallow compressive residual stress due to LP into thin plate parts [4].

In this study, low-energy short-pulse microchip LP was applied to butt-welded A5083 aluminum alloy sheets 2 mm thick, and improvements in surface residual stress was confirmed. The results of LP treatment on A5083 weld samples are shown in Fig.1 and Fig. 2.

Fig. 1 shows the results of the residual stress measurements on A5083 after low-energy short-pulse LP treatment (1 to 4 in the graph indicate arbitrary measurement points.). The welded sample was subjected to low-energy LP in 5 mm wide areas covering the weld-toe on both sides. The laser irradiation energy is 1.0 mJ, the focusing diameter is 0.18 mm, and the irradiation pulse duration is 0.43 ns. Fig. 1 shows that the surface residual stress of the samples after LP treatment is significantly improved compared to that before LP. Fig. 2 shows the results of residual stress depth profile measurement of A5083 base metal after LP (The laser parameters used were the same as described above.). X-ray diffraction and electrolytic polishing were repeated alternately to obtain RS depth profiles of peened and unpeened areas. It was shown that sufficient residual compressive stress was introduced to a depth of about 0.1 mm.

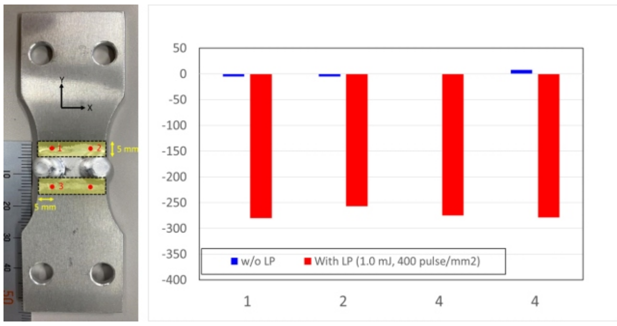

Figure 1. Surface Residual stress of A5083 welded sample

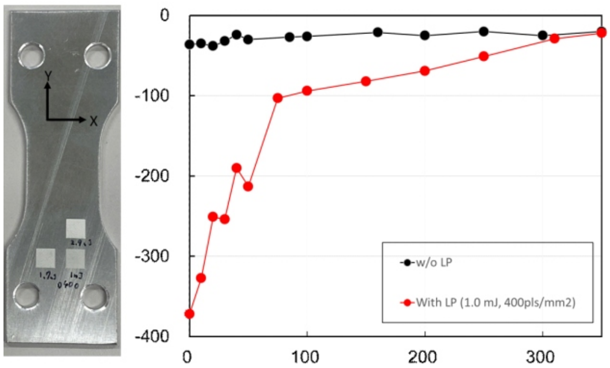

Figure 2. Residual stress depth profile of A5083 base metal.

From the above, low-energy short-pulse microchip LP is effective in improving the surface residual stress of thin metal materials. In the presentation, detailed fatigue test results will be presented in addition to the residual stress results.

1. R. Fabbro, J. Fournier, P. Ballard, D. Devaux, and J. Virmont, *J. Appl. Phys*., **68** (1990), 775-784.
2. Y. Sano, *Metals*, **10** (2020), 152.
3. T. Taira, *Opt. Mater. Express*, 1, **5** (2011), 1040-1050.
4. Y. Sano, K. Masaki, Y. Mizuta, S. Tamaki, T. Hosokai, T. Taira., *Metals* **11**(2021), 1716.

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S9 - 2

EFFECT OF HFMI TREATMENT ON RESULTING WELD TOE GEOMETRY AND RESIDUAL STRESSES

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Efficient and environmentally friendly use of materials in mobile machines often means the design of structures with the performance-to-weight ratio at the highest possible level. In the case of steels, this means, for example, steels produced with minimum emissions and reduction of steel consumption, favouring high-strength steels. In practice, stress levels can be assumed to arise, leading easily to fatigue failure, the well-known failure criterion in welded structures under cyclic and fluctuating loads. Comprehensive and powerful finite element software have enabled the use of advanced assessment methods, making it possible to take fatigue into account already at the design stage. In addition, fatigue assessment methods are constantly studied and compared e.g. with new materials and manufacturing methods, making latest information well available. In addition, new assessment methods are taking in the field, such as the 4R method developed by the Laboratory of Steel Structures, LUT University [1, 2].

The establishment of both new post-weld treatments and efficient fatigue assessment methods in the scope of international standards and guidelines is time-consuming and requires comprehensive research studies. One of the latest updates in the field is the high-frequency mechanical impact (HFMI) treatment of the weld toe. Research on the technique increased in the late 1900s. In 2016, the HFMI treatment was embedded in a guideline by the International Institute of Welding (IIW) [3] and will probably be recognized in the next version of Eurocode 3 [4]. The technique is based on impacting locally weld toe with a needle-like tool. As a result, the process modifies smoother transition geometry, improves surface quality, and introduces compressive residual stresses. Finally, an increase in the fatigue strength capacity of HFMI-treated welds can be obtained. Although the principle of the HFMI treatment itself is commonly known, the uniform treatment parameters are not possible to provide due to the great influence of structures being treated as well as different HFMI devices unique features. From the fatigue viewpoint, this is not in great importance, since despite the variation in the outcome, a considerable increase in the fatigue life can be achieved, by following the general and equipment manufacturer's introductions as well as by performing quality control [3]. However, variation in residual stress distribution and local geometry poses a challenge to the use of more accurate assessment methods [1, 2] and finite element (FE) based assessments [5], demanding uniform treatment quality and detail-based verified parameters.

In this study, the effect of HFMI treatment on the fillet weld toe geometry and surface residual stresses was studied. Both as-welded (AW) and HFMI-treated specimens were examined, however, more focus in this work was put on comparing results obtained using different HFMI pin radii. The HFMI treatments were performed using a pneumatic HiFIT device, applying three different standard pins with diameters of 3, 4, and 5 mm offered by the manufacturer. The treatments were carried out by an experienced laboratory person according to the manufacturer's instructions, recording all process parameters. In addition to the pin diameter, different base material grades, namely S355, S700, and S1100, were studied. In all studied cases, structural steel in plate thickness of 8 mm and Böhler Welding Union X96 solid wire was applied. The test specimens were manufactured in the LUT Laboratory of Steel Structures.

The local weld toe geometries were studied by measuring weld toe after and before HFMI treatment using the Keyence VR-3000 profilometer. Subsequently, the fitted curves could be compared both numerically and graphically, as shown in Figure 1. Transversal residual stresses at the treated toe and plate surface were measured before and after the HFMI treatment, measuring the stress distribution along the specimen

As expected by the observations in the previous studies [3], HFMI treatment can clearly modify local geometry and residual stress conditions at the weld toe. In all cases, the shape of the weld toe at the critical point became smoother, and the radii changed considerably (Figure 1). Both weld and base metals were processed, and a clear boundary between the base material and filler metal could not be observed after the HFMI treatments. Before the treatment, measured toe radii were roughly 0.1 to 0.5 mm, while after it, they corresponded to the pin geometry. The material strength was not found to have a significant effect on the results, although there was a slight variation in the results. It could also be noted that after the treatment, the weld geometrical starting point was slightly repositioned due to the

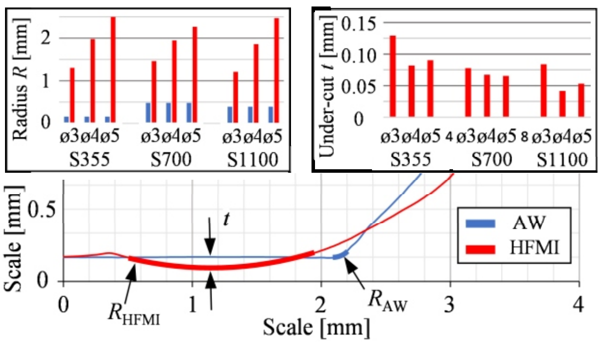

Figure 1. Weld toe rounding in as-welded and HFMI-treated condition.

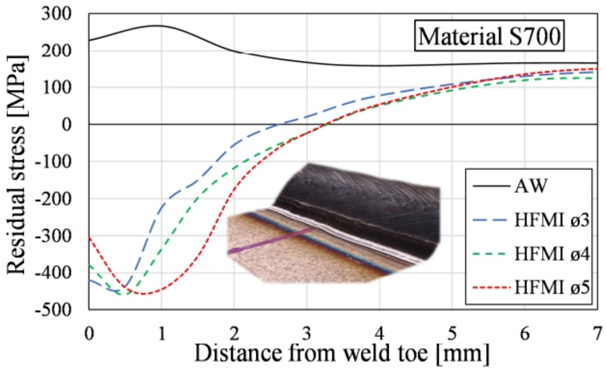

Figure 2. Distribution of stresses perpendicular to weld, measured from the surface of the plate.

treatment groove. The depth of the groove varied from 0.13 mm to 0.04 mm, depending on the material strength and pin radius.

Compressive residual stresses at weld toe were introduced by the HFMI treatments. In the AW state, the residual stresses varied between tensile 300 MPa to compressive -50 MPa, while after HFMI treatment stresses were compressive side from -200 to -700 MPa. The maximum compressive stress depended on the steel grade, with the pin size only affecting the shape of residual stress distribution (Figure 2).

Based on this study, the effect of HFMI treatment on local geometry and residual stress distribution should be studied in more detail. From the fatigue viewpoint, a lot of research work on HFMI treatments has been carried out over the past decades. However, increasing use of simulation-based assessment methods necessitates more accurate data from actual structure. For example, in the 4R method, both the shape and the residual stresses of the local detail are utilized in fatigue assessment. For details in the as-welded state, this is usually not a problem, as geometry and residual stress data are well available, or they can be conservatively approximated. In addition, manufacturing critical details are often carefully instructed and controlled, ensuring consistent weld quality. However, HFMI treatment is rarely as controlled, resulting in large variations and rough assumptions, finally making approximation methods inaccurate.

1. T. Nykänen, H. Mettänen, T. Björk & A. Ahola. (2017). Fatigue assessment of welded joints under variable amplitude loading using a novel notch stress approach. *International Journal of Fatigue*, **101**, 177–191.

2. T. Björk, H. Mettänen, A. Ahola, M. Lindgren & J. Terva. (2018). Fatigue strength assessment of duplex and super-duplex stainless steels by 4R method. *Welding in the World*, **62**(6), 1285–1300.

3. G. Marquis & Z. Barsoum. IIW Recommendations for the HFMI Treatment For Improving the Fatigue Strength of Welded Joints. Singapore: *Springer Singapore.* 2016.

4. prEN-1993-1-9 (2023) Eurocode 3: Design of steel structures - Part 1-9: Fatigue.

5. T. Pesonen, J. Riski, K. Lipiäinen, A. Ahola, & T. Björk (2024). Framework for a finite element method-based simulation approach to fatigue assessment of welded joints using the 4R method. International Journal of Fatigue, **181**, 108148-. https://doi.org/10.1016/j.ijfatigue.2024.108148.

S9 - 3

COMBINATION OF SYNCHROTRON EDXRD AND DILATOMETRY TO DETERMINE STRESS DEVELOPMENT IN STRUCTURAL STEELS AS A RESULT OF LASER BEAM WELDING

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Localised heat input into metallic materials, such as from a laser beam, and subsequent rapid cooling leads to distortion and residual stresses in components. As well as a loss of geometric accuracy, this can also lead to catastrophic failure of the design if the loads applied to the component in service are superimposed on the residual stresses and the yield point is exceeded.

The determination of residual stresses in components made from engineering alloys is therefore the basis for application-oriented design. Using high flux density white synchrotron radiation, the present work compares specimens of structural steel grade S235 with specimens of chromium-nickel steel material number 1.4304 with respect to their residual stresses in the component after laser bead-on-plate welding. The experiments were carried out at the German Electron Synchrotron (DESY) in Hamburg. The high-energy wiggler beamline P61A of the Petra III electron accelerator was used. The white X-ray beam used at the beamline is characterised by a broad energy spectrum from 0 keV to 200 keV applied to an energy dispersive diffraction setup. [1]

Lattice distortions due to local heat input from the laser beam will result in a peak shift towards different energy levels in the experimental setup. This peak shift behaviour can be observed with the realised transmission-based diffraction setup. In contrast to surface diffraction methods, this approach using the high flux synchrotron beam allows measurements to be made at a depth of 4 mm within a sample of 8 mm thickness. Even with this material thickness, exposure times of only 1 s could be achieved. The resulting

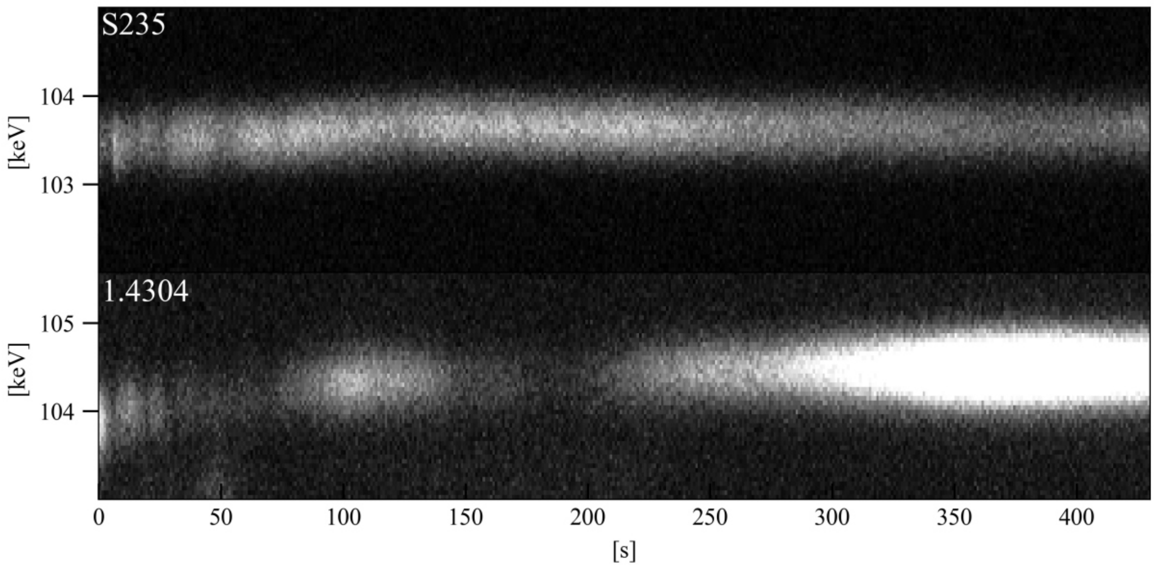

Figure 1. Diffraction peak shift over time for the *(400)* peak.

peak shift to different energies can be observed as the sample cools. In addition, grain growth within the measurement can be observed as well as the phase transformation in the mild steel sample from austenite to ferrite due to cooling. (**Figure 1**) Particular attention is paid to the differences in stress development due to the different phase transformation behaviour and the different coefficients of thermal expansion.

The basis of the comparison is a precise knowledge of the phase transformations and thermal expansions which are determined by quenching dilatometry. Small cylindrical specimens of the material, 10 mm in height and 3 mm in diameter, are prepared and heated to above the austenitisation temperature. The material is then quenched using a stream of inert gas and the change in length of the sample is accurately recorded. Repeating this procedure gives a precise knowledge of the correlation between cooling rate and phase transformation temperature. The superposition of dilatometry tests with diffraction-based strain measurements allows a quantitative conclusion to be drawn about the metal physical processes that contribute mainly to the development of residual stresses in the selected engineering alloys.

The finding of a higher strain in the bead-on-plate welded specimen of the alloyed 1.4304 steel compared to S235 indicates a reduction in stresses possibly due to the austenite-ferrite phase transformation. This finding was supported by the determination of the phase transformation in the dilatometer. One way of explaining this behaviour is the interference of the stresses developed with the phase transformation in the welding experiment. [2]

1. B. Kämpfe, F. Luczak, B. Michel, *Particle & Particle Systems Characterization*, **22**(6), (2005), 391–396.
2. S. Denis, A. Simon, G. Beck, *Transactions of the Iron and Steel Institute of Japan*, **22** (7), (1982), 504–513

The investigations presented here were carried out in the context of the Collaborative Research Centre SFB1120-260036980 “Precision Melt Engineering” at the Welding and Joining Institute (ISF) of RWTH Aachen University and were funded by the German Research Foundation (DFG). We would like to take this opportunity to thank them for their funding and support.

We thank DESY (Hamburg, Germany), a member of the Helmholtz Association HGF, for providing the experimental facilities. Parts of this research were carried out at PETRA III and we thank all collaborators for their support in using the beamline P61A. The measurement time was provided for the application I-20210328.

S9 - 4

ANALYSIS OF THE EFFECT OF RESIDUAL STRESS FORMED BY SURFACE TREATMENT PROCESS ON THE MECHANICAL PROPERTIES OF S45C WELDED JOINTS

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Residual stresses generated during the welding process of homogeneous parts affect the fatigue life and fatigue crack growth of the parts. Defects and residual stresses that occur during welding cause cracks in the weld area and lead to fracture. To solve these problems, research is being conducted to control the residual stress occurring in welds and evaluate the effects of residual stress. In this study, we measured and compared the effects of residual stress on the occurrence of cracks in the weld zone and mechanical properties of S45C material, a medium-carbon steel used in automobile parts. To measure and analyze residual stress, three measurement techniques, the Contour method, hole-drilling method, and XRD method were used to conduct comparative analysis and establish a measurement technique suitable for welded parts. Residual stresses in three directions were analyzed using the multiple method method. In addition, three surface treatment processes were used to apply compressive residual stress to the weld zone: laser shock peening (LSP), ultrasonic nanocrystalline surface modification (UNSM), and shot peening (SP). Changes in residual stress for each process were analyzed and optimal post-process conditions were derived. Fatigue tests were conducted before and after applying compressive residual stress to evaluate mechanical properties, and the effect of changes in microstructure caused by post-processing on hardness was analysed.

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ASSESSMENT OF THE ULTRASONIC IMPACT TREATMENT (UIT) FOR IMPROVING LIFETIME OF IN-SERVICE METALLIC WELDED STRUCTURES

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Welded assemblies are widely used in infrastructure projects, including bridges, buildings, and offshore structures like oil platforms and wind turbines. However, assessing the integrity and ensuring the long-term durability of welded structures remain challenging due to fatigue and environmental effects like corrosion. Fatigue leads to crack initiation and propagation, especially in stress concentration areas at the weld toe. The crack growth kinetic is strongly influenced by the stress state, including residual stress and stress concentration [1]. To enhance the lifespan of welded assemblies, various surface treatment and post-weld finishing methods are employed to reduce tensile stress concentrations at the weld toe. These processes involve releasing stresses locally, modifying local geometry to reduce the stress concentration factor, or more likely introducing beneficial compressive stresses in the weld region. Previous research on fatigue in high-strength steel (HSS) welded assemblies, has focused on studying the effects of post-weld treatments on the fatigue performance of welded joints [2]. However, existing literature often focuses on experimental results for initial post-weld treatment before the structure is put into service and ages. Moreover, most studies involve shot peening or grinding as a post-weld finishing method [3, 4], but alternative methods with fewer implementation constraints, such as no need for a confinement area, less bulky and costly equipment, are equally notable.

This paper presents an ongoing study on HSS welded T-joint aiming to complement existing research by considering the impact of the ultrasonic impact treatment (UIT) as a post-weld finishing operation at different stages of the welded assembly's lifespan. The study includes both experimental and numerical works. The finite element (FE) model is used to simulate various loading conditions and predict the thermomechanical behaviour from the welding phase, including cooling and the generation of initial residual stresses, to the post-weld treatment and the resulting residual stresses. Experimental tests are conducted to determine the fatigue lifespan of each specimen series, corresponding to different stages of their service life before post-weld finishing. The reference specimens, labelled "TW", correspond to the welded T-joint without any post-weld treatment and having a FAT90 fatigue class according to Eurocode 3 part 1-9 [5]. The levels of fatigue ag-

Table 1. Details of each series of tested specimens.

Series	Level of fatigue ageing	Post-weld treatment	Number of specimens
TW	reference specimens	No post-weld treatment	9
TW-UIT-PF0	0 %	UIT	9
TW-UIT-PF25	25 %	UIT	2
TW-UIT-PF50	50 %	UIT	3
TW-UIT-PF75	75 %	UIT	3

ing, before post-weld treatment, are expressed as a percentage of the lifespan obtained for the reference specimens under a given stress range and a stress ratio R set at 0.1. Table 1 provides details on the levels of fatigue aging, nomenclature, and the number of tested specimens in each series.

The S-N curve obtained for the TW-UIT-PF0 series shows the improvement in fatigue life achieved by applying UIT to the welded joint at the initial stage. Compared to the reference specimens, both the cut-off limit (endurance limit) and the slope of the S-N curve are modified. Applying UIT at different fatigue aging levels also appears beneficial for specimens with initial fatigue aging of 25 % and 50 % of the fatigue life. For TW-UIT-PF75 specimens, with 75 % initial fatigue aging, a service life equivalent to that of the reference specimens was achieved. This suggests that the fatigue aging of the specimens has already caused significant damage to the welded T-joint, rendering post-weld treatment ineffective. In conclusion, this study shows that ultrasonic impact treatment significantly enhances the fatigue life of unaged and aged welded T-joints. These initial tests establish a database of results applicable to fatigue design, calculation, and verification of welded structures. Further investigations are required to optimize treatment parameters while considering practical constraints. A comprehensive understanding of the underlying mechanisms responsible for the observed fatigue life enhancement will be achieved through analysing residual stresses before and after UIT and comparing them with data from FE models. Finally, exploring the effects of environmental aging would be of interest in future studies.

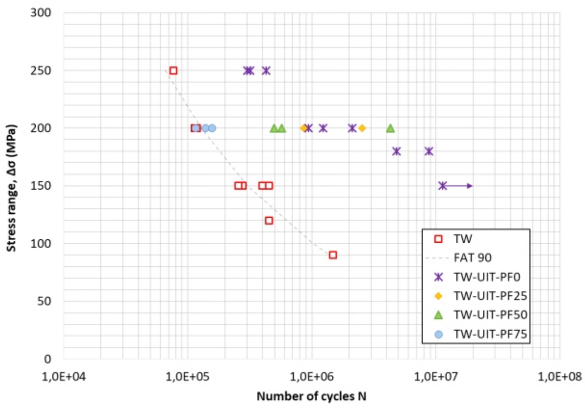

Figure 1. Fatigue life for each series of specimens.

1. N. T. Ninh, M. A. Wahab, The effect of residual stresses and weld geometry on the improvement of fatigue life. *Journal of Materials Processing Technology*, **48(1–4)**, (1995), pp. 581–588.
2. I. Sas, J. Lukács, Post-treatment of welding joints of high strength steels I.: improving weld geometry - overview, *Multidiszciplináris tudományok*, **12**. kötet, 1. sz. (2022), pp. 13–27.
3. L. Dieng, D. amine, Y. Falaise, S. Chataigner, Parametric study of Finite Element modelling of shot peening on welded joints, *Journal of constructional steel research*, **130**, (2017), pp. 234-247.
4. A. V. Hansen, H. Agerskov, J. Bjřrnbak-Hansen, Improvement of fatigue life of welded structural components by grinding, *Welding in the World*, **51**, (2007), pp. 61–67.
5. EN 1993-1-9; Eurocode 3: Design of Steel Structures – Part 1-9: Fatigue. CEN: Brussels, Belgium. 2005.

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Session X - Welding, Fatigue and Fracture 2

S10 - 1

INFLUENCE OF THE WELD GEOMETRY ON THE RESIDUAL STRESS REDUCTION USING LOW TRANSFORMATION TEMPERATURE WELDING CONSUMABLES

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Low transformation temperature (LTT) welding consumables offer an innovative approach to increasing the fatigue strength of welded high-strength steel structures, apart from the conventional methods of post-weld heat treatment [1] . LTT welding consumables are characterized by a martensitic phase transformation near ambient temperature, which generates compressive residual stresses in the weld and heat affected zone (HAZ) [2]. The aim is to achieve a weld geometry, which generate high compressive residual stresses at the fatigue crack critical weld toe.

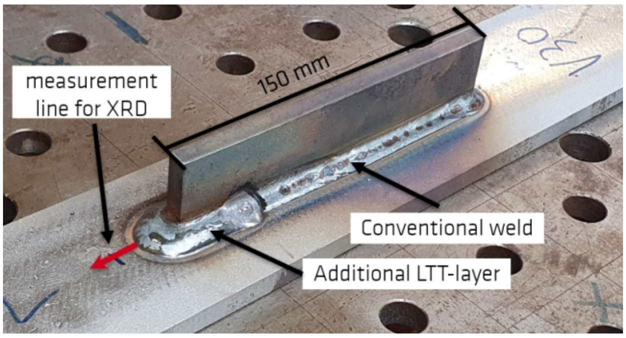

Figure 1. Longitudinal stiffener with additional LTT.

Longitudinal stiffeners were gas metal arc welded using a conventional welding consumable; the base material was a high strength steel S700M. A chromium-nickel alloyed LTT consumable was deposit subsequently just on front sides of the stiffeners (Figure 1). Different welding speeds and offsets led to varying cross sections of the weld (Figure 2). The residual stresses were determined using X-ray diffraction (XRD) in the crack critical HAZ [3].

When using only the conventional, the HAZ is characterized by high tensile residual stresses of about 350 MPa.

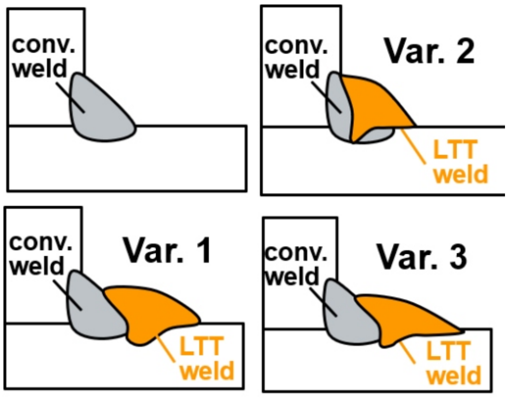

Figure 2. Cross section of the geometries.

The additional application of the LTT alloy leads to a significant reduction of the tensile residual stresses. Depending on the weld geometry, even compressive residual stresses up to -150 MPa can be observed at the weld toe.

1. Hensel, J., et al., Capability of martensitic low transformation temperature welding consumables for increasing the fatigue strength of high strength steel joints. *Materials Testing*, 2020. **62**(9): p. 891-899.
2. Kromm, A., J. Dixneit, and T. Kannengiesser, Residual stress engineering by low transformation temperature alloys-state of the art and recent developments. *Welding in the World*, 2014. **58**(5): p. 729-741.
3. Hobbacher, A.F., The new IIW recommendations for fatigue assessment of welded joints and components - A comprehensive code recently updated. *International Journal of Fatigue*, 2009. **31**(1): p. 50-58.

S10 - 2

NON-DESTRUCTIVE ESTIMATION OF THREE-DIMENSIONAL RESIDUAL STRESSES IN SPOT-WELDED JOINTS USING X-RAY DIFFRACTION AND EIGENSTRAIN THEORY

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As the residual stress has a relatively large influence on the crack growth rate, the fatigue life can be predicted if the three-dimensional residual stress distribution can be assessed non-destructively in the field. However, the X-ray diffraction method can only measure residual stresses on the surface of a component. Neutron diffraction can be used for the non-destructive measurement of 3D residual stresses down to a depth of several tens of millimetres; however, it can only measure them discretely and requires considerable time. Therefore, Korsunsky et al. used the eigenstrain reconstruction method [1-3] to determine the residual stress distribution over an entire structure from the elastic strains measured by neutron diffraction. However, neutrons are only available in dedicated facilities, making it difficult to perform measurements at manufacturing sites. Therefore, there is a need for the practical application of a three-dimensional residual stress estimation method using X-ray diffraction and the eigenstrain theory [4]. In this method, the three-dimensional eigenstrains [5] are estimated from the non-destructively measured surface elastic strain using inverse analysis [6]. The estimated eigenHstrains are then applied to a finite element model to reproduce the three-dimensional residual stress distribution. The aim of this study was to demonstrate via numerical analysis that the residual stress distribution in spot welds can be evaluated with a relatively high accuracy using this method, even assuming errors in X-ray diffraction measurements.

The eigenstrain, as defined in this study, is the inelastic strain that causes residual stress and does not necessarily correspond to the actual physical inelastic strain. The relationship between the elastic strain at the surface of the structure and the three-dimensional eigenstrain $\{\ ^*\}$ can be expressed as follows:

$$\{\ _e\} \quad [R]\{\ ^*\}, \tag{1}$$

where $[R]$ is the elastic response matrix relating the surface elastic strain to the eigenstrain over the entire structure, which can be determined if the Young's modulus, Poisson's ratio, and geometry (including constraint conditions) of the target member are known. Based on the relationship in Eq. (1), the inverse analysis used to estimate the three-dimensional eigenstrain using the surface elastic strain can be expressed as follows:

$$\{\quad\} \quad [R] \quad \{\quad\}, \tag{2}$$

where $[R]^+$ is the Moore-Penrose general inverse [7, 8] of the matrix $[R]$. As the elastic strain of the component surface can be measured non-destructively using X-ray diffraction, the eigenstrain of the entire component can be obtained non-destructively. The three-dimensional residual stress distribution was determined by inputting the estimated eigenstrain into a finite element model.

The inverse analysis in this study estimates the three-dimensional eigenstrain from two-dimensional surface information. To obtain a relatively high estimation accuracy, the number of unknowns should be reduced such that it is sufficiently smaller than the amount of measured information. In this study, the eigenstrain distribution decaying radially from the weld point was approximated using a function that multiplied a Gaussian function and a Chebyshev polynomial. Seven functions of order 0 to 6 were approximated for the *r-, -, and z*-directional components of the eigenstrain contributing to the residual stresses. The distribution width of the Gaussian function was determined using the response surface method, where the difference between the measured and estimated surface elastic strains was the objective function. Furthermore, Tikhonov's optimisation method [9] was used for the solution in the inverse analysis.

The finite element model used in this analysis is shown in Fig. 1. The model consists of two disks of 2 mm thickness with spot welding at $r = 0$ mm, closely welded from $r = 0$ mm to $r = 4$ mm at $z = 2$ mm. The model is 1/36 from 0 to 10° due to the axisymmetric boundary conditions. The elastic strains used for the estimation are the values of the *r*- and -directional components at all nodes on the surface of the plate ($z = 4.0$ mm). Values following a normal distribution with a standard deviation of 20 MPa were determined using random numbers and added to each surface elastic strain as a measurement error.

A comparison between the correct and estimated residual stresses is shown in Fig. 2 and Fig. 3. The estimated residual stresses using elastic strains on the top surface ($z = 4$ mm) without and with measurement error are shown as black dotted and yellow dashed lines, respectively. The results of the estimation of the residual stresses using this method showed that the residual stresses could be estimated with a relatively good accuracy. The estimation accuracy of this method was found to be less sensitive to measurement errors.

1. A. M. Korsunsky, *Journal of Mechanics and structures*, **1**, (2006), pp. 259–277.
2. A. M. Korsunsky, *A Teaching Essay on Residual Stresses and Eigenstrains*, Butterworth-Heinemann, (2017).
3. F. Uzun and A. M. Korsunsky, *Finite Elements in Analysis and Design*, **155**, (2019), pp. 43–51.

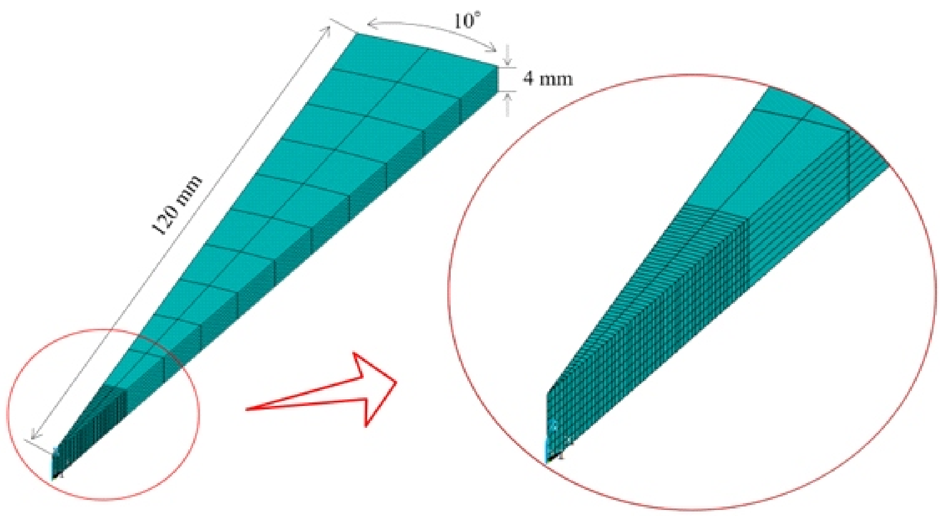
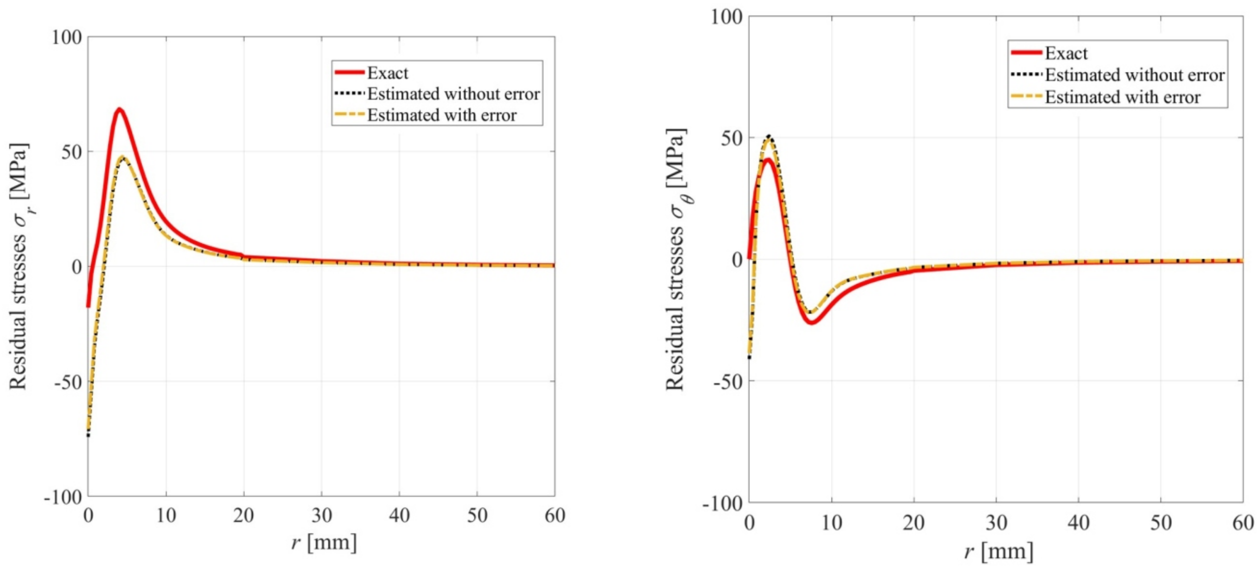


Figure 1. Finite element model of resistance spot welding with 1/36 due to axial symmetry conditions.



(a) r

(b)

Figure 2. Comparison of the exact and estimated residual stresses at = 0 degree and z = 0 mm.

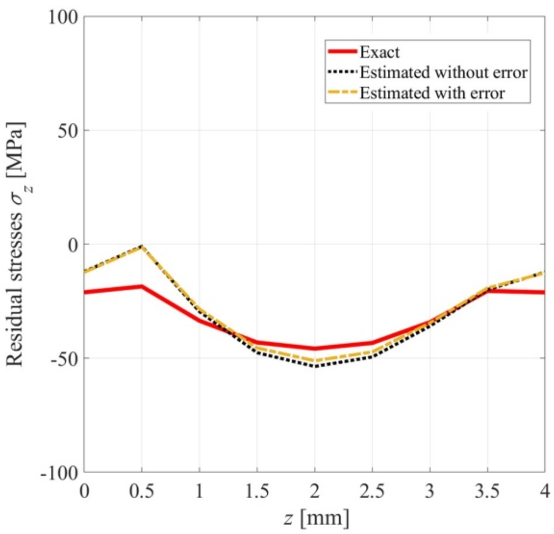


Figure 3. Comparison of the exact and estimated residual stresses in the thickness direction at r = 0 mm.

4. M. Ogawa, *Transactions of the Japan Society of Mechanical Engineers*, **80**, (2014), SMM0195.

5. T. Mura, *Micromechanics of defects in solids*, Martinus Nijhoff Publishers, 1987.

6. S. Kubo, *Inverse Problem*, Baifukan, 1992.

7. E. H. Moore, The American philosophical society, **1**, (1935).

8. R. Penrose, *Mathematical Proceedings of the Cambridge Philosophical Society*, **51**, (1955), pp. 406-413.

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S10 - 3

RESIDUAL STRESS FORMATION DURING REPEATED GOUGING AND REPAIR WELDING CYCLES OF HIGH-STRENGTH STEELS

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The construction of foundation and erection structures for wind power plants requires the use of modern, sustainable and resource-efficient high-strength fine-grained structural steels. Weld defects due to the welding process are unacceptable. To overcome this issue, local thermal gouging followed by re-welding is a common and cost-effective method. The high shrinkage restraint of the gouge by the surrounding structure can cause crack initiation when design and re-weld induced residual stresses are superimposed. This risk is intensified by the progressive degradation of the microstructure and mechanical properties of high-strength steels during the weld repair process.

This investigation focuses on high-strength steels S500MLO for offshore applications and S960QL for mobile crane applications. The reduction and development of residual stresses caused by local thermal gouging and re-welding was investigated. Digital Image Correlated (DIC) stress-strain analysis was performed during preheating, welding and cooling. The results of the global DIC analysis and local longitudinal and transverse residual stresses of the weld determined by X-ray diffraction were found to be in good agreement. Furthermore, different stress levels were identified during gouging and welding. Repeated repair cycles led to an increase of longitudinal and transverse residual stresses in the weld metal as well as a hardness increase in the heat affected zone.

S10 - 4

INVESTIGATION OF RESIDUAL STRESSES IN HOLE FILLING REPAIR WELDS BY TENSILE TESTING AND DIGITAL IMAGE CORRELATION

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Yield strength and tensile strength are mechanical properties of materials often used as a basis for design and construction. A simple method commonly used to determine these properties is the tensile test, where a force regulation or a constant strain rate is applied to the specimen. The applied force is measured, and the effective bulk stress can be deduced from geometric measurements of the specimen. Using Digital Image Correlation (DIC), which detects changes in an superficially applied speckle pattern, the local strain on the surface of the specimen can be calculated. The resistance determined using a load cell during the tensile testing is a superposition of residual stresses resulting from manufacturing processes such as rolling and welding, and the testing load, which is applied to maintain the desired strain rate. The local strength distribution in superposition with residual stresses have an impact on the fracture resistance of metallic parts.

Hole welds or fillet welds in holes offer a way to induce thermal stresses in flat specimen. Historically, these types of weldments have been regulated by [1] American AWS D1.1 and [2] European DIN EN 1993-1-8, with the European standard allowing residual stresses in the part to be ignored. However, depending on the size of the voids filled with filler material, the stress may not be negligible, so the effects and magnitudes of the residual stresses are investigated.

In the present work, flat tensile test specimens of defined geometry were produced. These specimens contain holes of different diameters and hole patterns which are additively filled using a gas metal arc welding (GMAW) process with varying infill strategies. Before welding the prepared specimen, as well as undamaged specimens of the same material, were stress-relief annealed. After annealing the specimens were clamped onto a tilt-turntable with the hole in flat position. Using spacers, a steel plate was positioned beneath the hole to prevent fusion with the table. After preparation the hole was filled up layer by layer using different infill strategies. Following, the plate was removed, and the bottom site welded with an additional layer to close eventual fusion defects. The welded and the undamaged reference specimens were than milled to an uniform geometry. Afterwards tensile tests were carried out using full-field three-dimensional strain measurement. This allowed conclusions to be drawn about the residual stress distribution and the effects of different infill strategies on the residual stress distribution. The stress values obtained were verified using Electronic Speckle Pattern Interferometry (ESPI). An exemplary ESPI measurement is

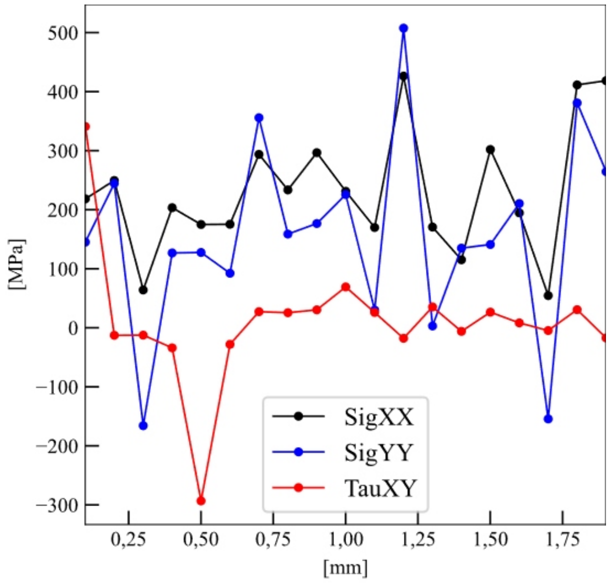

Figure 1. Exemplary ESPI residual stress measurement.

shown in figure 1. The presented measurement was taken next to the melt line on the base material site. It can be seen, that aside from two outliers the shear stress xy is close to zero over the entire depth. This specimen had a dimension of x = 250 mm, y = 50 mm and z = 10 mm, where it was milled on both sides to 8 mm after welding. The welding path used was alternating line welds along the x-axis of the specimen. The measurement shows tensile stress in both the x- and y-axis, with the stress the welding direction being slightly higher. The average tensile stress for both axes is close to the minimum yield strength of the base material.

In figure 2 an exemplary strain plot is shown. The DIC image shows a strain concentration along the centreline of the x-axis outside of the welding zone. The fracture observed resulted at a fusion defect. The weld metal, due to the higher tensile strength shows a reduced strain rate. Close to the weld metal, around the area where the ESPI-measurement was performed, the strain rate is lower than farther away form the weld. This may be due to the tensile stress measured with the ESPI-method, reducing the ductility and thus the strain reserves of this area.

The findings of these tests indicate a correlation between reduced strain rate seen during the tensile test and the residual stresses concluded by the ESPI-method.

1. AWS D1.1: Structural Welding Code-Steel, American Welding Society 2000.
2. DIN EN 1993-1-8, Eurocode 3: Bemessung und Konstruktion von Stahlbauten - Teil 1-8: Bemessung von Anschlüssen 2010.

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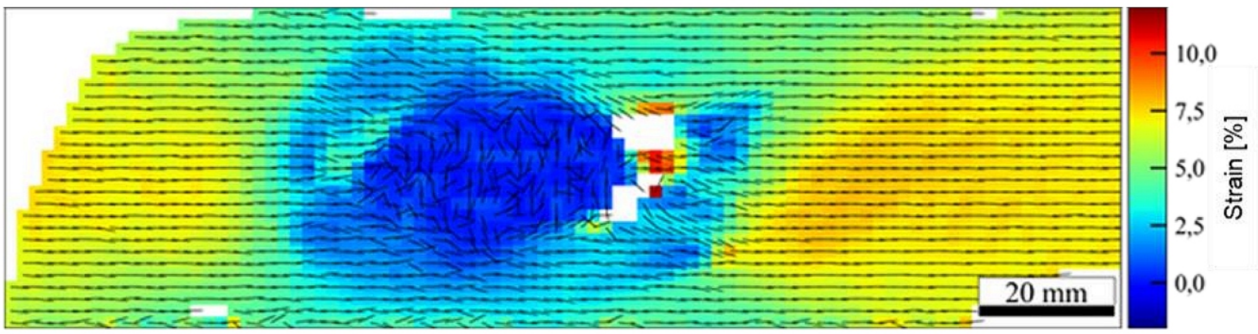

Figure 2: This is a figure caption.

S10 - 5

RESIDUAL STRESS EVALUATION IN LASER WELDED PLATES: WAVE-LIKE VS. LINEAR

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Welding of metallic material is a flexible method for achieving the joining of multiple parts. Although the structural performance of welded structures has significantly improved in the last decades, yet, there is still room for enhancement as testified by the gap in the performance when compared with the base material. The authors seek to reduce such a gap by introducing macroscopic unevenness to the weld profile, mimicking interlocking mechanisms often found in nature. To methodically explore this possibility, two sets of laser-welded plates were manufactured. The former is manufactured by using the traditional linear welding profile, while the latter by following a wave-like path.

Although several characteristics and properties can be studied concerning these welds, the focus of this specific study is the experimental evaluation of residual stress. Residual stress is one of the key causes of mechanical performance decay of weldments – especially when subjected to fatigue loadings – along with other microstructural heterogeneities found in welds, such as defects, texture, grain size and phase change. For this reason, a correct characterisation of residual stress in weldments is a mandatory prerequisite for understanding whether possible actions can be taken to minimise its impact and to accurately predict the mechanical performance of the part when in service.

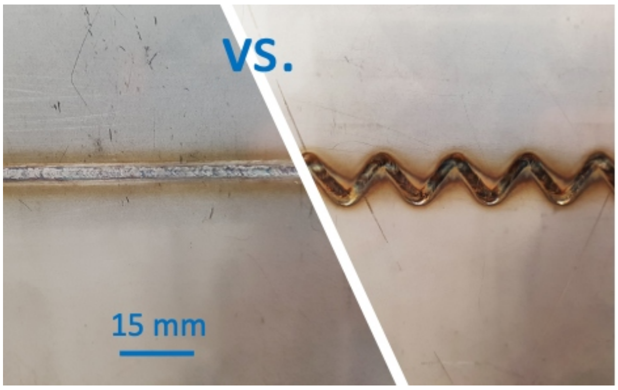

Figure 1. Overview of the analysed weldments.

The residual stress evaluation is carried out by exploiting the Neutron Diffraction technique, specifically concerning the longitudinal and transverse components of stress, with respect to the welding direction. This method is systematically employed for each studied weld, with additional measurements carried out at specific locations of the wave-like weld.

The exploitation of these results is widely discussed in the context of structural integrity and optimisation.

S10 - 6

EVALUABILITY OF X-RAY DIFFRACTION STRESS ANALYSES FOR HIGHLY DEFORMED HIGH MANGANESE STEELS

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Steel grades used for constructing car bodies have seen significant advancements in recent years, driven by the need for economic and environmental efficiency in modern individual transportation. These improvements have become possible due to the creation of steels, which combine excellent formability with high strengths, enhancing both passenger safety and vehicle performance and efficiency. Steels with increased manganese content are particularly important in the category of automotive body sheet materials. These materials achieve their final mechanical properties through deformation-induced twinning (TWIP effect) and the deformation-induced martensite formation in individual residual austenite grains (TRIP effect). TWIP and TRIP steels are characterized by their high tensile strengths after forming and their very high deformation reserves [1]. In the context of automotive construction, structural steel components are to this day typically joined using welding methods. The specifics of welding TWIP and TRIP steels are covered elsewhere [2-3]. The interrelation between weld seams and structural steel is complex. Firstly, the microstructure of the steel is locally melted during welding. Upon solidification, the microstructure within the weld seams changes significantly from its original state, displaying either a coarse dendritic or a fine martensitic structure, depending on the alloying elements. Secondly, the constraint during cooling between the weld seam and the adjacent base material results in high residual stresses along the length of the weld seams and in case of multi-phase microstructures it can be necessary to determine the phase-specific residual stresses using X-ray diffraction methods. The intricate microstructures found in both the weld seams and the typically pre-deformed base material make the assessment of stresses as basis of X-ray diffraction stress analysis more challenging. As the weld seam of these structural steels is a safety-critical element in vehicle construction and the interaction has not been conclusively clarified, high safety factors must be used in the design planning processes.

Within the present study, X40MnCrVAl 19-2 (mat.-no. 1.7401), HCT690T (mat.-no.1.0947) and S355MC (mat.-no.1.0976) metal sheets with a thickness of approx. 2 mm were examined. While the common ferritic welding-steel S355MC merely serves as a reference material, the study's scientific focus is on the TWIP steel X40MnCrVAl 19-2 as well as the TRIP steel HCT690T. The TWIP steel is purely austenitic and shows no martensite formation during deformation. The mainly ferritic TRIP steel exhibits a microstructure with approx. 15 vol% austenite in its initial state.

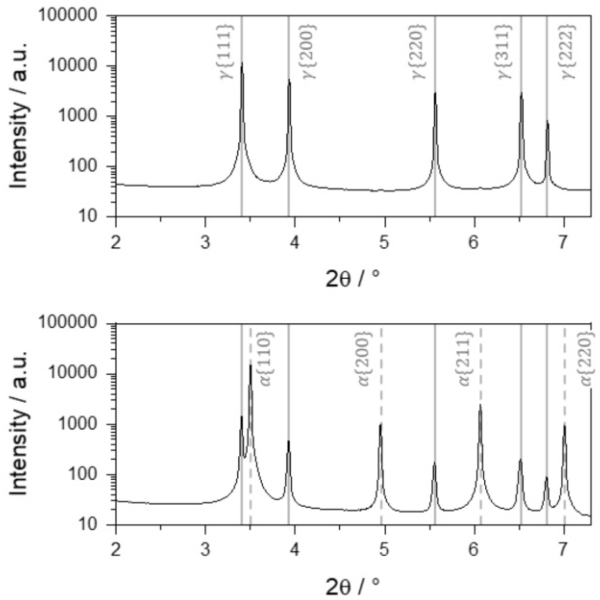

Figure 1. Measurable interference lines, exemplarily listed for the austenitic TWIP steel (top) and the TRIP steel (bottom).

To take the multi-phase structure of the local material states into account, in situ X-ray diffraction methods were used to investigate how plastic deformation and the welding of joints influence the development of residual stresses on both macroscopic and microscopic scales, as well as the hardening conditions. Starting with analyzing the initial states of the two materials, specimens that had undergone pre-deformation were also investigated. This analysis revealed that as deformation increases, the material's anisotropy becomes increasingly pronounced, posing challenges to standard evaluation methods such as the sin^2 method. Synchrotron experiments are particularly suitable for investigating deformation behavior, as they allow real-time insights into the development of material anisotropy. In particular, synchrotron experiments enable the analysis of several interference lines as shown in figure 1 and the associated investigation of the transformation kinetics resulting from the TWIP and TRIP effect, respectively. To gain a substantial comprehension on the effect of large plastic deformations on the evaluability of X-ray diffraction stress analysis for the materials of interest, continuous and discontinuous tensile loading experiments were performed at the DESY beamline P21.2@PETRA-III. For this purpose, specimen cut in rolling direction from the metal sheets were deformed to a total strain of up to 40 % using the custom-built miniature tensile testing rig shown in figure 2, which was specifically designed for these in situ investigations.

Understanding the changes in crystallographic texture and intergranular strains aids in analyzing load and residual stresses and in case of multi-phase material also the load partitioning behavior on the phases involved by means of X-ray diffraction, even when the material is in an anisotropic state. The information gathered from these studies will be valuable for improving evaluation of X-ray stress analysis in samples affected by anisotropy effects and by this means also enhancing the accuracy of future welding simulations, since finally more meaningful experimental data can be provided for validation of simulation results.

1. W. Bleck, *Mat.-wiss. u. Werkstofftechn*., **31**/2, (2000), 162-168.

2. S. Russo, M. de Maddis, G. D'Antionio, F. Lombardi, *Metals*, **6**/11, (2016), 270.

3. A. Buchwalder, K. Rüthrich, R. Zenker, H. Biermann, *Adv. Eng. Mater*., **15**/7, (2013), 566-570.

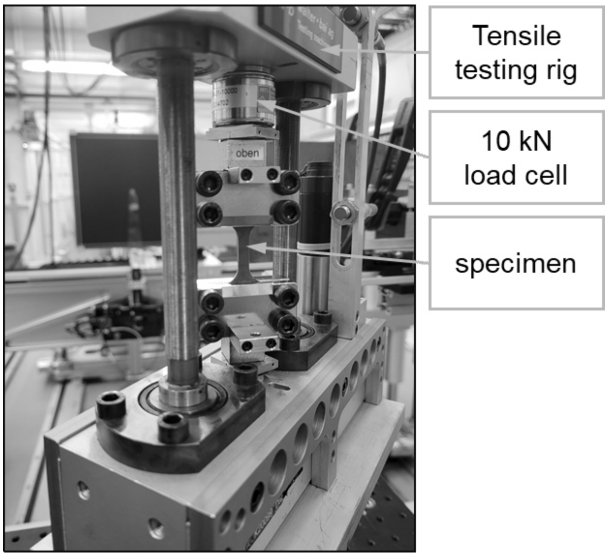

Figure 2. Experimental setup at the DESY beamline P21.2@PETRA-III.

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Session XI - Microelectronics, Thin Films and Coatings

S11 - 1

NANOSCALE GRADIENTS OF RESIDUAL STRESSES AND MICROSTRUCTURE IN PERFORMANCE-CRITICAL REGIONS OF HARD CERAMIC THIN FILMS

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The functional properties of various high-demanding applications in microelectronics, aviation, automotive, as well as in the metalworking industry are based on the application of thin films. Despite tremendous effort over the last decades to unveil the complex relationship between the film's microstructure, stress gradients, process parameters and material selection, little is known about the properties of thin films at performance-critical regions such as curved surfaces, edges and pits.

In this submission, we clarify the complex relationship between deposition conditions and both lateral and depth gradients of residual stresses and microstructure in nanocrystalline protective thin films. Our primary focus is on *the edge region of cutting tools*. The results document that the residual stress state, microstructure, and mechanical properties are significantly influenced by the non-planar substrate shape. These factors are intricately interconnected with the functional properties of the cutting inserts.

Microstructural properties and residual stress gradients across the cutting edge area of an AlTiN thin film, applied on a cemented WC-Co insert, were characterized by cross-sectional synchrotron X-ray nanodiffraction (CSnanoXRD) at the ID13 beamline of the ESRF in Grenoble. The interface between the thin film and substrate was precisely aligned parallel to an X-ray beam, which was collimated to a cross-section of 75×75 nm^2 using multilayer Laue lenses (MLL). Two-dimensional diffractograms were collected from approximately 40 µm thick cross-sectional lamellae using an Eiger 4M detector (Fig. 1). The data acquisition involved a field-of-view of 20×20 µm^2, scanned with a step of 50 nm. The collected Debye-Scherrer rings were analysed using the pyFAI software package and evaluated according to the methodology outlined in [1].

The results from the synchrotron experiments reveal that the cross-sectional residual stress gradients within the thin film are more pronounced at the cutting edge with a curved substrate interface than at the planar substrate-thin film regions located adjacent to the cutting edge area. Compressive residual stresses up to -5 GPa were measured at the cutting edge, implying that the residual stresses are increased up to 100% in comparison to the adjacent regions. Additionally, the results revealed a presence of strong lateral gradients of microstructure and stress, which indicate different thin film growth conditions at the cutting edge. In addition, the collected synchrotron results were correlated with the data from complementary scanning and transmission electron microscopy, *cf.* Fig. 2. Finally, the mechanical properties of the thin film were assessed at the cutting edge using nanoindentation.

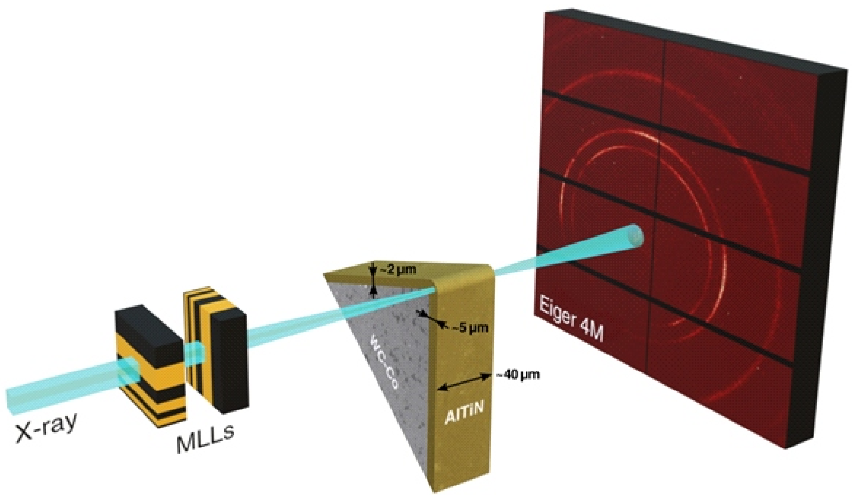

Figure 1. The CSnanoXRD setup in shows the cross-sectional lamellae prepared from a cutting insert, the sample is scanned in transmissional geometry. Multilayer Laue lenses enabled a beam size of 75×75 nm^2 on the sample, a step of 50 nm was used to map the thin film microstructure and stress at the cutting edge.

The integrated approach of synchrotron X-ray nanodiffraction, electron microscopy analysis and mapping of mechanical properties with nanoindentation enabled a successful correlation of the structure-function relationship directly at the cutting edges of nanoceramic inserts.

Illustrated through the nanoscale characterization of the cutting edge of an AlTiN thin film, we have demonstrated that the CSnanoXRD method serves as a unique tool for resolving gradients in microstructure and residual stresses within performance-critical regions of thin films. We contend that this approach is pivotal for elucidating the mechanical and functional properties at critical points in thin films, not only within cutting tools but also in various microelectronic applications.

1. M. Meindlhumer, N. Jäger, S. Spor, M. Rosenthal, J.F. Keckes, H. Hruby, C. Mitterer, R. Daniel, J. Keckes, J. Todt, Scripta Materialia, **182**, (2020), pp. 11-15.

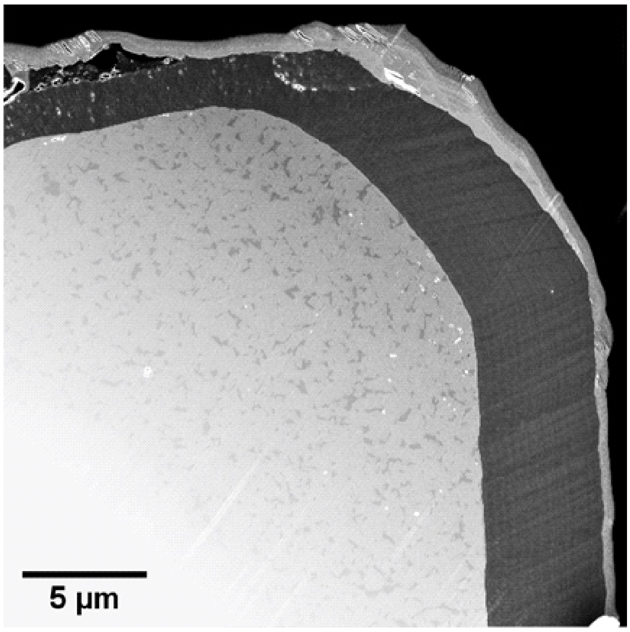

Figure 2. SEM micrograph of a FIB-prepared cross-section of the cutting edge reveals substantial differences in the thin film microstructure at the curved and planar substrate regions, respectively

S11 - 2

RESIDUAL STRESS IN COLD GAS SPRAYED TITANIUM COATINGS – ROLE OF SUBSTRATE MATERIAL AND PROCESS PARAMETERS

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Cold gas spraying (CGS), is a material deposition process executed by spraying the feedstock material particles on the substrate at supersonic velocities. In CGS, the material particles are not melted and the deposition occurs in a solid-state. Therefore, it is highly suitable for temperature and oxidation sensitive materials. The bonding of particles however can be attributed to adiabatic shear instabilities at the interfaces with the substrate or already deposited material caused by the high-strain-rate plastic deformations of high kinetic energy particles upon impact [1,2]. The current study is a part of the collaborative project CORE (Computerized Refurbishment) funded by dtec.bw aiming to utilize CGS as an advanced repair technique for aerospace applications [3]. The residual stresses developed during the CGS process play an important role in the performance and mechanical integrity of CGS-repaired parts. In this early stage of the project, the main focus is paid to adjust the CGS process parameters and possible boundary conditions contributing to a residual stress build-up and as a consequence to obtain a knowledge-based assessment option to specifically influence residual stresses induced through processing.

In the current study, the influence of substrate material and substrate thickness on the development of residual stresses in Titanium coatings deposited by means of CGS was of specific interest. Furthermore, the variation of relevant process parameters was in the focus of our investigations. For this means, grade 1 Titanium coatings were deposited by CGS on various substrate materials, i.e. on grade 2 Titanium, on Steel AISI304, on commercially pure Copper and on AlMg3 substrates. The further investigation for studying the influence of process parameters such as process gas temperature, nozzle traverse speed, etc. and of geometric boundary conditions (i.e. the global stiffness of the substrate) was undertaken by spraying grade 1 Titanium powder on AlMg3 substrate (see Figure 1) of different thicknesses under varying gas temperatures and nozzle traverse speeds. For analysing residual stress depth distributions in above-mentioned CGS deposited specimens, mainly the incremental hole-drilling method was employed and the results were partially complemented by X-ray diffraction according to the sin^2 -method in combination with a stepwise layer-removals.

In addition, as a preview for future activities, deep-rolling was performed as a post-treatment on specific specimens. The aim was to achieve an effective reduction on surface roughness after CGS and a densification of the coatings. As a side effect it is expected that through the choice of appropriate deep-rolling parameters, manageable

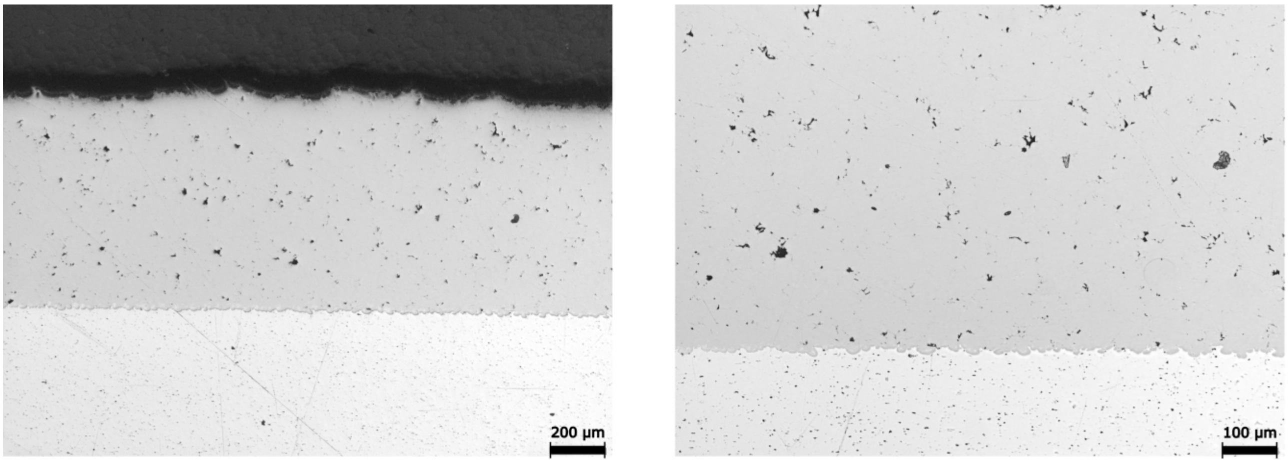

Figure 1. Typical microstructure of grade 1 Titanium coatings deposited on AlMg3 substrates.

compressive residual stress distributions can be induced in the CGS deposits.

In general, the results indicate that the residual stresses through the deposited layer thickness are mostly compressive in nature. Moreover, an influence of the CGS process parameters on the residual stresses could be distinguished for all coating systems mentioned above. As an example, it has been found that high velocity impacts of solid particles cause compressive peening stresses. On the other hand, these peening stresses are superimposed by tensile contributions caused by quenching of impacted particles, which are at relatively higher temperature than the substrate material. Furthermore, thermal stresses developed due to different CTEs (Coefficients of Thermal Expansion) of coating and substrate materials also contributes to the final residual stress state [4, 5]. The geometrical boundary conditions such as substrate thickness were observed to have a notable influence on the residual stress states. In particular, it was found that a lower dimensional stability of the specimens, provided by thinner substrates, support that the substrates tend to bend significantly. This in turn results in a noticeable influence on the residual stress distributions that develop. The respective contributions to the finally resulting internal stress distributions are discussed with regard to or in connection with local temperature distributions, the thermal expansion of the spray material and possible stress relaxations, when the effective specimen temperatures are reached, and components geometric stiffness defined by the substrate thickness.

1. H. Assadi, F. Gärtner, T. Stoltenhoff, H. Kreye, *Acta Materialia*, **51**, (2003), pp. 4379-4394.
2. H. Assadi, H. Kreye, F. Gärtner, T. Klassen, *Acta Materialia*, **116**, (2016), pp. 382-407.
3. F. Gärtner et al., *dtec.bw-Beiträge der Helmut-Schmidt-Universität / Universität der Bundeswehr Hamburg: Forschungsaktivitäten im Zentrum für Digitalisierungs- und Technologieforschung der Bundeswehr*, **1**, (2022), pp. 9-19. DOI: 10.24405/14524.
4. T. Suhonen, T. Varis, S. Dosta, M. Torrell, and J. M. Guilemany, *Acta Materialia*, **61**, (2013), pp. 6329–6337.
5. S. Kuroda, T. Dendo, S. Kitahara, *Journal of Thermal Spray Technology*, **4**, (1995), pp. 75-84.

S11 - 3

MACROSCOPIC AND MICROSCOPIC RESIDUAL STRESSES IN NICKEL-ALUMINUM BRONZE MATRIX COMPOSITE SURFACE DEPOSITS MANUFACTURED VIA LASER MELT INJECTION

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Nickel-aluminum bronze (CuBz) alloys have found widespread applications across various industries due to their excellent combination of mechanical properties and corrosion resistance. To facilitate the utilization of nickel-aluminum bronze alloys in high-value applications and reduce the need for part replacements and overall costs, one key area of interest is enhancing their surface wear resistance. For this purpose, metal matrix composite (MMC) coatings have proven highly valuable [1].

A notable example is the deposition of spherical fused tungsten carbide comprising WC and W_2C (sFTC) via laser melting injection (LMI for surface reinforcement of CuBz substrates. This process has demonstrated the potential to reduce wear by approximately 80 % in CuBz samples [2, 3]. However, macro and micro residual stresses develop in the MMC coatings, leading to geometric distortion and reducing fatigue strength [4, 5] as well as service life [6]. Therefore, it is crucial to investigate and understand the development of residual stresses in these MMC coatings.

In this contribution, we employed neutron diffraction to determine the residual stress profiles in sFTC/CuAl10Ni5Fe4 surface deposits manufactured through LMI. A thermo-mechanical finite element model was also developed to predict the temperature and residual stresses in the re-melted CuAl10Ni5Fe4 bronze. The effects of single/multiple laser tracks and pre-heating temperature on the residual stress state were assessed. In addition, the microstructure was characterized in detail using various microscopic methods.

This investigation provides a comprehensive understanding of the residual stress state in the MMC coatings. The findings from this study have significant implications for optimizing the manufacturing process, reducing residual stresses, and ultimately enhancing the performance and extending the service life of the MMC coatings.

1. Y. Feng, X. Pang, K. Feng, Y. Feng, Z. Li, Journal of Materials Research and Technology 15 (2021) 5597-5607.
2. H. Freiße, A. Langebeck, H. Köhler, T. Seefeld, Dry Metal Forming Open Access Journal 2 (2016) 001-006.
3. X.X. Zhang, J.R. Kornmeier, M. Hofmann, A. Langebeck, S. Alameddin, R.P. Alessio, F. Fritzen, J.R. Bunn, S. Cabeza, Strain (2023) e12457.
4. H. Köhler, K. Partes, J.R. Kornmeier, F. Vollertsen, Physics Procedia 39 (2012) 354-361.
5. H. Köhler, V. Jayaraman, D. Brosch, F.X. Hutter, T. Seefeld, Physics Procedia 41 (2013) 502-508.
6. G.A. Webster, A.N. Ezeilo, International Journal of Fatigue 23 (2001) S375-S383.

S11 - 4

RESIDUAL STRESS FIELD IN CIGS PHOTOVOLTAIC SOLAR CELL

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To accelerate the energy transition, the development of photovoltaic (PV) solar energy is an interesting solution. PV solar panels are currently made with single-junction crystalline silicon cells whose performance is close to the theoretical limit of around 30%. Promising new emerging technologies are based on thin films (solar absorbers) that can be applied to different substrates. They allow low-cost massive production but have much more complex microstructures than silicon. One of the existing possibilities is the use of copper, indium, gallium, selenium alloys (CIGS) thin films to produce semi-transparent photovoltaic cells on flexible substrates. Commercial solutions are being developed and this type of solution is increasingly being used in agriculture, transport, marine and space applications. The success of their use depends on their reliability and resistance to atmospheric conditions and mechanical stress.

The residual mechanical states generated during the fabrication of these multilayer coatings will have an influence not only on their mechanical behavior but also on their possible coupling with chemical effects, two phenomena that play a role in cell life. This work is dedicated to the determination of the initial stress state field of this multilayer coating.

The CIGS PV cells used in this study was made up of multiple thin layers using physical vapor deposition (PVD). A soda lime glass was used as a substrate on which a first back contact Mo layer with a thickness of 0.6 µm was deposited by magnetron sputtering. A 1.8 µm thick CIGS ($CuGa_xIn_{1-x}Se_2$) absorber layer was then deposited on top of the Mo layer by co-evaporation at a temperature of 550 °C. An image of the cross section of the multilayer coating acquired on backscattering electron mode (BSE) is shown in Figure 1.The contrast on the BSE image shows both polycrystalline layers deposited on an amorphous glass substrate. A complete CIGS PV cell is completed by several other very thin layers (about 20-300 nm) deposited on top of the CIGS to have a functional cell (anode, drain grid, etc...).

X-ray diffraction was used to characterize the two main layers. An X-ray beam (1 mm diameter), using a four-circle diffractometer (Seifert MZ VI) in the laboratory, irradiates the cell surface from above. The deep penetration of X-rays

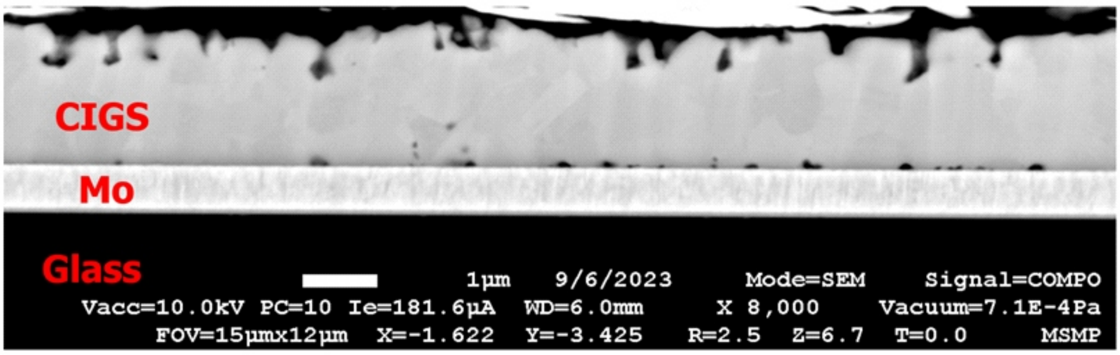

Figure 1. CIGS PV cross section, BSE SEM image.

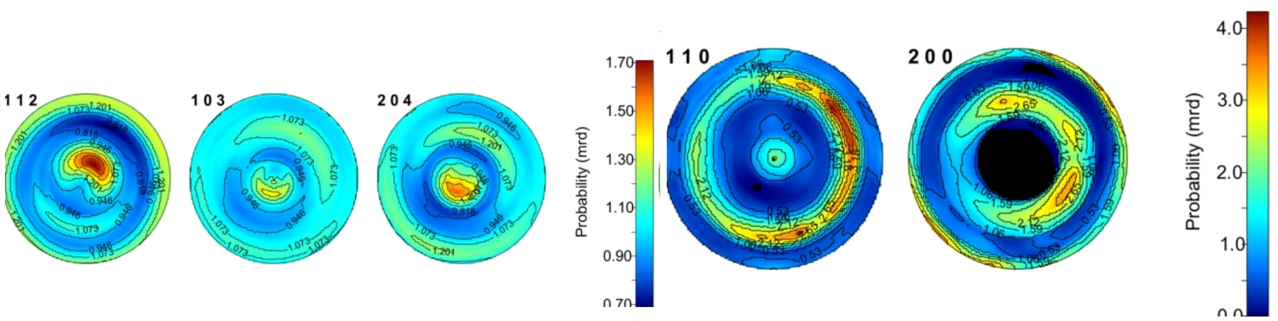

Figure 2. Pole figures: a) CIGS, b) Mo

(Cr-K radiation) allows both CIGS and Mo layers to be detected for texture and residual stress analysis. Figure 2 shows a very low fiber texture in the CIGS layer and a more pronounced texture in the Mo layers. The residual stresses were calculated using the method, considering the anisotropy of the material on the X-ray elastic constant using the Kröner-Eshelby approach [1].

To complete these measurements, a very simple model of stress generation was carried out using an elastic analytical method, considering only the thermal incompatibility strain related to the difference in expansion coefficients between the layers and the substrate. An agreement was found between the modelling and the experimental results.

1. H. Behnken, V. Hauk, (1986), *Z. Metallkde*, **77**, (1986), pp. 620-626.

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S11 - 5

SUBSURFACE CHARACTERIZATION OF FEMTOSECOND-LASER PEENED ALUMINIUM

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Laser shock peening (LSP) is used in the industry to improve the mechanical properties of metals. Due to the inhomogeneous deformation resulting from a dislocation density gradient below the surface compressive residual stresses are created, which improve the fatigue and corrosion resistance of the peened parts. Femtosecond laser shock peening (fs-LSP) is a novel method that also induces compressive residual stresses below the surface, but it due to the smaller amount of absorbed energy (compared to nano-LSP) does not necessitate a protective layer. The treated surface also has a lower roughness [1].

In this study, aluminium samples were peened with fs-LSP as shown in Fig.1a, using different fluences from 2 to 30 $J.cm^{-2}$ and pulse durations from 160 fs to 3 ps.

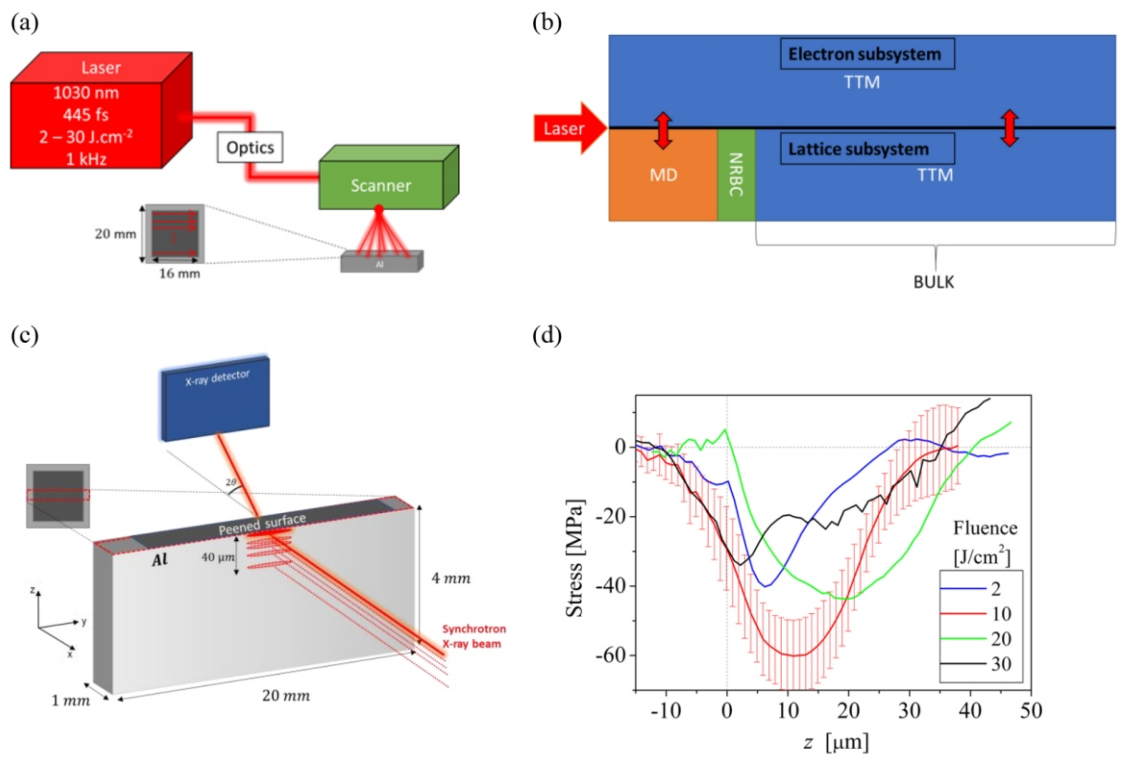

Figure 1. Schematic drawing of (a) the experimental setup of laser peening, (b) the geometry of numerical simulations, (c) and the synchrotron X-ray measurements. (d) Evolution of the measured residual stresses below the irradiated surface for various incident fluences.

Sub-surface modifications were characterized at synchrotron source (Fig.1b) using micro-beams and by analysing the X-ray diffraction peak profiles. The latter allowed not only determining the dislocation density and the associated strain energy, but also the residual stresses (Fig.1d). In order to optimize the understanding of this process, a numerical approach was developed, based on a coupled two-temperature model for electrons and ions within the frame of molecular dynamics (TTM-MD, following eqs. (1) and eq. (2) [2, 3]. The computations give access to all thermodynamic variables, atomic positions and their evolution during peening. The experimental dislocation density values were compared to the prediction of the Meyers' model applied to simulations results [4].

$$C_e(T_e)\frac{T_e}{t} \quad [K_e(T_e,T_l) \; T_e] \quad G(T_e)(T_e \quad T_l) \quad S(\vec{r},t) \tag{1}$$

$$m_j \frac{v_j}{t} \quad {}_jU(r_1,\ldots,r_n) \quad F_j^{lang}(T_e \quad T_l) \quad \frac{P_e}{n_i} \tag{2}$$

1. A. Nakhoul *et al.*, "Energy feedthrough and microstructure evolution during direct laser peening of aluminum in femtosecond and picosecond regimes," *J Appl Phys*, vol. 130, no. 1, Jul. 2021, doi: 10.1063/5.0052510.

2. A. Borbély, A. Aoufi, D. Becht, and J. Keckes, "X-ray methods for strain energy evaluation of dislocated crystals," J Appl Crystallogr, vol. 56, pp. 254–262, Feb. 2023, doi: 10.1107/S1600576722012262.

3. D. Ivanov and L. Zhigilei, "Combined atomistic-continuum modeling of short-pulse laser melting and disintegration of metal films," Phys Rev B Condens Matter Mater Phys, vol. 68, no. 6, Aug. 2003, doi: 10.1103/PhysRevB.68.064114.

4. L. V Zhigilei, D. S. Ivanov, E. Leveugle, B. Sadigh, and E. M. Bringa, "Computer modeling of laser melting and spallation of metal targets," 2004. [Online]. Available: http://www.faculty.virginia.edu/CompMat/.

5. Meyers, M.A., Gregori, F., Kad, B.K., Schneider, M.S., Kalantar, D.H., Remington, B.A., Ravichandran, G., Boehly, T., Wark, J.S., 2003. "Laser-induced shock compression of monocrystalline copper: Characterization and analysis". Acta Materialia 51, 1211–1228.

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Session XII - Neutrons

S12 - 1

INTRODUCTION TO THE ENGINEERING AND SCIENTIFIC STRESS DIFFRACTOMETER AT CHINA ADVANCED RESEARCH REACTOR AND ITS APPLICATIONS

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A new neutron residual stress instrument-Engineering and Scientific Stress Diffractometer (ESSD) at China Advanced Research Reactor was built and put into service. Here the neutron optic and the main components of ESSD will be introduced including neutron guide, doubly focusing Si (400) monochromator, monochromator shielding, sample stage, first slit/radial collimator , area detector and sample environment. The flux at the sample position was accurately measured using neutron Au activation method. The neutron flux at the sample position is 3.0×10^7 n cm^{-2} s^{-1} at wavelength 1.64 Å with full reactor power, which indicates that ESSD is a world-class instrument.

ESSD has been used to measure the 3D residual stresses in typical engineering components. The day-one experiment was measuring the inner residual stresses of the full-size high-speed train wheels. The largest penetrating thickness was 41 mm with gauge volume 3 mm × 3 mm × 3 mm. 3D residual stress mapping were obtained, which was used to assess the structural integrity. Another typical experiment is SiCp/Al matrix composites. The macro stress measured by user showed to be about zero. Neutron diffraction illustrated the micro stress of aluminum and SiC, especially the total macro stress of aluminium phase and SiC phase was near to be zero, which proved the advantages of thermal neutrons. Also large aluminium alloy forging pipe, superalloy disks, superalloy ring pieces and steel welding will be introduced.

S12 - 2

HIGHLIGHTING THE CAPABILITIES OF NEUTRON DIFFRACTION FOR RESIDUAL STRESS DETERMINATION IN INDUSTRIAL RELEVANT ALUMINIUM CAST COMPONENTS

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The manufacturing stages of a cylinder head in a motor block comprise casting and solution heat treatment followed by a quenching step and finally artificial ageing. This can result in tensile residual stress (RS) in the component which overlay with thermo-mechanical loads during operation and may reduce the lifetime of the component. It is thus necessary to optimize the production stages for residual stresses, especially the subsequent heat treatment, by adjusting related process parameters. Simulations have been the main tool for the optimization of process parameters. Simulated residual stresses during heat treatment frequently deviate from measured values. This discrepancy results from insufficient material modelling, since phenomenological material definitions inadequately describe plastic flow and creep behavior during heat treatment. Therefore, a new physical material model for Al cast alloys in the form of a user subroutine has been introduced at Nemak [1]. It includes state variables like dislocation density or precipitation fractions, which form the basis for modelling strengthening mechanisms such as dislocation or precipitation hardening. This enables the flow stress for each time step to be calculated and returned to the solver for residual stress calculation. However, to confirm the results from the simulations, the residual stresses need to be determined and confirmed through experiments. In the past, destructive residual stress measurements by sectioning method were used for the cylinder head. However, as this method is only applicable for easily accessible areas on the surface, the predictive capabilities of the physical material model for failure-critical areas within the bulk could not be validated. It is therefore necessary to resort to a non-destructive measurement technique to identify potential weaknesses of the physical material model approach with regard to the simulation of residual stresses. Due to the

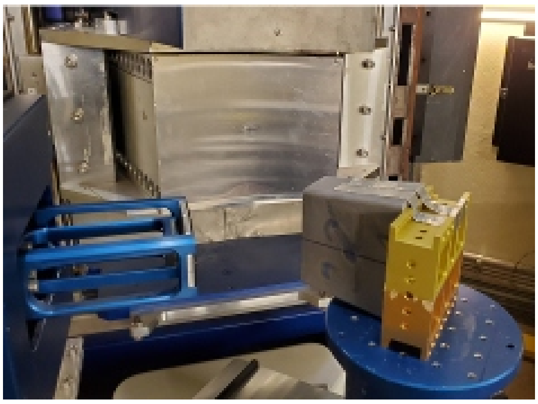

Figure 1. Neutron measurement of the wedge-shaped sample with POLDI at PSI.

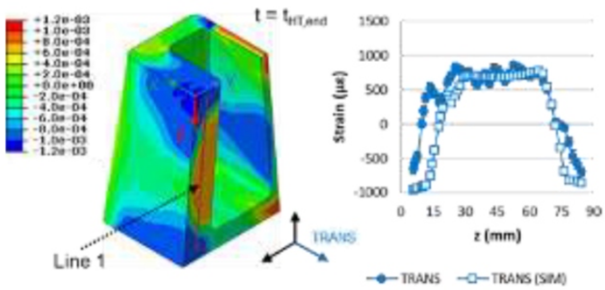

Figure 2. Validation of the elastic strain model in transverse direction for the wedge-shaped sample (T6).

Figure 3. Neutron measurement of the cylinder head sample with SALSA at ILL.

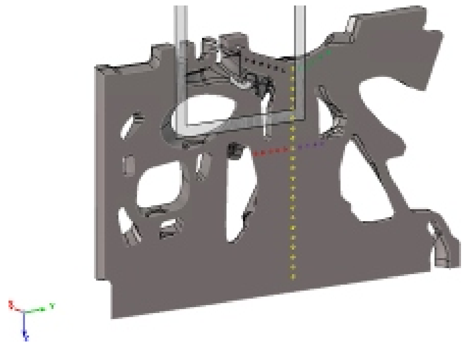

Figure 4. Measurement points highlighted in a section within the cylinder head samples.

microstructural inhomogeneity and dendritic microstructure, X-ray diffraction techniques (SXRD, LXRD) were deemed to not be suitable. Therefore, the method of choice here is neutron diffraction (ND) since it allows to probe the residual stresses even in the bulk of the sample without altering its shape or dimension, hence, without potentially influencing its RS distribution due to cutting.

As a first stage, small Al cast (AlSi7Cu0.5Mg) wedges were produced as test cases with a similar microstructure and measured (Fig. 1 and 2) to ensure the applicability of ND for the cylinder head. The results were used to develop an optimal scan strategy for measuring the real size Al cast cylinder heads (Fig. 3 and 4). The production process of the cylinder heads studied with this project consists of three parts. First, casting of the material and then two different heat treatment stages, one followed directly by the other. This way, three different samples were produced each representing a step in the production process (further denoted AC, T4, T6). The wedges were measured at three different instruments (neutron sources): POLDI [2] (Paul Scherrer Institut), SALSA [3] (Institut Laue-Langevin), and Engin-X [4] (ISIS). The latter two instruments were also used to measure the cylinder heads.

While each source has its advantages, the Time-of-Flight (TOF) method allows the stress determination through full pattern analysis to obtain the lattice parameters. Considering the cross-sectional dendrite diameters sizes in the Al cast samples, determining the lattice parameter can provide a more accurate representation of the bulk behavior of the material in this study. We will further highlight the differences in the reference measurement approaches used (Al powder vs comb samples) and how they influence the RS results.

With the results from the neutron measurements of the wedges, a preliminary validation of the new modelling approach at NEMAK could be achieved. Additional destructive RS measurements (e.g. contour method) were done on the wedges to evaluate the results from the neutron measurements. The cross-correlation of RS characterization methods with the simulation is still ongoing.

This study proves the direct industrial impact of RS characterization methods with neutrons and their complementarity. The successful validation of the model will open the possibility to virtually layout cast Al components including e-housings or battery trays in terms of heat treatment induced stresses. The results also serve as input to the EASI-STRESS EU project [5], which aims to standardize the workflows and output of non-destructive residual stress methods at large-scale research facilities (LRIs).

1. https://www.nemak.com/

2. Stuhr, U.; Spitzer, H.; Egger, J.; Hofer, A.; Rasmussen, P.; Graf, D.; Bollhalder, A.; Schild, M.; Bauer, G.; Wagner, W. Time-of-flight diffraction with multiple frame overlap Part II: The strain scanner POLDI at PSI. *Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment* **2005**, *545* (1-2), 330-338.

3. Pirling, T., Bruno, G., and Withers, P. J., *Materials Science and Engineering: A*, **437**, (2006), 139-144.

4. Santisteban, J.; Daymond, M.; James, J.; Edwards, L. ENGIN-X: a third-generation neutron strain scanner. *J Appl Crystallogr* **2006**, *39* (6), 812-825.

5. https://cordis.europa.eu/project/id/953219

S12 - 3

STUDY OF RESIDUAL STRESSES ON THE HK4-STRAIN SCANNER INSTRUMENT

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Diffraction based methods have proven to be effective techniques for studying residual stresses in materials. However, different diffraction techniques can provide results from different depths of the sample under investigation. Due to the large penetration lengths of neutrons in most engineering materials, neutron diffraction is an extremely useful method for non-destructively probing materials deep below their surface. However, unlike laboratory scale XRD, the source of neutron radiation for materials research is only available in large facilities such as research reactors or spallation source facilities with dedicated instruments on neutron beamlines. The aim of this contribution is to introduce the neutron strain scanner instrument at the horizontal channel #4 of the research reactor LVR-15 in Řež, Czech Republic.

The HK4 strain scanner is a two-axis neutron diffractometer employing an elastically bent perfect Si crystal as a focusing monochromator and a 6-axis robotic arm for accurate sample positioning. The instrument is also equipped with a two-dimensional position sensitive neutron detector, a cadmium slit system and a radial collimator to shape the incident and diffracted beam (Fig. 1).

The interpretation of the measured strain distribution is not straightforward due to the presence of pseudo-strains (PS) (Fig. 2). They are most pronounced in near-surface measurements and in materials with large neutron attenuation coefficients. Therefore, in the first part of the presentation, the simulation [2] and treatment of PS during data analysis will be briefly discussed

In the second part, the capabilities of the instrument are demonstrated by the results [3,4] of measurements on samples produced by additive manufacturing and on complex shaped engineering parts.

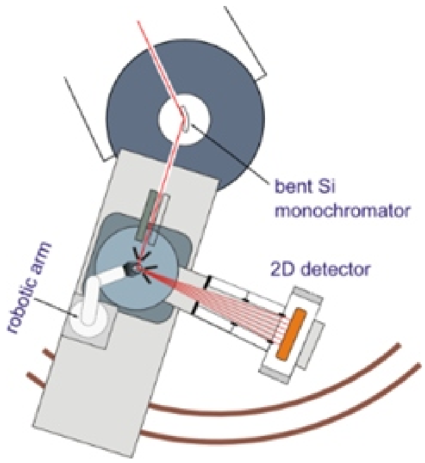

Figure 1. The layout of the HK4 strain scanner [1]

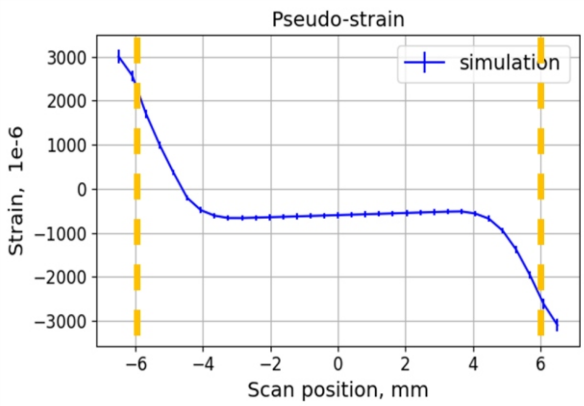

Figure 2. A simulated pseudo-strain distribution (sample surface is marked with yellow dashed line). The PS magnitude and distribution depends on the actual sample geometry, material and experiment geometry.

1. https://www.ujf.cas.cz/en/departments/department-of-neutron-physics/instruments/lvr15/HK4.html
2. https://github.com/NPLtools/stressfit
3. L. Kunčická, R. Kocich, G. Németh, K. Dvořák, M. Pagáč, Additive Manufacturing, Volume 59, Part A, (2022), 103128.
4. R. Kocich, L. Kunčická, M. Benč, A. Weiser, G. Németh, International Journal of Bioprinting, 10(1), (2024), 1416.

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S12 - 4

THE ACCURACY OF NEUTRON DIFFRACTION STRESS DETERMINATION

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Neutron diffraction methods for the determination of stress fields within a materials sample or an engineered component is today a well-established and mature technique. International collaborations between neutron sources and industry within VAMAS and RESTAND projects [1] during more than two decades have led to this achievement. This work culminates in an international standard first established in 2005 and a revised version in 2019 [2]. It describes ways of instrument and sample alignment and gives instructions for measuring, reporting and stress calculation.

However, benchmarking exercises between stress-diffractometers [3] have shown that reported measuring uncertainties are underestimating the real scatter in results. This happens when only uncertainties from the peak fit are taken into account for error calculation. This is often done for the simple practical reason that it is not obvious of how to estimate the magnitude of other error sources such as alignment inaccuracies and systematic errors.

Experimental factors that influence the accuracy of stress determination are precision of sample manipulation and reproducibility of sample alignment, the position of the gauge volume with respect to instrument axes and stress gradients.

The positioning accuracy of sample stages is normally well determined by the manufacturer and therefore known. It is often negligible. Sample alignment depends on the tools used for the setup (cameras, theodolites…). 3D-coordinate measuring tools can provide < 5 micrometres precision and are very helpful when it comes to the alignment of complex components. There exist many precision tools today to help alignment. But the best proof of the *real* position of the gauge volume inside the sample is an entry scan, where the sample surface is entered in small steps into the gauge volume. The resulting intensity profile provides a measure of the relative position between gauge volume and sample surface. Its analysis requires a mathematical model that takes into account the shape of the surface and requires precise parameters of the gauge volume.

For the instrument SALSA at the Institut Laue Langevin, we have developed a procedure which provides all that. It is used at the same time for instrument alignment, determination of gauge volume parameters and determination of systematic errors. A thin foil of polycrystalline material (i.e. 0.3 mm thick steel-foil) is scanned in different orientations across the gauge volume and the diffraction pattern is analysed. These scans provide the dimension of the gauge volume, intensity distribution and its absolute position with respect to the instrument centre and the zero position of the alignment system. These parameters can be treated as systematic errors and used for the correction of sample coordinates especially when the sample is rotated by for instance the for strain scanning typical 90°.

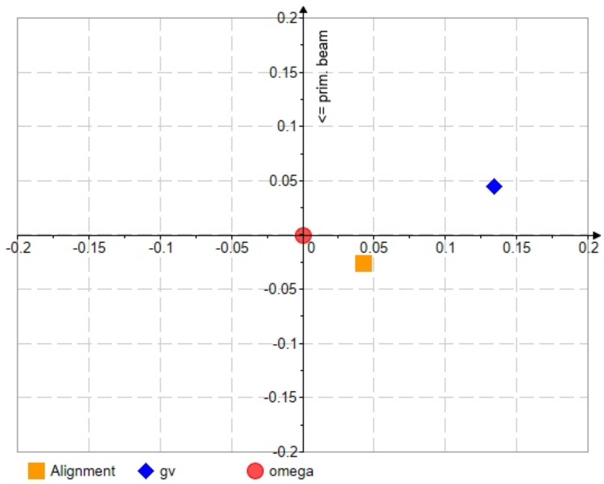

Figure 1. Positions of sample stage rotation centre (red), zero position of the alignment system (orange) and gauge volume position (blue) as determined by our alignment procedure. The scale is in mm.

Parts of this method are already embedded in the Neutron Quality Label (NQL), supported the BrightneSS-project [4].

The steeper stress gradients are in a sample the higher are the requirements on positioning accuracy. The other way round, positioning errors lead to larger uncertainties of strain and stress values in the vicinity of stress gradients. This should be taken into account in the calculation of error propagation.

In this paper we describe the above mentioned alignment procedures, ways to reduce positioning errors and a more complete error propagation calculation that takes into account all the above described uncertainties.

1. Youtsos, A., Ohms, C. European standardization activities on residual stress analysis by neutron diffraction . Appl Phys A 74 (Suppl 1), s1716–s1718 (2002).

2. ISO 21432:2019 - Non-destructive testing; Standard test method for determining residual stresses by neutron diffraction.

3. R.C. Wimpory, C. Ohms, M. Hofmann, R. Schneider, A.G. Youtsos, Statistical analysis of residual stress determinations using neutron diffraction, International Journal of Pressure Vessels and Piping, Volume 86, Issue 1, 2009, Pages 48-62, ISSN 0308-0161, https://doi.org/10.1016/j.ijpvp.2008.11.003.

4. R.S. Ramadhan, S. Cabeza, T. Pirling, S. Kabra, M. Hofmann, J. Rebelo Kornmeier, A.M. Venter, D. Marais, Quantitative analysis and benchmarking of positional accuracies of neutron strain scanners, *Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment, Volume 999, 2021, 165230, ISSN 0168-9002*

S12 - 5

NEUTRON DIFFRACTION LINE PROFILE ANALYSIS ON QUENCHED MEDIUM CARBON STEEL WITH TEMPERING AT DIFFERENT TEMPERATURES

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As one of the most vital materials for mechanical components, middle-carbon martensite steels have attracted interest due to their mechanical properties. The hardness of quenched ones is beyond 700 HV (~60 HRC) ; however, one puzzle that remains to be solved is the decrease in the elastic limit of as-quenched materials . Thus, this aims to quantitatively characterise the dislocation density and its arrangement for modelling to drop elastic limits in as-quenched martensitic steels. Uchima *et al.* proposed to correct dislocation density using the dislocation arrangement parameter obtained by line profile analysis (LPA) .

In this work, 0.56%C steel (AISI 1552 equivalent) was used after being heated to 830 C for 3 h and quenched into oil. Followed by quenching, tempering was performed at several temperatures from 160 °C to 490 °C. The tensile test of each specimen and 0.2% proof stresses were determined as the elastic limits. Neutron diffraction experiments were performed at ML20 in MLF, J-PARC. The obtained line profiles were analysed by convolutional multiple whole profile (CMWP) software . The details of specimen shapes and experiment conditions are shown in the paper .

Figure 1 shows the obtained dislocation density ( ) and dislocation arrangement parameter (M^*) via LPA for each tempering temperature. The dislocation density in the as-quenched specimen was the highest and monotonically decreased with tempering as a function of tempering temperature. On the other hand, the M^* values show different trends. The variation of M^* values was small until the tempering temperature was under 250 °C and dropped the value simultaneously around the temperature. Since the M^* values indicate the magnitude of the arrangement of dislocations, the result can show that the dislocations, which were randomly arranged in the as-quenched specimen, became arranged during tempering around 250 °C.

In **Figure 2**, the relationship between 0.2% proof stress and the square root of dislocations is shown. In general, the elastic limit or yield stress is proportional to the square root of dislocation density, known as the Bailey-Hirsch relationship. It is interesting to note, however, that the stresses did not increase proportionally as the square root of the dislocation density increased. Therefore, we employed a correction function that depends on the M^* values. At first, the relationship between M^* and tempering temperature, T, was shown as follows,

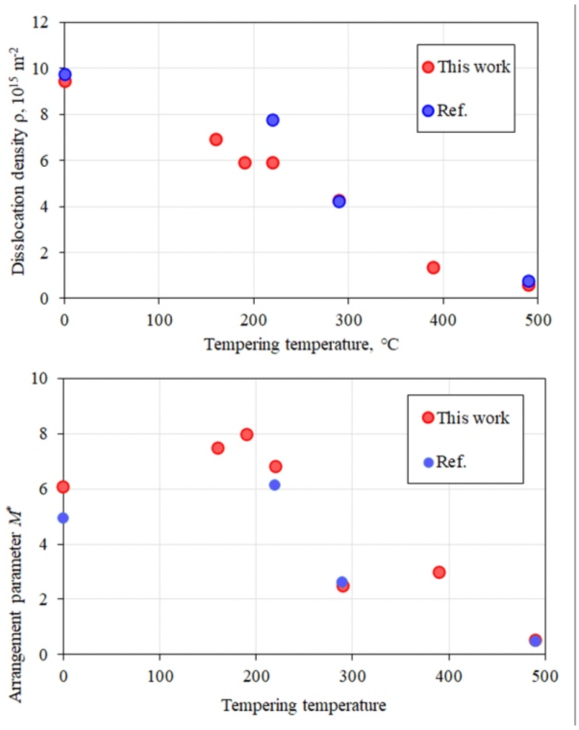

Figure 1. Obtained dislocation density (above) and arrangement parameter (bottom) via LPA as a function of tempering temperature

$$M(T)^* \quad \frac{\quad}{1 \quad \exp(\quad (T \quad T))} \quad M_0, \qquad (1)$$

here, and M_0 are constants. The effective dislocation density ($_{\mathrm{eff}}$) was introduced using eq. (1) as follows

$$_{eff} \quad \frac{1}{M(T)/M_0}. \qquad (2)$$

Dislocations in effective dislocation density impact yielding thresholds due to obstacle movement. The corrected relationship between 0.2% proof stress and the square root of dislocations is given by the eq. (2), are also shown in **Figure 2**. As a function of the square root of dislocation density, the 0.2% proof stress aligns on the approximated line.

Overall, we introduced a correction based on the dislocation arrangement parameter as an effective dislocation density parameter to address the puzzle. Despite having a higher dislocation density, as-quenched or low-temperature tempered materials have a lower 0.2% proof strength. Thus, by examining the arrangement of dislocations and correcting the total dislocation density to the effective dislocation density, one can determine the general relationship between yield strength and dislocation density.

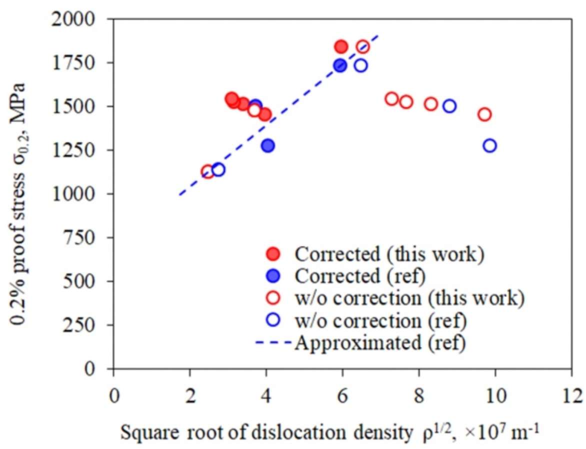

Figure 2. 0.2% proof stress obtained by tensile test as a function of the square root of dislocation density. Results in the past work [4] and this work were agreed. The corrected plots in both the past and this work were aligned on the approximated line.

1. J. Shimbe, M. Kumagai, A. Tanabe, Y. Mizuno, Variation of tensile strength and elastic limit due to tempering at low temperature on quenched medium carbon steel, Trans. JSME 87 (2021) 21-00082.
2. Y. Mizuno, M. Kumagai, A. Tanabe, J. Shimbe, Effect of work-hardening on the relationship between compressive strength and hardness of quenched medium carbon steel, Trans. JSME 90 (2024) 23-00242.
3. B. Hutchinson, P. Bate, D. Lindell, A. Malik, M. Barnett, P. Lynch, Plastic yielding in lath martensites - An alternative viewpoint, Acta Mater. 152 (2018) 239-247.
4. H. Uchima, M. Kumagai, J. Shimbe, A. Tanabe, Y. Mizuno, Y. Onuki, Impact of Dislocation Density and Mobility on Yielding Behavior in Quenched Medium-carbon Martensitic Steel Tempered at Low Temperature, ISIJ Int. 62 (2022) 998-1003.
5. G. Ribárik, B. Jóni, T. Ungár, Global optimum of microstructure parameters in the CMWP line-profile-analysis method by combining Marquardt-Levenberg and Monte-Carlo procedures, J. Mater. Sci. Technol. 35 (2019) 1508-1514.

Presentations of exhibitors

EL - 1

MRX PRODUCTS FOR RESIDUAL STRESS EVALUATION THROUGH X-RAY DIFFRACTION

A. Sprauel

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MRX is a company that develop equipments in the field of residual stress evaluation and quantitative analysis through X-ray diffraction. X-ray diffraction permits non-destructive control of crystalline material surface. The purpose of this presentation is to introduce the company and its products.

MRX main product is the X-Raybot, the first goniometer on a 6-axis collaborative robot. The robot allows to program and run automatic measurements on multitude of points, on the same or on different parts. The equipment is portable and can be used either on-site with a tripod or in a lab on a heavy-duty table. The XRaybot features several other innovations such as an air-cooled tube for low noise, an ultra-sensitive pure Si detector, and a laser triangulation module to position the goniometer at the right distance and orientation on complex parts shape, thus reducing error in normal and shear stress evaluation.

Great efforts are made on research and development of new products. A high-power version is now available for the X-Raybot. It uses water-cooled X-ray tube that can be powered on up to 30kV and 10mA, for a more intense signal.

1st in market 3D scanner, a more convenient positioning system is available as option. Based on a compact laser line triangulation module mounted directly on the goniometer (cf. Figure 1), this system can make accurate 3D scan of complex surface. Measurements points, lines or maps can then be selected on the 3D scan (cf. Figure 2) for a faster set-up. This system also permits scans matching to measure on the same points on identical parts, or a part that has been moved (e.g. after electro polishing).

To answer rising customer demands, MRX is also working on new equipments, such as a device dedicated to quantitative analysis of retained austenite or other phases with a 2D detector.

Figure 1. 3D scanner mounted on the X-Raybot.

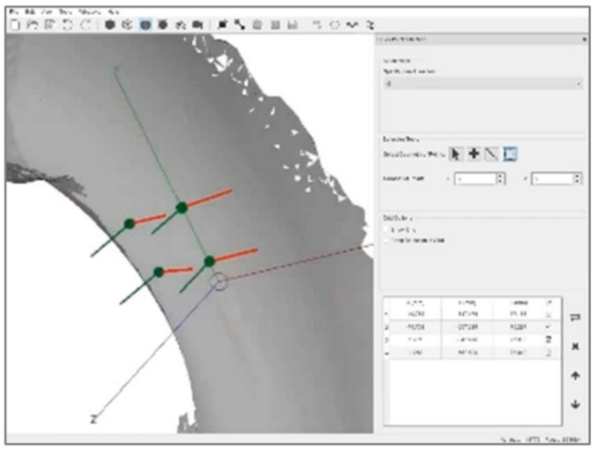

Figure 2. Measurement points definition in AdaptiveXRD software

EL - 2

ANTON PAAR – X-RAY ANALYSIS SOLUTIONS

Benedikt Schrode

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Anton Paar develops, produces, distributes and provides support for analytical instruments used in research, development and quality control worldwide. This presentation will give an overview of Anton Paar with a focus on X-ray based technologies we offer. From the latest addition to our portfolio, the multipurpose X-ray diffractometer XRDynamic 500, over the well-established non-ambient XRD attachments to small-angle X-ray scattering (SAXS). You will get an introduction to the history of X-ray analysis at Anton Paar and the current portfolio.

EL - 3

RESIDUAL STRESS ANALYSIS WITH A NEW TABLE TOP MULTIPURPOSE XRD INSTRUMENT (D6 PHASER)

Kurt Erlacher

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This presentation will discuss the use of a multipurpose table top XRD instrument with emphasis for residual stress measurements.

We will discuss the benefits of using such an instrument, and how it compares to traditional setups. The work will provide an overview of the instrument's features and capabilities, as well as discuss the advantages and disadvantages of using such a table top XRD instrument for residual stress measurements.

In Fe-based materials the amount of retained austenite is very often of great interest. As such, we also discuss the capabilities for retained austenite determination.

Finally, we will present several case studies demonstrating the successful application of this instrument.

EL - 4

INNOVATIONS IN RESIDUAL STRESS MEASUREMENT: RIGAKU'S CUTTING-EDGE X-RAY SOLUTIONS

Tom Faske

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Rigaku Corporation, the world's premier provider of X-ray instrumentation, is proud to present its latest advancements in residual stress analysis at the 11th European Conference on Residual Stresses (ECRS-11). Our talk, titled "Innovations in Residual Stress Measurement: Rigaku's Cutting-Edge X-ray Solutions," will delve into the challenges and breakthroughs in the field of residual stress measurement. As industry leaders, we have continuously pushed the boundaries of X-ray technology to meet the evolving needs of material science. At ECRS-11, we will showcase how our state-of-the-art instruments, including the renowned SmartLab series, provide unparalleled precision and reliability. Our presentation will cover the application of diffraction methods, advanced modeling techniques, and the integration of synchrotron and neutron approaches in our instrumentation. We will also discuss the role of X-ray technology in emerging sectors such as additive manufacturing and microelectronics, where managing residual stresses is critical for ensuring product quality and longevity. Join us at ECRS-11 to explore how Rigaku's innovations are shaping the future of residual stress analysis and contributing to the advancement of material sciences.

EL - 5

RESIDUAL STRESS MEASUREMENT ON A PITCH CIRCLE OF A GEAR TOOTH FLANK

Andrzej Wojtas

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This paper describes a procedure proposed for Residual Stress Measurement on a pitch circle of a gear tooth flank without removing the neighbouring tooth i.e. in a non-destructive measurement. The procedure can be applied to a $\sin^2$ method in omega geometry of an X-ray diffractometer.

The $\sin^2$ method, commonly used in stress diffractometers requires the knowledge of the d_0 – unstrained lattice parameter for the analysed material. It is usually taken from the first exposure at Psi = 0 i.e. perpendicular to the surface whereby the diffracting planes are parallel to the surface.

An exposure at = 0 in a measurement point on the pitch circle of a tooth flank would usually be obstructed by the neighbouring tooth and therefore using the conventional approach, such measurements are not possible without removing one tooth hence making the procedure destructive. In the proposed method the d_0 value is obtained also at = 0 but in a location only slightly moved towards the tooth head – accessible at = 0. It is assumed that the material composition and microstructure a few mm’s apart are identical and that the d_0 is exactly the same.

With the d_0 obtained from the point A a $\sin^2$ measurement can be performed in Point B without the first inclination at 0 . which would be obstructed by the neighbouring tooth.

The accuracy of this modified procedure should be verified, in each case, by performing both measurements in the same point. Furthermore the alignment of the data points on the $\sin^2$ plot will also be indicative of the correctness of the selected d_0 value.

EL - 6

STRESSTECH SOLUTIONS IN X-RAY DIFFRACTION, BARKHAUSEN NOISE ANALYSIS AND IN ESPI HOLE DRILLING

Mikko Palosaari[1], Sebastian Send[2], Carlo Scheer[2]

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In this presentation Stresstech as a company is introduced. The companys 40-years experience in manufacturing measurement equipment for Barkhausen noise analysis, X-ray diffraction and in ESPI hole drilling has made the company a reliable partner in grinding burn detection and in residual stress analysis. Stresstech solutions will be show-cased along with the latest advancements in the methodologies.

EL - 7

NOVEL RESIDUAL STRESS MEASUREMENT APPLICATIONS

E. Kingston

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VEQTER was incorporated 20 years ago as a spin-off from the University of Bristol providing Deep-Hole Drilling (DHD) residual stress measurement services to the Engineering Industry worldwide. Since then, VEQTER has diversified the techniques provided to become one of the world leaders in residual stress measurement services, offering the widest range of techniques available anywhere globally. To that end, VEQTER has carried out a wide variety of residual stress measurements for a wide variety of industries and within a wide variety of components, both at its headquarters in Bristol and on-site worldwide. Therefore, the presentation accompanying this abstract will review a small selection of novel applications using the DHD and other techniques to show what has been possible, the current state-of-the-art and the diverse requirements of the Engineering Industry.