

Poster sessions - group 1 Diffraction, Synchrotron radiation and Neutrons, Instruments

P1 - 1

BEER – THE NEUTRON DIFFRACTOMETER FOR MATERIALS ENGINEERING RESEARCH AT THE EUROPEAN SPALLATION SOURCE

J. Šaroun¹, P. Beran^{1,3}, J. Fenske², G. Nowak², D.J. Siemers², R. Kiehn², J. Burmester², P. Lukáš¹, B. Peric³

¹Nuclear Physics Institute of CAS, Husinec - Řež 130, 25068 Řež, Czech Republic ²Helmholtz-Zentrum Hereon, Max-Planck-Str. 1, 21502 Geesthacht, Germany ³European Spallation Source ERIC, P.O. Box 176, 22100 Lund, Sweden saroun@ujf.cas.cz

The European Spallation Source (ESS) is the high brilliance pulsed neutron source under construction in Lund, Sweden, which will provide new opportunities for research employing neutron scattering and imaging methods in Europe. Among the suite of 15 instruments [1] to be available for users by 2028, the *Beamline for European Materials Engineering Research* (BEER) will be unique in addressing the needs of researchers from both academic and industrial sectors for non-invasive lattice strain measurements, such as fast residual stress mapping of engineering components or in-situ and in-operando material studies under in-

dustrially relevant thermo-mechanical conditions and time scales

BEER has been designed as a time-of-flight diffractometer with a very long flight path (160 m) to take maximum advantage of the ESS 2.8 ms long pulse with high integral neutron flux. The neutron guide system with bi-spectral extraction optics [2] will deliver a broad neutron spectrum from both the thermal and cold surfaces of the moderator, yielding neutron flux of more than 10⁸ n/s/cm² over 0.17 nm wide wavelength band usable for neutron diffraction measurements (see Table 1 for more details).





Figure 1. Left: Experimental area of BEER, CAD view from detailed design. Right: Installed neuron guides.

Table 1. Performance characteristics of BEER for ESS source at 2 MW (simulation results).

Operation mode	Flux, n/s/cm ²	Resolution d/d [%]
High resolution diffraction (pulse shaping)	2.0 × 107	0.20
High flux diffraction (pulse shaping)	1.6 × 108	0.48
High resolution strain scanning (modulation)	4.3 × 107	0.16
High flux strain scanning (modulation)	2.3 × 108	0.37



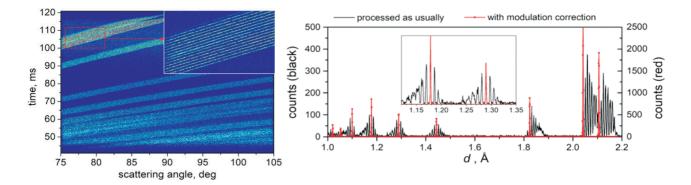


Figure 2. Simulated experiment (duplex steel). *Left*: modulated diffraction data. *Right*: Corresponding diffractogram before (black, left scale) and after reconstruction (red, right scale).

A pair of 1 m² area detector banks at +- 90° scattering angles will be available for simultaneous measurements of two perpendicular strain components (Figure 1). Variable input slit system and exchangeable radial collimators attached to the detector will allow to define gauge volume within large samples with spatial resolution down to sub-mm scale as suited for mapping of residual stresses. Accurate positioning of large samples or sample environment devices (e.g. stress rigs) up to the weight of 2 t will be provided by a hexapod motion system, while a robotic arm will be available for texture measurements or automatic exchange of smaller samples.

Unlike instruments at existing short pulse sources, BEER employs a cascade of choppers which can define wavelength resolution in a variety of optional resolution modes. For strain mapping experiments, where the crystal structure of dominant phases is known, modulation choppers can be used for diffraction peak multiplication [3]. This technique allows to multiply the data acquisition rate by a factor of up to more than 5 while preserving high resolution for lattice strain determination. The effect of modulation on simulated diffraction data and reconstruction of unmodulated diffractograms is illustrated in Figure 2. For diffraction measurements of unknown or low symmetry structures where the modulation method cannot be used, a set of pulse shaping choppers can define the wavelength resolution.

Present state and schedule

Many of the BEER components have already been produced or will begin production in 2024. Most of the 156 m long neutron guide system have already been installed and aligned in the ESS experimental hall. It is expected that the BER construction will be finished during 2026, followed by the hot commissioning phase and transition to user operation by 2028. The instrument capability will be further extended during subsequent upgrade stages by installing choppers needed for high-flux operation modes and adding detectors which allow for simultaneous strain and texture measurements and extend the accessible size scale during in-situ thermo-mechanical loading. Installation of smallangle scattering detector is also envisaged in the instrument design in order to provide additional information on microstructure evolution of materials under investigation.

- K. H. Andersen et al., Nucl. Instr. and Meth. A 957 (2020) 163402.
- J. Šaroun, J. Fenske J., M. Rouijaa, P. Beran, J. Navrátil,
 P. Lukáš, A. Schreyer, M. Strobl, J. Physics: Conf. Series,
 746 (2016) 012011.
- M. Rouijaa, R. Kampmann, J. Šaroun, J. Fenske, P. Beran, M. Müller, P. Lukáš, A. Schreyer, *Nucl. Instr. and Meth.* A 889 (2018) 7-15.

The BEER project is a common in-kind contribution to the ESS construction realized by the Helmholtz- Zentrum Hereon (Germany) and Nuclear Physics Institute, CAS (Czechia). The project is supported by the German Bundesministerium für Bildung und Forschung and the Czech Ministry of Education, Youth and Sports (LM2023057).



HIGH-RESOLUTION NEUTRON DIFFRACTION FOR FINER STUDIES OF POWDER DIFFRACTION LINES

P. Mikula, V. Ryukhtin, P. Strunz

Nuclear Physics Institute CAS, 250 68 Řež, Czech Republic mikula@ujf.cas.cz

Feasibility of focusing high-resolution three axis diffractometer with the polycrystalline sample between the monochromator and the analyzer for studies of finer effects of diffraction lines is routinely used at the medium-power reactor LVR-15 in Řež. The focusing three-axis set-up equipped with bent perfect crystal monochromator and analyzer exploits both focusing in real and momentum space and provides the intensity and resolution parameters for measurements within a reasonable measurement time [1-3]. It offers the sensitivity in determination e.g. of macro-strains in polycrystalline materials $\varepsilon = \Delta d/d$ close to 10^{-5} and $FWHM_A$ of the analyzed diffraction lines down to 5x10⁻² deg when obtained on virgin samples of the diameter of a few millimeters. Together with special tasks of strain/stress studies related, namely, to plastic deformation [4,5], it also permits to study a finer substructure of individual diffraction lines which can appear, namely, in the case of polycrystalline alloys where more phases having very close values of lattice spacing could exist. It will be documented on several experimental results [6,7]. Fig. 1 shows the schematic drawing of the diffractometer setting used for different width of the samples, when the samples of a rather large width (up to about 20 mm) are schematically pictured by a cylindrical solid situated in the horizontal position. Figs. 2 and 3 demonstrate the resolution properties of the setting for different α -Fe(110) samples. Then, when taking the samples of the Inconel 718, Figs. 4 and 5 demonstrate examples of observations of the accompanying phases γ' and γ'' having very close lattice spacings with respect to the γ matrix. The differences of the lattice spacings of the individual phases can be determined by using the relations: $\Delta\theta_A \approx -\Delta(2\theta_S)$ (for large values of R_A) and $\Delta\theta_S =$ $-(\Delta d_{\rm S}/d_{\rm 0,S})\cdot \tan \theta_{\rm S}$.

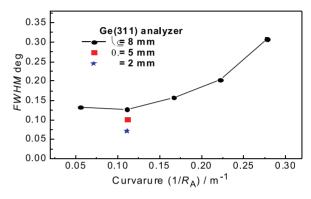
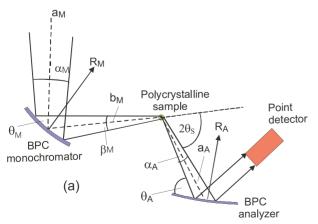


Figure 2. *FWHM* dependence of the analyser rocking curve for 3 diameters of α -Fe(110) standard samples situated in vertical position.

Finally, it can be stated that the presented neutron diffraction setting can offer an additional support to complement the information achieved by using the other conventional characterization methodologies. In this way we would like to inform possible external users that the beam time on the high-resolution diffractometer is offered through submission of the experiment proposal within the CANAM project:

 $\frac{http://www.ujf.cas.cz/en/research-development/large-r}{esearch-infrastructures-and-centres/canam/about-the-proj}{ect/}$

M. Vrána, P. Lukáš, P. Mikula, J. Kulda, *Nucl. Instrum. Methods in Phys. Research*, A 338, (1994), 125.
 DOI:10.1016/0168-9002(94)90172-4.



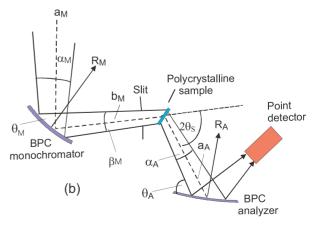
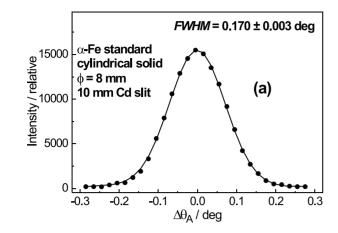


Figure 1. Schematic drawing of the 3-axis diffractometer setting using BPC monochromator and analyzer and a polycrystalline sample of a small width - (a), as well as the configuration of the setting for a sample of a rather large width - (b).





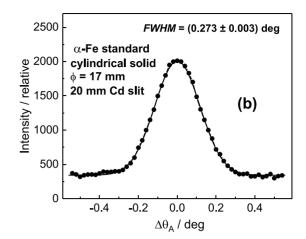


Figure 3. Examples of two analyser rocking curves for the α -Fe(110) samples of $\phi = 8$ mm - (a) and 17 mm - (b), respectively, both situated in horizontal position when simulating a large width of the irradiated gauge volume.

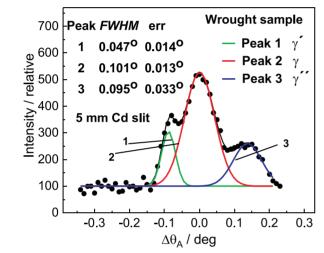


Figure 4. Analyser rocking curve rela-ted to the wrought Inconel 718 sample.

- P. Mikula, J. Šaroun, V Ryukhtin, and J. Stammers, *Powder Diffraction*, 35, Issue 3, (2020), 185.
 DOI:10.1017/S0885715620000329.
- 3. P. Mikula, J. Šaroun, V. Ryukhtin and P. Strunz, Materials MDPI, 13, (2020), 5449. DOI: 10.3390/ma13235449.
- R. Delhez, T. H. De Keijser, E. J. Mittemeijer, Fresenius' Zeitschrift für analytische Chemie, 312(1), (1982), 1. DOI:10.1007/BF00482725.
- K. Macek, P. Lukáš, J. Janovec, P. Mikula, P. Strunz, M. Vrána, M. Zaffagnini, *Mat. Sci. Eng.* A 208, (1996), 131. DOI:10.1016/0921-5093(95)10047-4.
- 6. P. Mikula and V. Ryukhtin, *Procedia Structural Integrity*, *43*, *(2023)*, *119*. DOI: 10.1016/j.prostr.2022.12.245.
- 7. P. Mikula and V. Ryukhtin, *On a Possibility of High-Resolution Neutron Diffraction Observation of Inconel 718 Substructure Phases*, Proc. of the EAN 2023 Conf., 5. 6. -8. 6. 2023, Košice, Slovakia. In print.

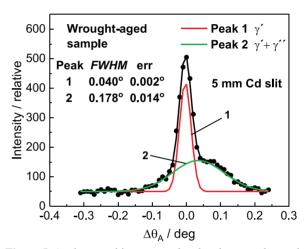


Figure 5. Analyser rocking curve related to the wrought-aged Inconel 718 sample.

Measurements were carried out at the CANAM instrument of NPI CAS Řež installed the CICRR infrastructure, which is financially supported by the Ministry of Education and Culture - project LM2023041. The authors acknowledge support from ESS participation of the Czech Republic – OP (CZ.02.1.01/0.0/0.0/16_013/0001794) and from the project ESS Scandinavia-CZ II (LM2018111), respectively. Furthermore, they acknowledge support from the CAS in the frame of the program "Strategie AV21, No. 23". The authors thank B. Michalcová from NPI CAS for significant help with measurements and data elaborations.



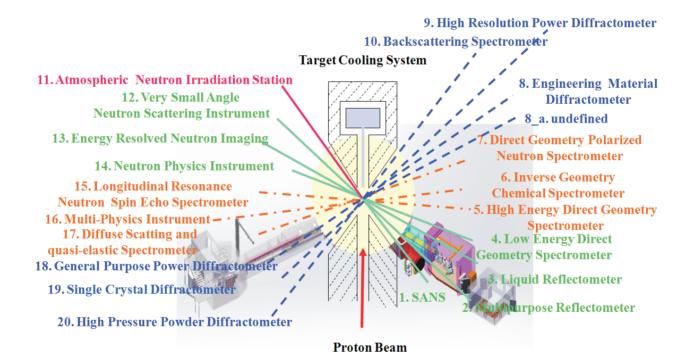
THE ENGINEERING MATERIALS DIFFRACTOMETER "EMD" AT THE CHINA SPALLATION NEUTRON SOURCE

Liang Zhou $^{1,2^*}$, Wenting Du 1,2 , Chao Ding 1,2 , Xiaodong Zhang 1,2 , Chen Wang 1,2 , Wenli Song 1,2 , Chunming Hu 1,2 , Xiaohu Li 1,2

¹Institute of High Energy Physics, China Academy of Science, Beijing, 100049, China ²Spallation Neutron Source Science Center, Dongguan, 523803, Guangdong, China

The China Spallation Neutron Source (CSNS) is the fourth global pulsed spallation neutron source, officially launched in 2018. Currently, CSNS operates at 140KW, with plans to upgrade to 500KW in the future. CSNS intends to build a total of 20 neutron instruments, with 8 already accessible to the public and 3 currently being constructed. One of these instruments is the Engineering Material Diffractometer (EMD), which specializes in studying strain mapping, microstructure, phase transformations, texture, and Bragg-edge transmission imaging. Currently, EMD is in the trial phase, offering a maximum neutron flux of 9*10⁶

n/s/cm² and a best resolution of 0.25%. It operates in high-FOM (figure of merit) mode for most cases, high-intensity mode for texture, and high-resolution mode for tiny structural changes. Notably, EMD has successfully conducted tests on residual stresses of standard samples, such as Vamas and TG4, consistently producing reliable results that compare well with other diffractometers worldwide. Additionally, EMD has both uniaxial and biaxial tensile devices for conducting *in-situ* tensile experiments. In the future, it will be upgraded with more diverse sample environments to enable more complex experiments.





OPTIMIZED XRD MEASUREMENT CONFIGURATIONS FOR RESIDUAL STRESS ANALYSIS WITH XRDYNAMIC 500

Benedikt Schrode, Andrew O. F. Jones, Marius Kremer, Praveen Vir

Anton Paar GmbH, Austria
Benedikt.schrode@anton-paar.com

With the launch of the XRDynamic 500 automated multipurpose powder X-ray diffractometer, Anton Paar is breaking new ground in XRD and taking materials research to the next level. The core of XRDynamic 500 is the TruBeamTM concept, comprising a large goniometer radius and evacuated optics units, automatic change of the beam geometry and all optics components, and automated instrument and sample alignment routines. All of these features combine to deliver outstanding data quality that can be measured with high efficiency in a straight-forward manner.

The high level of automation means that you can perform measurements on various sample types and shapes without any user interaction, even with different beam geometries and instrument configurations. Due to the easy exchange of the X-ray source and the automated instrument alignment, fluorescence issues can be treated at their root instead of only the symptoms.

In addition to the key instrument features and benefits, application examples highlighting residual stress measurements will be presented.

P1- 5

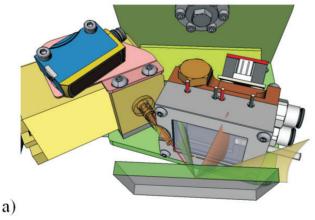
ASSESSMENT OF ACCURACY OF A PORTABLE DIFFRACTOMETER FOR RESIDUAL STRESS MEASUREMENT

F. Cazes¹, H. Sankaran¹, B. Bacroix¹, G. Michaud¹, C. Jeannot², C. Lebreton²

¹LSPM–CNRS, UPR 3407, Université Sorbonne Paris Nord, F-93430 Villetaneuse, France ²ADR, groupe ALCEN, Thomery, France fabien.cazes@lspm.cnrs.fr

We present residual stress measurements obtained on steel using an InelInnov [1] (formerly Inel) X-Solo portable diffractometer presented in Fig. 1a. The diffractometer is equipped with a chromium target generator and a 2D detector. Measures from the 2D detector, as represented in Fig. 1b, are analysed using Rietveld software Maud [1] and house made tools in Python for image analysis in the fashion of strategies proposed in [3] and [4]. A sheet bending device was developed for the study, in order to validate the measurements made with the diffractometer. The curvature of the sheet is measured beforehand using a comparator or from image analysis of photographs of the specimen in the

bending device. This enables to evaluate the deformation along the surface of the specimen that will be used as a reference to be compared with the diffraction measurements. As the diffractometer possesses no sample holder, parts are designed for the different specimen geometries to be investigated and manufactured with 3D printing. An optical device is developed in order to determine to location of the irradiated zone in the sample. Experimental difficulties such as the positioning of the sample in with respect to the diffractometer, determining the cell parameter of the material, and correcting measurement artefacts will be presented. The aim of this preliminary work is to validate the



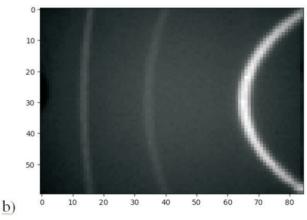


Figure 1. X-Solo device for residual stresses measurements (a) and diffraction rings observed for an iron powder (b).



experimental protocol and to assess stress measurement accuracy of the device. In the longer term, the aim is to assess the nature of residual stresses present in ball bearing rings manufactured by project partner ADR-Alcen, which can lead to distortions following material removal and heat treatment. It is important to control these distortions, which can jeopardize the correct assembly and operation of bearings in service.

- InelInnov https://inelinnov.com/.
- 2. Lutterotti, L., Matthies, S., & Wenk, H. (1999). MAUD: a friendly Java program for material analysis using diffraction. *CPD NEWSLETTER*, 21, 14-15.
- Francois, M. (2023). X-ray stress analysis with a 2D detector. Part 1. Data acquisition and treatment strategies. J. Appl. Cryst. 56, 48-60.
- He, Bob B. Two-dimensional X-ray Diffraction. John Wiley & Sons, 2018.

P1 - 6

FUNDAMENTAL STUDY ON A 3D RESIDUAL STRESS ESTIMATION METHOD USING X-RAY DIFFRACTION FOR MACHINED SURFACE MATERIALS.

R. Okui, T. Kitazawa, Y. Sano, M. Ogawa

Kogakuin University, 1-24-2 Nishishinjuku, Shinjuku-ku, Tokyo 163-8677, Japan ogawa-masaru@cc.kogakuin.ac.jp

Residual stresses generated during machining affect the dimensional accuracy and fatigue strength of structures. On the other hand, adding compressive residual stress through surface modification processing such as laser peening can improve fatigue strength. Understanding the relationship between various surface processing conditions and residual stresses makes it possible to consider high-precision processing methods and assess fatigue life. However, the X-ray diffraction method can only measure the surface residual stress. Repeating electropolishing and X-ray diffraction to measure the residual stress at deeper depths does not allow the measurement of the original residual stress values, as the residual stress is released by electropolishing. Neutron diffraction is the ideal method, but requires extensive facilities and relatively long measurement time. Therefore, there is a relatively simple method to estimate three-dimensional residual stress using the eigenstrain theory [1-3] and the X-ray diffraction method [4] (hereafter referred to as 'this method'). The aim of this study is to assess the three-dimensional residual stress distribution for surface worked materials with relatively high accuracy using this method. The estimation accuracy of this method is demonstrated by numerical analysis using a relatively sim-

The relationship between the surface elastic strain $\{\epsilon_e\}$ and the three-dimensional eigenstrain $\{\epsilon^*\}$ [5] can be expressed by the following equation

$$\{\varepsilon_e\} = [R]\{\varepsilon^*\},\tag{1}$$

[R] is the elastic response matrix that relates the surface elastic strain to the three-dimensional eigenstrain and can be obtained if the Young's modulus, Poisson's ratio and geometry of the component are known. The inverse analysis [6] to estimate the three-dimensional eigenstrain using the surface elastic strain can be expressed by the following equation.

$$\{\varepsilon^*\} = [R^+]\{\varepsilon_\varepsilon\},\tag{1}$$

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

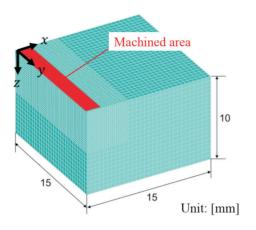
The inverse analysis in this study estimates the three-dimensional eigenstrain from the two-dimensional elastic strain on the surface. Therefore, the number of unknowns must be reduced appropriately to improve the estimation accuracy of this method. In this study, it was assumed that the eigenstrain is distributed constant in the working direction. The thickness-directional distribution of the eigenstrain in each direction was approximated by a linear combination of several functions that multiply a Gaussian function and a Chebyshev polynomial.

The FE model is shown in Fig. 1. This model is a quarter model symmetrical with respect to the x=0 mm plane and the y=0 mm plane. A laser shock peening process is assumed, and the machined area is assumed to be $0 \le x \le 2$ mm, $0 \le y \le 15$ mm and z=0 mm. The material is stainless steel with a Young's modulus of 200 GPa and Poisson's ratio of 0.3, and the number of nodes and elements are 43,331 and 39,000, respectively. The elastic strains of the x- and y-directional components of all nodes at z=0 mm obtained using exact eigenstrains were used as measurement data to estimate three-dimensional eigenstrains. The measurement error of the X-ray diffraction method was expressed by a random number following a normal distribution with a standard deviation of 30 MPa and added to the value of the surface elastic strain.

A comparison of the exact and the estimated residual stresses is shown in Fig. 2. The red bold line is the exact distribution, and the eight dotted lines are the estimated values with different measurement errors. The results show that relatively high estimation accuracy was achieved.

1. A. M. Korsunsky, *Journal of Mechanics and structures*, 1, (2006), pp. 259–277.





Exact

Estimated with error1

Estimated with error3

Estimated with error3

Estimated with error3

Estimated with error4

Estimated with error4

Estimated with error4

Estimated with error6

Estimated with error6

Estimated with error7

Estimated with error7

Estimated with error7

Estimated with error8

Figure 1. FE model used for numerical analysis.

Figure 2. Comparison between correct and estimated residual stresses.

- 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.
- 4. M. Ogawa, Transactions of the Japan Society of Mechanical Engineers, 80, (2014), SMM0195.
- T. Mura, Micromechanics of defects in solids, Martinus Nijhoff Publishers, 1987.
- 6. S. Kubo, Inverse Problem, Baifukan, 1992.

Part of this study was funded by the Japan Welding Engineering Society.

P1 - 7

THE SIN²PSI AND ALL SIMILAR METHODS SHOULD BE REPLACED BY SOMETHING BETTER

175

B. Ortner

Retired from University Leoben, Leoben, Austria Balder.Ortner@aon.at

The $\sin^2\psi$ method has been in use for about one century. Its advantge in times without modern computers are clear: one needed no more than a piece of paper, a pencil, a ruler and a keen eye to draw regression lines. Its disadvantage, not been realized for a long time, is simply this: it is not a least squares method. The consequence is a loss in accuracy of the results – the calculated stress tensors. Now, since we all have computers available at any time, this advantage is no more valid, the disadvantage still exists. Therefore we all should forget the sin psi method and replace it by something better which meets the requirements of a least squares method. The prerequisit for such method is already known for nearly half a century. This is the famous equation, discoverd by Dölle and Hauk [1, 2]:

$$\varepsilon(\varphi, \psi, hkl) = F_{ij}(\varphi, \psi, hkl)\sigma_{ij}$$
 $i, j = 1..3$

 $\varepsilon(\varphi, \psi, hkl) = F_i(\varphi, \psi, hkl)\sigma_i$ i = 1..6; according to Voigt's or Kelvin-Mandel's notation

or

$$d(\varphi, \psi, hkl) = d_0 (1 + F_i(\varphi, \psi, hkl)\sigma_i)$$

 $F_{ij}(\varphi, \psi, hkl)$ are called x-ray elastic factors. They are easily calculated if s_1 and s_2 are known (the case of a quasiisotropic polycrystal) and also easily calculated for a single crystalline material.

For a polycrystalline material with texture, the methods to calculate F_{ij} (ϕ , ψ , hkl) are also known, one only must have the orientation distribution function.

With Dölle-Hauk's equation we can establish a system of linear equations, the solution of it is a matter of only one or two commands in a computer programme.

In the case of a textured material or a single crystal, in some cases with special circumstanced for data acquisition, the $\sin^2 \psi$ method is not applicable or is applied with strong loss of reliability.

Therefore, many methods were invented to overcome all these shortcomings of the sin, psi method. And most of these methods rely on one or more linear regressions and therefore are in a similar way erronious as the sin, psi method.

In former papers [3-6] we discussed these errors by regarding standard deviations. In the actual contribution we are taking a different approach to explain the flaws of all



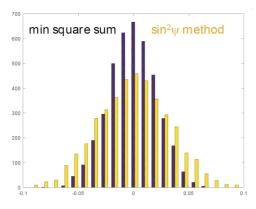


Figure 1.

these linear regression methods. An approach which is less theoretical and abstract. This is the simulation of measurements (measurements with measurement errors, generated by random numbers) followed by one of the linear regression methods or the correct method as described above. We then looked at the probability of the results to be more or less close to the correct values. An example is shown here for the template of all these methods, the sin psi method. We assumed a concrete stress state, calculted strains at $\varphi =$ 0° , 60° , 120° , at ψ so that $\sin^2 \psi$ is in equal distances, assumed measurement errors and then calculated the σ_x , σ_y , σ_{xy} . In the histogram of Fig 1 one can see how often the result deviates a certain amount from the exact value. Obviously, if using the correct method the result is more often close to the correct value as when the sin psi method is used. In Fig 2 are the same results depicted in a different way. There are the tops of the histogram bars plotted and conneted with lines. Therefore one can even more immediately see that the chance for a accurate result is much higher when using the least squares method instead of the sin psi

Quite similar results are obtained for all other linear regression methods we analysed. Results will be presented for some of these methods, the Dölle-Hauk method [7], φ -integral method [8], crystallite group method [9], g-method [10], $\cos \alpha$ -method [11].

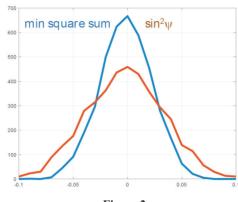


Figure 2.

- 1. H. Dölle, V. Hauk, Z. Metallkde. 70 (1979), 682-685.
- 2. H. Dölle, V. Hauk, Z. Metallkde. 69 (1978), 410-417.
- 3. B. Ortner, Advances in X-ray Analysis, Volume 50, (2007) 117-127.
- 4. B. Ortner, Advances in X-ray Analysis, Volume 52, (2009) 763-772.
- 5. B. Ortner, Powder Diffraction 24-2-sup (2009) S16-S21.
- 6. B. Ortner, Materials Science Forum Vol. 681 (2011) pp 7-12, Trans Tech Publications, Switzerland.
- 7. V. Hauk Ed., Structural and Residual Stress Analysis by Nondestructive Methods: Elsevier 1989, p. 148.
- 8. Ibid, p. 171.
- 9. Ibid, p. 205.
- 10. A. C. Vermeulen, Mat. Sci. Forum, 404-407 (2002) 35-42.
- S. Taira, K. Tanaka, T. Yamazaki, J. Soc Mat. Sci. Japan, 27–294 (1978) 251.



RESIDUAL STRESS MEASUREMENTS USING DIFFERENT TECHNIQUES, SIN, PSI AND COS-ALPHA TECHNIQUE

Mohammed Belassel¹, James Pineault¹, Michael Brauss²

¹PROTO MANUFACTURING LIMITED, 6150, Morton Industrial Parkway. Lasalle, Ontario, Canada. mbelassel@protoxrd.com, jpineault@protoxrd.com

²PROTO MANUFACTURING INC, 12350 Universal Drive, Taylor. Michigan, USA. mbrauss@protoxrd.com

A sound approach for residual stress measurements in crystalline materials have been widely developed and applied for many decades. It was shown that the development of the techniques successfully passed many crucial steps to produce the best theory and practice. These steps are well documented and available for review in many published books and scientific papers.

In this paper we feel the urge to review these techniques and spell the theory and practice developed until present to help the users adhere to the best path in order to achieve the best results that can help science and industry.

The techniques include the Sin²Psi and Cos-Alpha techniques. We will emphasize the known and the unknown form theory and practice point of view. In addition, we will review the theory from the physics and the mechanics point of view where divergence sometimes is obvious in particular the difference between the strain measured using a strain gauge and the strain measured using x-ray diffraction

P1 - 9

GFAC NATIONAL AND INTERNATIONAL ACTIVITIES ON RESIDUAL STRESS EVALUATION

K. Wegerhoff¹, T. Bergey², A. Cartier², E. Wasniewski¹, GFAC members³, F. Lefebvre¹

¹CETIM, 52 Avenue Felix Louat, Senlis, 60304, France ²ArianeGroup, Foret de Vernon, Vernon, 27208, France ³SF2M and AFM, French association, France kerian.wegerhoff@cetim.fr

1. Abstract

Residual stresses are self-balancing static multiaxial stresses existing in an isolated system of uniform temperature and in the absence of any external loading. They have a crucial role in industry by influencing the performance of materials, particularly in terms of fatigue, cracking and deformation. Accurately assessing residual stresses remains a complex issue. Conventional techniques such as X-ray diffraction, incremental hole drilling method or contour method are commonly used but have many limitations.

2. Introduction

The GFAC (French Group for residual stress analysis) works since several years on projects like external reference samples in relation with EN 15305-2009 standard or material removal correction. This is a group of industrialists and academics affiliated to the « Société française de Métallurgie et de Matériaux » (SF2M) and the « Association Française de Mécanique » (AFM). Its natural position is at the crossroads of materials and mechanics. The French industrial and university laboratories that use the various methods of residual stress analysis participate in this group.

3. GFAC current projects

The aim of the GFAC is to carry out research and development work of common interest to its members and to pool their resources in order to work on the following items:

- Reference samples [1]
- Material removal correction
- Standardisation work
- 2D Detector

The challenge is to improve best practice in industrial and university laboratories in the determination of residual stresses by X-ray diffraction through specific research work. Standardisation work is underway with the French standardisation agency (AFNOR). The latest French standard proposed concerns the quantification of phases using X-ray diffraction (NF A09-282). Work to revise existing standards EN15305 [2] and NFISO 21432 [3] on the determination of residual stresses is due to begin in the next few years at European level. Best practices are exchanged between laboratories at quarterly meetings. In addition, reference samples [4] have been created in recent years, and round robin tests are carried out to provide each laboratory taking part in the tests with a reference sample validated by an accredited certificate.



4. CETIM activities for the Easi-Stress project

On a national level, CETIM is part of the GFAC consortium. CETIM is also one of the members of the European Easi-Stress project. The Easi-Stress project has the aim to strengthen industrial access to large-scale research infrastructure of non-destructive synchrotron X-ray and neutron diffraction-based for residual stress characterization.

One of the studies carried out as part of the Easi-Stress project is a comparison of residual stress evaluation using the contour method, neutron diffraction, high energy synchrotron X-ray diffraction, laboratory X-ray diffraction and hole drilling. The aim is to examine a series of components with industrial relevance with complex geometries, material systems and residual stress fields.

The residual stress analyses presented in Figure 1 have been obtained on a sample produced by laser powder bed fusion (L-PBF) additive manufacturing using stainless steel 316L powder. The residual stresses are evaluated in three directions (x,y and z). Various stress analyses were also carried out on welded components, Cast wedge and U-forms samples.

- E. Wasniewski, B. Honnart, F. Lefebvre, E. Usmial, "Material removal, correction and laboratory X-ray diffraction" Advanced Materials Research, vol. 996, 2014, pp. 181-186.
- NF, NF EN 15305: 2009 Non-destructive testing Test method for residual stress analysis by X-ray diffraction.
- 3. NF ISO 21432: 2020 Non-destructive testing Test method for residual stress analysis by neutron diffraction.
- F. Lefebvre, E. Wasniewski, M. Francois, J. Cacot, P. Le-bec, E. Baumhauer, D. Bouscaud, T. Bergey, "External reference samples for residual stress analysis by X-ray diffraction", Advanced Materials Research, vol. 996, 2014, pp. 221-227.

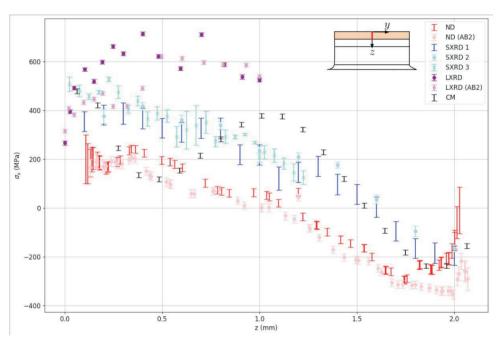


Figure 1. Results for as-built components across methods showing stresses acting in the x direction.



TRIAL OF NEW TREATMENT IN CASE OF BIG GRAINS

Gy. Török, M. Markó

HUN-REN Centre for Energy Research, Konkoly-Thege Miklós út 29-33., Budapest, 1121, Hungary Torok.gyula@ek.hun-ren.hu

In case of residual stress measurement in classic procedure we need to choose a gauge volume where the number of grains are some 1000 to achieve the proper average the peak form.

This peak shift will give us the strain parameters. The disturbance of normal peak form leads to the unwanted shift of peak position, leading to the systematic error in the strain measurement.

In reality the number of grains are less due to request of space resolution from customers and the real grain size of material.

In neutron case usually in steels the problem is solvable with tilting of sample +/- 5 degrees, however in multi pass welding the grain size can grow such way that tilting not help. Also we have a problem with the preferred orientation where the elastic constant parameters deviate from the averaged one. In our treatment we do not try to correcting all of systematic errors only try to reduce it.

In existing peak do a differential one, in order to find the maximum. From differential curve choose the part what closer to the resolution of setup.

We suppose that the resolution is known from other sources. (a measurement with good average of grain). Then make a fit with "good" points only.

That results less fluctuation in data supposing that the resolution function does not depends substantially from the investigated gauge volume.

(That is often correct for neutron measurement, because of SANS contribution is small and usually the adsorption is small)

The correction is not eliminating all of errors but reduces in our case a factor of 2.

The automatic correction is possible with program.

We will get information not only the strain/stress in the material, but the train/stress fluctuation too.

P1 - 11

ROLE OF THE SECOND ORDER PLASTIC INCOMPATIBILITY STRESSES ON PLASTIC DEFORMATION IN DEFORMED MAGNESIUM

S. Wronski¹, S. Szostak¹, M. Wronski¹, A. Baczmanski¹, M. Marciszko-Wiąckowska², M. Klaus³, Ch. Genzel³

¹AGH University of Science and Technology, WFilS, al. A. Mickiewicza 30, 30-059 Kraków, Poland ²AGH University of Science and Technology, ACMIN, al. A. Mickiewicza 30, 30-059 Kraków, Poland ³Abteilung fürMikrostruktur- und Eigenspannungsanalyse, Helmholtz-Zentrum Berlin fürMaterialien und Energie, Albert-Einstein-Str. 15, Berlin 12489, Germany wronski@agh.edu.pl

In the presented research, the second-order plastic incompatibility stress in magnesium was investigated by means of diffraction. The lattice strains were measured by the multiple reflection method using high energy X-rays diffraction during uniaxial in situ tensile tests. One of the important reasons for the formation of residual stresses in polycrystalline materials is the anisotropy of the plastic deformation process. Different slip systems activity leads to different plastic deformations of polycrystalline grains. The resulting misfit (incompatibility) between neighboring grains is the source of the second order incompatibility stresses. The second-order residual stresses [1], characterizing the heterogeneity of the stresses on the scale of polycrystalline grains, may affect the plastic deformation process of the material. The stress measurements were performed in situ during the tensile test were made using synchrotron ED diffraction at BESSY (EDDI beamline HZB, Berlin) using a white beam (wavelength in the range λ : 0.18–0.3 Å). The geometry of measurements were pre-

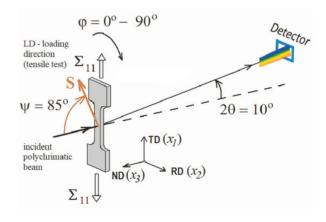


Figure 1. An experimental setup used for lattice strain measurement by ED diffraction.



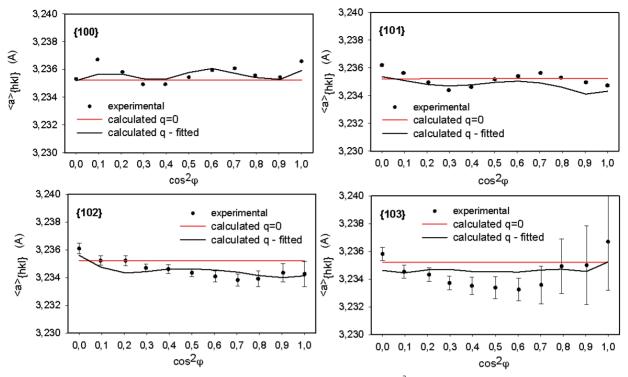


Figure 2. Measured lattice parameters (points) and theoretical results (lines) vs. $\sin^2 \psi$ for unloaded sample. Black lines for $q \neq 0$ (the second order stresses are taken into account) and red lines for q = 0 (the influence of second-order stresses is neglected).

sented in figure 1. Diffractograms were collected with the steps of 0.1 vs. $\cos 2\varphi$, within the range of $\varphi = (0^{\circ}, 90^{\circ})$, in symmetrical transmission mode for a constant $2\theta = 10^{0}$ scattering angle.

It can be shown [2] that an average lattice parameter, measured in the direction of the scattering vector, can be expressed as:

$$\left\langle a(\phi, \psi) \right\rangle_{\{hkl\}} = \left[F_{ij} \left(hkl, \phi, \psi \right) \sigma_{ij}^{I} + q \left\langle \varepsilon^{II, model} \left(\phi, \psi \right) \right\rangle_{hkl} \right] a_0 + a_0$$
(1)

where; $F_{ij}(hkl, \phi, \psi)$ are diffraction elastic constants, σ^{I}_{ij} - macroscopic stresses, a_0 - the equivalent lattice parameter in a stress-free material. The $<\epsilon^{II,model}(\phi,\psi)>_{hkl}$ tensor characterises incompatibility stresses which remain after the unloading of macro-stresses $(\Sigma_{ij}\rightarrow 0)$ and are caused by inter-grain plastic deformation incompatibility. The $<\epsilon^{II,model}(\phi,\psi)>_{hkl}$ strain remains after unloading of the macrostresses and it can be calculated by the self-consistent model. The anisotropy of the incompatibility stresses can be correctly predicted by the model if the experimental texture is used as the input data. To predict the evolution of lattice parameter during tensile test the Elastic-Plastic

Self-Consistent (EPSC) model developed by Lipiński and Berveiller [3]. The experimental $< a(\phi, \psi)>_{\{hkl\}}$ vs. $\sin^2\psi$ curve for unloaded sample was presented in Figure 1. As shown by black line, the quality of the fit improves significantly when the second order stresses are taken into account in the analysis and q is determined from Eq. (1). This important improvement of fitting quality – when the q parameter is adjusted – proves that the analysis is carried out correctly and the significant second order stresses are generated during plastic deformation in the studied sample.

- E. Macherauch, H. Wohlfahrt, U. Wolfstieg, Zur zweckm" aßigen Definition von Eigenspannungen, HTM J. Heat Treatment Mater. 28 (1973) 201–211, https://doi. org/10.1515/htm-1973-280305.
- A. Baczmański, M. Wroński, P. Kot, S. Wroński, A. Łabaza, K. Wierzbanowski, A. Ludwik, M. Marciszko-Wiąckowska, Mater. Des. 202 (2021) 109543.
- P. Lipinski, M. Berveiller, E. Reubrez, J. Morreale, Arch. Appl. Mech. 65 (1995) 291–311.

This work was supported by grants from the National Science Centre, Poland (NCN) No. 2019/35/O/ST5/02246 and 2023/49/B/ST11/00774.



DEPTH-RESOLVED RESIDUAL STRESS ANALYSIS OF MECHANICALLY PROCESSED WC-Co HARDMETALS BY MEANS OF A MULTI-WAVELENGTH XRD METHOD

S. Send¹, C. Scheer¹, M. Kipp², D. Biermann², M. Palosaari³

¹Stresstech GmbH, Konnwiese 18, 56477 Rennerod, Germany ²TU Dortmund, Institute of Machining Technology, Baroper Straße 303, 44227 Dortmund, Germany ³Stresstech Oy, Tikkutehtaantie 1, 40800 Vaajakoski, Finland sebastian.send@stresstech.com

Machining of WC-Co cemented carbides, e.g. by means of blasting, grinding and finishing, gives rise to a characteristic residual stress depth profile in the near surface region of the material extending to a depth of a few um. The magnitude and the depth gradient of the generated normal stress affect the performance of the hardmetal in industrial applications. Under usual laboratory conditions, the capabilities of monitoring residual stress depth profiles in WC-Co are limited due to the lack of an appropriate procedure for material removal. In this work various WC-Co samples, mechanically treated with different processing parameters, were investigated as per DIN EN 15305 [1] based on a multi-wavelength XRD method. For that purpose, an Xstress G2R equipped with a Ti tube and an Xstress DR45 equipped with Cr, Mn and Cu tubes served for data acquisition in modified-γ geometry. The Xstress DR45 uses two-dimensional XRD [2, 3] enabling residual stress measurements with short exposure times and was operated in standard mode [4]. In Tab. 1 the analysed Bragg peaks of the WC phase generated by the respective applied radiation are listed with the associated diffraction angles 2θ and mean penetration depths τ . Consequently, the information accessible by the XRD method is limited to a depth range up to about 1.6 µm below the sample surface.

In order to derive correct residual stress values from the recorded strain data, the X-ray elastic constants of the investigated WC-Co material were determined experimentally. Mounting a WC-Co test specimen in a four-point bending device, strain measurements could be carried out in a compressive bending mode with appropriate load cycles as defined in DIN EN 15305. Fig. 1 shows the results obtained for the slope of the $\varepsilon(\sin^2\psi)$ curves for the various hkl reflections as a function of the external load stress. The corresponding microscopic X-ray elastic constants $(1/2)S_2^{hkl}$, which are given by the slopes of the dashed lines

Table 1. Experimental conditions for depth-resolved residual stress measurements of WC-Co.

Radiation	Τί Κα	Cr Kα	Mn Kα	Cu Kα
WC {hkl}	110	102	201	212
20	142.2°	135.8°	132.3°	154.8°
τ	0.35 μm	0.53 μm	0.63 μm	1.62 μm

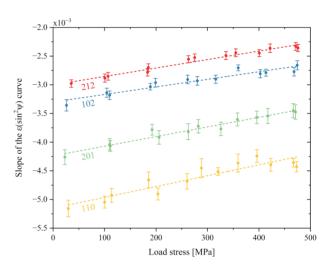


Figure 1. Measurement results after four-point bending to determine the X-ray elastic constants $(1/2)S_2^{hkl}$ of WC-Co.

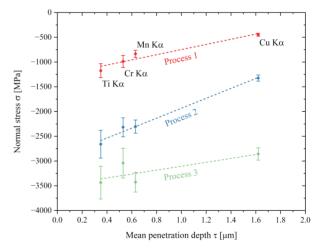


Figure 2. Measured and fitted normal stress depth profiles of WC-Co obtained after different machining processes.

fitted through the data points, could be extracted with relative standard deviations between 7 % and 14 %.

In the next step, applying the experimental $(1/2)S_2^{hkl}$ values to strain data, the residual stress depth profiles of the defined set of mechanically processed WC-Co samples were measured under the conditions of Tab. 1. Fig. 2 displays the depth-resolved normal stress in the grinding direction of the material in Laplace space for three different



states after machining of WC-Co. The dashed curves are linear least-squares fits of the data points providing identical normal stress profiles after re-transformation to real space [5]. In the depth range probed by the multi-wavelength XRD method it could be demonstrated that the types of mechanical processing give rise to characteristic normal stress levels and gradients. The data revealed that the normal stress gradient generated by process 2 is about twice as large as the one generated by processes 1 and 3.

1. DIN EN 15305, Non-destructive testing – Test method for residual stress analysis by X-ray diffraction, 2008.

- B. B. He, Two-Dimensional X-Ray Diffraction, second edition (Hoboken: John Wiley & Sons), 2018.
- 3. B. B. He, Proceedings of ICRS 10, Material Research Proceedings 2, (2016), 265-270.
- 4. S. Send, D. Dapprich, M. Palosaari, ICRS 11 conference papers, hal-04015041, 2022.
- C. Genzel, postdoctoral thesis, Humboldt University of Berlin, 1999.

P1 - 13

EVOLUTION OF SHEAR RESIDUAL STRESSES IN STEEL SPECIMENS DURING THE TORSION TEST

D. Perez-Gallego^{1,2}, J. Ruiz-Hervías^{1,2}, D.A. Cendón Franco^{1,2}, A. Valiente^{1,2}

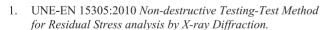
¹Centro de Investigación de Materiales Estructurales (CIME)

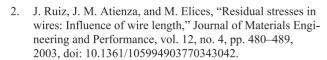
²Departamento de Ciencia de Materiales. ETSI Caminos, Canales y Puertos.

Universidad Politécnica de Madrid

d.perez@upm.es

The test method for residual stress analysis by X-ray diffraction [1] proposes the use of reference samples with a known value of residual stresses for the qualification and verification of the equipment. As the authors have explained in previous studies, the stress-reference samples that are usually employed have a known value of the normal residual stresses, although the shear stress is also included in the qualification process. Consequently, it would be necessary to have samples with known tangential residual stresses as well, so that the test method could be validated independently for each of the residual stress components. Previous work by the authors has validated the torsion test as a method for generating residual shear stresses in a controlled and reproducible manner. In the present work, the evolution of residual stresses in a eutectoid wire rod (with pearlitic microstructure [2]) will be studied experimentally at different stages of the torsion test. For this purpose, unloading at different twist angles will be carried out and the residual stresses on the surface of the samples will be evaluated by X-ray diffraction (Figure 1). Preliminary results indicate that above a certain value of the twist angle the value of the residual stresses remains stable. This would allow the fabrication of standard samples with known tangential residual stresses in a reproducible way.





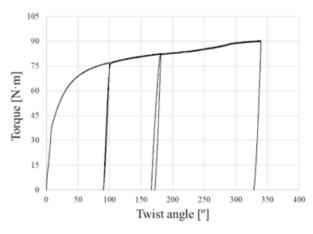


Figure 1. Torsion test in a steel sample (load-unload cycles at 100°, 180° and 340°).

Table 1. Residual stresses at the surface of the sample after load-unload cycle at 100° twist angle.

Residual stresses [MPa]	$ \begin{pmatrix} 3 & -227 & 17 \\ -227 & -8 & -8 \\ 17 & -8 & 0 \end{pmatrix} $
Uncertainty [MPa]	$\pm \begin{pmatrix} 15 & 13 & 3 \\ 13 & 15 & 3 \\ 3 & 3 & 11 \end{pmatrix}$



EVALUATION OF TENSILE LOAD STRESS AND X-RAY ELASTIC CONSTANT OF CRYSTALLINE RESIN MATERIAL BY X-RAY DIFFRACTOMETRY.

A. Nezu¹, T. Kikuchi¹, and Y. Akiniwa²

¹Rigaku Corp., 3-9-12 Matsubara-cho, Akishima 196-8666, Tokyo, Japan ²Yokohama National Univ., 79-5 Tokiwadai, Hodogaya-ku, Yokohama 240-8501, Kanagawa, Japan nezu@rigaku.co.jp, t-kiku@rigaku.co.jp, and akiniwa-yoshiaki-xd@ynu.ac.jp

The development of lightweight material is progressing in various fields, including the automobile industry, with the aim of saving energy and reducing environmental impact. Especially, synthetic resin is highly expected to be a lightweight material used for structural parts, thus, the evaluation of material strength is very important. Here, measurement results of tensile load test using X-ray diffractometry for crystalline resin materials are introduced.

Figure 1 shows the measurement results of (a) POM: polyacetal and (b) PPS: poly phenylene sulphide, obtained with chromium X-ray tube. Left one shows the diffraction peaks observed in each ψ -angle by $\sin^2 \psi$ method, middle one shows the line diagrams between the diffraction angle 2θ and the function of $\sin^2 \psi$ in each tensile load stress, and right one shows the line diagram between the slope of the 2θ - $\sin^2 \psi$ diagram M and the applied stress σ_{app} .

Looking at Fig. 1, it can be seen that the 2θ - $\sin^2 \psi$ diagrams in each tensile load stress intersect at around one point and that the M- σ_{app} diagram shows high linearity, for both POM and PPS resins. These results indicate that X-ray stress measurement for crystalline resin materials can be accurately performed.

In addition, X-ray elastic constant can be obtained from the slope of the M - σ_{app} diagram and calculated through equation (1), where E is Young's modulus, vi is Poisson's ratio, and θ_0 is Bragg angle under no strain. Table 1 shows

Table 1. X-ray elastic constants of POM unannealed and PPS annealed at 230 $^{\circ}$ C.

X-ray elastic constant	POM 1 0 0	PPS 2 0 0 + 1 1 1
$2/S_2 = E/(1+v)$ [MPa]	1,569	2,240

X-ray elastic constants of POM unannealed and PPS annealed at 230 °C.

In this poster presentation, the evaluation of the degrees of crystallinity and preferred orientation by X-ray diffractometry will be also reported, and a new tensile loading attachment, for a multipurpose X-ray diffractometer, is introduced.

- 1. M. Kakudo, *Japanese journal of polymer science and technology*, *Kobunshi*, **12**, No.140, (1963), pp.814-819.
- 2. T. Ito, Japanese journal of polymer science and technology, Kobunshi, 13, No.142, (1964), pp.30-37, 48.
- 3. K. Asai, Journal of the Society of Materials Science, Japan, 39, No.441, (1990), pp.782-789.
- K. Takasa, N. Miyashita, S. Tachibana and K. Takeda, Journal of the Society of Materials Science, Japan, 54, No.1, (2005), pp.51-55.
- K. Shimizu, K. Kameyama, M. Saito, K. Kimura, E. Kokuryu and K. Tanaka, *Journal of the Society of Materials Science, Japan*, 72, No.4, (2023), pp.324-331.
- K. Tanaka and Y. Akiniwa, Journal of the Society of Materials Science, Japan, 47, No.12, (1998), pp.1301-1307.

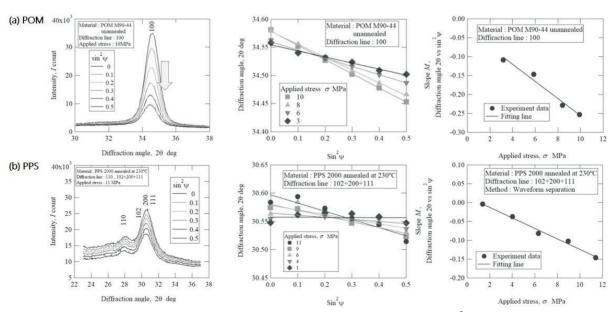


Figure 1. Measurement results of (a) POM and (b) PPS; Left is the diffraction peaks observed by $\sin^2 \psi$ method, middle is the $2\theta - \sin^2 \psi$ diagrams in each tensile load stress, and right is the M - σ_{app} diagram.



RESIDUAL STRESS OF EV BEARING COMPONENT WITH DIFFERENT OPERATING CONDITION

D. O. Kim^{1*}, Y. J. Choi¹, J. P. Kim¹, S. W. Park¹, S. H. Yoon², J.K Choi³, T.S Choi⁴

¹Korea Automotive Technology Institute, 303 Pungse-ro, Pungse-myeon, Cheonan, Chungnam, Republic of Korea

Bearing is the core component of motor and gear box in electric vehicles (EV). While the rotational speed of internal combustion engine is about 2000~7000rpm, the bearing of electric motor in EV need to endure over 15,000rpm [1]. In recent, the maximum rotational speed of EV motor has been increased up to 20,000rpm in order to improve driving performance and energy efficiency. [2] Since the residual stress affect the fatigue life of bearing and race, it is important to maintain the residual stress in specified value [3-5]. However, as the rotational speed increases in EV motor, the temperature of bearing may increase during operation. Thus, it can affect the residual stress on the surface of bearing. Therefore, in this research, the residual stress has been measured on ball bearing, inner and outer race of EV with different operating time and load, and compared with new one. The residual stress has been measured by x-ray stress measurement system by determination of linear slope determination of 2θ vs $\sin^2 x$. By rotating the goniometer, directional stresses of $\sigma_{0^\circ},\,\sigma_{45^\circ},\,\sigma_{90^\circ}$ have been measured and used to calculate the planar principal stresses and its axis. From the experiment, it is found that the compressive residual stress of ball bearing, inner and outer race after 170h operating time with radial direction load of 1,020kgf and axial direction load of 30kgf decreased by 10~20%. It is assumed that even though the temperature of oil bath which bearings were placed in is reached to 80°É, the surface of bearing becomes higher. Thus, by using this residual stress results, the temperature of bearing may be predicted conversely. Also, the X-ray elastic constant has been measured and compared with elastic modulus with different heat treatment condition, and it has been used to calculate residual stresses.

- 1. H. J. Stadtfeld, GEARTECHNOLOGY (2020).
- 2. S. Brauer, High speed electric vehicle transmission (2017).
- 3. T. Coors, F. Pape, G. Poll, Material Research Proceedings 6 (2018), 215-220.
- M. Šafář, J. Valíček, M. Harničárová, M. Šajgalík, H. Tozan, M. Kušnerová, M. Drbúl, M. Kadnár, A. Czán, Frontiers in Materials (2023), 1-17.
- W. Guo, Residual stress evaluation in bearing (2018), 22-23.

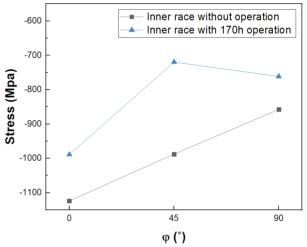


Figure 1. Residual stresses of inner-race bearing.

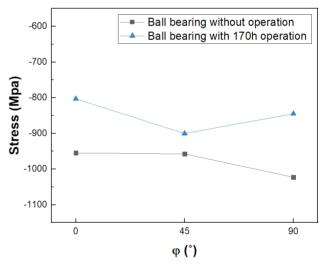


Figure 2. Residual stresses of ball bearing.

This work was supported by the Technology Innovation Program (No.20016117) funded by the Ministry of Trade, Industry and Energy, Republic of Korea.

Yeonhap System, 26 Hwanggeum-ro 323beon-gil, Gimpo-si, Gyeonggi-do, Republic of Korea
 ILJIN Global, 527 Samseong-ro, Gangnam-gu, Seoul, Republic of Korea
 Hyundai Steel, 1480 Songak-eup, Bukbusanup-ro, Dangjin, Chungnam, Republic of Korea dokim@katech.re.kr



LONG-TIME SURVEY OF LASER PROCESSED CALIBRATION SAMPLES FOR BARKHAUSEN NOISE METHOD

S. Santa-aho¹, A. Sorsa², M. Vippola¹

¹Material Science and Environmental Engineering, P.O.Box 589, 33104 Tampere University, Finland ²Environmental and Chemical Engineering, University of Oulu, P.O. Box 4300, 90014 University of Oulu, Finland suvi.santa-aho@tuni.fi

Barkhausen noise (BN) measurements are commonly utilised in industrial quality control after machining processes such as grinding. Different power transmission components are ground to meet the final tolerances. The BN method enables to locate grinding burns which might arise during grinding and compromise the quality of the manufactured component. The proper, accurate and verified operation of the BN equipment is valuable. One tool to verify the operation of the BN equipment is the use of calibration samples with artificial damages. Artificial thermal damages will simulate the excess heat generation in the grinding process, and they have been processed with different heating systems for example: induction and laser processing [1], temperature-controlled laser processing [2] and hydrogen-oxygen flame burn processing [3]. Nowadays, the laser systems seem to be the most popular method for creating these artificial grinding burns which have been found suitable for BN calibration purposes.

This study presents the results of long-term changes in residual stresses associated with the artificially laser-produced calibration samples. The two inspection methods are the magnetic Barkhausen noise and X-ray diffraction (XRD) based residual stress (RS) measurements. Two different sets of steel samples were measured with laser-processed thermal damages. Both materials were hardened steels, and the surface was normally ground prior the laser processing. The first set was laser processed with 4 kW Haas HL4006D laser and the detailed processing information can be found in [1]. The first set of samples were manufactured in 2010. This set was measured right after the manufacturing, 6 months and 12 months after the manufacturing and 14 years after the manufacturing. The second set of samples were manufactured with laser processing in 2022 with the similar type of temperature-controlled laser procedure as described in [3] and measured after the manufacturing and 2 years after the manufacturing.

Studies concerning the time dependence of the laser-processed sample BN measurements indicated that no major changes were observed in the BN root-mean-square (RMS) values during up to one year for the first set of samples. However, the RS measurements revealed some changes. The longer survey times are still under characterization and are to be reported during the conference. The issue of time-dependent changes should be considered when determining the optimal utilization time for calibration samples. Temperature changes could be one contributing factor for RS changes so keeping calibration blocks at constant temperature may be beneficial.

In addition, discussion is raised on the topics related to the calibration sample manufacturing. At the moment, there are no standards or guidelines for the calibration samples. The industries using the artificially produced calibration samples have their own internal guidelines and practices how to use them. The questions are how often the calibration samples should be changed and how to verify that the calibration sample needs to be replaced. Some issues are more related to the BN measurement system itself and others to the calibration samples.

- S. Santa-aho, M. Vippola, A. Sorsa, J. Latokartano, M. Lindgren, K. Leiviskä, T. Lepistö, *J Mat Proc Tech*, 212 (2012), pp. 293-298.
- 2. S. Santa-aho, A. Sorsa, J. Latokartano, K. Leiviskä, L. Suominen, M. Vippola, 11th International Conference on Barkhausen Noise Measurements and Micromagnetic Testing ICBM11 (2015).
- 3. S. Santa-aho, M. Deveci, S. Savolainen, M. Vippola, *Proceedings of the 12th ECNDT* (2018).

Support of TEKES for NOVEBARK research project is gratefully acknowledged. Authors would like to thank Mr. Jyrki Latokartano and Mr. Pekka Ala-Mäyry for laser sample preparation.



RESIDUAL STRESS MEASUREMENT OF TITANIUM WELDED BLADE BY NEUTRON AND SYNCHROTRON X-RAY DIFFRACTION TECHNIQUES

Shilei LI¹, Yang LI¹, Lunhua HE², Ke YANG³, Yandong WANG¹

¹University of Science and Technology Beijing, Beijing 100083, China ²Spallation Neutron Source Science Center, Dongguan 523803, China ³Shanghai Synchrotron Radiation Facility, Shanghai 201210, China lishilei@ustb.edu.cn

A systematic assessment of the three-dimensional residual stress distribution in welded, heat-treated, and machined titanium alloy blades was conducted using the General Purpose Powder Diffractometer at the China Spallation Neutron Source and BLS12W beamline Station of Shanghai Synchrotron Radiation Facility. During the welding process, the temperature of the blade weld area is significantly higher than that of the base body. During the subsequent cooling process, the weld seam shrinks. Due to the obstruction of the base material, a high residual tensile stress is formed, up to more than 760MPa. The residual stress decreases rapidly away from the weld, and the residual stress in the matrix area is very low. During the heat

treatment process, the metastable β phase undergoes phase transformation under the action of residual tensile stress and temperature, and the volume expansion of the α phase offsets the residual tensile stress. Measuring neutron diffraction stresses in titanium alloy blades poses several challenges, including weak diffraction signals and difficulties in positioning. Through preliminary literature research, microstructural analysis, optimization of measurement parameters, and meticulous data processing, better results for residual stresses can be obtained, showing good consistency with results obtained from other neutron sources.



Poster sessions - group 2 Mechanical Relaxation Methods, Additive Manufacturing, Welding

P2 - 1

EXPERIMENTAL VALIDATION OF A PLASTICITY CORRECTION PROCEDURE FOR HOLE-DRILLING RESIDUAL STRESS MEASUREMENTS

A. Benincasa¹, D. Halabuk²

¹SINT Technology srl, Via delle Calandre 63, 50041 Calenzano (FI), Italy
²Institute of Solid Mechanics, Mechatronics and Biomechanics, Brno University of Technology, Technická
2896/2, 616 69, Brno, Czech Republic
alessio.benincasa@sintechnology.com

The hole-drilling method, a widely used technique for measuring near-surface residual stresses, assumes linear elastic behaviour of the measured material [1]. However, when high residual stresses are present, local yielding can occur around the drilled hole due to stress concentration, leading to overestimated residual stresses in states with plastic deformation [2]. In cases where the residual stresses approach or exceed 80% of the material yield stress, measurement accuracy is significantly affected [3, 4].

In order to correctly evaluate high uniform residual stresses, a procedure for a correction of the plasticity effect was introduced [5]. The procedure relies on nearly 8 million simulated states with plastic deformation and is capable of correcting any combination of uniform residual stresses with magnitudes up to the material yield stress. It also covers a wide range of material parameters, hole diameters, and strain gauge rosettes and it is independent of the orientation of the strain gauge rosette.

The correction procedure has undergone extensive numerical testing, producing very promising results. However, validation through experiments is necessary before its practical application. Therefore, several measurements of

high residual stresses were conducted and the correction procedure was applied during the evaluation process. The obtained results from the experiments indicate that the procedure can effectively mitigate the plasticity effect, making it highly valuable for practical applications.

- G. S. Schajer, Practical residual stress measurement methods. Chichester: Wiley. 2013.
- M. Beghini, L. Bertini, P. Raffaelli, An Account of Plasticity in the Hole-Drilling Method of Residual Stress Measurement. *J. Strain Anal. Eng.*, 30, (1995), pp. 227–233.
- 3. J. Gibmeier, M. Kornmeier, B. Scholtes, Plastic Deformation during Application of the Hole-Drilling Method. *Mater. Sci. Forum*, **347-349**, (2000), pp. 131–137.
- Y. C. Lin, C. P. Chou, Error Induced by Local Yielding Around Hole in Hole Drilling Method for Measuring Residual Stress of Materials, *Mater. Sci. Tech.*, 11, (1995), pp. 600–604.
- 5. D. Halabuk, T. Navrat, Universal Procedure for Correction of Plasticity Effect in Hole-Drilling Uniform Residual Stress Measurement. *Exp Mech*, **62**, (2022), pp. 1267–1287.



P2 - 2

INFLUENCE OF TURNING AND DEEP ROLLING PROCESSES ON BEARING FATIGUE

S. Dechant¹, H. Nordmeyer², F. Pape¹, B. Breidenstein², B. Denkena², G. Poll¹

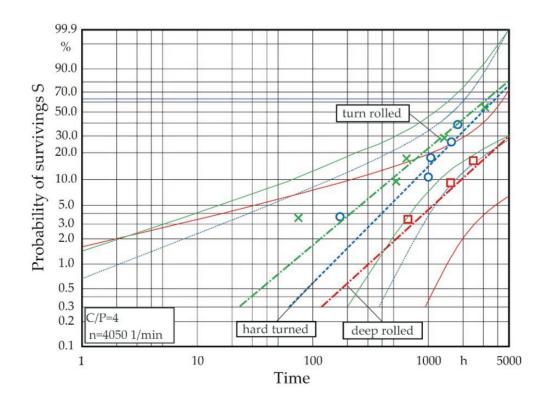
¹Leibniz Universität Hannover, Institute of Machine Design and Tribology, An der Universität 1, 30823 Garbsen, Germany

²Leibniz Universität Hannover, Institute of Production Engineering and Machine Tools, An der Universität 2, 30823 Garbsen, Germany dechant@imkt.uni-hannover.de

Due to the worldwide economic and ecological change, the application of innovative technologies that protect the environment and conserve resources is of importance. Particularly in manufacturing technology, there is potential for process optimization that is not yet fully understood or fully exploited. For example, inducing compressive residual stresses through manufacturing can enhance a component's (subsurface) fatigue strength. The residual stress depth profile can be specifically modified by the innovative hard turn-rolling process, in which the heat input generated by turning is used for a simultaneously performed deep rolling process. An application of the hard turn-rolling process to rolling-element bearings achieves longer bearing fatigue lives as determined by calculation and previous experiments. Furthermore, bench tests showed that, despite a very high maximum and a large penetration depth of the compressive residual stresses, the service life was not increased. A possible reason could be material effects caused by the heat input of the process, which is generated by dissipative processes and cannot be specifically controlled. In previous investigations, no holistic view of the

component subsurface area and its effects on component service life was carried out. It could be shown that deep rolling improves bearing fatigue life, while in case of the turn rolling process more work has to be done to achieve a beneficial influence on the bearing fatigue life.

For bearing fatigue life, the subsurface properties of bearings have a significant impact. The term subsurface refers to the volume area of the workpiece whose properties are influenced by a machining process [1]. The most important attributes of the subsurface are hardness, texture, microstructure, residual stresses, and defects like cracks or material faults. Studies have shown, that the functional component behavior under cyclic and quasi-static loading is significantly influenced by these subsurface properties [2-5]. According to Sollich, the wear resistance and service life of highly loaded components are significantly influenced by the residual stresses [6]. Thus, compressive residual stresses can lead to an increase in operational strength, as they counteract crack initiation and propagation. Tensile residual stresses, on the other hand, have a negative influence on component life [6-9]. Denkena et al. were able to





show that the service life of rolling bearings can be increased by the targeted induction of compressive residual stresses. However, it should be noted that the achievable increase in service life depends on the strength of the material and the specific residual stress state [10].

- B. Breidenstein, Oberflächen und Randzonen hoch belastbarer Bauteile, Habilitationsschrift, Tewiss, Leibniz Universität Hannover, 2011.
- E. Brinksmeier, J.T. Cammett, W, König P. Leskovar, J. Peters, H.K. Tönshoff, Residual stresses – measurement and causes in machining processes, CIRP Annals-Manufacturing Technology 31 (1982) 491-510.
- E. Brinksmeier, Prozeß- und Werkstückqualität in der Feinbearbeitung, Habilitationsschrift, Hannover, VDI Düsseldorf, 1991.
- K.H. Kloos, Über die Bedeutung der Oberflächen- und Randschichteigenschaften für die Bauteilhaltbarkeit, VDI -Berichte, 506 (1984) 1-14.

- D. Radaj, M. Vormwald, Ermüdungsfestigkeit Grundlagen für Ingenieure, Dordrecht, Springer, Berlin, 2007.
- A. Sollich, Verbesserung des Dauerschwingverhaltens hochfester Stähle durch gezielte Eigenspannungserzeugung, Fortschrittsberichte VDI, 5 (376), VDI-Verlag, Düsseldorf, 1994.
- 7. M. Habschied, B. de Graaff, A. Klumpp, V. Schulze, Fertigung und Eigenspannungen, HTM Journal of Heat Treatment and Materials 70(3) (2018) 111-121.
- 8. F. Klocke, W. König, Fertigungsverfahren 1 Drehen, Fräsen, Bohren, 8. Edition, Springer, Berlin, 2008.
- K. Röttger, G. Wilcke, S. Mader, Festwalzen eine Technologie für effizienten Leichtbau, Materialwissenschaft und Werkstofftechnik 36 (6) (2005) 270-274.
- B. Denkena, G. Poll, O. Maiß, T Neubauer, Affecting the Life Time of Roller Bearings by an Opti-mal Surface Integrity Design After Hard Turning and Deep Rolling, Advanced Material Research 967 (2014) 425 – 434.

P2 - 3

THE RESIDUAL STRESS DETERMINATION OF THE LABORATORY SPECIMEN BY SEMI-DESTRUCTIVE RELAXATION METHOD

P. Dlhý¹, J. Rakušan¹, O. Peter², R. Fajkoš², P. Hutař¹, M. Jambor¹

¹Institute of Physics of Materials of the Czech Academy of Sciences, v.v.i., Žižkova 513/22, Brno, Czech Republic ²BONATRANS GROUP a.s., Revoluční 1234, 735 94, Bohumín, Czech Republic dlhy@ipm.cz

The measurement of residual stress often involves methods that can either partially or entirely destroy the sample, which is problematic when preservation is necessary for further analysis. Various techniques, including destructive, semi-destructive, and non-destructive methods, are employed depending on the requirements and constraints of the study [1, 2]. The relaxation techniques associated with destructive methods are straightforward but result in the loss of the sample. Non-destructive methods, while preserving the sample completely, can be costly and sometimes impractical.

To address these challenges, the modified slotting method, a semi-destructive technique, offers a balanced solution. This method enables the determination of the residual stress profile across the entire specimen width while preserving enough material for subsequent analysis, such as fatigue crack growth rate testing.

The analysed samples are manufactured directly from the induction-hardened railway axle. The axial residual stress is mainly preserved after the sample is cut out. Due to the specimen geometry, the tangential residual stress is relaxed. As mentioned, the axial residual stress is predominant, leading to an assumption of a uniaxial stress state.

This contribution deals with residual stress measurement, which involves attaching strain gauges in a single direction, creating a slot, and measuring the relaxed strain. Numerical simulations are employed to accurately estimate the original residual stress field, making the modified slotting method effective for studies requiring detailed stress analysis and sample preservation.

- G. S. Schajer. Practical Residual Stress Measurement Methods. Chichester, UK: John Wiley & Sons, Ltd, 2013. ISBN 9781118402832. doi:10.1002/9781118402832.
- 2. F. A. Kandil, J. D. Lord, A. T. Fry and P. V. Grant. *A Review of Residual Stress Measurement Methods A Guide to Technique Selection*. 2001.

This research has been supported by grant No. FW03010149 "New wheel design for freight transport with higher utility properties" of The Technology Agency of the Czech Republic and the equipment and the base of research infrastructure IPMinfra were used during the research activities.



P2 - 4

HARNESSING RESIDUAL STRESSES AND MICROSTRUCTURE IN LASER POWDER BED FUSION BUILT 316L STAINLESS STEEL THROUGH HIGH-TEMPERATURE HEAT TREATMENT AND SEVERE SHOT PEENING

T. Gundgire¹, S. Santa-aho¹, T. Rautio², M. Vippola¹

¹Materials Science and Environmental Engineering, Faculty of Engineering and Natural Sciences, Tampere University, Tampere, Finland

²Kerttu Saalasti Institute, University of Oulu, Pajatie 5, 85500 Nivala, Finland tejas.gundgire@tuni.fi

Laser powder bed fusion (LPBF) stands out as a highly promising metal additive manufacturing technology, particularly valued for its capacity to fabricate intricate geometries using materials such as 316L austenitic stainless steel. However, the as-printed components exhibit significant tensile residual stresses and anisotropic microstructures which could lead to premature failure and thus reduced mechanical performance. Consequently, a typical approach involves conducting a solution annealing heat treatment at temperatures ranging from 1050 to 1100 °C. This process serves to eliminate the anisotropy by inducing recrystallization and mitigating the residual stresses. However, recent reports indicate that high-temperature solution annealing can diminish stress corrosion cracking resistance [1]. Thus, in this study, severe shot peening (SSP) was employed alongside solution annealing to enhance surface and subsurface properties, aiming to bolster stress corrosion cracking resistance and mechanical performance.

The as-printed LPBF 316L samples were subjected to solution annealing at 1100 °C. This solution-annealed specimen was then subjected to the air blast SSP. SSP refers to an intensive shot peening protocol with increased exposure time. The impact of these post-processing methods on residual stresses was assessed through X-ray dif-

fraction-based measurements on both the surface and along the depth. Results indicated that solution annealing effectively alleviated over 90% of residual stresses from the as-printed state. Subsequent SSP induced beneficial compressive residual stresses of more than - 700 MPa on the surface. Residual stress depth profiles revealed significant compressive stresses extending up to 300 µm from the surface. Moreover, SSP-induced work hardening in the near-surface region notably elevated surface hardness values. Additionally, the bombardment of shot-peening media contributed to a smoother surface, effectively reducing surface roughness by half compared to the initial measurements. All the aforementioned surface and subsurface modifications in residual stresses, hardness as well as surface roughness are recognized to enhance stress corrosion cracking resistance and mechanical performance [2]. These beneficial enhancements establish SSP as a dependable technique for altering surface and subsurface in LPBF-fabricated 316L stainless steel parts.

- Z. Que, T. Riipinen, S. Goel, A. Revuelta, T. Saario, K. Sipila, A. Toivonen, J. Corros. Sci., 214, (2023), 111022.
- D. Kumar, S. Idapalapati, W. Wang, S. Narasimalu, J. *Materials*, 12, (2019), issue 16, 2503.

P2 - 5

NUMERICAL STUDY OF THE DEEP-HOLE DRILLING METHOD

Bruno Levieil¹, Baptiste Lamothe¹, Pierre Faucheux¹, Cédric Doudard¹, Sylvain Moyne¹, Florent Bridier²

¹ENSTA Bretagne, IRDL—UMR CNRS 6027, 2 Rue François Verny, 29200 Brest, France²

²Naval Group, Bouguenais, France

bruno.levieil@ensta-bretagne.org

The development and shaping of structural components as well as their assembly generate significant residual stress fields in the structures. These stresses are superimposed on the in-service loads and influence fatigue durability and buckling. When these residual stresses are not known, implementing a conservative design approach will lead to oversizing which can be both costly and limiting in terms of performance, with a particular increase in the weight of the structures. Beyond optimising existing designs, using new materials and innovative manufacturing processes (additive manufacturing in particular) also requires charac-

terizing the residual constraints generated. Finally, the improvement of the knowledge of the internal stresses of components already in service can allow the extension of their service life.

Residual stress fields are by nature heterogeneous and most often multiaxial. Therefore, their knowledge requires the characterization at different points and in different directions in space. For this purpose, different techniques differ in the possible directions of measurement, the volume of measurement, and the depth of measurement.



The standardized and controlled methods are currently reserved for measurements near the surface. Few methods can be used to characterize the stresses in the core of thick and large structures. The contour method and the deep hole drilling. These methods rely on strain measurements during stress redistribution due to material removal.

This work focuses on the deep-hole drilling method and discusses two particular post-treatment hypotheses:

- The stress redistribution is supposed to occur without plastic deformation
- The strain measured at a location is solely due to the stress redistribution at this same location.

It is shown through a numerical study how these two assumptions can lead to stress extrema underestimation and how they compare quantitatively for different stress profiles.

P2 - 6

STRESS EVALUATION IN A CROSS-PLY GFRP-STEEL LAMINATE BY THE INCREMENTAL HOLE-DRILLING TECHNIQUE

J.P. Nobre¹, T.C. Smit², R.G. Reid², T. Wu³, T. Niendorf⁴

¹Univ Coimbra, CFisUC, Department of Physics, Coimbra, Portugal ²School of Mechanical, Industrial and Aeronautical Engineering, University of the Witwatersrand, Johannesburg, South Africa

³Fraunhofer Institute for Ceramic Technologies and Systems, Dresden, Germany ⁴Institute of Materials Engineering, University of Kassel, Moenchebergstr. 3, 34125 Kassel, Germany joao.nobre@dem.uc.pt

The incremental hole drilling technique (IHD) has shown its importance to evaluate through-thickness non-uniform residual stresses in composite laminates. The validation of the obtained results, however, is still an important issue [1]. Samples of a cross-ply GFRP-steel laminate were subjected to axial loading and, consequently, due the different mechanical properties of each layer, a pulse variation of stress at fibre-fibre and fibre-metal interfaces are generated. The determination of such steep stress discontinuity, which can be predicted by the Classical Lamination Theory (CLT) [2], is challenging to be experimentally determined by the incremental hole-drilling technique. Based on a differential method [3], it was possible to eliminate the initial residual stresses in the laminate and the strain relaxation curves due to the applied loading only could be determined. The experimentally measured strain-depth relaxation curves are then compared with those determined numerically using the finite element method (FEM) to simulate IHD. Both are used as input for the unit pulse integral method for stress determination by IHD [4-5]. Using CLT stress results as reference, despite the discrepancy observed at the deep fibre-metal interface, it was concluded that IHD can be further developed to accurately determine

the residual stress distribution through the composite plies of fibre metal laminates (FML) in particular, and fibre reinforced polymers in general, including near the interfaces.

- T.C. Smit, J.P. Nobre, R. Reid, T. Wu, T. Niendorf, D. Marais, A. Venter, *Exp. Mech.*, 62, (2022), 1289.
- 2. M.W. Hyer, *Stress analysis of fiber-reinforced composite materials*. Lancaster: DEStech Publications. 2009.
- 3. J.P. Nobre, J. Stiffel, A. Nau, J. Outeiro, A. Batista, W. Paepegem, B. Scholtes, *CIRP Annals*, 62(1), (2013), 87.
- 4. S. Akbari, F. Taheri-Behrooz, M. Shokrieh, Composites Science and Technology, 94, (2014), 8.
- 5. T.C. Smit, R. Reid, Exp. Mech., 64, (2024), 275.

This work received funding from German Research Foundation (DFG) under Grant Agreement No 399304816 and from the National Research Foundation of South Africa (NRF) under Grant Agreement No 106036. This work was also financed through national funds by FCT - Fundação para a Ciencia e Tecnologia, I.P. in the framework of the projects UIDB/04564/2020 and UIDP/04564/2020, with DOI identifiers 10.54499/UIDB/04564/2020 and 10.54499/UIDP/04564/2020, respectively.



P2 - 7

EFFECT OF SMAT ON MULTIAXIAL FATIGUE PROPERTIES OF A 7075 ALUMINIUM ALLOY

P. Gao, Z. Sun, D. Retraint*

LASMIS, University of Technology of Troyes (UTT), 12 rue Marie Curie, CS 42060, 10004 Troyes, France delphine.retraint@utt.fr

Surface Mechanical Attrition Treatment (SMAT) is a promising surface treatment technique for enhancing the mechanical properties of metallic materials. Thanks to high-frequency multidirectional impacts, SMAT can generate a nanostructured layer at the surface of a part, as well as a high compressive residual stress (CRS) profile and a work hardening gradient in the subsurface area [1]. It has been demonstrated that mechanical properties, especially fatigue strength, can be enhanced by SMAT for various materials under different loading conditions [2-6]. The aim of this work is to investigate the fatigue properties of a 7075 Aluminium Alloy (AA7075) treated by SMAT under combined tension-compression/torsion loading.

Dumbbell-shaped fatigue specimens are machined from raw bars. To improve the treatability/ductility of the precipitation-hardened material, a non-standard heat treatment used in our previous work [7] is applied to the specimens. SMAT is performed on the fatigue specimens after mechanical polishing, with various process parameters determining the treatment intensity. The residual stress profile is measured iteratively by X-ray diffraction (XRD), coupled with electrolytic polishing to remove material layer by layer. Multiaxial fatigue tests are conducted for non-treated specimens (mechanically polished: MP), and SMATed specimens under different loading conditions. It is noteworthy that several SMATed fatigue specimens are electrolytically polished (referred to as SMAT-EP) before fatigue test to remove a superficial layer of about 15~20 μm. Residual stress relaxation induced by fatigue loading is determined using XRD, and fractographic observation is

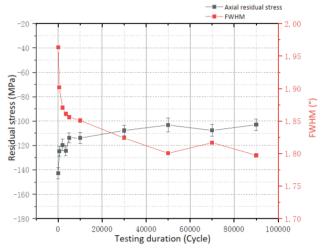
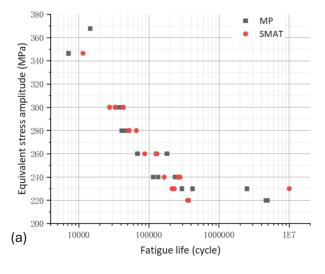


Figure 1. S-N plot for (a) MP and SMATed specimens, and (b) MP and SMAT-EP specimens.

carried out to analyse the effect of SMAT and the associated damage mechanisms.

The fatigue test results are presented in the form of S-N plots. A minimal difference can be seen between SMATed and untreated (MP: mechanically polished) specimens at relatively high load levels, and at low load levels the SMATed specimens show even shorter fatigue lives (see Fig. 1a). An intermittently interrupted test is conducted to measure and analyse the relaxation of residual stress during fatigue loading. The evolution of residual stress at the top surface is shown in Fig. 2, which shows that relaxation occurs mainly at the early stage of cyclic loading, followed by



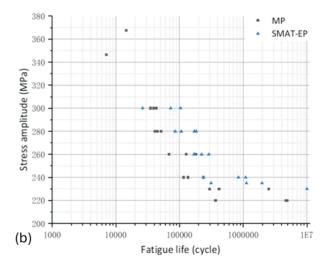


Figure 1. S-N plot for (a) MP and SMATed specimens, and (b) MP and SMAT-EP specimens.



a stabilisation period during which no apparent relaxation occurs. Approximately 40% of the initial residual stress is released after cyclic loading. As for the SMAT-EP specimens, they exhibit much longer fatigue life at all load levels, compared to the MP specimens (Fig. 1b).

Due to the comparable surface roughness of the MP and SMAT-EP specimens, it is reasonable to conclude that the CRS layer induced by SMAT, which is not fully relaxed during fatigue, plays a dominant role in increasing the fatigue strength of this material.

- Z. Sun, J. Zhou, D. Retraint, *Mater. Trans.*, **64**, (2023), 1739.
- T. Roland, D. Retraint, K. Lu, J. Lu, Scr. Mater., 54, (2006), 1949.

- D. Li, H. N. Chen, H. Xu, Appl. Surf. Sci., 255, (2009), 3811.
- 4. C. Dureau, M. Arzaghi, R. Massion, Y. Nadot, T. Grosdidier, *Mater. Sci. Eng. A*, **840**, (2022), 142916.
- T. Gao, Z. Sun, H. Xue, D. Retraint, *Int. J. Fatigue*, 139, (2020), 105798.
- L. Brasileiro, Z. Sun, C. Mabru, R. Chieragatti, G. Proust, D. Retraint, *Procedia Struct. Integr.*, 38, (2022), 283.
- 7. Y. Li, D. Retraint, P. Gao, H. Xue, T. Gao, Z. Sun, *Metals* (Basel)., **12**, (2022), 785.

P2 - 8

DEVELOPMENT OF A NUMERICAL APPROACH FOR THE ASSEMENT OF ACCURACY OF RELAXATION METHODS AND APPLICATION TOWARDS A RING GEOMETRY.

H. Sankaran¹, F. Cazes¹, B. Bacroix¹, C. Jeannot², C. Lebreton².

¹LSPM – CNRS, 99 Av. Jean Baptiste Clément, Villetaneuse, 93430, France ²ADR-ALCEN, 12 Chem. des Prés, 77810 Thomery harihara.sankaran@Jspm.cnrs.fr

Relaxation methods like hole drilling method, contour methods, sectioning methods, etc. for measuring residual stress are widely used in the industry due to its apdaptability and versatility [1]. The theory behind each of the methods arise from 2 main concepts, namely, Eigenstrains [3] and Buckner's super position principle [2,4]. However, all the methodologies contain a set presuppositions. Thus making the results obtained from the methods an approximation. A full numerical approach for relaxation method is developed to estimate the error associated with the methods, independent of experimental errors.

Relaxation methods can be decomposed into 3 basic stages: Equilibrium state of reference denoted as *stage 0*, modified stress state after material removal and relaxation referenced as *stage 1* and finally reconstructed stress state through numerical simulation referenced as *stage 2*. All three stages are simulated in this contribution using FEniCS in order assess the accuracy of the methods. This is

done by comparing the stress at stage 0 considered as reference and the stress calculated using the relaxation method based on the superposition of stages 1 and 2. Error plots will be show for applications based on ring geometries.

A starting point for the application of the proposed method is an assumption of the eigenstrain components as a function of the depth in the material. In this work, we obtain this eigenstrain profile through a simplified approach of machining simulation in the fashion of [5]. Initially a equivalent thermo mechanical simulation that simulates turning is performed using salome-meca and code_aster and the non-elastic strains or eigenstrains are obtained and a spline interpolation is used to fit the point data.

Finally the proposed apprach is composed of two parts:

1. First, a numerical simulation of the process is performed to estimate the components of the eignenstrain as a function of the depth in the material.

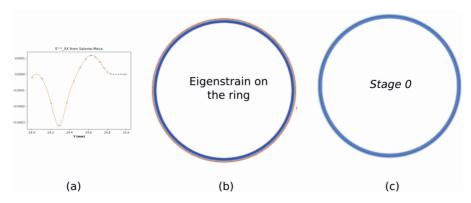


Figure 1. Methodology to obtain stage 0.



2. Second, the three stages of a relaxation method are simulated for a given geometry with the obtained eignestrain and an estimation of the error is obtained by comparing the residual stress at stage 0 with the one reconstitued based on stages 1 and 2.

As an illustration of this approch, Fig. 1a shows the eigenstrain obtained from process simulation and Fig. 1b and Fig. 1c show the eigens strain in the form of stresses to the ring geometry and the resultant residual stress respectively. The proposed approach provides a fully numerical framework towads improving the results obtained using sectioning method and also helps us understand the relation between eigenstrains and resulting residual stresses for a given geometry and the relaxation method in use.

Our objective is to prepare the estimation of residual stresses in more complicated situations such as thin ring for which a multiple sectionning will be performed in order to estimate the residual stresses as in [2]. In this approach, the first cut will be used to analyse the linear components of the stresses throughout the thickness of the ring, and the following cuts will provide the stress fluctuations through the

thickness of the ring considering that ring to be in the form of bernoulli beam.

- L. Margilies, M. J. Kramer, R. W. McCallum, S. Kycia, D. R. Haeffner, J. C. Lang, A. I. Goldman, Rev. Sci. Instrum., 70, (1999), 3554.
- 2. Daulton D. Isaac, Michael B. Prime, and Nagaraj Arakere. Residual stress measurement of full-scale jet-engine bearing elements using the contour method. Volume 9, pages 69–81, Cham, 2017. Springer International Publishing.
- Toshio Mura. Micromechanics of defects in solids. Springer Netherlands, 1987.
- 4. Michael Prime. Cross-sectional mapping of residual stresses by measuring the surface contour after a cut. Journal of Engineering Materials and Technology, 123:162–168, 05 2001.
- Alexandre Mondelin, Frederic Valiorgue, Joël Rech, Michel Coret, and Eric Feulvarch. Hybrid model for the prediction of residual stresses induced by 15-5ph steel turning. International Journal of Mechanical Sciences, 58(1):69–85, 2012.35.

P2 - 9

OPTIMIZATION OF 3D PRINTING PARAMETERS TO MINIMIZE RESIDUAL STRESSES IN MARAGING STEEL

K. Trojan¹, I. Zetková², J. Čapek¹, N. Ganev¹, Š. Houdková³

¹Faculty of Nuclear Sciences and Physical Engineering, Czech Technical University in Prague, Trojanova 13, 120 00 Prague, Czech Republic

²Regional Technological Institute, Faculty of Mechanical Engineering, University of West Bohemia, Universitní 8, 306 14 Pilsen, Czech Republic

³Výzkumný a zkušební ústav Plzeň s.r.o., Tylova 1581/46, 301 00 Pilsen, Czech Republic karel.trojan@fjfi.cvut.cz

Fatigue crack initiation and propagation play an important role in fatigue properties, where they are shown to be strongly associated with surface roughness, microstructure parameters (dislocation density, crystallite size, microcracks) and last but not least macroscopic residual stresses [1]. During additive manufacturing using the selective laser melting technology, a complex residual stress distribution is created that can significantly affect the printing itself and also the mechanical properties of the final product. The magnitude of these stresses may even approach the yield strength of the material [2]. Thus, research has been carried out to optimize 3D printing parameters to minimize residual stresses in MS1 maraging steel.

It was found that the preheating temperature of the build platform significantly affects both the residual stresses and microstructure parameters as well as the mechanical properties. The results of macroscopic residual stresses obtained from the centre of the cylindrical test samples are shown in Fig. 1. For both samples, the tensile residual stresses near the surface still dominate, but a rapid decrease in residual stresses can be observed for the sample with platform preheating to 120 °C, while only compressive residual stresses have been described below the surface.

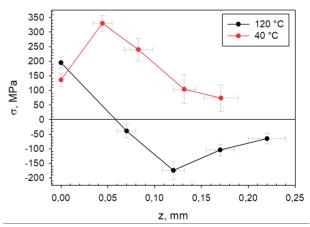


Figure 1. Depth profile of macroscopic residual stresses in the axial direction for samples with different building platform preheating

We are still working on the problem, we are looking for what other printing parameters affect the residual stress state, and we will present further advances in the talk. Further, depth profile of macroscopic residual stresses obtained using X-ray diffraction and gradual electrolytic



removal of surface layers will be complemented by the results of the drilling method and the mechanical properties will be compared.

 I. Černý, J. Čapek, J. Kec, K. Trojan, N. Ganev, S. Němeček in *Comprehensive Structural Integrity*, edited by F. Aliabadi & W. Soboyejo, (Elsevier Science), 2023, pp. 391-415. 2. J. L. Bartlett & X. Li, Addit. Manuf., 27, (2019), 131-149.

The work was supported by Technology Agency of the Czech Republic "Development of "3D print-thermal spray" systems for dynamically and cyclically loaded applications", grant No. TH75020003. The work of CTU staff was supported by the Grant Agency of the Czech Technical University in Prague, grant No. SGS22/183/OHK4/3T/14.

P2 - 10

RESIDUAL STRESS ANALYSIS ON A DED-ARC ADDITIVE MANUFACTURED HIGH-STRENGTH STEEL COMPONENT USING THE CONTOUR METHOD

G.A. Shabdali¹, K. Wandtke*¹, L. Engelking¹, A. Kromm¹, D. Schroepfer¹, T. Kannengiesser¹, R. Scharf-Wildenhain², A. Haelsig², J. Hensel²

¹Bundesanstalt für Materialforschung und -prüfung, BAM, 12205 Berlin ²Technische Universität Chemnitz, Professur Schweißtechnik, 09126 Chemnitz *karsten.wandtke@bam.de

Direct Energy Deposition with arc (DED-arc) or wire arc additive manufacturing (WAAM) has significantly transformed the manufacturing paradigm in recent years by the virtue of its capability to fabricate intricate, large scale metallic parts owing to high deposition rates, high efficiency, and cost effectiveness. Subsequent enhancement in efficiency can be achieved through the utilization of the high-strength structural steels. The fabrication of the intricate geometries possesses challenges in regulating the residual stresses (RS), representing a significant concern in the realm of additive manufacturing (AM). High residual stresses contribute to an increased risk of cold cracking particularly in the welding of the high strength steels arising from complex interactions among the material, process conditions and component design. Reliable residual stress evaluation is vital in the structural integrity assessment of the welded components. Therefore, in the present study, the contour method was used to analyse the full field longitudinal residual stresses in an open hollow cuboid specimen fabricated by DED-arc. In this method, the specimen is cut along a desired plane of interest and the deformation caused by the cut surface is measured using the coordinate measuring machine and an industrial non-contact 3D scanner. A different cutting and restraint methodology was adopted and its influence on the residual stresses was analysed. The results indicate that the maximum tensile residual stresses around 600 MPa occurred in the left wall of the DED-arc structure exactly two layers below from the top. Additionally, the stresses at the bottom layer of the base plate demonstrate tensile in longitudinal direction and the corresponding balancing compressive residual stresses occurred at the top layer of the base plate. The contour approach is efficient and precise way for generating a two-dimensional residual stress map. The results obtained from the contour method was further validated using the X-ray Diffraction and both sets of findings demonstrated similarity.



P2 - 11

A NOVEL CUTTING METHOD FOR CONTOUR METHOD MESUREMENTS - A PROOF OF PRINCIPLE

Jeferson Araujo de Oliveira

StressMap, The Open University, United Kingdom; jeferson.oliveira@open.ac.uk

Residual stresses are a key to understanding how the manufacturing processes influence the structural integrity of safety-critical mechanical components. There are several residual stress measurement techniques available, among which, the contour method stands out as capable of generating a cross-sectional map of the residual stresses after introducing a cut into the test components of interest. This cut has a very particular set of requirements, such as: already cut surfaces cannot be re-cut and the cut width must be uniform throughout the cross-section. Because of these stringent requirements, to date, only electro-discharge machining (EDM) has been successfully used to map residual stresses across a test sample. Attempts to use other tech-

niques, such as waterjet or laser cutting have failed to produce cut surfaces with high enough quality.

In this work, I present the evaluation of the use of abrasive diamond wire cutting for contour measurements. Identical aluminium alloy specimens were cut using wEDM and abrasive diamond wire cutting and then analysed using the contour measurement to assess and compare the 2-D maps of cross-sectional residual stresses from the two cutting techniques. Results show a good agreement between the two cutting methods, suggesting that abrasive diamond wire cutting is a suitable substitute for wEDM cutting in the contour method in certain applications. This breakthrough enables the application of the contour method to non-conductive materials, which was previously not possible.

P2 - 12

HIGH AND CONVENTIONAL STRAIN RATE MECHANICAL RESPONSE OF TITANIUM SUBJECTED TO SEVERE PLASTIC DEFORMATION

M. Saferna¹, L. Farbaniec¹, A. Baczmański¹, K.L. Wong², M.A. Lopez², D.E. Eakins², K. Wierzbanowski¹

¹Fac of Physics and Appl. Computer Science, AGH Univ. of Kraków, Mickiewicza 30, 30-059 Kraków, Poland ²Department of Engineering Science, University of Oxford, Parks Road, Oxford OX1 3PJ, UK Andrzej.Baczmanski@fis.agh.edu.pl

Mechanical properties of polycrystalline Titanium (grade 2), which was previously subjected to multi-stage hydrostatic extrusion technique (one of the methods of Severe Plastic Deformation), were studied. The main research goal was to understand the influence of strain rate on the compressive behaviour of the material, as well as to compare its characteristics obtained in the conventional tensile and compression tests. In the case of high rate-induced strain deformation under dynamic compression, a strong accumulation of residual stresses was created, which in turn plays an important role in the resultant mechanical characteristics. In the course of the research, the plastic yield points, material strengthening, maximum strength and the role of residual stress accumulation was analysed in detail. The agreement of the material parameters with the Hall-Petch law was also found. Finally, significant differences in the yield points were observed between tension and compression tests, especially in the case of dynamic compression – Fig. 1. The maximum strength of the material in the tensile tests showed a noticeable increase as a function of cumulative strain.

In summary, the presented work provides new insights to the understanding of the mechanical properties of Titanium grade 2 after Severe Plastic Deformation, considering the role of accumulated residual stress. Suggestions for fur-

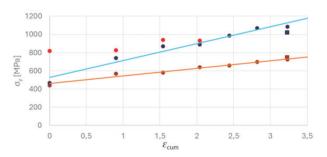


Figure 1. Example comparison of yield thresholds as a function of cumulative strain for conventional tension and compression tests; points corresponding to dynamic compression were also

marked (•compression, •tension, •dynamic compression).

ther research include the analysis of microstructural features, such as grain structure, texture, morphology, and size using transmission and scanning microscopy, which can expand our understanding of this material.

This work was partly financed by grant from the National Science Centre, Poland (NCN), No. UMO-2023/49/B /ST11/00774 and supported by the program "Excellence initiative – research university" for the AGH University of Science and Technology.



P2 - 13

IMPACT OF HYDROGEN-INDUCED EFFECTS AND RESIDUAL STRESS ON THE PROPERTIES OF 34CRMO4 IN SLOW STRAIN RATE TEST

A. Bolender¹, P. Zeisberg², O. Maißł, A. Liehr¹, T. Niendorf¹

¹Institute of Materials Engineering (University of Kassel), Mönchebergstr. 3, 34125 Kassel, Germany ²sera Hydrogen GmbH, sera-Straße 1, 34376 Immenhausen, Germany ³ECOROLL AG Werkzeugtechnik, Hans-Heinrich-Warnke Str. 8, 29227 Celle, Germany

As an energy carrier of the future [1], green hydrogen also requires appropriate conditioning of the materials in the relevant component surface layers. [2] In 2018, the project partner sera Hydrogen proposed a new technology for clean piston compressors based on dry-running reciprocating systems. High-pressure compression of hydrogen is used in filling stations for hydrogen-powered vehicles. The result was a prototype design that has already been successfully tested in initial laboratory and field trials. The primary aim of this study is the analysis of microstructures in the surface layer of the gas cylinder having a significant effect on lifetime. The hydrogen environment embrittlement can be supported by tensile stress or residual tensile stress in the entire component or in characteristic areas. [3] Therefore, these effects should be minimized by superposition with residual compressive stresses. Most relevant effects have been studied by numerous research groups from around the world. Most of these studies aimed at preventing or reducing a hydrogen contamination. In our research project, we focused on the challenge of finding a suitable

iron-based alloy together with a surface condition in order to make it more resistant to hydrogen-induced embrittlement. Special tensile test under hydrogen load like in [4] and depth resolved residual stress measurements were the main tool for characterizing the cylinder surface.

- HYDROGEN Roadmap Europe: A Sustainable Pathway for the European Energy Transition, Publications Office of the European Union, Luxembourg, 2019.
- American Society of Mechanical Engineers, 2019 ASME boiler & pressure vessel code: An international code, 2019th ed., American Society of Mechanical Engineers, New York, N.Y., 2019.
- 3. J. Woodtli, R. Kieselbach, Damage due to hydrogen embrittlement and stress corrosion cracking, Engineering Failure Analysis 7 (2000) 427–450.
- Toshio Ogata (Ed.), Simple Mechanical Testing Method to Evaluate Influence of High Pressure Hydrogen Gas, American Society of Mechanical Engineers, 2018.



Presentations of exhibitors/sponsors

EL - 1

MRX PRODUCTS FOR RESIDUAL STRESS EVALUATION THROUGH X-RAY DIFFRACTION

A. Sprauel

MRX Mesures Rayons X, 6 rue de Dingolfing, Brumath, France adrien@mrxrays.com

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 X-Raybot 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.

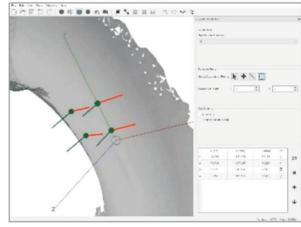


Figure 2. Measurement points definition in AdaptiveXRD



EL - 2

ANTON PAAR - X-RAY ANALYSIS SOLUTIONS

Benedikt Schrode

Anton Paar GmbH, Austria; benedikt.schrode@anton-paar.com

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

Bruker AXS, Germany kurt.erlacher@bruker.com

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

Rigaku Europe SE, Germany tom.faske@rigaku.com

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 re-

nowned 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

PROTO Manufacturing Europe Spzoo, Poland awojtas@protoxrd.com

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 $\sin^2\!\psi$ method in omega geometry of an X-ray diffractometer.

The $\sin^2 \psi$ 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 $\psi = 0$ 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 with-

out removing one tooth hence making the procedure destructive. In the proposed method the d_0 value is obtained also at $\beta=0$ but in a location only slightly moved towards the tooth head – accessible at $\beta=0$. 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 $\sin^2 \psi$ 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 $\sin^2 \psi$ plot will also be indicative of the correctness of the selected d_θ value.

EL - 6

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

Mikko Palosaari¹, Sebastian Send², Carlo Scheer²

¹Stresstech Oy, Finland ²Stresstech GmbH, Germany mikko.palosaari@stresstech.com

In this presentation Stresstech as a company is introduced. The company's 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

VEQTER Ltd., 8 Unicorn Business Park, Whitby Road, Bristol, BS4 4EX, UK ed.kingston@veqter.co.uk

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 in-

dustries 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.



EL -8

THREADING DISLOCATION'S STRAIN FIELDS VISUALIZED AND CLASSIFIED IN SCANNING ELECTRON MICROSCOPE (SEM)

M. Čalkovský, E. Baťková, S. Čagala Hu, J. Dolenský, L. Strakoš, T. Vystavěl

Thermo Fisher Scientific, Vlastimila Pecha 12, Brno 627 00, Czech Republic martin.calkovsky@thermofisher.com

Crystalline defects metrology plays a critical role in the power segment of the semiconductor industry, from the lab level to the fabrication level. In particular, the presence of strain fields around threading dislocations (TDs) in GaN has been found to have negative effects on the performance of (opto)electronic devices. TDs are crystal lattice defects that induce strain in the crystal and act as centers of non-radiative recombination, leading to current leakage and a decrease in device effectiveness and lifetime. Recognizing the significance of these concerns, there is a need within the semiconductor industry to quickly and comprehensively investigate the presence and type of TDs on a large scale (100 µm²). To address this need, we offer a unique solution: a fully automated, non-destructive, high-throughput, and quantitative crystalline defect metrology system for GaN.

Our method is based on Electron Channelling Contrast Imaging (ECCI) technique [1], which is performed in a scanning electron microscope (SEM). In SEM an electron beam scans surface of a crystalline specimen and as a result of the beam-specimen interaction, strain fields around TDs can be visualized on backscatter-electron (BSE) images under specific crystal orientations. The experimental setup is schematically described in Figure 1. Electron channelling refers to the diffraction of the electron beam on its way in the specimen and enables visualisation of the crystal defects as they change the phase of the incident electron wave function. As a result, the backscattering probability changes and yields a contrast variation in the BSE image around TDs. Figure 2a depicts a typical ECCI image, where the visible dots represent TDs and the strain fields around TDs are expressed by characteristic black-white

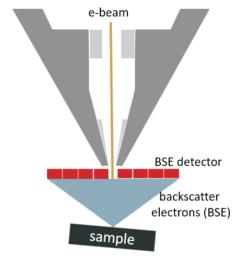


Figure 1. Schematics of Electron Channelling Contrast Imaging (ECCI) experimental setup performed in a SEM. Under specific specimen orientation the strain fields around threading dislocations are visualized by detection the backscattered electron using a BSE detector.

contrast variation. Moreover, SEM enables the setup of a precise sample orientation using the electron channelling pattern as shown in Figure 2b. Classification of edge, screw, and mixed types the TDs requires acquisition of at least two ECCI datasets, each acquired in different diffraction conditions, marked in Figure 2b as g1 and g2. Figures 2a and 2c show the same defects imaged at the two orientations. By analyzing the contrast around TDs, it is possible

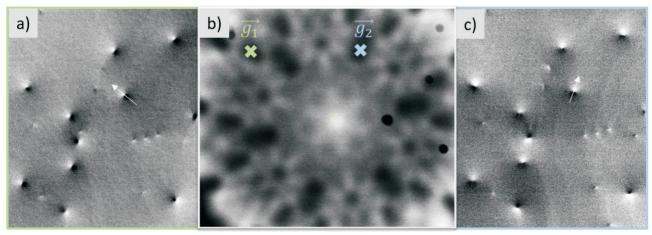


Figure 1. a) 15 keV electron channelling contrast image (ECCI) from a GaN specimen revealing threading dislocations, i.e. spots with black-white contrast. b) The specimen orientation is revealed from the electron channelling pattern (ECP)m where two diffraction conditions are identified (g1 & g2). Same defects are imaged a) and c) under these two diffraction conditions, showing contrast change that enable defect classification.



the semiconductor industry to quickly and comprehensively investigate the presence and type of TDs on a large scale ($100 \, \mu m^2$). To address this need, we offer a unique solution: a fully automated, non-destructive, high-throughput, and quantitative crystalline defect metrology system for GaN.

Our method is based on Electron Channelling Contrast Imaging (ECCI) technique [1], which is performed in a scanning electron microscope (SEM). In SEM an electron beam scans surface of a crystalline specimen and as a result of the beam-specimen interaction, strain fields around TDs can be visualized on backscatter-electron (BSE) images under specific crystal orientations. The experimental setup is schematically described in Figure 1. Electron channelling refers to the diffraction of the electron beam on its way in the specimen and enables visualisation of the crystal defects as they change the phase of the incident electron wave function. As a result, the backscattering probability changes and yields a contrast variation in the BSE image around TDs. Figure 2a depicts a typical ECCI image, where the visible dots represent TDs and the strain fields around TDs are expressed by characteristic black-white contrast variation. Moreover, SEM enables the setup of a precise sample orientation using the electron channelling pattern as shown in Figure 2b. Classification of edge, screw, and mixed types the TDs requires acquisition of at least two ECCI datasets, each acquired in different diffraction conditions, marked in Figure 2b as g1 and g2. Figures 2a and 2c show the same defects imaged at the two orientations. By analyzing the contrast around TDs, it is possible to classify the defects. The identification, counting, and quantification of TDs are performed using AI methods [2], ensuring a fully automated process. Further, the implementation of AI denoising techniques to the ECCI data enables faster data acquisition and enhances throughput compared to the current defect metrology techniques used in the semiconductor industry. With the new development in electron detection technology, particularly in pixelated semiconductor detectors [3], it is alternatively possible to visualize and quantify defect's strain fields from EBSD patterns on large semiconductor wafers. This opens a new opportunity for strain analysis in SEM.

- G. Naresh-Kumar, B. Hourahine, P.R. Edwards, A.P. Day, A. Winkelmann, A.J. Wilkinson, P.J. Parbrook, G. England, and C. Trager-Cowan: *Rapid Nondestructive Analysis of Threading Dislocations in Wurtzite Materials Using the Scanning Electron Microscope*. Phys. Rev. Lett. 108 (2021) 135503.
- L. Strakos, O. Machek, T.Vystavel, A. Schulze, H. Han, M. Caymax, R. Young: Electron Channeling Contrast Imaging for Beyond Silicon Materials Characterization. Proceedings of the ISTFA 2018, (pp. 363-367). ASM. https://doi.org/10.31399/asm.cp.istfa2018p0363
- T.Vystavěl, P. Stejskal, M. Unčovský, Ch. Stephens, Tilt-free EBSD, Microscopy and Microanalysis, Volume 24, Issue S1, 1 August 2018, Pages 1126–1127, https://doi.org/10.1017/S1431927618006116

The ALL2GaN Project (Grant Agreement No. 101111890) is supported by the Chips Joint Undertaking and its members including the top-up funding by Austria, Belgium, Czech Republic, Denmark, Germany, Greece, Netherlands, Norway, Slovakia, Spain, Sweden and Switzerland.

Views and opinions expressed are however those of the author(s) only and do not necessarily reflect those of the European Union, Chips JU or the national granting authorities. Neither the European Union nor the granting authorities can be held responsible for them.