

Measurement of residual stress by diffraction techniques

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Abstract. Residual stresses present themselves as a two axed sword in material science applications. Compressive residual stresses offer beneficial conditions in applications where fatigue performance is required with it being able to mitigate crack initiation and propagation, whilst tensile residual stresses are generally considered to be detrimental. Non-destructive assessment of these stresses has become an essential requirement in high performance components where residual stress tailoring can render substantial improvements in the optimisation of component design. Strain scanning with diffraction techniques enables accurate investigation of the depth dependence by direct probing of the lattice plane spacing of the material microstructure. Complementary diffraction techniques are discussed and their application illustrated with results from a number of studies.

1. Introduction

The total stress that a component experiences in practical use is the vector sum of the applied and residual stresses. The applied stresses result from the loading forces in use and can be calculated to high precision. Residual stresses on the other hand result from the processing and manufacturing treatments of the material and are mostly only approximated qualitatively. Residual stresses in solids are defined as those stresses that are present in the material without it being subjected to an external force. They can be as high as the in situ yield stress of the material, and are subject to an internal force balance over the volume of the part. Macroscopic internal stresses in polycrystalline materials, which are homogenous over a large number of crystalline grains (micrometer to millimeter size), lead to local strains and stresses.

Diffraction based techniques enable accurate non-destructive measurement of the prevailing residual strain tensors from which the stress fields can be calculated in conjunction with the material elastic moduli [1-3]. Diffraction refers to the constructive interference of short wavelength radiation from the microstructure of a crystalline material as governed by the Bragg law $n\lambda = 2d^{hkl} \sin \theta^{hkl}$ where λ is the wavelength of the probing radiation, d^{hkl} is the interatomic spacing, θ^{hkl} the specular angle of diffraction with reference to the crystallographic lattice planes hkl . A limiting condition for diffraction to take place from crystal lattice planes is $\lambda \leq 2d^{hkl}$, thus requiring wavelengths 1-3 Å in magnitude. Such wavelengths are offered by X-rays and thermal neutrons. X-rays are generated as low energy monochromatic beams from target tubes such as Cu, Co, Cr, etc. Each target tube has its own characteristic wavelength (energies smaller than 8 keV). Higher energy X-rays, 15-200 keV, are generated in synchrotron light sources. Thermal neutrons are generated in steady state research reactors or spallation sources. From a residual strain measurement perspective, each of these radiation

types present different penetration depths that enables depth selective non-destructive investigation. The penetration depths are governed by their fundamental interactions with matter. X-rays, having an interaction with the electron cloud of the material, have penetration depths that are proportional to the atomic number. In typical engineering materials the penetration depths are limited to 10 μm , i.e. presenting a near surface probe. Synchrotron X-rays are substantially more penetrating due to their higher energies. As an example 2 mm penetration into mild steel is achievable for energies larger than 60 keV. Thermal neutrons present a probe that is generally 1000 times more penetrating than normal X-rays for the transmission metal element series. Typically 40 mm steel can be penetrated. By defining a gauge volume with beam apertures, depth resolved investigations can be performed by systematic translation of the sample in a step-wise manner through the gauge volume.

Strain scanners are special derivatives of powder diffractometers that are devoted to the three-dimensional mapping of internal strains, by very precise measurement of the local lattice plane spacing deviations $\Delta d^{hkl} / d^{hkl}$ induced by these strains and stresses:

$$\Delta d^{hkl} / d^{hkl} = (\Delta\lambda / \lambda) - \cot\theta\Delta\theta$$

Constant wavelength strain scanners correspond to $\Delta\lambda = 0$ and pulsed instruments to $\Delta\theta = 0$. The strains determined in the laboratory coordinate system is related to the unknown strain components in the sample coordinate system using the transformation relation $\varepsilon'_{ij} = \alpha_{ik}\alpha_{jl}\varepsilon_{kl}$ where α_{ij} are the direction cosines between the coordinate systems. By orientating the specimen such that the diffraction vector is along the X'_3 coordinate axis in figure 1 the strain $\varepsilon_{\phi\phi}$ in that direction can be determined

$$\begin{aligned} \varepsilon_{\phi\phi} = \frac{d_{\phi\phi} - d_0}{d_0} &= \varepsilon_{11} \cos^2 \phi \sin^2 \varphi + \varepsilon_{22} \sin^2 \phi \sin^2 \varphi + \varepsilon_{33} \cos^2 \varphi + \varepsilon_{12} \sin 2\phi \sin^2 \varphi \\ &+ \varepsilon_{13} \cos\phi \sin 2\varphi + \varepsilon_{23} \sin\phi \sin 2\varphi \end{aligned}$$

This equation relates the quantities measurable with diffraction, $d_{\phi\phi}$ and d_0 (strain-free lattice parameter), to the unknown components of strain ε_{ij} in the material. Having determined the strain tensor, the stress tensor is obtained from Hooke's law

$$\sigma_{ij} = \frac{1}{1/2S_2} \left[\varepsilon_{ij} - \delta_{ij} \frac{S_1}{1/2S_2 + 3S_1} \varepsilon_{ii} \right]$$

where S_1 and $1/2S_2$ are the diffraction elastic constants.

Strain scanning with diffraction techniques is widely applied in materials characterization studies. This stems from the solid fundamentals in technique benchmarking employed and continuous expansion programs [4,5]. Diffraction furthermore enables investigation of different chemical phases in multi-phased materials. Examples from investigations that have been performed using X-rays, synchrotron light and neutrons are presented to illustrate potential applications. The investigations have been done at the Necsfa facilities, supplemented with investigations at leading international facilities. The neutron strain scanner at Necsfa is currently upgraded to offer capabilities of international standard to the South African research community.

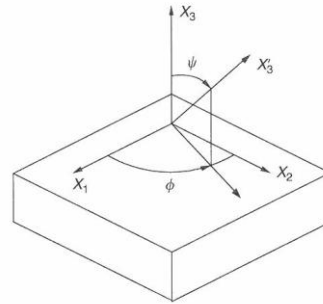


Figure 1: Coordinate systems to measure stresses with diffraction. The sample coordinate system, X_i , is oriented by angles ϕ and φ to the sample coordinate system. X'_3 is the laboratory axis coordinate system.

2. Examples of applications

Examples are given of residual stress investigations in practical samples owing to their production / processing steps, supplemented by the characterization of a surface conditioning treatment deliberately applied to introduce a beneficial residual stress field rendering improved fatigue performance.

2.1. WC-Co coated substrates

Tungsten-carbide based *cermet* coatings with metallic cobalt binders, e.g. WC-Co, are frequently used when wear resistance, high surface hardness and low coefficient of friction similar to sintered carbide materials are required. In the industry, WC-Co coatings are typically deposited on substrates by the high-velocity oxygen-fuel, (HVOF) spraying process. In coated substrate systems, residual stresses are a superposition of contributions from the sample production history (cold work related to forging, rolling, extrusion), surface preparation (roughening through grit blasting) and the coating deposition process. Inherent to the nature of the HVOF coating deposition process, residual stresses due to the coatings arise from the impact, cooling, solidification and solid-state cooling of the splats, first onto the substrate and subsequently onto existing splats as the coating is built up layer by layer.

By combining the penetration depths of laboratory X-rays (Co-tube), high energy synchrotron radiation (80 – 200 keV white beam synchrotron X-rays from the ID15A instrument at the ESRF in energy dispersive mode) and thermal neutrons (KOWARI neutron strain scanner of the Bragg Institute, ANSTO) [11], depth resolved strain information was performed. Results are shown in figures 2-4.

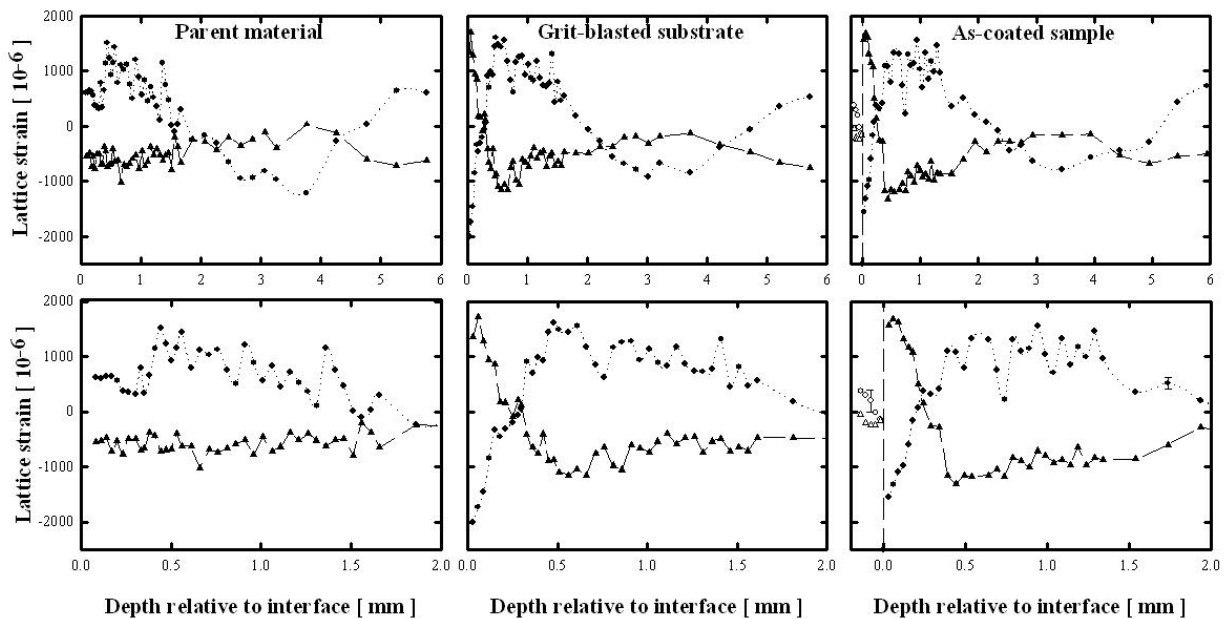


Figure 2: In-plane residual stress depth dependence determined from the lattice strain results of the (311) Cu and (101) WC Bragg peaks measured with synchrotron X-ray diffraction on a brass sample set. Results are for the parent material reference state (left set), the grit-blast substrate (centre set) and HVOF as-coated sample (right set) [6]. The near surface residual stress of the WC determined with laboratory X-rays (Co radiation) is also indicated [7]. The substrate coating interface is at 0 mm. The bottom sets of figures show an enlarged view of the results at the near-surface region. The error bars indicate the systematic error associated with the measurements. The lines connecting the symbols are guides to the eye. Legend: ●, substrate in-plane stress; ○, WC in-plane stress; ▲, WC near surface stress (laboratory X-rays).

Owing to the extremely small coating thickness of 200 μm (in sense of neutron diffraction stress experiment) measurements were done employing a fine through-thickness measurement mesh strategy with the aim to obtain the coating stress indirectly, i.e. through stress balance in the coating/substrate system. In addition, an attempt has been made to measure stresses in coatings directly. The two approaches gave a good countercheck on each other.

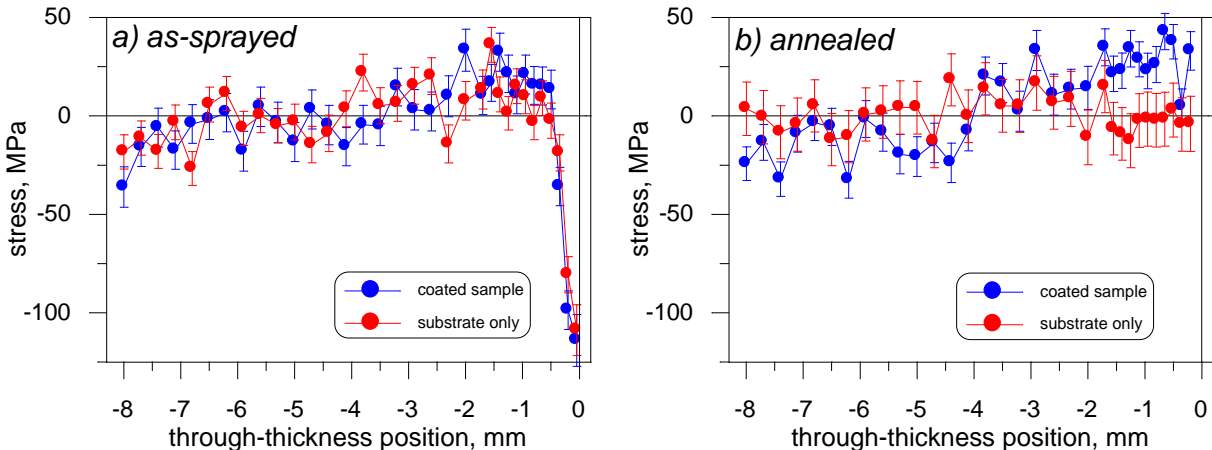


Figure 3. Experimentally measured through-thickness stress profiles for steel samples in a) as-sprayed and b) annealed conditions. Profiles measured in substrate only samples are also shown. [8]

Results of the stress profiles measured in a mild steel substrate series are shown in figure 4 as a comparison between the residual stress distributions observed from the as-coated and substrate-only samples, respectively for as-coated and annealed counterpart series. The differences between each set of profiles yield the stress purely due to the elastic effect from the WC coating.

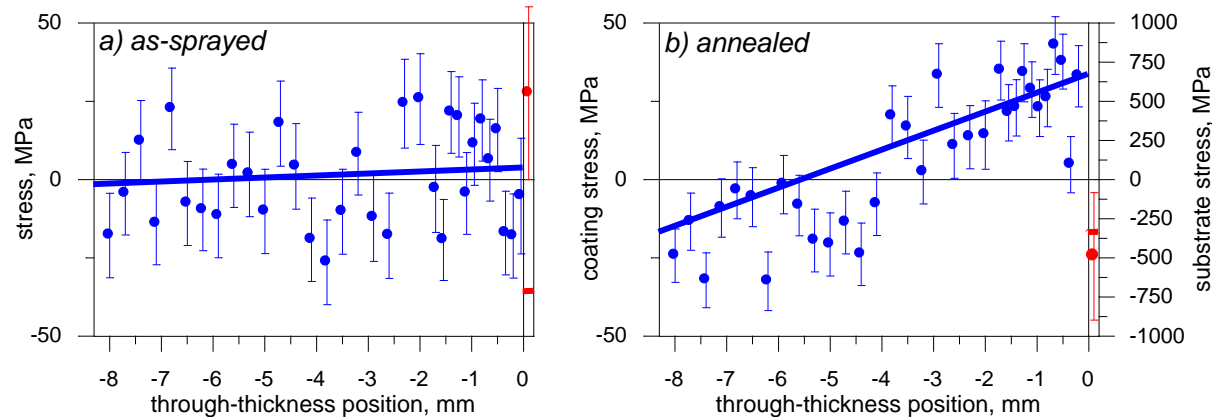


Figure 4. Experimentally determined through-thickness stress profiles for steel samples in a) as-sprayed and b) annealed conditions and model fit of the experimental data. [8]

2.2. Characterisation of production steps of cold cooled automotive undercarriage springs

Different approaches are followed with the manufacture of coil springs in the automotive industry today. Lately springs is manufactured using the cold coil forming process in which the feeder rod material is heat treated in advance, off line, and the coiling operation done at much lower temperature. By reducing the time the material has to be processed at high temperatures, excess of 900 $^{\circ}\text{C}$, this leads to substantially lower manufacturing costs. The input material to the coiling process is thus

already in a fully tempered martensitic state that gives the material its strength and hardness. The coiling is done at room temperature followed by a low temperature stress annealing (380 °C) to reduce the detrimental effects associated with the cold working process. Figure 5 shows the residual stress components mapped across the diameter of the coil. Investigations have been performed on the KOWARI neutron strain scanner (Bragg Institute, ANSTO). To measure the interplay between the various production stages on the residual stress fields, the interior stresses were directly measured with the spring geometry intact to negate possible relaxation effects that may result from sectioning or application of other destructive approaches.

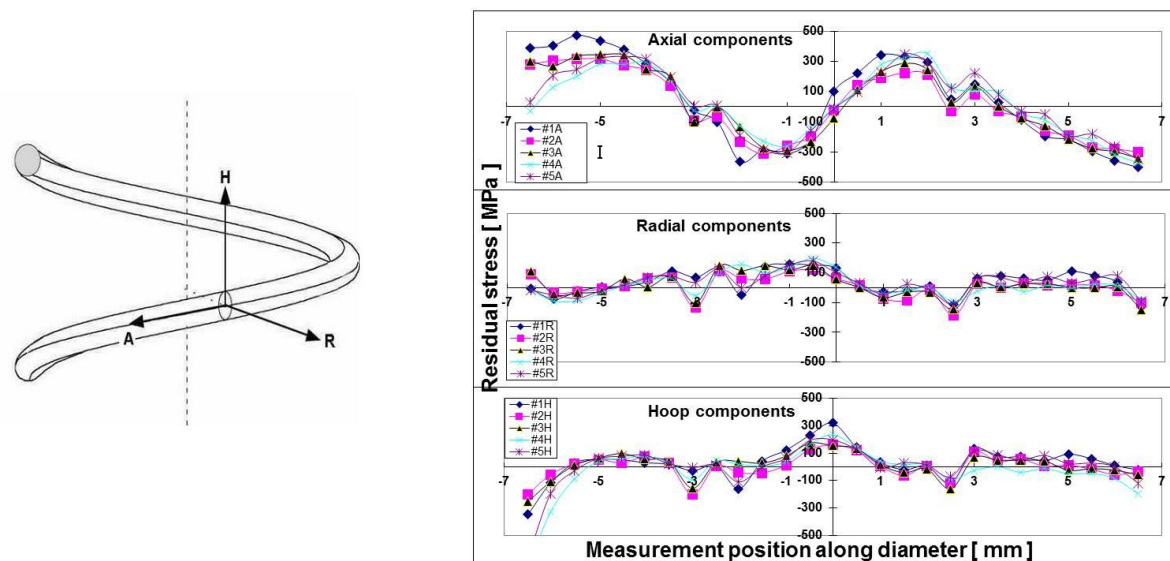


Figure 5: Results of the residual stresses measured along the centre line of cold coiled helical springs used in the motor vehicle industry. Samples have been extracted from sequential production steps: 1) Cold coiled from the fully tempered martensite state; 2) Low temperate tempering; 3) Hot setting; 4) Hot shot peening; 5) Cold shot peening.

2.3. Shot peened surfaces

Shot peening a surface treatment technique that is routinely used to introduce beneficial compressive residual stresses within the near-surface regions of components to mitigate against stress corrosion cracking and improves wear resistance. The technique comprises bombarding the sample surface with high speed pellets causing localised plastic deformation from which the restoring force captures a substantial compressive stress in the near-surface regions. The residual strain depth dependence was investigated using the 65 keV synchrotron beam at the ESRF on instrument ID31. Figure 6 show the in-surface residual strain fields existing at different regions in a specific sample geometry chosen to enable studying the influence of the underlying elastic material volume on the compressive residual stress conditions [9].

Conclusion

The complementary application of diffraction techniques using different radiation types to investigate various material systems have been presented. The examples show the capabilities to provide insight into the performance of materials from the characterisation of the inherent residual stresses. The neutron strain scanning technique is furthermore ideally suited to the study of in-situ loading response [10] with the investigations even possible at elevated or low temperatures to mimic the application conditions of materials in practical use.

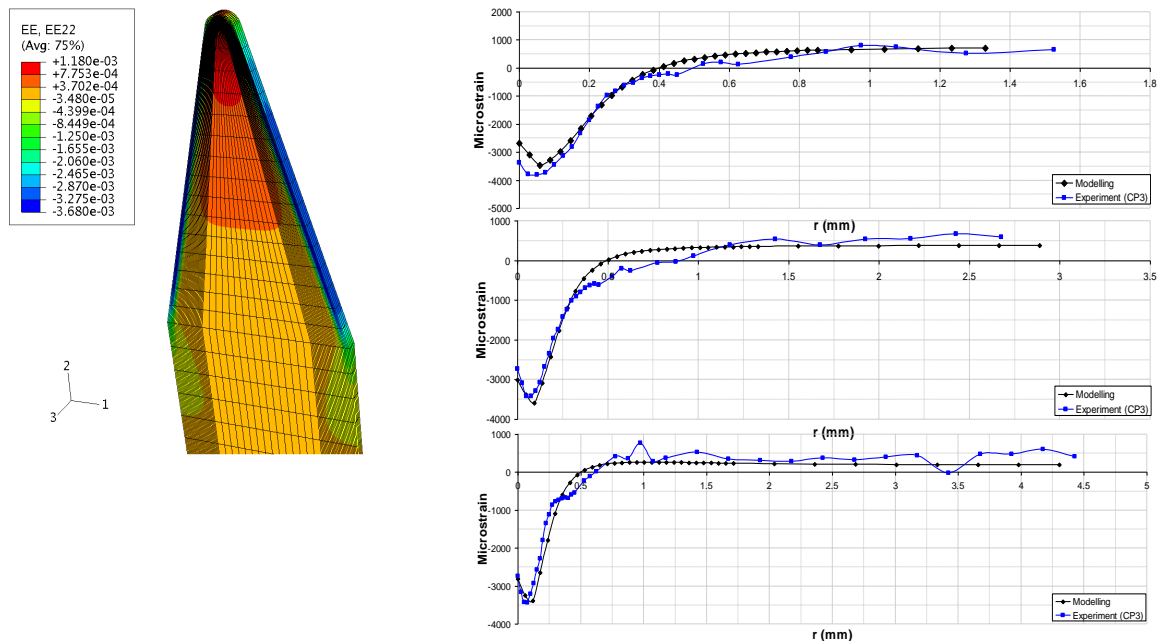


Figure 6: Depth dependence investigations of the in-plane residual strains in a shot peened sample with specifically geometry chosen to investigate the influence of the underlying elastic material volume on the beneficial compressive strains [9]. The depth dependence investigations have been performed with 65 keV synchrotron X-rays.

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References

- [1] *Introduction to the Characterization of Residual Stress by Neutron Diffraction*, Eds. MT Hutchings, PJ Withers, TM Holden and T Lorentzen, 2005, Taylor and Francis, ISBN 0-415-31000-8
- [2] *Analysis of residual Stress by Diffraction using Neutron and Synchrotron Radiation*, Eds. ME Fitzpatrick and A Lodini, 2003 Taylor and Francis, ISBN 0-415-30397-4
- [3] *Neutrons and Synchrotron Radiation in Engineering Materials Science*, Eds. W Reimers, AR Pyzalla, A Schreyer, H Clemens, 2008, Wiley-Vch Verlag GmbH & Co, ISBN 978-3-527-31533-8
- [4] GA Webster (ed.), "Neutron Diffraction Measurements of Residual Stress in a Shrink-fit Ring and Plug" 2000 *VAMAS Report No. 38* ISSN 1016-2186
- [5] C Ohms, RV Martins, O Uca, AG Youtsos, PJ Bouchard, M Smith, M Keavey, SK Bate, P Gilles, RC Wimpory, L Edwards 2008 *Proceedings of ASME PVP 2008* ASME, CD Publication, Order No. I795CD, ISBN 0-7918-3828-5
- [6] AM Venter, T Pirling, T Buslaps, OP Oladijo, A Steuwer, TP Ntsoane, LA Cornish, N Sacks 2102 *Surface and Coatings Technology* **206** 4011
- [7] OP Oladijo, AM Venter, LA Cornish, N Sacks 2012 *Surface and Coatings Technology*. In Press.
- [8] AM Venter, V Luzin, D Hattingh 2012 *Material Science Forum*. Submitted for publication.
- [9] TS Jun, AM Venter, AM Korsunsky 2011 *EXPERIMENTAL MECHANICS* **51** Issue: 2 165
- [10] AD Krawitz, AM Venter, EF Drake, SB Luyckx, B Clausen 2009 *Int. Journal of Refractory Metals & Hard Materials* **27** 317
- [11] A Brule, O Kirstein 2006 *Physica B* 385-386 1040