Stress Engineering for Earth & Space



Measurement of Awkward Components using X-Ray Diffraction

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The Measurement of Residual Stress using Laboratory Based X-Ray Diffraction Instruments; the Positives, the Pitfalls and a Round Robin, 27 September 2023, NPL



www.stress-space.com



1. Geometric

- Surface normal is not vertical
- Curved surfaces
- Limited X-ray goniometer access (including shading)
- High stress gradients (positioning is critical)
- Component too large or too heavy to fit on diffractometer test table
- 2. Material
 - Rough surface
 - Large grain size
 - Texture
- 3. Place
 - Installed infrastructure or plant
 - Production line (Industry 4.0 applications)
 - Hazardous environment





A robotic X-ray diffractometer designed by MRX-rays for surface residual stress analysis





Vision-guided X-Raybot





Stress-Space Ltd developed an Adaptive X-Raybot System (2021)

- Laser scans the surface geometry of interest (to within \pm 25 μ m)
- Positions the measurement lateral coordinates (to within 100 μ m)
- Defines the measurement surface normal & direction vector
- Adapts to curved surfaces where the normal is non-vertical.
- Remote control of measurements
- Over 10x faster set-up
- Input CAD/externally scanned model
- Audit trail with graphics



Acknowledgements

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Dr Stephen Nneji Dr Graham Appleby





Ringweld residual stresses measured by XRD





Distance from disk centre (mm)

11111 LLLL.

Stresstech and Pulstec results courtesy of the Open University

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Awkward Geometry: AM Bracket





Acknowledgements:	Architectural	bracket	supplied t	for residual	stress studies	, UCL
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1.	Complex geometry
2.	Non-planar surfaces
3.	Positioning
4.	Surface roughness ?
5.	Grain size ?
6.	Texture ?

AM Bracket



Laser guided adaptive system applied to define the X-ray optics for the positions and directions of interest



Awkward Geometry: Tee – Elbow Weld





XRD measurement lines 1mm beneath surface



- 1. Restricted angular access for XRD goniometer
- 1 mm sub-surface line scans defining stress profile up to fusion boundary required
- 3. Hoop & axial curvature plus weld cap features

Tee-Elbow Electro-polishing





- Laser system measured profile of ECM excavation
- Measurement positions located to within about 0.25 mm of the edge of the weld bead.
- Double curvature of Tee to Elbow captured together with uneven weld topography.



Tee-Elbow XRD Goniometer Access





6 o'clock position (left) $-40 \ to \ 0^o \ \psi$ range for axial $\pm \ 30^o \ \psi$ range for hoop Used 25 ψ tilts

X-Raybot source stand-off is 120 mm which helps access

9 o'clock position (right) -40 to $0^{\circ} \psi$ range for axial No restriction for hoop Used 25 ψ tilts



Significant Surface Roughness (Ra)



When Ra < d (penetration depth) XRD gives accurate stress results

penetration depth average roughness R

When Ra > d (penetration depth) XRD gives increasingly erroneous results





Mitigation: If R_a/d > 1.0, then gently smooth with SiC papers followed by an electrolytic polish.

Acknowledgements: Dr Richard Moat (Open University); research funded by Stress-Space (SSL) & SPRINT

A. Li, V. Ji, J. Lebrun, and G. Ingelbert, Exp. Tech., vol. 19, pp. 9–11, 1995

Grain Size – SPRINT Project Outcome

Materials having a large grain size challenge the validity of determining accurate measured stresses using laboratory XRD.

Inconel 718 beams (13.5 x 12.0 x 142) mm were solution heat treated at 955, 1050 and 1150 $^{\circ}$ C in an inert gas atmosphere and quickly cooled for 1h to obtain microstructures with the following grain sizes:

Small= $24 \pm 3 \mu m$ Medium = $56 \pm 16 \mu m$ Large= $117 \pm 33 \mu m$

The beams were then bent under 4-point loading and released to introduce tensile top and compressive bottom surface residual stresses.

The stresses were measured by XRD at 10 locations on each specimen using a range of test conditions: PSI and PHI oscillations, translations and multiple measurements.





Grain Size – SPRINT Project Outcome





Residual stresses in the bent IN718 beams with 3 different grain sizes were determined using two XRD instruments with different acquisition settings:

- Stresstech
- > X-Raybot

Results were compared against ICHD measurements.

Note: max bending stress dependent IN718 yield properties associated with different grain sizes

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Grain Size - SPRINT Project Outcome







Conclusion:

X-Raybot multiple exposure method eliminated grain size measurement errors for the medium and large grain size IN718 beams

Texture



Microstructures with strong crystallographic texture are found in additive manufactured parts (worse than welding). e.g. in FCC austenitic steels, Al and Ni alloys.

Strong texture creates anisotropic bulk elastic properties.

At the **grain scale** the deformation response of crystallographic planes used for XRD (**diffraction constant**) is affected by both elastic and plastic anisotropic effects.

Thus E_{xxx} varies strongly with direction and a single value cannot be used for $\sin^2\psi$ fitting in laboratory XRD.



Typical microstructures and texture present in AM of FCC material taken from Lakshmi L. Parimi, Ravi G. A., Daniel Clark, Moataz M. Attallah, Materials Characterization, Volume 89, 2014, Pages 102-111

Texture - introduces error



Colour coded texture: 0 = random, 1=2, 2=3, 3=5, 4=11, 5=42 (unit of strain 10⁻³%).



Texture anisotropy effects were examined using selfconsistent modelling for materials with varying severity of texture. 1000s scenarios were simulated varying the applied stress & associated plasticity for 10 different levels of synthetic texture.

Simulation Results

- Large stress errors in sin² psi method for stainless steel owing to texture along the (001) fibre direction assuming random texture bulk modulus.
- Errors in apparent XRD measured stress increase with degree of texture.

Dealing with Texture



Colour coded texture: 0 = random, 1=2, 2=3, 3=5, 4=11, 5=42 (unit of strain 10^{-3} %).



- Stress error much less using modulus calculated from slope of modelled stress-strain curve
- XRD residual stress measurements are potentially feasible for stresses acting in the 2 directions orthogonal to the <001> fibre direction in FCC materials, if an appropriate elastic modulus is used and angular sweeps are made around this fibre direction.
- 3. The transverse isotropic modulus can be determined by EPSC modelling for given texture, or from a columnar grain aggregate model or by measurement (ASTM standard method).

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Awkward Location







ENGIN-X Neutron Diffractometer

- 1. In-situ "field" measurements
- 2. Restricted access
- 3. Remote control essential

Can surface residual stresses be measured by XRD at the same time as bulk stresses by neutrons?



STFC Proof of Concept Grant awarded (13 Jan 2023)



Proof of Concept Experiment on ENGIN-X (March 2023)

Supported by: Graham Appleby, Saurahb Kabra, Tung Lik Lee and Joe Kelleher (STFC) and Adrien Sprauel (MRX-Rays)

Experimental plan: Measure CTE of test sample



- 1. 20 mm dia cylindrical test coupon
- 2. Extracted from A508 Cl 3 steel ringweld (after stress relief).
- Top surface was electrochemically milled to a depth of 100 microns to remove any residual stresses introduced by EDM machining.
- 4. Cylinder was thermo-coupled at the top and bottom.
- Temperature was raised in approx. 20°
 C increments from room temperature to 240 °C using a hot plate.



CTE is the mean coefficient of thermal expansion

Remote In-situ Measurements







Laser scan

Reference XRD measurements



Birds eye view of adaptive laser scan of surface for defining measurement points and vectors

	Stress-fre	ee powder	Stressed reference (-509 ± 35) MPa		
Alignment	Direct stress (MPa)	Shear stress (MPa)	Direct stress (MPa)	Shear stress (MPa)	
+ 45°	-6 (17)	-7 (3)	-533 (19)	1 (3)	
-45°	-12 (10)	7 (2)	-537 (17)	-1 (3)	







Bird's eye video clip showing X-Raybot {211} lattice parameter measurements using $Sin^2 \psi$ method

Mean CTE measured by XRD and neutron diffraction compared with ASME A508 Class 3 code data



Awkwardness	Some Solutions		
Location: structure/plant/production line cannot be moved (or hazardous environment)	Measure in-situ using portable XRD equipment (use remote controlled robotic XRD system for hazardous cases)		
Material: surface roughness (Ra/d > 1)	Smooth surface (light abrasive) then electro-polish		
Material: large grain size	Use larger gauge area, oscillation and/or sum multiple exposures		
Material: texture	Measure normal to the <001> fibre direction in FCC alloys using transverse isotropic diffraction elastic constants.		
Geometry: Complex 3D curved surfaces	Define measurement position, surface normal and measurement vectors using a laser guided adaptive XRD system (e.g. X-Raybot)		
Geometry: Restricted XRD goniometer access	Try different mounting, other diffraction planes, or restrict angular range of tilts (compensate for by maximizing number of tilts).		
Geometry: High stress gradients	Use smaller gauge size & high resolution global/local positioning system (e.g. laser guided adaptive X-Raybot)		