

The Benchtop Platform

POWERFUL SPEED, ACCURACY AND SENSITIVITY

- 1.2 kW with internal cooling
- Time-tested goniometer
- LYNXEYE XE-T detector
- Motorized beam optics

- VERSATILE MEET EVERY NEED

- Reflection and transmission Powder XRD
- Non-ambient diffraction
- GID, XRR, Stress, Texture

ACCESSIBLE AUTOMATIC, AFFORDABLE, EASY-TO-USE

- Dynamic Beam Optimization
- Touch panel operation
- Stage and optics exchange



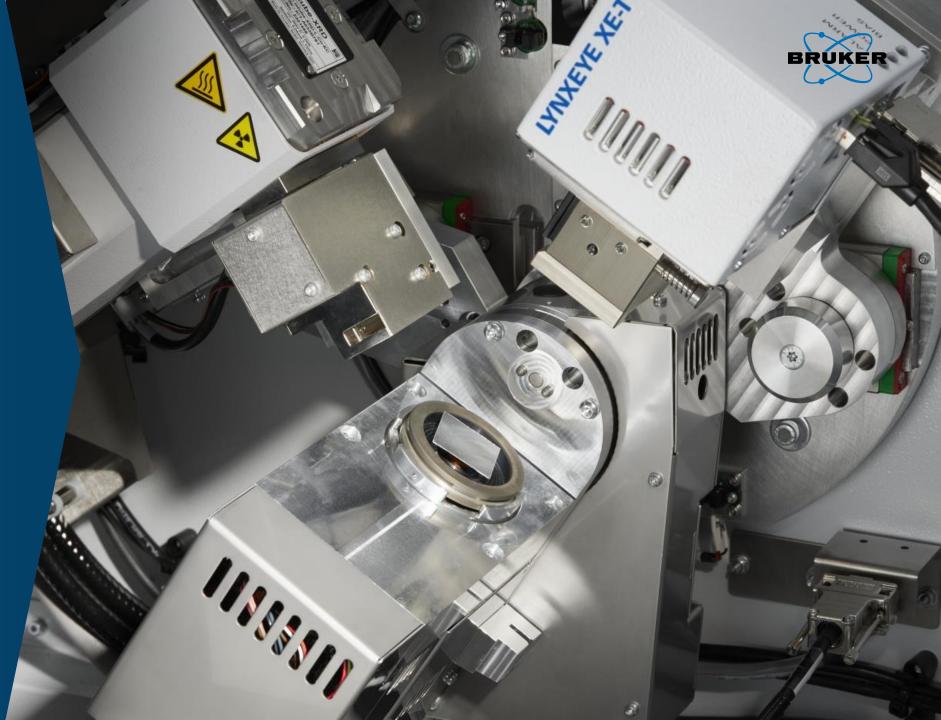


D6 PHASER - THE BENCHTOP PLATFORM



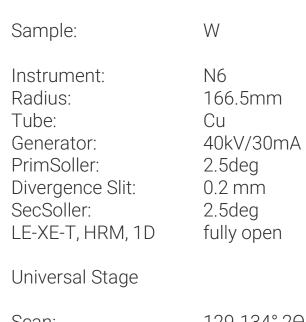
Residual Stress Analysis

Generator 1200W X-ray Tube Cu or Cr Incident Optic Advanced Optic with Axial Soller Sample Stage Universal Stage with Phi Attachment Air Scatter Control None Diffracted Beam Optic Axial Soller Detector LYNXEYE XE-T



DIFFRAC.WIZARD FOR D6

Data collection - Uniaxial STRESS DIFFRAC.WIZARD



Scan:	129-134 20
Psi tilt:	± 45°, 17 steps
Incr:	0.03° 20
Time:	0.2s/step
Total:	19 min

•
$\uparrow \downarrow \uparrow$

WIZARD settings as for D8

 Tilt Psi is achieved with Universal Stage





Data collection DIFFRAC.WIZARD

Sample: W Stress Free Reference Instrument: N6 Radius: 166.5mm Tube: Cu Generator: 40kV/30mA PrimSoller: 2.5deg Divergence Slit: 0.2 mm SecSoller: 2.5deg fully open LE-XE-T, HRM, 1D Universal Stage Scan: 129-134° 20 Psi tilt: ± 45°, 17 steps 0.03° 20 Incr: Time: 0.2s/step Total: 19 min

LEPTOS 7.14 (W reference)

E 🔗 Iso still psi17 10s

🗄 🔗 Iso still psi17 30s

So psi17 0.1s
 So psi17 0.1s
 So psi17 0.2s
 So psi17 0.2s
 So psi17 0.2s
 So psi17 0.2s

Project
 Project

<u>File Edit Sample Simulation Stress Scripts Options Help</u>

7.5*10

6.5*10³

5.5*10³

6*10

7*10³

다 좀 다 물 만 참 다 비 빤 빵 좀 한 봐 있 수 있는 것, 다 한 것, 가 이 이 것, 많은 않 때 때 이 있

5°10³ 131 057 4.5*10³ 131.028 4*10³ Ê 3.5*10³ 3*10 0.3 0.5 0.2 0.4 2.5*103 2*10³ Psi 30.0 34.0 37.8 41.4 45.0 4 • 1.5*10³ 1*10 Phi Angle -9999 💌 5*10 Peak Evalu Stress mode Crustal Norma 129 129.5 130 130.5 131 131.5 132 132.5 133 133.5 C Sliding Gravity C Gravity Normal + Shear Standard Pearson VII 🔲 Use as inpu range2 range3 range4 - range5 range11 range13 — range14 — range15 · range16 Parabolic C Multiple Pr d0 -/lerged Plot Dataset Reduction/Fit Results Ellipsoid Lame Tailored Report Merged Plot Dataset Reduction/Fit Results Ellipsoid Lame Tailored Report Correction Peak Evaluation Absorption 🖌 Gravity 30 👻 Threshold Stress tensor (MPa) ▼ Sliding Gravity ... 10 20 30 40 50 60 70 80 ▼ Background 5 points at edges Polarisation ✓ Parabolic 70 ▼ K-alpha 2 ratio 0.50 Fit Standard 🔽 Smooth 🔽 Fit Pearson VII 🔲 Fit Multiple Peaks 👘 K-alpha 2 Sample H K L Wavelength 2Theta Poisson Young S1 1/2 S2 Arx σm 131.160 0.290 401606 -7.221E-7 3.212E-6 Stress orientatio -Scalar stress (Phi, Psi) ± 0.5 Normal -9.1 ± 2.7 Shear -4.8 < Save as default Clear >12:38:41 - Project file saved: W referen >12:38:44 - New Stress object created: S PsHyd -/-± -/-12:44:14 · Project file saved: W refere >13:28:02 - New Stress object created: >13:29:12 - Project file saved: W referen Merged Plot Dataset Reduction/Fit Results Ellipsoid Lame Tailored Report >

According EN15305 the instrument is qualified for residual stress measurements if the stress free W powder gives a result of $|\sigma| \le 31$ MPa with uncertainty $|u_{(\sigma)}| \le 31$ MPa



20.7 25.7 30

14.5

20 ·

131.171

131 142

131.114

131.0857



– ø ×

- Psi>0 → Psi<0 — Regression

37.8 41.4

34

STRESS Analysis DIFFRAC.LEPTOS – Normal/Shear stress only positive ψ



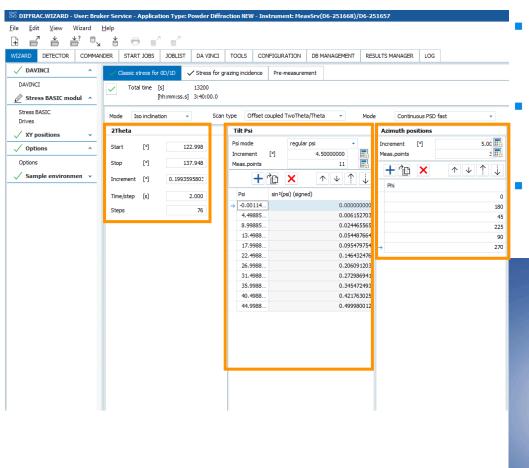
W Kr Sputtered LEPTOS 7.12 (Stress-W-2GPa) Sample: Ð File Edit Sample Simulation Stress Scripts Options Help on Glass E-- Stress6 N5 Instrument: - stress-W-2GP_tensor (range 1) stress-W-2GP_tensor (range 2) - Psi>0 5 stress-W-2GP_tensor (range 3) Radius: 166.5mm Ar Psi< 0</p> 131 053 -🕂 stress-W-2GP_tensor (range 4) 2.2*10 - Regression To stress-W-2GP_tensor (range 5) 130,9246 2*10³ Tube: tress-W-2GP_tensor (range 6) Cu 130,795 To stress-W-2GP tensor (range 7) 1.8*10 stress-W-2GP_tensor (range 8) 130,666 1.6*10 Generator: 40kV/15mA T stress-W-2GP tensor (range 9) ≩ 1,4*10³· 130,536 • stress-W-2GP_tensor (range 10) To stress-W-2GP tensor (range 11) Ĕ 1,2*10³ PrimSoller: 130,407 2.5deg stress-W-2GP_tensor (range 12) 1*10³ 130,278 To stress-W-2GP tensor (range 13) 8*10² 0.2mm - 🕂 stress-W-2GP_tensor (range 14) Divergence Slit: 130,149 stress-W-2GP_tensor (range 15) 6*10 0,2 0,3 0,4 0,5 T stress-W-2GP tensor (range 16) Sin^2(Psi) 4*10 Phi 0°/180° Stress-W-2GP_tensor (range 17) 5 stress-W-2GP_tensor (range 18) 134 124 126 128 130 132 Psi -45.0 -42,1 -39,2 -36,3 -33,2 -30,0 -26,6 **∢** Þ stress-W-2GP_tensor (range 19) SecSoller: 2.5deg range1 - range2 range3 - range4 - range5 range6 - range7 - range8 - range9 To stress-W-2GP tensor (range 20) range10 - range11 - range12 - range13 - range14 - range15 - range16 range17 - range18 Phi Angle -359,9! 🔻 - stress-W-2GP_tensor (range 21) range19 -- range20 - range21 - range22 - range23 - range24 - range25 - range26 — range27 Peak Evaluation Stress model Crystal 5 stress-W-2GP_tensor (range 22) LE-XE-T, HRM, 1D 4.95mm range28 - range29 - range30 - range31 - range32 - range33 - range34 - range35 - range36 O Normal range37 -- range38 - range39 - range40 - range41 - range42 - range43 - range44 - range45 2Theta0 130,993 Sliding Gravity C Gravity Normal + Shear range46 — range47 — range48 — range49 — range50 — range51 — range52 — range53 — range54 C Standard Pearson VII C Biaxial range55 - range56 - range57 - range58 - range59 - range60 - range61 Use as input - range62 range63 Biaxial + Shear - range64 - range65 - range66 C Parabolic d0 0,0847 nm C Triaxial Universal Stage Merged Plot Dataset Reduction/Fit Results Ellipsoid Lame Tailored Report Merged Plot Dataset Reduction/Fit Results Ellipsoid Lame Tailored Report Correction Peak Evaluation 🔽 Gravity 30 👻 Absorption Threshold sigma Scan: ▼ Sliding Gravity ... 10 20 30 40 50 60 70 80 123-138°20 points ▼ Background 5 sigma II at edge Polarisation ▼ Parabolic 70 sigma III Psi tilt: +45°, 11 steps ▼ K-alpha 2 ratio 0,50 🔽 Fit Standard -Scalar stress (Phi, Psi)-Smooth Fit Pearson VII 0.2°20 Incr: Sample Normal -2098,5 ± 39,0 ± 7,7 1-32,7 Shear Material НКГ Wavelength 2Theta Poisson Young S1 1/2 S2 Arx Time: 2s/step >9:12:14 · LEPTOS 7.12 started 321 0,1540549 131,160 0,290 401606 -7,221E-7 3,212E-6 1,000 >9:12:14 · Device database loaded 9:12:14 - Opened MDB: default.dbm Total[.] 22x152s 9:12:14 · Opened Stress MDB: stress.dbm PsHyd -/-± -/->9:12:27 · Project file opened: Stress-W-2GPa Merged Plot Dataset Reduction/Fit Results Ellipsoid Lame Tailored Report

Data collection - Stress Tensor DIFFRAC.WIZARD

Sample: W Ar Sputtered on Glass Instrument[.] Ν5 Radius: 166.5mm Tube: Cu Generator: 40kV/15mA PrimSoller: 2.5deg Divergence Slit: 0.2mm 1) 0°/180° Phi 2) 45°/225° 3) 90°/270° SecSoller: 2.5deg 4.95mm LE-XE-T, HRM, 1D

Universal Stage with Phi Attachement

Scan:	123-138°2 0
Psi tilt:	+45°, 11 steps
Incr:	0.2°2 0
Time:	2s/step
Total:	66x152s



- WIZARD settings as for D8
- Tilt Psi is achieved with Universal Stage
- Azimuth positions set with Phi attachment









Sample:

Instrument[.] Radius: Tube: Generator. PrimSoller: Divergence Slit: Phi

SecSoller: LE-XE-T, HRM, 1D

Universal Stage with Phi Attachement

on Glass

166.5mm

40kV/15mA

Ν5

Cu

2.5deg

0.2mm

2.5deg

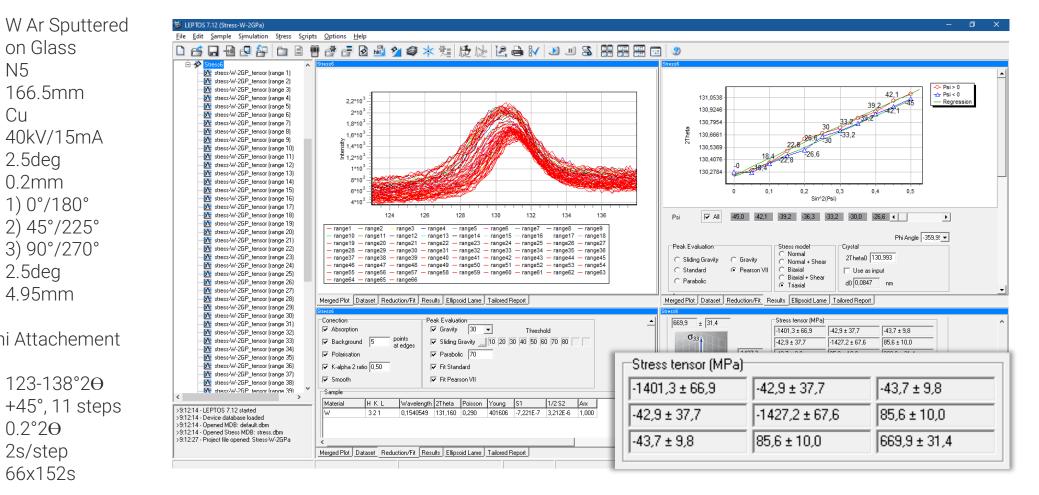
4.95mm

1) 0°/180°

2) 45°/225°

3) 90°/270°

Scan:	123-138°2 0
Psi tilt:	+45°, 11 steps
Incr:	0.2°20
Time:	2s/step
Total:	66x152s



Data collection – Parallel Beam Geometry DIFFRAC.WIZARD



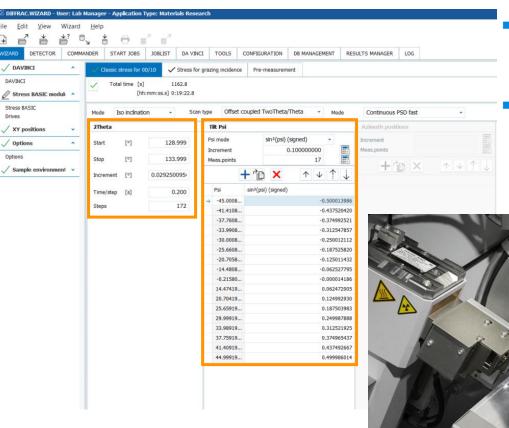
Sample: W Kr Sputtered Instrument: Radius: Tube: Generator: PrimSoller: Divergence Slit: SecSoller: LE-XE-T, HRM, OD

on Glass N6 166.5mm Cu 40kV/30mA 2.5deg 0.2 mm equatorial soller 0.4° fully open

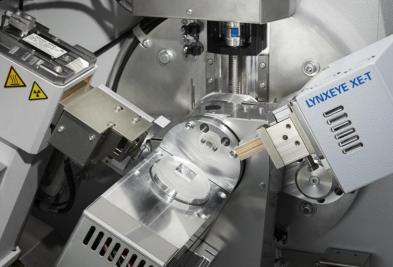
Universal Stage

Scan: Psi tilt: Incr: Time: Total:

129-134° 20 ± 45°, 17 steps 0.2°20 2s/step 17 min



- WIZARD settings as for D8
- Tilt Psi is achieved with Universal Stage



Data collection – Parallel Beam Geometry DIFFRAC.WIZARD



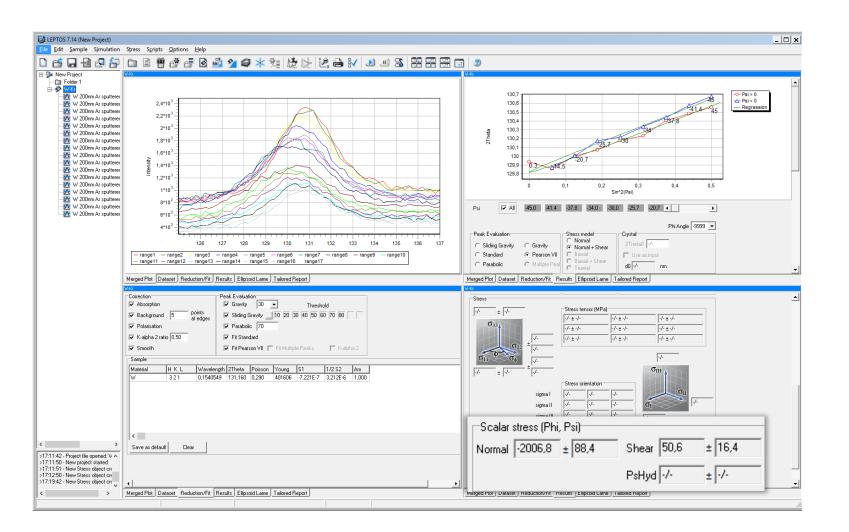
W Kr Sputtered Sample: on Glass Instrument: Radius: Tube: Generator: PrimSoller: Divergence Slit: SecSoller: LE-XE-T, HRM, OD

Ν6 166.5mm Cu 40kV/30mA 2.5deg 0.2 mm equatorial soller 0.4° fully open

Universal Stage

Scan: Psi tilt: Incr: Time: Total:

129-134°20 ± 45°, 17 steps 0.2°20 2s/step 17 min



MATERIALS RESEARCH **Residual Stress Analysis**

- Stress-free sample (W-Powder) Results according EN 15305
- W Thin Film (one sputtered under Aratmosphere the other under Kratmosphere)
- Residual Stress Analysis using the Iso-Inclination method is fully implemented!



X-RAY DIFFRACTION **D6 PHASER – Benchtop Residual Stress Analysis**

Application Report 40

The D6 PHASER is a multipurpose benchtop diffractometer that is uniquely suited for modern materials research characterization. In this report, we present the capabilities of this system in a reflection diffraction configuration for residual stress analysis.

Residual stress is the localized stress that remains in a material after it has undergone processes such as welding, casting, forming, machining, or thin film deposition. The analysis of residual stresses is important to understand how these stresses affect the performance and lifetime of a component. Additionally, the residual stress can be used to identify specific material properties and failure mechanisms which can be used in the design of components and parts.

Tungsten layers are components of thin-film transistors in TFT-LCD screens. They are used when large screen formats, high image definition, and optimized contrast are required. Tungsten is also used in microelectronics, for example for creating layers in frequency filters. Other applications for tungsten includes diffusion barriers made of tungsten-nitride, conductor tracks in microelectronic components as well as reactively sputtered transparent layers made from tungsten oxide for OLED displays and for use in electrochemistry. In this study the residual stress of a tungsten layer created by PVD under Ar atmosphere and one sputtered at Kr atmosphere is analyzed. The thickness of the films is in the range of 200nm



Universal stage with spring sample holder



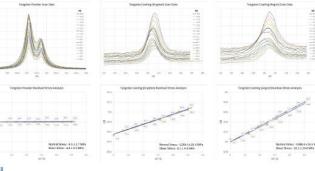
The (321) reflection of tungsten was chosen due to its high angle, 131' 29 for Cu radiation, resulting in high sensitivity to d-spacing changes. For the ISO-inclination method 17 psi steps were chosen between -45° and +45° with a constant step size in sin²Ψ.

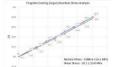
The Cu source was operated at 40kV/30mA, while the divergence was The CD source was operated at dor/work (with the directed was controlled with a 0.2 mm situat at a 2.5 m sixal Soller. The LYNXEV XE-T detector was used in high count rate mode with a 5° detector opening. Additionally, a 0.2 mm Ni filter was positioned in the beam path. The Universal stage was selected to mount the sample. The measurement was planned using the WIZARD plugin of DIFFRAC MEASUREMENT.

The diffractometer is verified for residual stress measurements based on the EN15305 by measuring a stress-free sample. According to this norm the equipment is certified if a stress-free tungsten specimen gives a normal stress smaller than +/-31MPa with an uncertainty of +/-31MPa and a shear stress smaller than +/- 15.6 MPa with uncertainty 15.6 MPa

Floure 2 Measurement geometry for residual stress measurement in the D6 PHASER. Shown is the optional phi attachment which enables blaxial stress measurements

The stress-free sample was found to exhibit a normal stress of -9.1+/-2.7 MPa with a shear stress of -4.8 +/-0.5 MPa. The film deposited in a krypton atmosphere exhibits a strong compressive stress of -1.3 GPa with a minimal shear stress of -8.1 MPa while the film deposited under an Ar atmosphere pushed the stress to -2.0 GPa with shear stress of -20 MPa





Raw scan data (Top) and sin³ analysis (Bottom) of stress-free tungsten powder (Left) and two tungsten coaling: deposited under krypton (Middle) and argon (Right) atmospheres

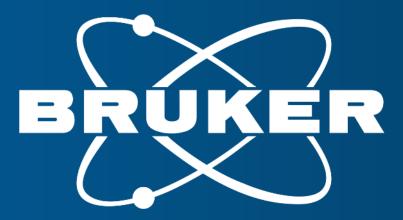


RESIDUAL STRESS ANALYSIS with D6 Conclusion





- Residual Stress Analysis with D6 can be performed according European Norm for Residual Stress measurements (EN 15305)
- Very good for flat samples
- Line focus allows high intensity -> short data collection
- Control of spot size with fixed sample illumination and height limiting slits



Innovation with Integrity

Innovation with Integrity