

D6 PHASER

# 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

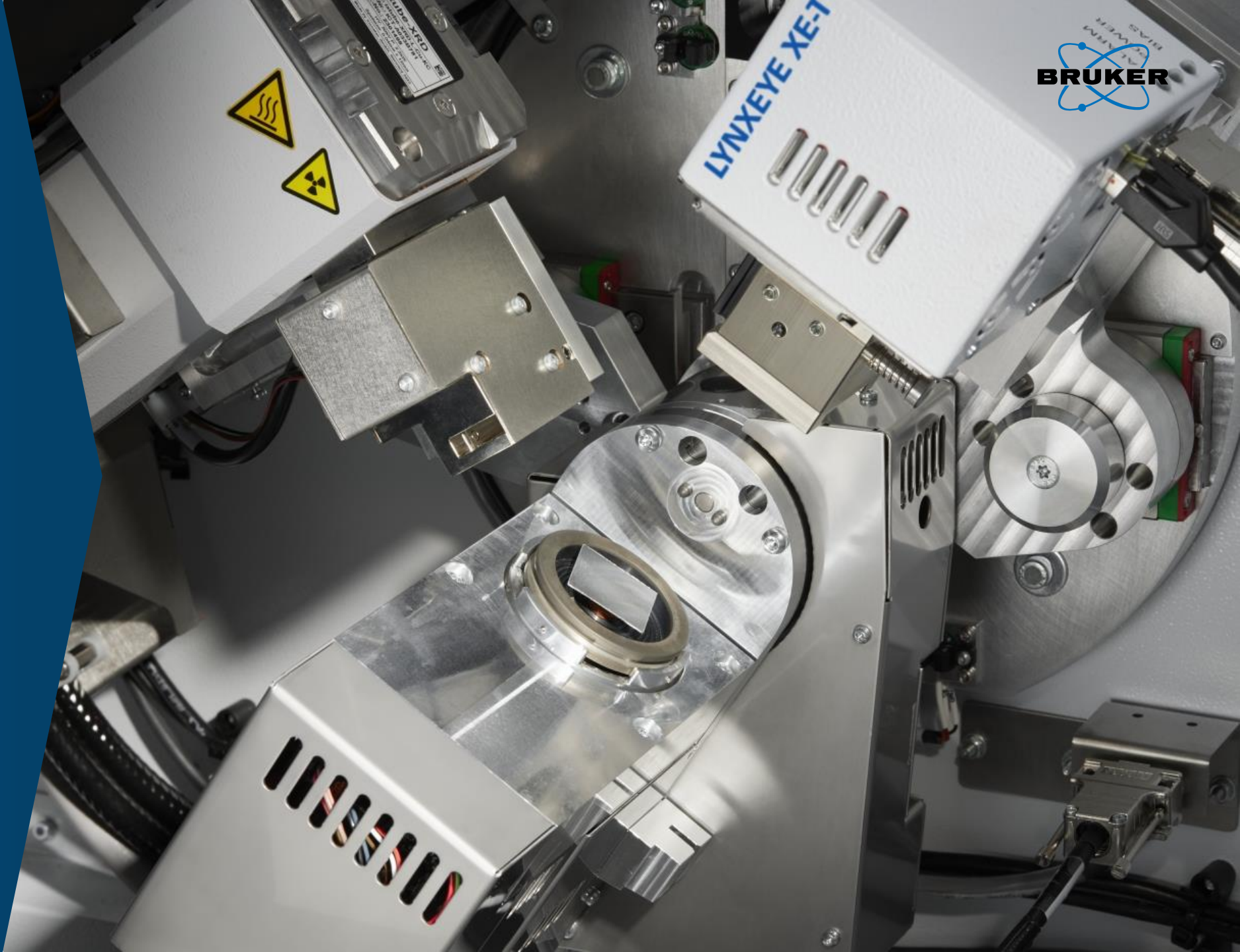
- Dynamic Beam Optimization
- Touch panel operation
- Stage and optics exchange

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  
**Diffacted Beam Optic** Axial Soller  
**Detector** LYNXEYE XE-T



# Data collection - Uniaxial STRESS

## DIFFRAC.WIZARD

Sample: W

Instrument: N6

Radius: 166.5mm

Tube: Cu

Generator: 40kV/30mA

PrimSoller: 2.5deg

Divergence Slit: 0.2 mm

SecSoller: 2.5deg

LE-XE-T, HRM, 1D fully open

Universal Stage

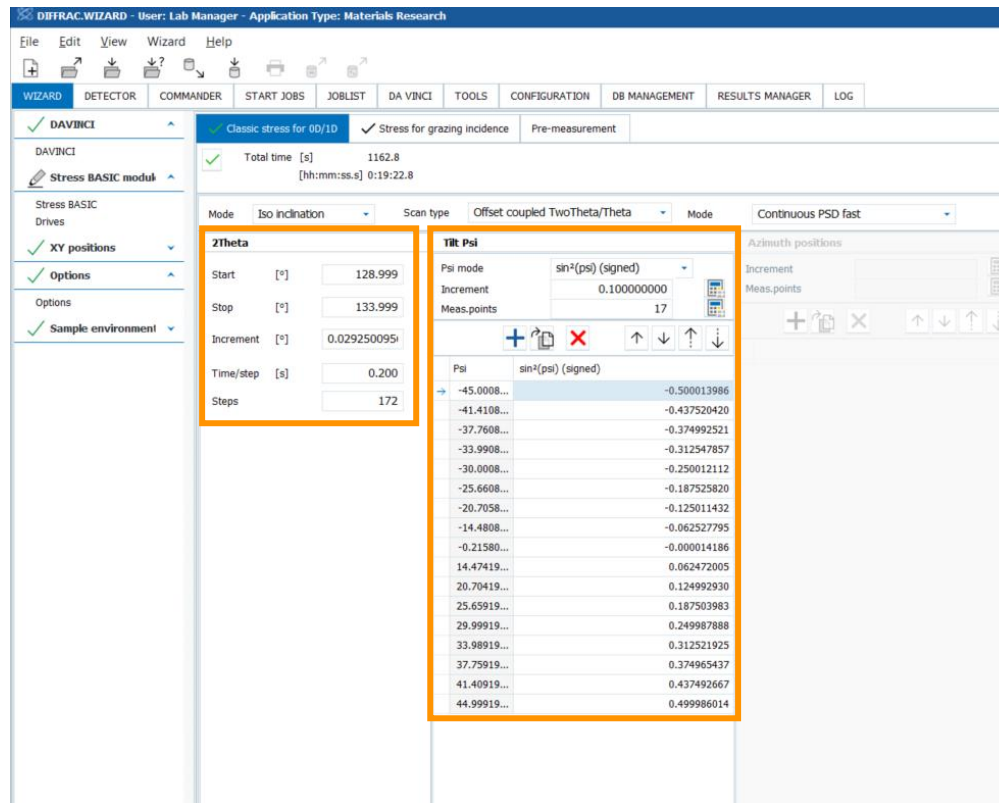
Scan: 129-134° 2θ

Psi tilt: ± 45°, 17 steps

Incr: 0.03° 2θ

Time: 0.2s/step

Total: 19 min



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



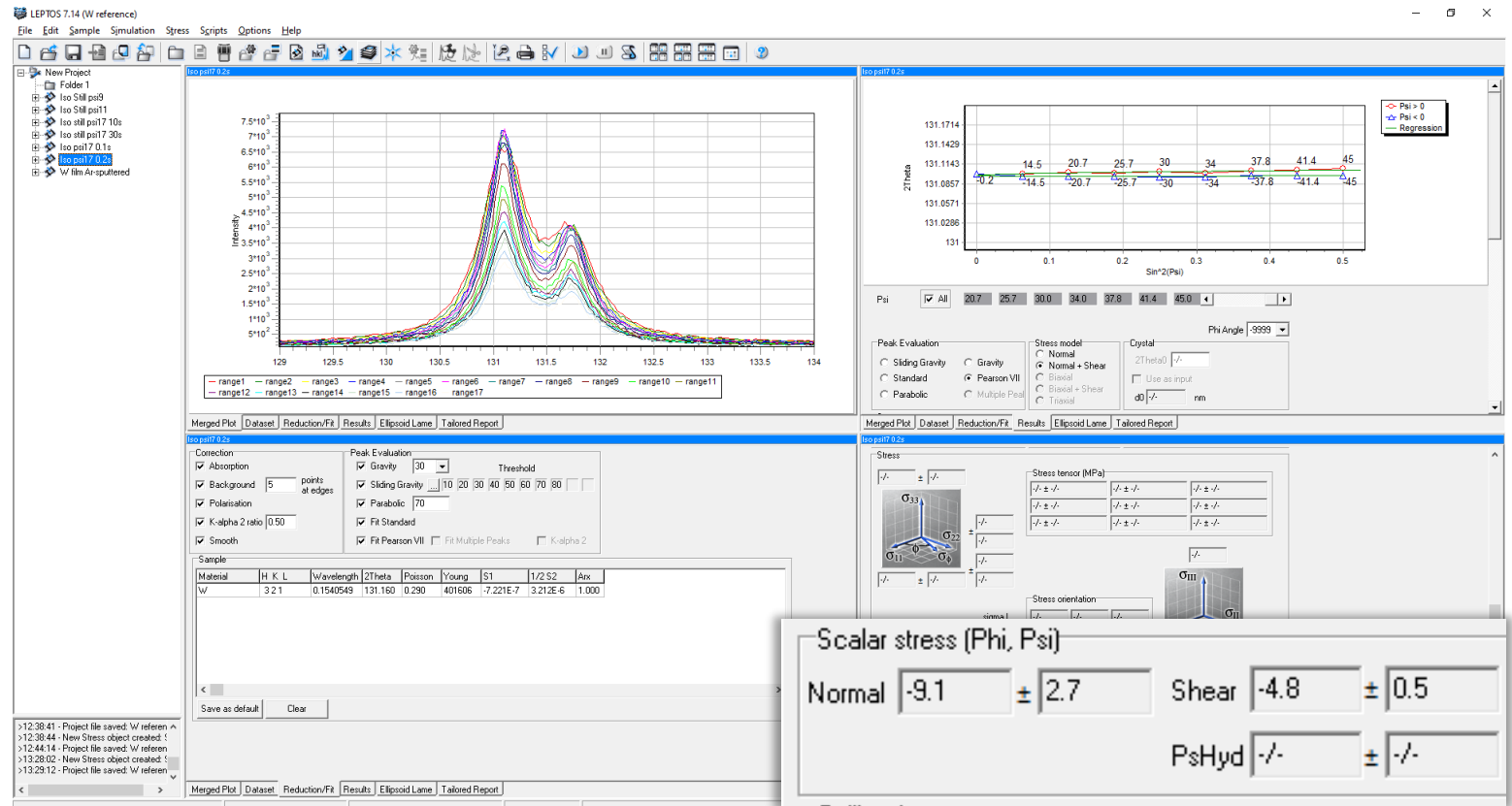
# Data collection DIFFRAC.WIZARD

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  
 LE-XE-T, HRM, 1D fully open

Universal Stage

Scan: 129-134° 2θ  
 Psi tilt: ± 45°, 17 steps  
 Incr: 0.03° 2θ  
 Time: 0.2s/step  
 Total: 19 min



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

# STRESS Analysis

## DIFFRAC.LEPTOS – Normal/Shear stress only positive $\psi$

Sample: W Kr Sputtered on Glass

Instrument: N5

Radius: 166.5mm

Tube: Cu

Generator: 40kV/15mA

PrimSoller: 2.5deg

Divergence Slit: 0.2mm

Phi: 0°/180°

SecSoller: 2.5deg

LE-XE-T, HRM, 1D 4.95mm

Universal Stage

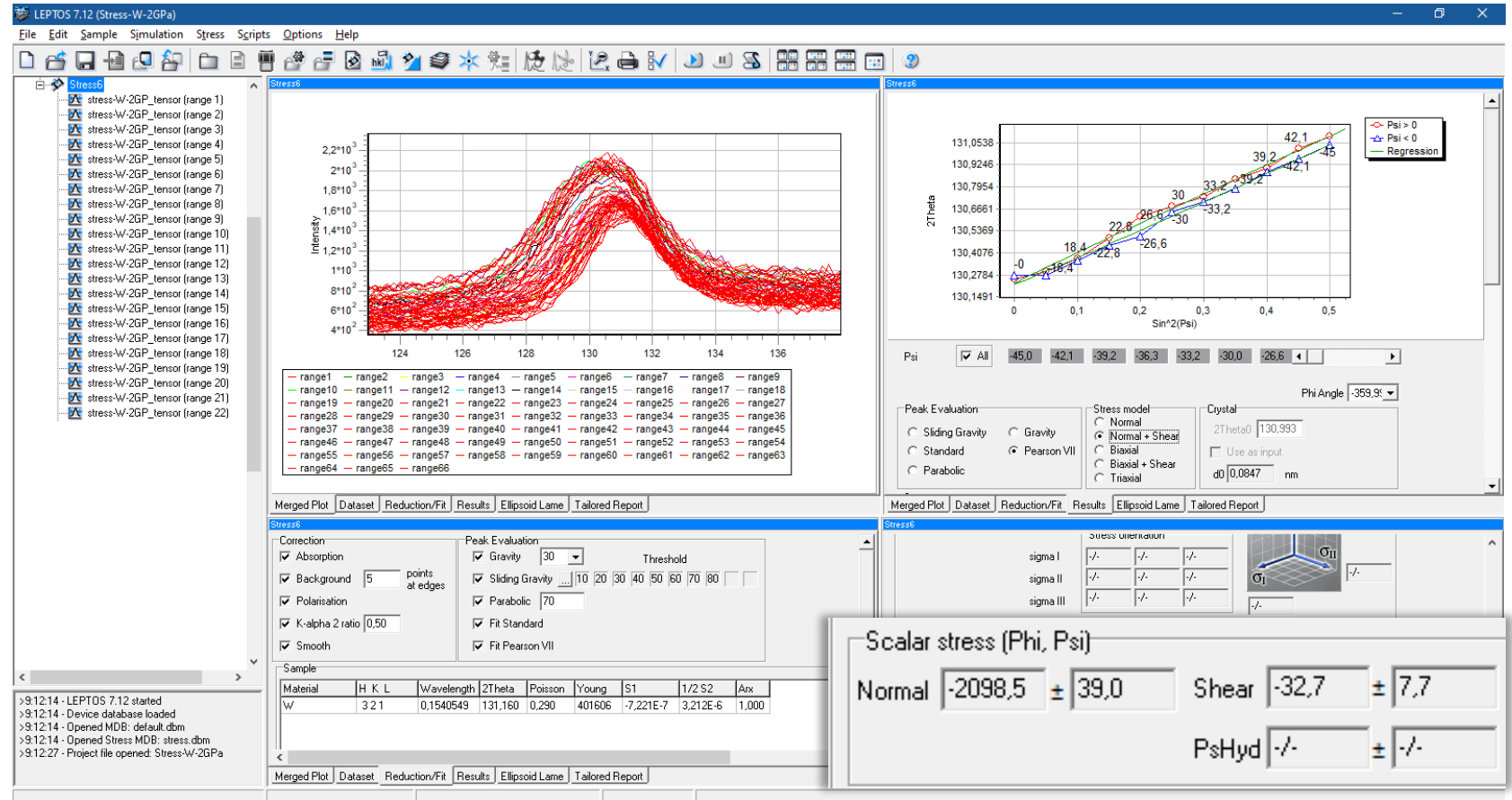
Scan: 123-138°2 $\theta$

Psi tilt: +45°, 11 steps

Incr: 0.2°2 $\theta$

Time: 2s/step

Total: 22x152s



# Data collection - Stress Tensor DIFFRAC.WIZARD

Sample: W Ar Sputtered  
on Glass

Instrument: N5

Radius: 166.5mm

Tube: Cu

Generator: 40kV/15mA

PrimSoller: 2.5deg

Divergence Slit: 0.2mm

Phi  
1) 0°/180°  
2) 45°/225°  
3) 90°/270°

SecSoller: 2.5deg

LE-XE-T, HRM, 1D 4.95mm

Universal Stage with Phi Attachment

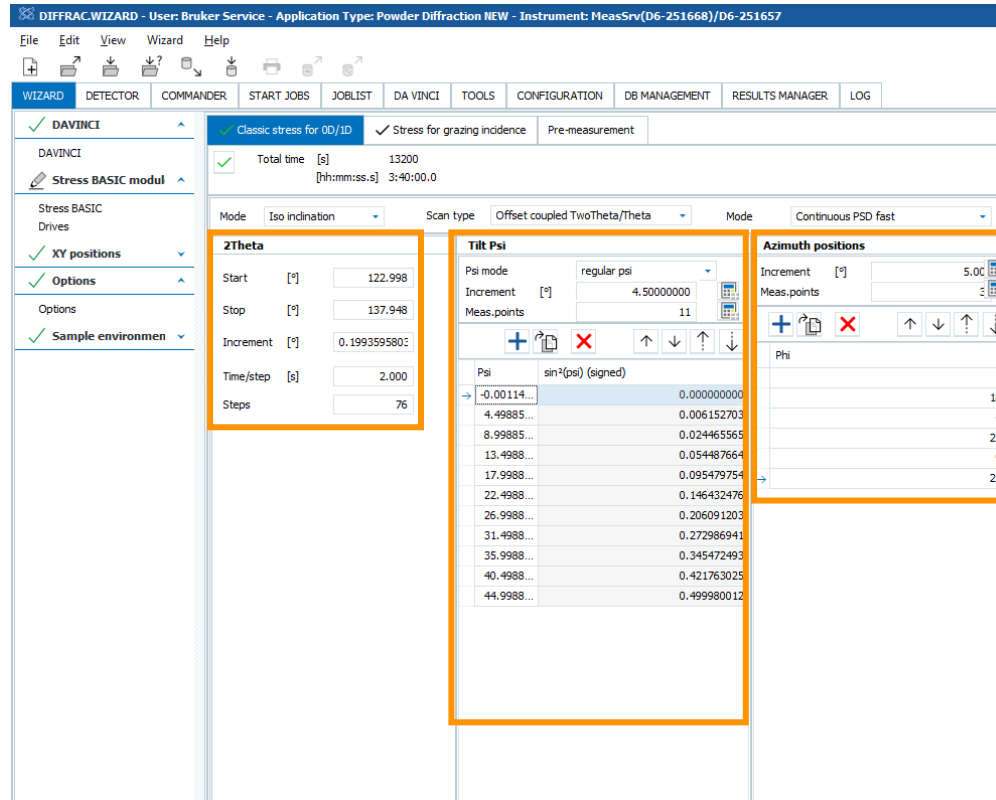
Scan: 123-138°2θ

Psi tilt: +45°, 11 steps

Incr: 0.2°2θ

Time: 2s/step

Total: 66x152s



Psi	sin²(psi) (signed)
-0.00114...	0.00000000
4.49885...	0.006152703
8.99885...	0.024465565
13.4988...	0.054487664
17.9988...	0.095479754
22.4988...	0.146432476
26.9988...	0.206091203
31.4988...	0.272986941
35.9988...	0.345472493
40.4988...	0.421763025
44.9988...	0.499980012

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



# STRESS Analysis

## DIFFRAC.LEPTOS - Stress Tensor only positive $\psi$

Sample: W Ar Sputtered on Glass

Instrument: N5

Radius: 166.5mm

Tube: Cu

Generator: 40kV/15mA

PrimSoller: 2.5deg

Divergence Slit: 0.2mm

Phi  
1) 0°/180°  
2) 45°/225°  
3) 90°/270°

SecSoller: 2.5deg

LE-XE-T, HRM, 1D 4.95mm

Universal Stage with Phi Attachment

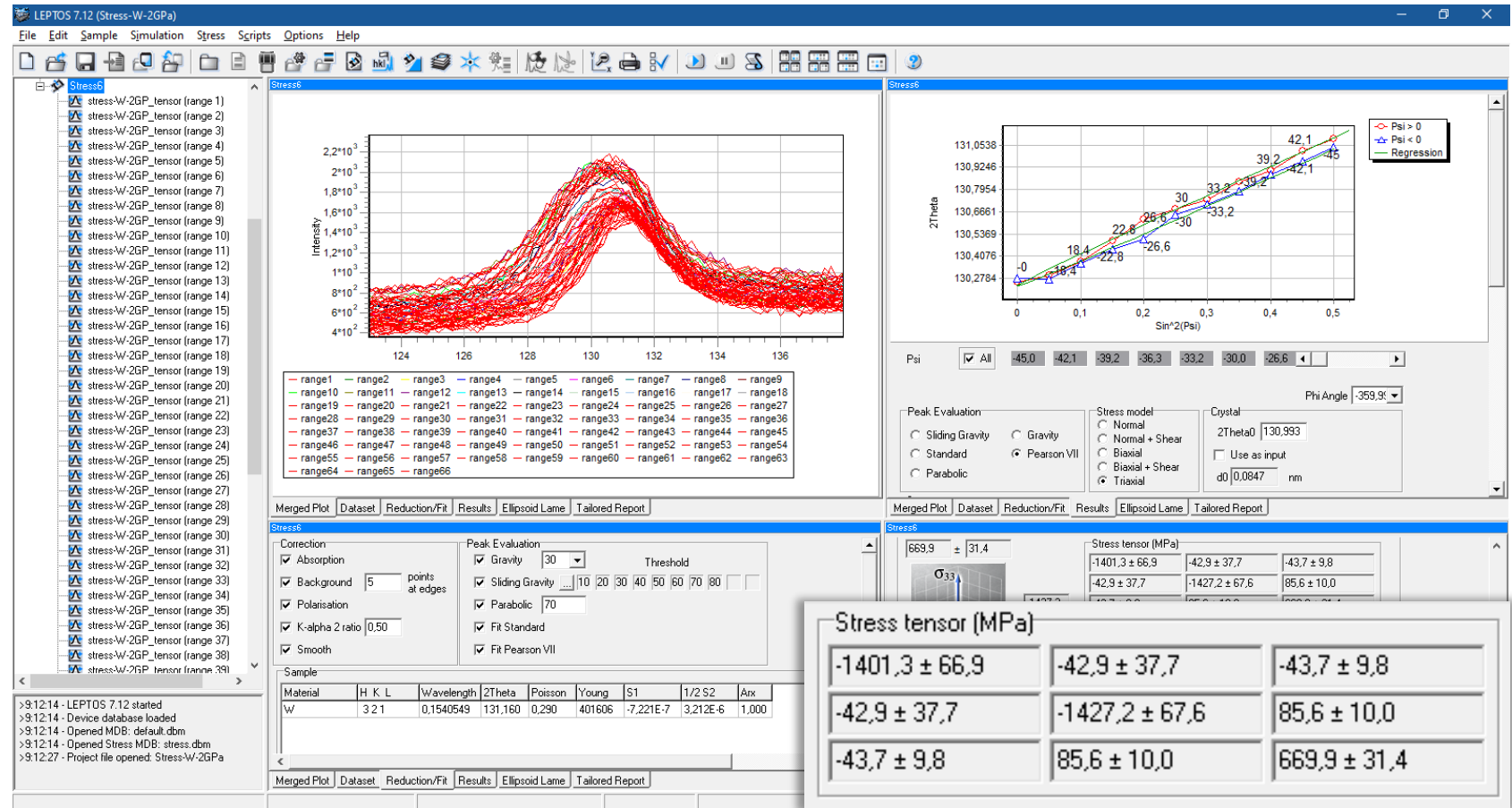
Scan: 123-138°2 $\theta$

Psi tilt: +45°, 11 steps

Incr: 0.2°2 $\theta$

Time: 2s/step

Total: 66x152s



# Data collection – Parallel Beam Geometry

## DIFFRAC.WIZARD

Sample: W Kr Sputtered on Glass

Instrument: N6

Radius: 166.5mm

Tube: Cu

Generator: 40kV/30mA

PrimSoller: 2.5deg

Divergence Slit: 0.2 mm

SecSoller: equatorial soller 0.4° fully open

Universal Stage

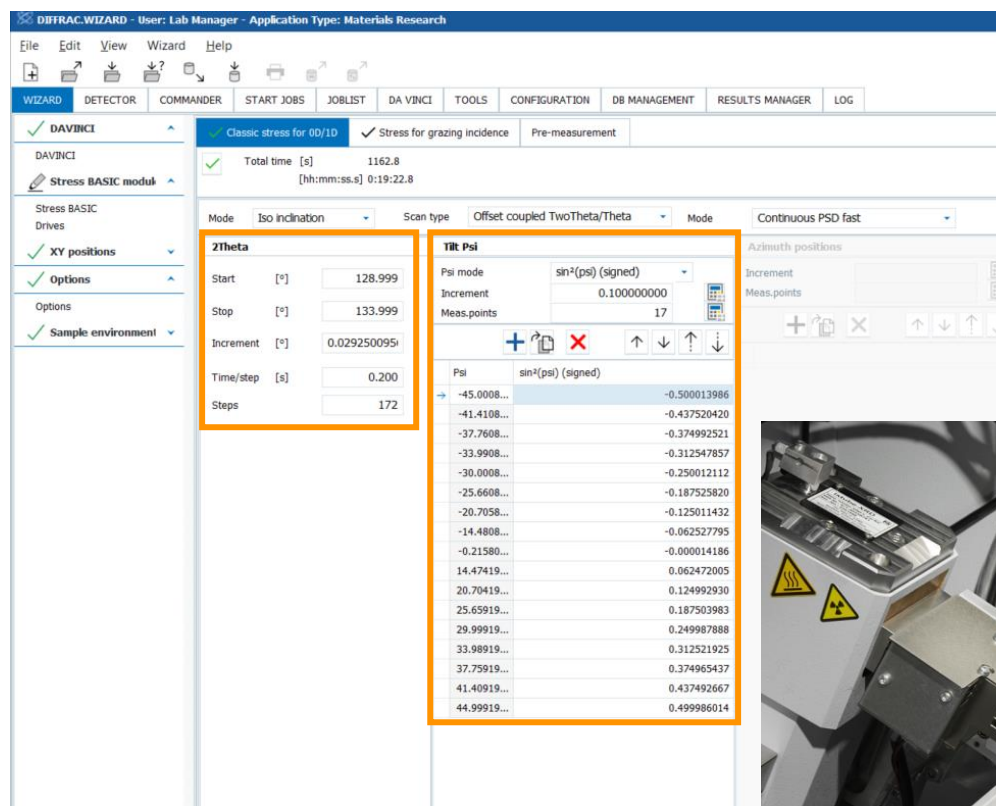
Scan: 129-134° 2 $\theta$

Psi tilt:  $\pm 45^\circ$ , 17 steps

Incr: 0.2° 2 $\theta$

Time: 2s/step

Total: 17 min



DIFFRAC.WIZARD - User: Lab Manager - Application Type: Materials Research

File Edit View Wizard Help

WIZARD DETECTOR COMMANDER START JOBS JOBLIST DA VINCI TOOLS CONFIGURATION DB MANAGEMENT RESULTS MANAGER LOG

DAVINCI

Stress BASIC modul

Stress BASIC Drives

XY positions

Options

Sample environment

Classic stress for 0D/1D Stress for grazing incidence Pre-measurement

Total time [s] 1162.8 [hh:mm:ss.s] 0:19:22.8

Mode Iso inclination Scan type Offset coupled TwoTheta/Theta Mode Continuous PSD fast

**2theta**

Start [°] 128.999

Stop [°] 133.999

Increment [°] 0.029250095

Time/step [s] 0.200

Steps 172

**Tilt Psi**

Psi mode sin<sup>2</sup>(psi) (signed)

Increment 0.100000000

Meas.points 17

Psi	sin <sup>2</sup> (psi) (signed)
-45.0008...	-0.500013986
-41.4108...	-0.437520420
-37.7608...	-0.374992521
-33.9908...	-0.312547857
-30.0008...	-0.250012112
-25.6608...	-0.187525820
-20.7058...	-0.125011432
-14.4808...	-0.062527795
-0.21580...	-0.000014186
14.47419...	0.062472005
20.70419...	0.124992930
25.65919...	0.187503983
29.99919...	0.249987888
33.98919...	0.312521925
37.75919...	0.374965437
41.40919...	0.437492667
44.99919...	0.499986014

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





# Data collection – Parallel Beam Geometry

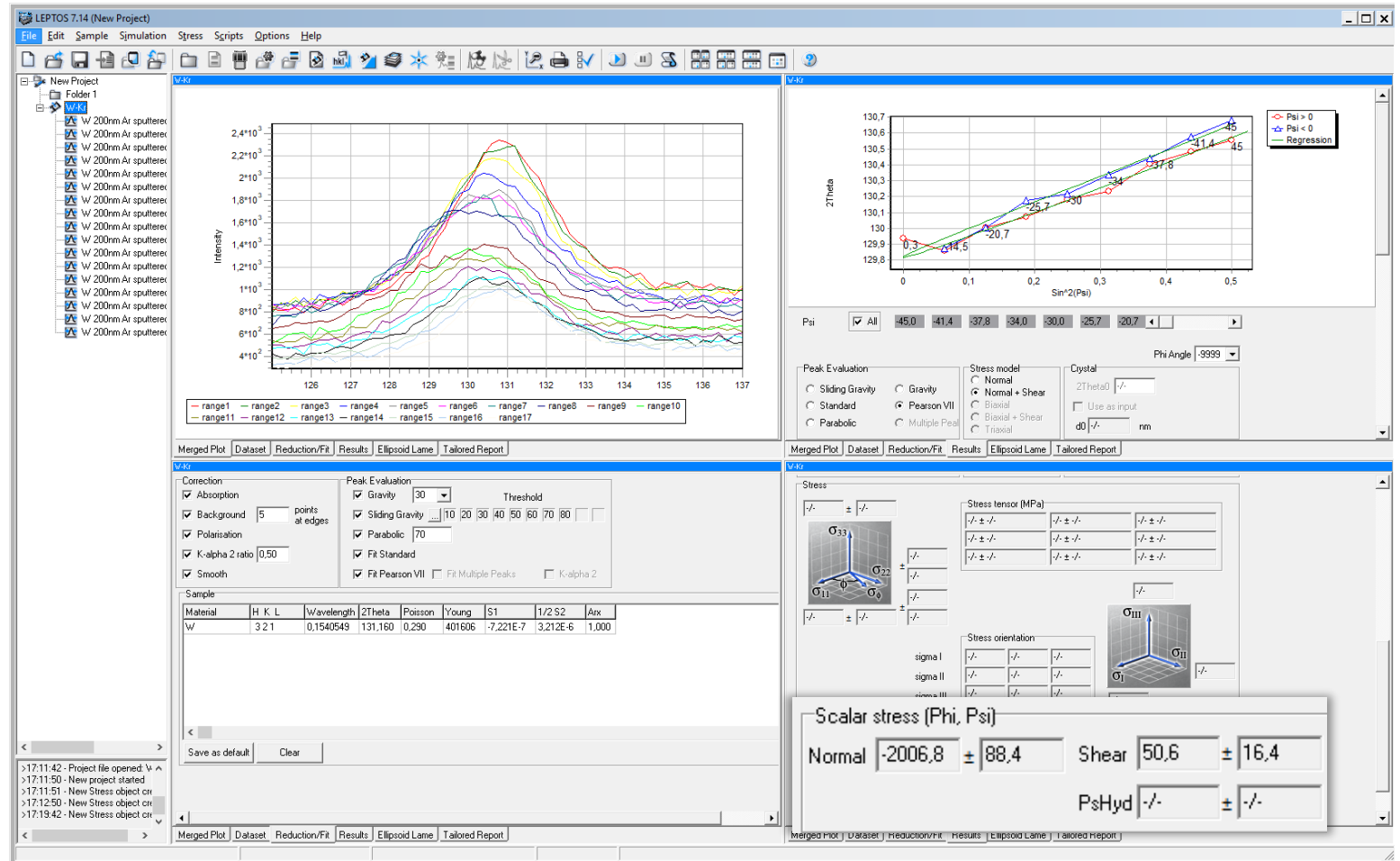
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SecSoller: equatorial soller 0.4°  
LE-XE-T, HRM, OD fully open

Universal Stage

Scan: 129-134° 2 $\theta$   
Psi tilt:  $\pm 45^\circ$ , 17 steps  
Incr: 0.2° 2 $\theta$   
Time: 2s/step  
Total: 17 min



# MATERIALS RESEARCH

## Residual Stress Analysis

- Stress-free sample (W-Powder)  
Results according EN 15305
- W Thin Film  
(one sputtered under Ar-atmosphere the other under Kr-atmosphere)
- 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.



Figure 1  
Universal stage with spring sample holder



The  $\{321\}$  reflection of tungsten was chosen due to its high angle,  $131^\circ 2\theta$  for Cu radiation, resulting in high sensitivity to d-spacing changes. For the ISO-inclination method 17 psi steps were chosen between  $-45^\circ$  and  $+45^\circ$  with a constant step size in  $\sin^2\psi$ .

The Cu source was operated at 40kV/30mA, while the divergence was controlled with a  $0.2\text{mm}$  slit and a  $2.5^\circ$  axial Soller. The LYNXEYE XE-T detector was used in high count rate mode with a  $5^\circ$  detector opening. Additionally, a  $0.2\text{mm}$  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  $\pm 3\text{MPa}$  with an uncertainty of  $\pm 31\text{MPa}$  and a shear stress smaller than  $\pm 15.6\text{MPa}$  with uncertainty of  $15.6\text{MPa}$ .

Figure 2  
Measurement geometry for residual stress measurements in the D6 PHASER. Shown is the optional phi attachment which enables biaxial stress measurements

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

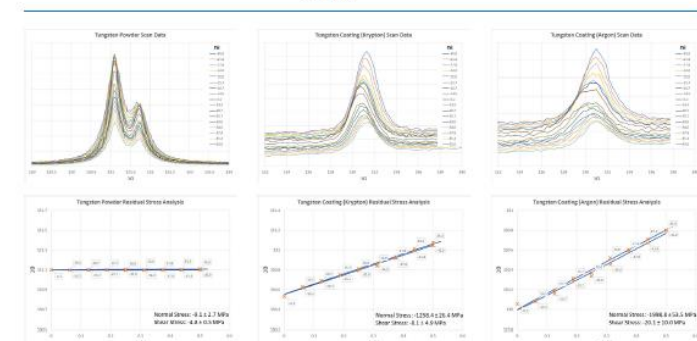


Figure 3  
Raw scan data (Top) and  $\sin^2\psi$  analysis (Bottom) of stress-free tungsten powder (Left) and two tungsten coatings deposited under krypton (Middle) and argon (Right) atmospheres.

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bruker.com

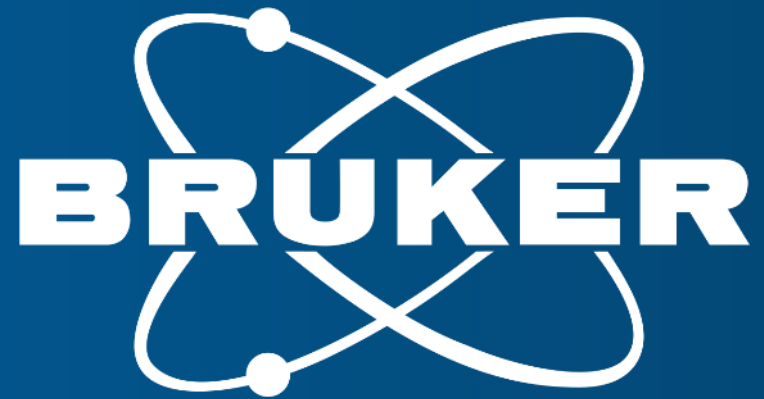
# RESIDUAL STRESS ANALYSIS with D6

## Conclusion

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- 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