X-RAY DIFFRACTION

D6 PHASER – Benchtop XRD 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.

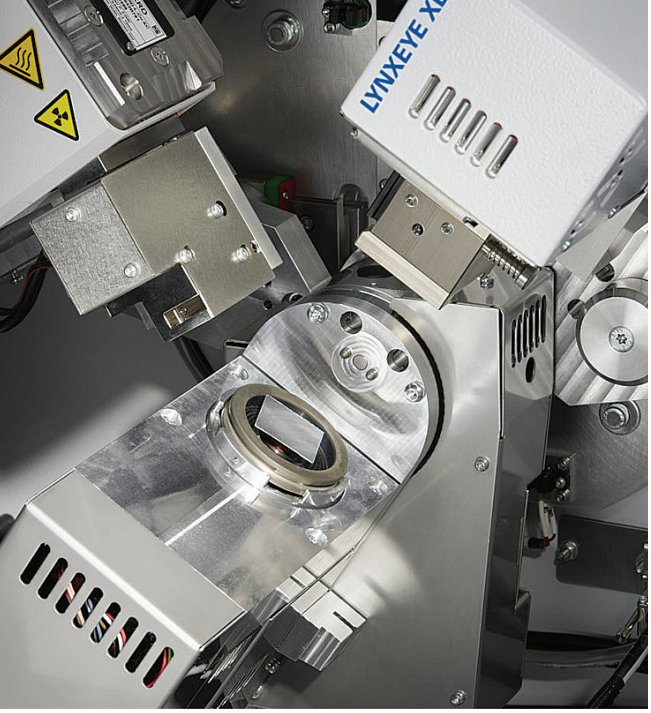

Figure 2
Measurement geometry for residual stress measurements in the D6 PHASER. Shown is the optional phi attachment which enables biaxial stress measurements.

The 321 reflection of tungsten was chosen due to its high angle, 131° 2θ for Cu radiation, resulting in high sensitivity to d-spacing changes. For the ISO-inclination method 17 psi steps were measured between -45° and +45° 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 mm slit and a 2.5° axial Soller. The LYNXEYE XE-T detector was used in high count rate mode with a 5° detector opening. Additionally, a 0.2 mm Ni filter to surpress Kß diffraction peaks 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 +/-31 MPa with an uncertainty of +/- 31 MPa and a shear stress smaller than +/- 15.6 MPa with uncertainty of 15.6 MPa.

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.

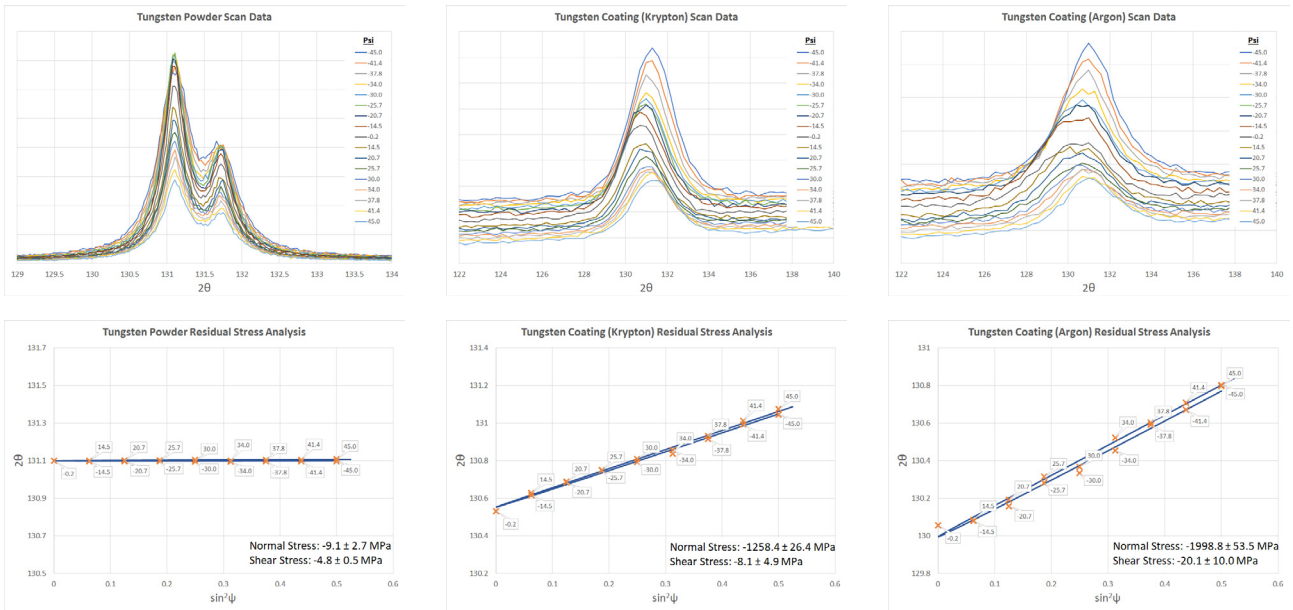

Figure 3
Raw scan data (Top) and $\sin^2\Psi$ analysis (Bottom) of stress-free tungsten powder (Left) and two tungsten coatings deposited under krypton (Middle) and argon (Right) atmospheres.

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