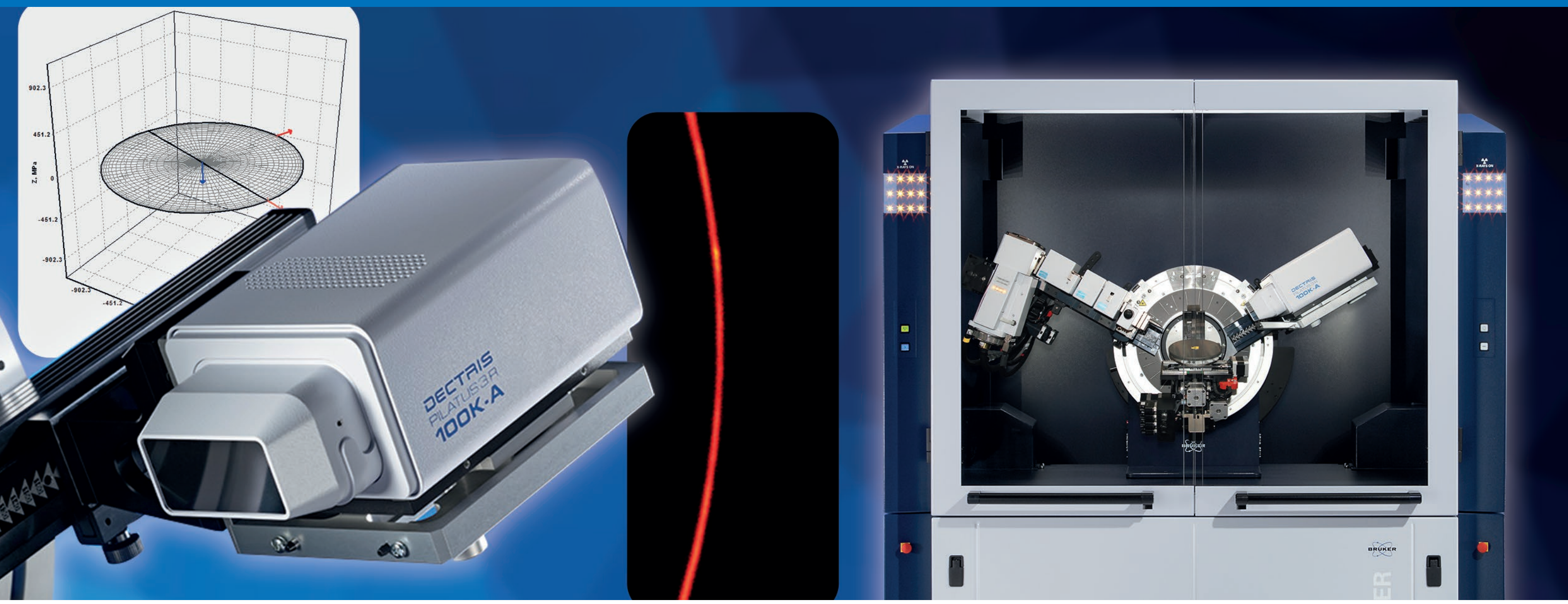


Application Report XRD 24

# D8 DISCOVER with PILATUS3 R 100K-A Residual Stress Analysis with XRD²

**The D8 family of diffraction solutions combined with the DECTRIS PILATUS3 R 100K-A detector is an innovative two dimensional x-ray diffraction (XRD²) solution that is uniquely suited for multipurpose modern materials research characterization. In this report, we present the capabilities of this system in a reflection diffraction configuration for residual stress analysis.**

## Introduction

Residual Stress is the strain remaining in a material when no active load is being applied. These strains can be present due to mechanical deformation, such as the process of machining a surface, or kinetically limited heating and cooling. Often times, it is beneficial to produce a compressive residual stress in a surface to increase resistance to fracture propagation. In coatings with a tensile residual stress present, a small scratch can lead to cracking and delamination, while a coating with a compressive stress would inhibit propagation of the fracture.

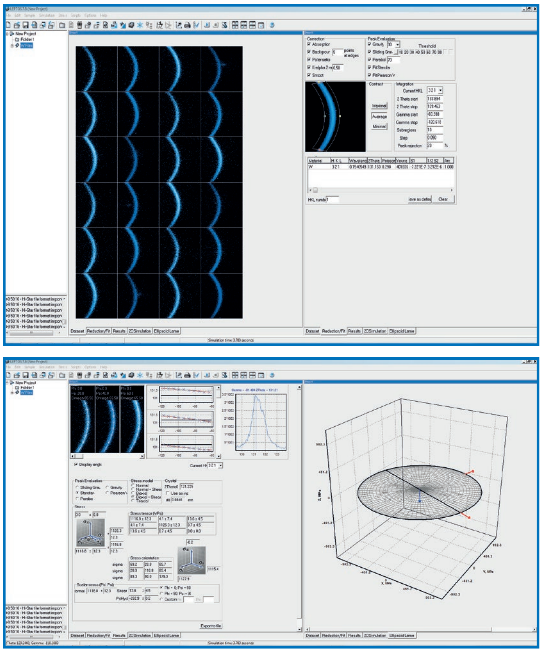

Figure 1: Residual Stress Analysis in DIFFRAC.LEPTOS. Analysis Setup (top), Analysis Result (bottom).

## Measurement

The sample consists of a 250 nm thin film of tungsten on a single crystal silicon substrate. The sample was measured with a D8 DISCOVER equipped with a unique copper IμS micro-focus source, 300 micron collimator, a centric Eulerian cradle and PILATUS3 detector positioned 10 cm from the sample. The detector was mounted in a gamma optimized orientation to increase the tilt coverage of the measurement.

The (321) reflection of tungsten was chosen due to its high angle, 131° $2\theta$ for Cu radiation, resulting in high sensitivity to d-spacing changes. To facilitate calculation of the full stress tensor, images were collected at $\phi = 0°$ to 315° in 45° increments, with $\Psi = 20°, 40°, 60°$. Each image was collected for 10 seconds, resulting in a total collection time of 5 minutes. The measurement was planned using the WIZARD plugin of DIFFRAC.MEASUREMENT.

## Results

Analysis was performed in DIFFRAC.LEPTOS. The raw images are directly loaded and basic information about the sample entered, shown in Figure 1. In order to facilitate accurate entry of the x-ray elastic constants, an extendable database for the x-ray elastic constants containing the commonly used materials is included in DIFFRAC.LEPTOS.

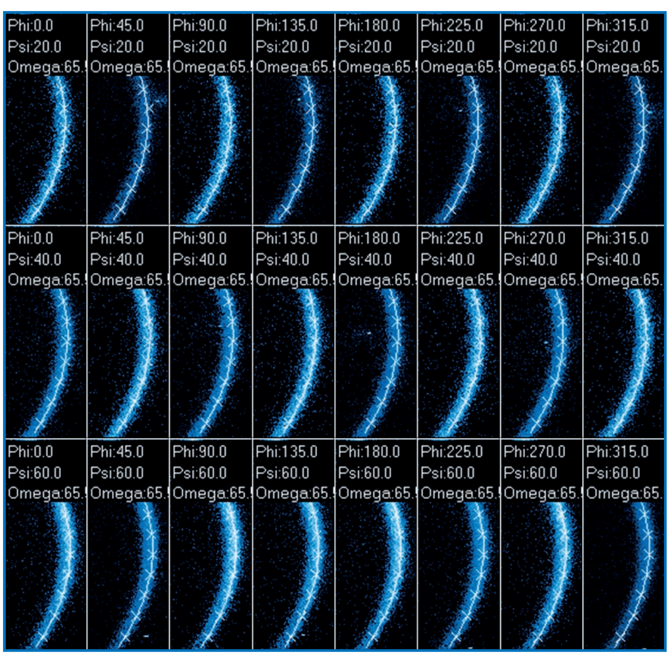


Figure 2: Fit of the 2 dimensional debye rings of the tungsten film. The white line represents the model and the white X's the actual peak positions.

The data was fit using a standard peak shape and a biaxial with shear model of the sample. DIFFRAC.LEPTOS utilizes the general model for residual stress, derived from the diffraction vector approach. Details of the method can be found in reference [1]. The result of the fit can be seen in Table 1 and Figures 2 and 3. As expected, a planar tensile stress, with $\sigma 11 = \sigma 22 = 1120$ MPa is found with error ~1%. Tensile stresses in surface coatings are not desirable due to the risk of fracture leading to delamination. By adjusting deposition parameters, such as the temperature of the sample or the pressure in the deposition chamber, the tensile stress can be lowered, or turned compressive.

By using the D8 Diffractometer with PILATUS3 detector for XRD² combined with DIFFRAC.MEASUREMENT and DIFFRAC.LEPTOS, residual stress data can be collected and analyzed rapidly with high precision.

## Author

Jon Giencke

## References

[1] Bob B. He, Two-dimensional X-ray Diffraction, John Wiley & Sons (2009)

| Biaxial with Shear Residual Stress Tensor (MPa) | | |
|---|---|---|
| 1116.8 ± 12.3 | 4.1 ± 7.4 | 13.6 ± 4.5 |
| 4.1 ± 7.4 | 1126.3 ± 12.3 | 0.7 ± 4.5 |
| 13.6 ± 4.5 | 0.7 ± 4.5 | 0.0 ± 0.0 |

Table 1. Calculated Biaxial with shear residual stress tensor for the tungsten film.

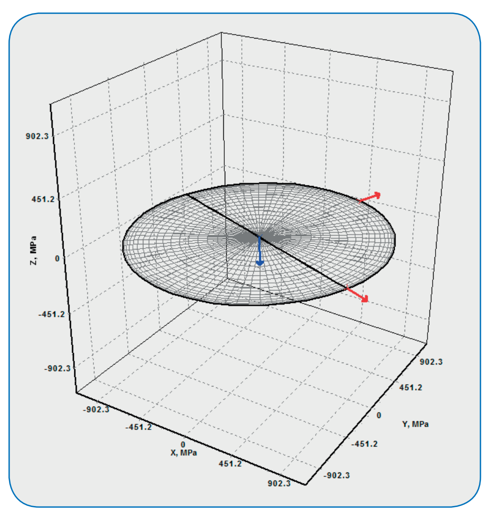


Figure 3: Three dimensional stress ellipsoid representation of the orientation of the stress in the tungsten film.

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