

Application Note XRD 614

Residual Stress with EIGER2 R 500K

- 0D, 1D, and 2D investigation of a polycrystalline tungsten coating on silicon

Many manufacturing processes leave residual stresses which can affect the performance of manufactured components. Compressive stress can be engineered into a metal coating to resist crack propagation, while tensile stress can be exploited to enhance conductivity in semiconductors. Strained materials exhibit changes in atomic spacing which can be detected by X-ray diffraction (XRD) and related to the stress via elastic constants.

Three common methods for measuring residual stress are the $\sin^2(\psi)$ method, the multi-HKL method, and the 2D method. The EIGER2 R 500K is a versatile hybrid photon counting X-ray detector which can be operated in 0D, 1D, and 2D modes, allowing measurements in all three ways. In this report we examine a 500 nm polycrystalline tungsten coating on a silicon wafer.

Report Summary

- All three methods yielded a similar result from the W film
 - Grazing Incidence multi-HKL method
 $\sigma_{11} = -1.8$ GPa (surface) to -2.0 GPa (200 nm depth)
 - Line beam $\sin^2(\psi)$ method
 $\sigma_{11} = \sigma_{22} = -2.0$ GPa
 - Spot beam 2D method
 $\sigma_{11} = \sigma_{22} = -2.0$ GPa
- Only change between methods was the beam masks
- Optimal method should be chosen based on the sample type and desired information

Introduction

There are three principal methods for measuring residual stress using XRD, with the optimal technique depending on the type of sample and information needed from the result. The traditional $\sin^2(\psi)$ method tracks the shift in 2θ of a single peak as the diffraction vector is tilted away from the surface normal. For large, homogeneous samples this can be done in iso-inclination mode with a line-focus beam and a 1D detector for fast data collection. This can also be performed in spot focus in either iso-inclination or side-inclination mode, with increased measurement time commensurate with the reduced flux.

A variation of the $\sin^2(\psi)$ technique is the multi-HKL method, in which the source is held at a fixed angle relative to the sample surface while the detector is scanned over a wide range. Each peak gives a measure of the stress in a different tilt of the diffraction vector, allowing stress to be calculated. Since the incident angle can be fixed at a grazing angle, this technique is well suited for thin films and depth profiling.

A third way to measure stress is the 2D method, in which the stress is measured with a 2D detector and calculated based on modeling of the complete Debye sphere. As this technique requires a spot focus beam it can be performed in either iso- or side-inclination mode and is well suited for measuring the local stress around small features.

Due to differences in the thermal expansion coefficient between film and substrate materials, coatings deposited on a substrate at high temperature often develop large residual stresses after cooling to room temperature. In this study we measure the residual stress of a 500 nm tungsten film on Si by all three methods using a D8 DISCOVER diffractometer equipped with Cu radiation (40 kV, 40 mA), Göbel mirror, Centric Eulerian Cradle, and EIGER R 500K detector, operating in 0D, 1D, and 2D modes.

Multi-HKL Method: Stress Depth Profile using GID Line Focus – 0D Detector Mode

For the multi-HKL method a 0.2 mm aperture slit, 0.4° equatorial Soller, and 0D detector mode was used. A series of 2θ scans from 35° to 135° were performed, with the incident angle varying from 0.6° to 5° . Data were brought into LEPTOS S where the stress was calculated as a function of depth, assuming a linear attenuation coefficient of $0.324 \mu\text{m}^{-1}$, a total external reflection angle of 0.55° , and a film thickness of 500 nm.

As seen in figure 3, the stress is fairly uniform near the surface, trending toward a bulk value of approximately -2.0 GPa.

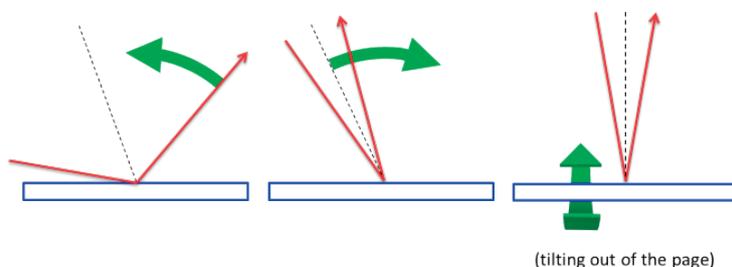


Figure 1. Three strategies for measuring residual stress used in this study: 0D Multi-HKL (Left), 1D $\sin^2(\psi)$ (Middle), 2D Method (Right)

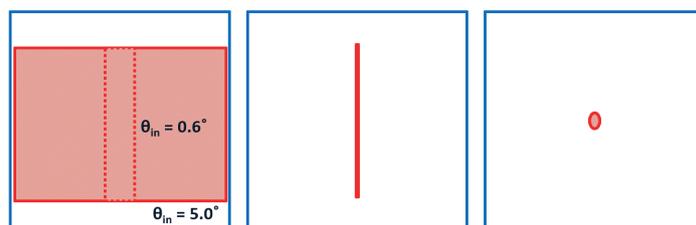


Figure 2. Comparison of the beam footprint for Multi-HKL (Left), $\sin^2(\psi)$ (Middle) and 2D Methods (Right)

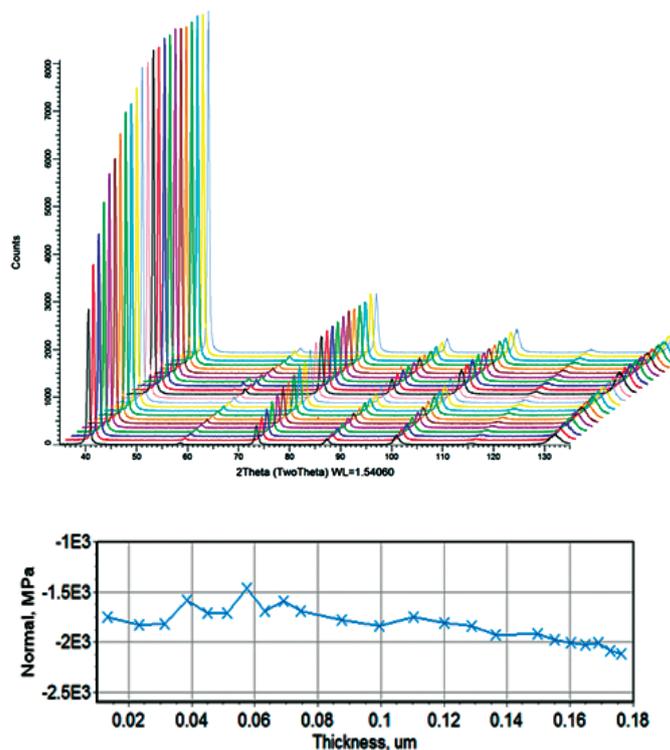


Figure 3. (Top) 2θ scans in grazing incidence mode. (Bottom) Normal stress as a function of depth in the tungsten film.

Sin²(ψ) Method

Line Focus – 1D Detector Mode

For the sin²(ψ) method a 0.2 mm aperture slit and a 1D detector mode covering 14° 2θ were used. Data were collected in iso-inclination mode, using 10 sec 1D still scans of the (321) W reflection taken at equal steps of 0.05 in sin²(ψ) from -0.5 to +0.5, at φ = 0°, 45°, and 90°. Total collection time was 10 min.

1D snapshot scan data for φ = 0° are shown in figure 4, showing a clear 2θ shift from sin²(ψ) = -0.5 to +0.5. The biaxial stress tensor indicates a uniform compressive stress of -2.0 GPa, with no significant shear. This result is in agreement with the bulk value of the film, as shown in the grazing incidence depth profile (fig. 3).

2D Method

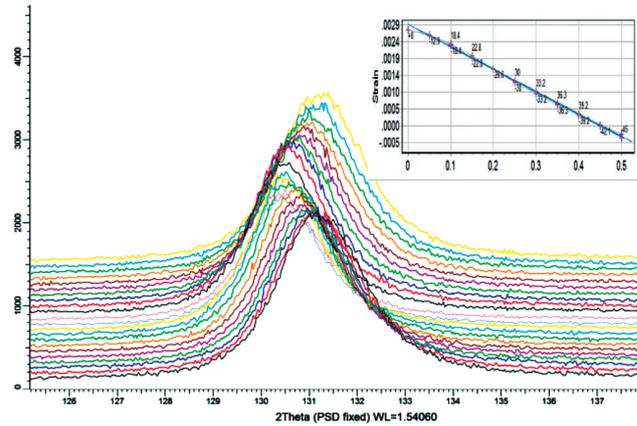
Point Focus – 2D Detector Mode

For the 2D method a 1.0 mm micromask, 1.0 mm magnetic collimator, and 2D detector mode were used. Data were collected in side-inclination mode using 25 sec frames of the (321) W reflection taken at 4 steps in ψ from 5° to 45°, at φ = 0°, 45°, 90°, 180°, 225°, and 270°. Total collection time was 10 min.

2D frame data for φ = 0° are shown in figure 5, overlaid with the fit result representing the distorted Debye ring. The biaxial stress tensor indicates a uniform compressive stress of -2.0 GPa with no significant shear. This result represents a local measurement, while the line-focus data represent the average over a large area; the close agreement between the two indicates a uniform stress over the film surface.

Conclusion

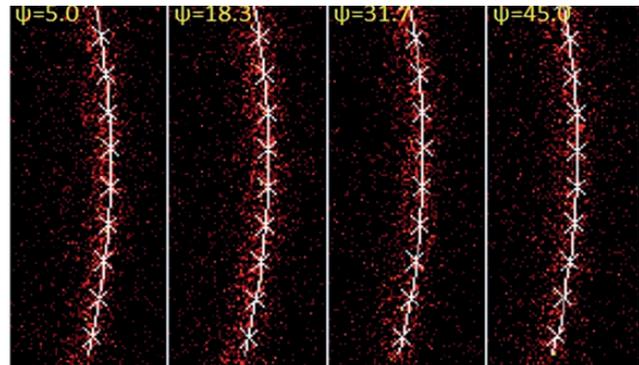
Stress analysis was performed on a 500 nm tungsten film following the multi-HKL (0D), sin²(ψ), and 2D methods, with a bulk value of -2.0 GPa obtained by all three methods. This close agreement gives confidence that the user is free to select the technique that best matches the type of sample being measured, without having to reconfigure or recalibrate the instrument.



Stress tensor (MPa)

-1995.3 ± 23.8	65.5 ± 20.5	-3.8 ± 5.3
65.5 ± 20.5	-2008.6 ± 23.8	6.3 ± 5.3
-3.8 ± 5.3	6.3 ± 5.3	0.0 ± 0.0

Figure 4. Stress result as measured by the traditional sin²(ψ) method. (Top) 1D snapshot scans for φ = 0°. Inset shows strain vs. sin²(ψ) plot for these scans. (Bottom) Measured biaxial stress tensor in the lab coordinates.



Stress tensor (MPa)

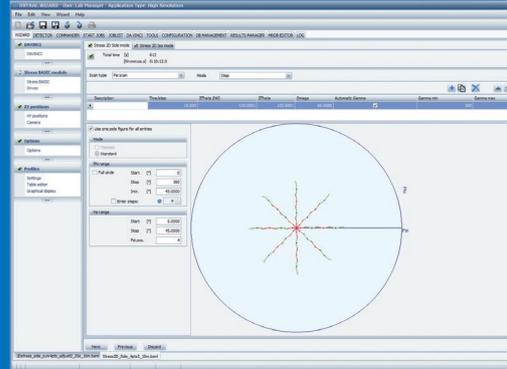
-2027.3 ± 19.2	10.3 ± 19.5	-1.6 ± 5.6
10.3 ± 19.5	-2027.7 ± 19.1	-0.1 ± 5.5
-1.6 ± 5.6	-0.1 ± 5.5	0.0 ± 0.0

Figure 5. Stress result as measured by the 2D method. (Top) 2D frames and the fit to the data for φ = 0°. (Bottom) Measured biaxial stress tensor in the lab coordinates.

DIFFRAC.SUITE Workflow for Residual Stress

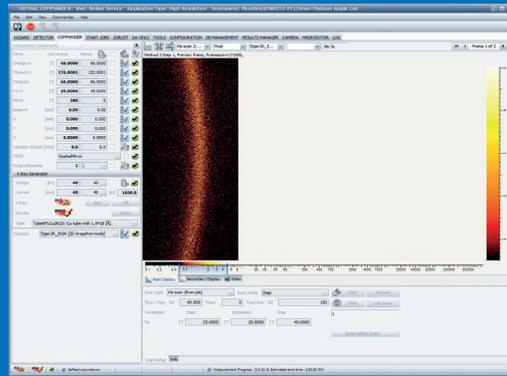
PLAN in DIFFRAC.WIZARD

- Measurement scheme optimization for 0D, 1D, and 2D detector modes
- Preview γ and 2θ coverage for a given detector distance and goniometer position
- Setup mapping experiments at specific locations or at a regular grid of points



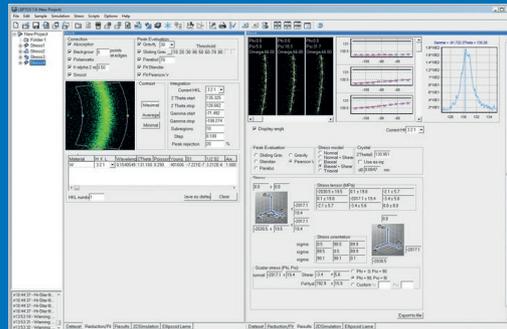
MEASURE in DIFFRAC.COMMANDER

- Direct measurement control or launch predefined experiment methods
- Real-time data monitoring
- Calibrated iso-lines cursor for real time observation



ANALYZE in DIFFRAC.LEPTOS

- Supports 0D, 1D, and 2D stress evaluation
- Single-HKL and multi-HKL methods
- Stress depth profiling and mapping
- Clear, concise report generation



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