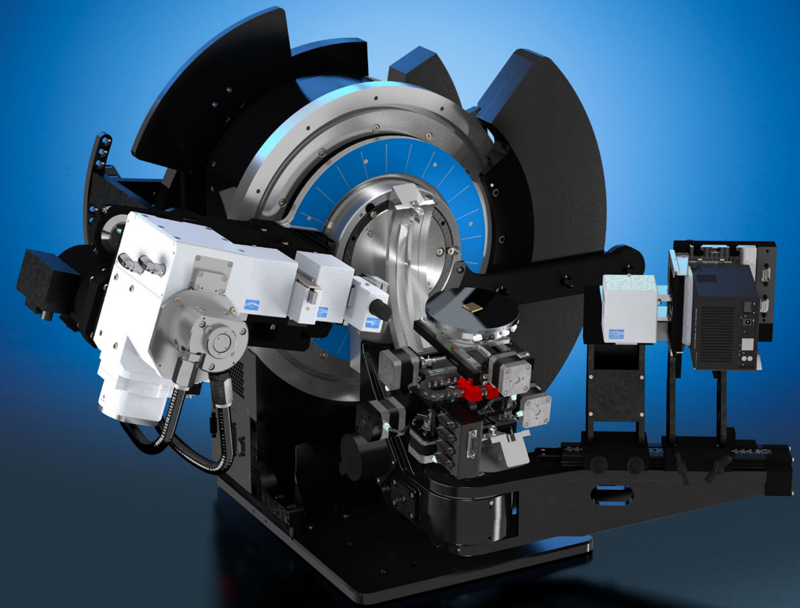


X-RAY DIFFRACTION

Characterization of ultra-thin metallic film using incidence diffraction geometries

Application Note 623

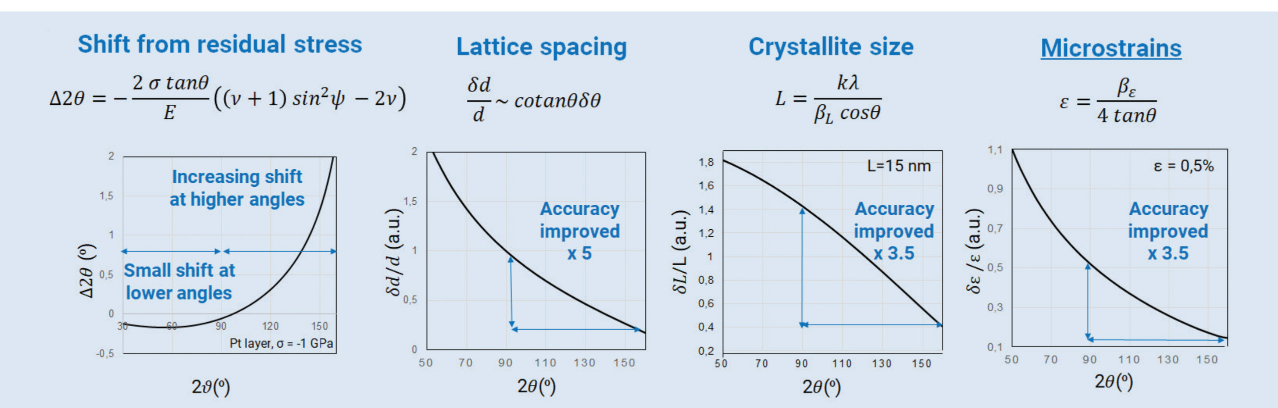
Introduction

For thin films, and down to ultra-thin films (<10 nm), the accessible diffraction volume is so limited that symmetric diffraction geometries (e.g. Bragg-Brentano) have reached their limits in providing useable data, due to a penetration depth of X-ray widely exceeding the film thickness.

Grazing incidence diffraction techniques become more appropriate and enable the enhancement of the signal through the control of the penetration depth. The confinement of the material into thin or ultra-thin films leads to highly anisotropic properties and favours the development of preferred orientation, residual stress, microstrain, or anisotropic crystallite size. Coplanar and non-coplanar grazing incidence geometries probe film properties in different directions and provide therefore complementary information for a better understanding of the film structure.

Why acquiring patterns on a wide angular range?

Grazing incidence experiments are often acquired on a limited angular range that provides however a number of diffraction peaks that is sufficient for the identification of the phase(s) that formed. Extending data acquisition up to higher angles is nevertheless mandatory to evidence and quantify residual stress in the film and significantly improves the accuracy on (micro)structural parameters.



Experimental details

A 10 nm ultra-thin metallic film of Pt on Si substrate has been investigated using a D8 DISCOVER Plus diffractometer equipped with an ATLAS™ goniometer and a non-coplanar arm. Angular resolution was achieved using 0,5° soller slits. Diffraction patterns were collected in both coplanar and non-coplanar diffraction geometries up to 160° to reach best accuracy on the evaluated parameters (highest accessible 2θ depending on configuration).

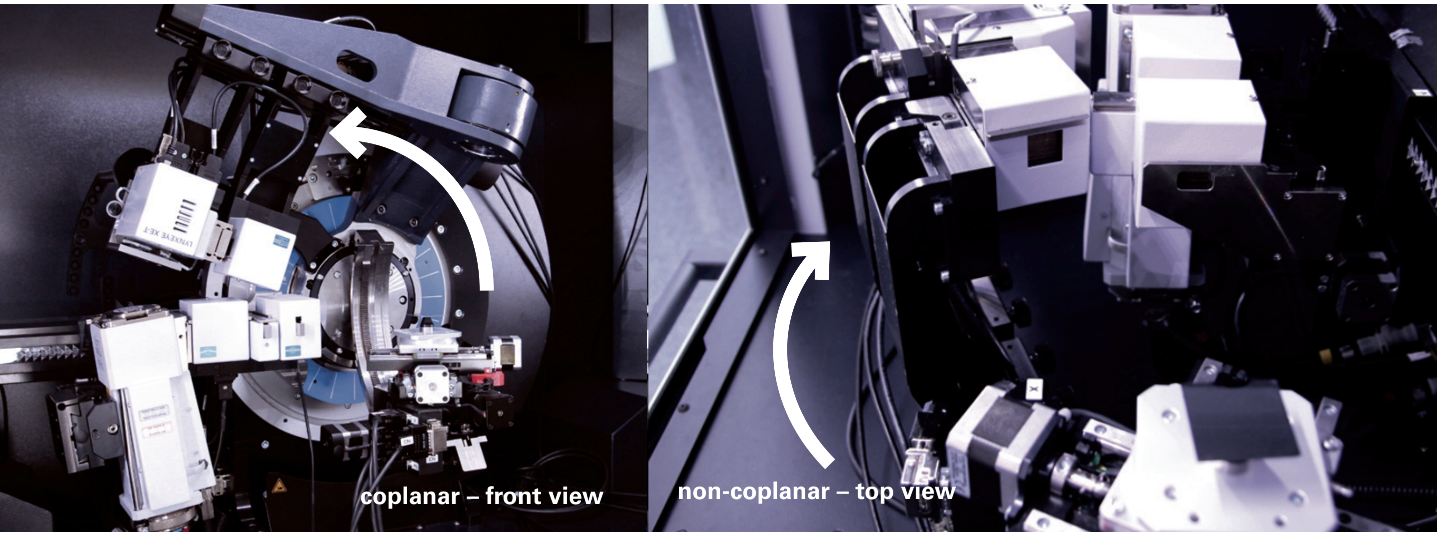


Fig. 1 Final drive positions in coplanar and non-coplanar scans.

Qualitative results

Diffraction peaks in the first half of the coplanar scan decently match the reference pattern of Pt, but significant deviations are observed at higher angles. Refining the lattice parameter does not lead to a satisfactory fit, which evidences the presence of residual stress. In fact, the position of the peaks is ruled by compounding influences having different angular dependencies: the surface refraction inducing a constant peak shift, the stress-free lattice parameter following Bragg's Law and residual stress following the law of theory of elasticity.

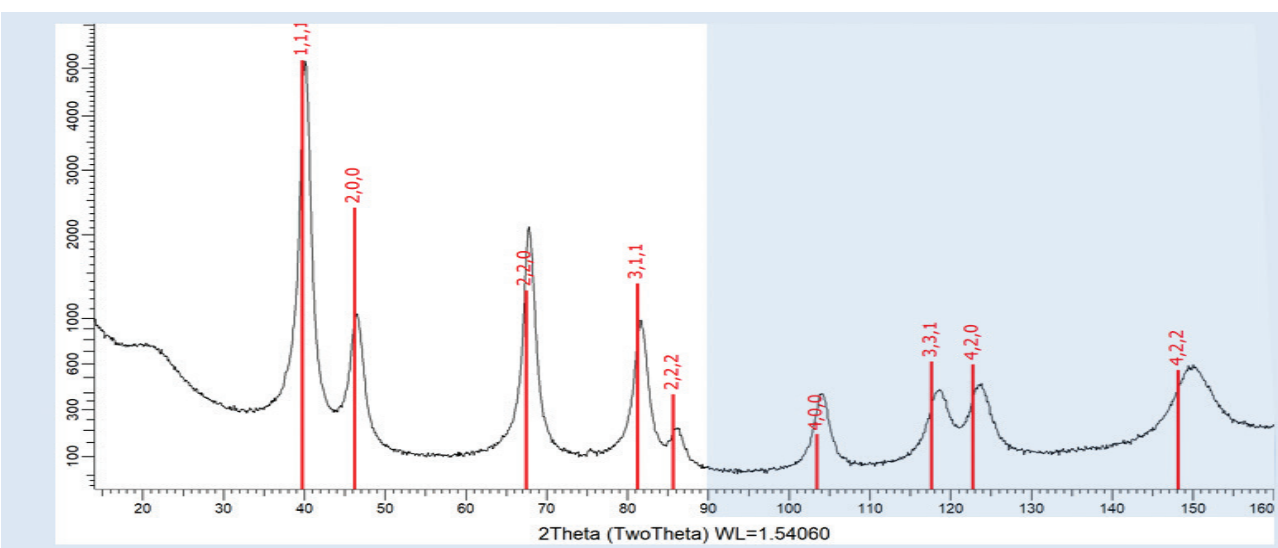
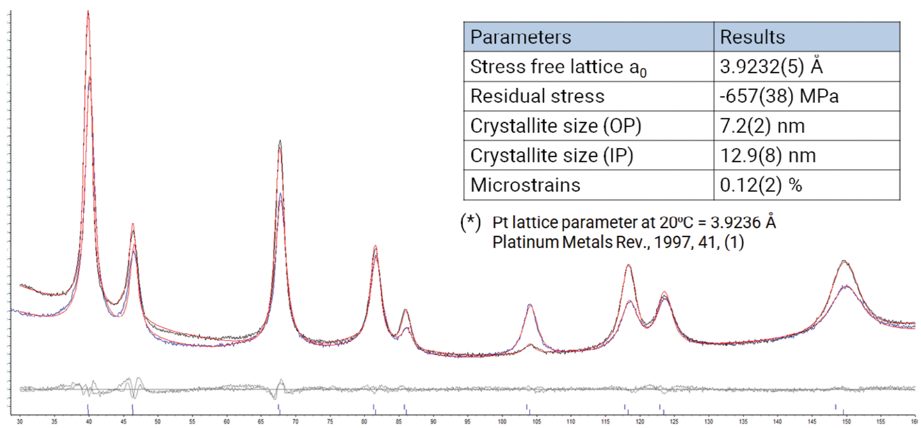


Fig. 2 Scan region with white background represents the typical GID scan range.
Increased accuracy is achieved by extending the measurent range (grey region).

Refinement model

The dataset from both coplanar and non-coplanar geometries are refined simultaneously using a Whole Powder Pattern Fitting (WPPF) approach in DIFFRAC.TOPAS. The wide angular range facilitates the separation of the various contributions to the peak positions using the following model with only 5 refinable parameters:

- the surface refraction reduces the effective penetration angle in coplanar grazing incidence and shifts all peaks towards higher angles by $\theta_i - (\theta_i^2 - \theta_c^2)^{½}$, where θ_i = incident angle, θ_c = critical angle of Pt. The refraction correction does not apply for in-plane diffraction.
- the peaks of cubic Pt are generated by a (hkl) phase in the space group Fm-3m with a refinable stress-free lattice parameter a_0.
- assuming a biaxial strain field uniform in-plane, the residual stress σ induces in coplanar geometry an angular shift $(-2 \tan\theta/E)\ ((\nu+1)\sin^2\Psi-2\nu)$ where E = Young modulus, ν = Poisson ratio and $\Psi = \theta_{hkl} - \theta_i$. In-plane, the atomic planes are uniformly strained and the lattice parameter is refined by $a_0 + \sigma\ (1-\nu)/E$.
- In-plane <L-Vol>$_{IP}$ and out-of-plane <L-Vol>$_{OP}$ crystallite sizes are refined independently and a microstrain ε_0 is a common parameter in both geometries.



Parameters	Results
Stress free lattice a_0	3.9232(5) Å
Residual stress	-657(38) MPa
Crystallite size (OP)	7.2(2) nm
Crystallite size (IP)	12.9(8) nm
Microstrains	0.12(2) %

Fig. 3 Simultaneous WPPF refinement of coplanar and non-coplanar measurements in DIFFRAC.TOPAS.

Conclusions

- The ATLAS™ goniometer enables high angle data acquisition for an accurate evaluation of structural parameters in thin films. Coplanar and non-coplanar geometries nicely complement each other for a better understanding of the film structure.
- A consistent model implemented in TOPAS discriminates the contribution of the residual stress and the stress-free lattice parameter to the peak positions, and the microstructural parameters to the peak width.

Bruker AXS
info.baxs@bruker.com

Worldwide offices
bruker.com/baxs-offices

Online information
bruker.com/xrd

bruker.com