A new device for fast and accurate X-ray texture analysis

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For X-ray diffraction experiments involving continuous tilting of a sample using Eulerian cradles, beam defocusing and over-irradiation of the sample during the measurements are important factors influencing data quality as well as the quality of analysis results. Applications that are mainly affected are e.g. Texture and Residual stress measurements during which samples are positioned to pre-calculated χ-positions, while scans are performed in φ, 2θ or ω-2θ. Conventionally, to avoid defocusing and over-irradiation, small beam dimensions have to be used. However, this also results in a significant loss of measurement intensity and hence measurement speed. In this contribution, we present a new, proprietary beam-shaping device that mitigates both, defocusing and over-irradiation, in combination with significantly larger beam dimensions. The results presented in this poster show the significant improvement of measurement intensity/speed, as well as counting and particle statistics.

Introduction

The collection of pole figure measurements or residual stress measurements for X-ray texture and stress analysis requires that the sample under study is tilted, in order to observe crystal orientations and strain at different orientations with respect to the normal vector of the sample. However, due to the measurement geometry, X-ray beam dimensions as well as the tilt of the sample, the shape and size of the beam projection on the sample surface will change significantly during these type of measurements. This effect is typically referred to as "defocusing". Depending on beam and sample dimensions, this defocusing effect may even result in part of the incident X-ray beam falling outside of the sample area. This effect is known as "over-irradiation" and leads to incorrect peak intensity measurements. The commonly used approach to deal with defocusing and over-irradiation is to reduce the size of the X-ray beam, so that even at high tilts the complete beam irradiates the surface of the sample. This, however, significantly reduces the incident beam flux and therefore diffraction intensity or measurement speed. In addition, the smaller spot size can have a significant negative effect on data quality and subsequent analysis due to the smaller irradiated volume and low particle statistics contributing to the overall diffraction intensity.

In this contribution we present results from various materials obtained using a new device that includes a motorized slit that "follows" the sample tilt during and during 2-axes measurements (Fig. 1). This significantly reduces defocusing and over-irradiation and provides higher beam intensity and larger irradiated surface, which in turn allows faster and more accurate measurements.

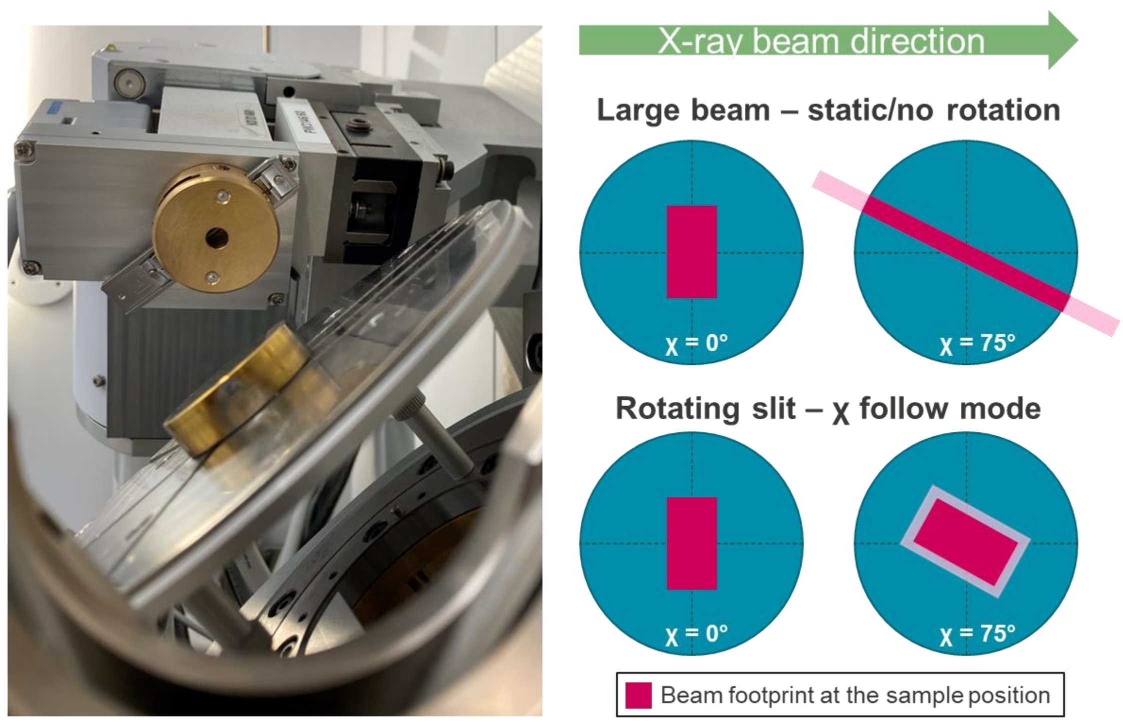

Figure 1 (left): Motorized rotatable slit during a pole figure measurement on the (111) reflection of a TiN coating on steel. The peaks collected at large tilts are broader, which is due to defocusing effects. (Right): Illustration of the resulting beam projection during tilting of a sample, with and without the new device.

Experimental

The X-ray powder diffraction was carried out using an Empyrean diffractometer with the following configuration:

Measurement configuration	
Geometry	Reflection & Parallel beam geometry
Scan modes	Symmetric & Grazing-incidence scans, Texture & Residual Stress measurements
Radius	240mm
Radiation	Cu Kα (λ = 1.54056A˚), Ni Kβ filter
Incident beam optics	X-ray Polycapillary optic, Rotating slit module
Diffracted beam optics	dCore module, Fixed anti-scatter slit
Detectors	1Der detector, Eiger2 R 500K Si
Sample stage	Three and Five-axis cradle
Samples	fluor. disc, rolled Cu, TiN on steel, Al-alloy

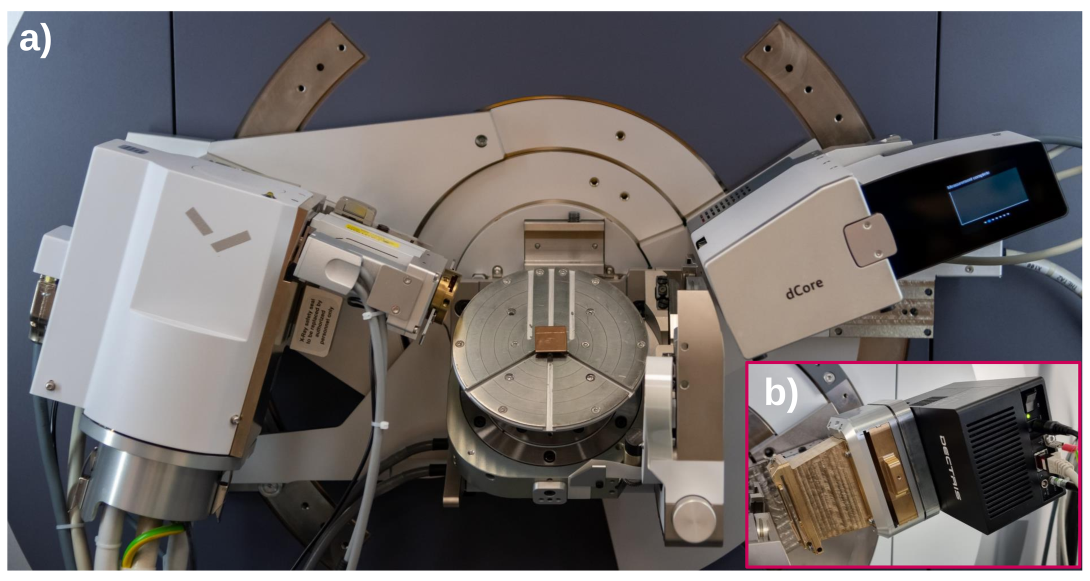

Figure 2: (a) Empyrean diffractometer equipped with the new rotating slit device, three-axis cradle, dCore optical module and a 1Der detector. (b) Diffracted beam path equipped with Eiger2 R 500k Si detector.

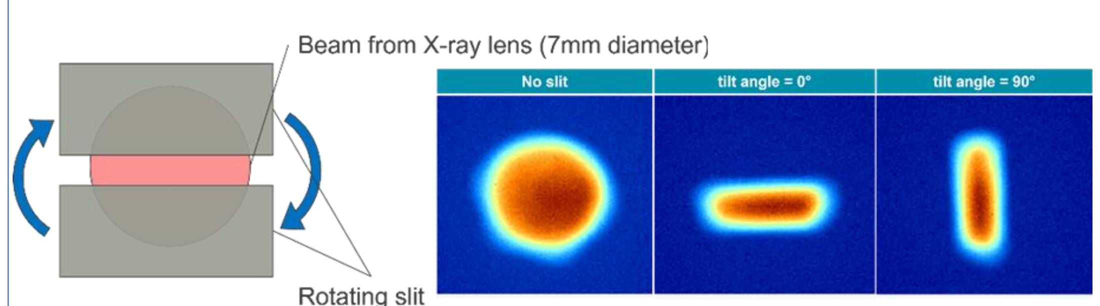

Figure 3: (a) Functionality of rotating slit and corresponding direct beam images without slit as well as a ¼° divergence slit at 0° and 90° tilt

Beam projection & defocusing with sample tilt (χ)

The footprint of the direct beam in dependence of the sample tilt, with and without the use of the rotating slit, was directly imaged using a fluorescent sample disc (Fig. 4). The incident angle (ω) for these measurements was 20° and the beam dimension was 7x1 mm². The obtained beam projections on the sample surface show a significantly higher defocusing of the beam and over-irradiation of the sample beyond χ=50° with a conventional static collimator (Fig. 4a-d). However, with the rotating collimator slit following the sample tilt ("follow mode"), defocusing is clearly contained and the full beam remains withing the sample area (Fig. 4e-f), even at sample tilts >75°χ. Even with a smaller beam, defocusing is more efficiently suppressed with the rotating slit (Fig.5).

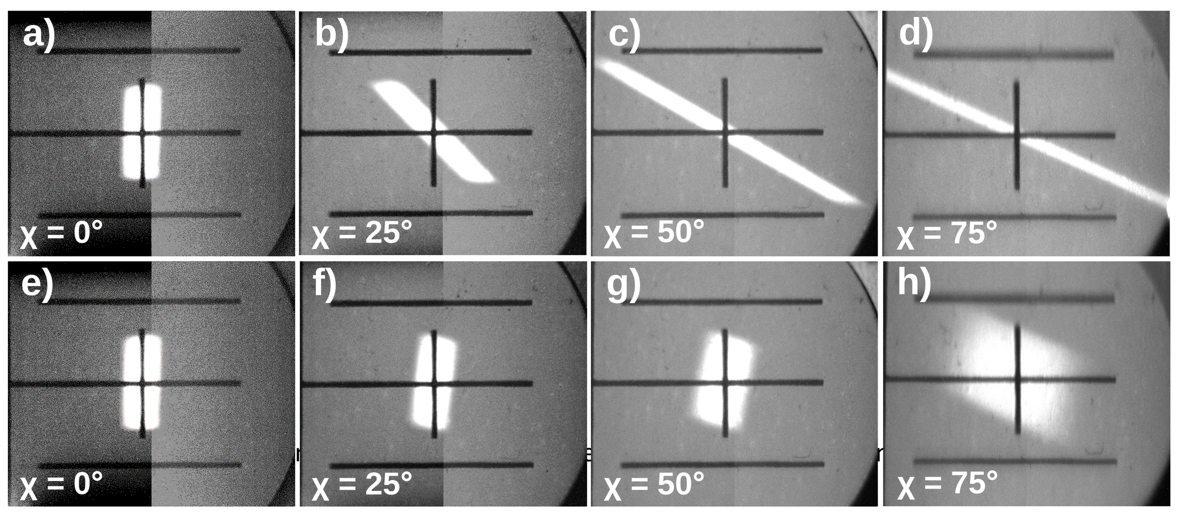

Figure 4 (a-d): Beam projections with an X-ray lens and a static collimator at χ=0°, 25°, 50°, and 75°. (e-f): Corresponding projections measured with the new rotating slit module.

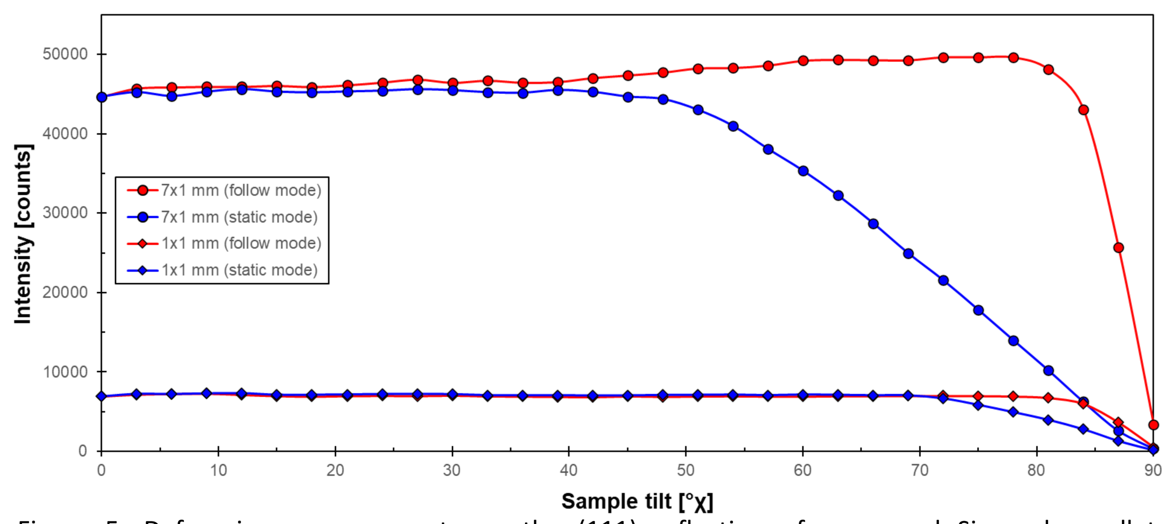

Figure 5: Defocusing measurements on the (111) reflection of a pressed Si-powder pellet performed with a 7x1mm² and 1x1 mm² beam, both with a static collimator and with the rotating slit following the sample tilt

In addition to over-irradiation, beam defocusing also results in a significant peak broadening even at higher sample tilts in measurement configurations involving 1D or 2D detectors, which are commonly used for residual stress or 2D-texture measurements, A desirable side effect of the rotating slit for these measurements, is a significant reduction of the tilt-induced increase in peak broadening even for large beam sizes (Fig. 6e-f). Therefore, in combination with the rotating slit, significantly larger spot sizes can be used for stress and texture measurements even in combinations with 1D/2D detector configurations.

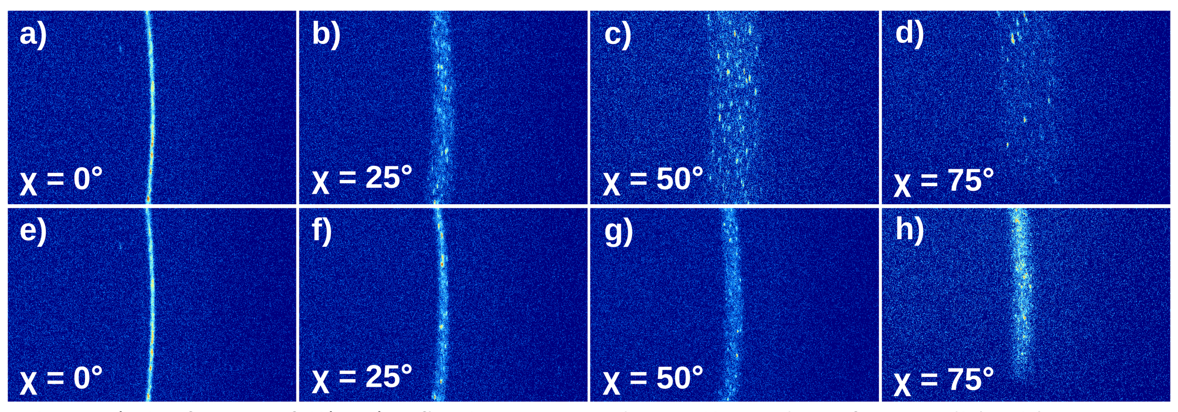

Figure 6): 2D frames of Si(111) reflection measured on a Si-powder reference disk with a 7 x 0.5 mm² beam at different at χ=0°, 25°, 50°, and 75° in combination with a Eiger2 R 500k Si. (a-d) 2D frames measured with a X-ray lens and a conventional, static collimator. (e-f) Corresponding projections measured with the new rotating slit module.

Results – Texture measurements & analysis

For standard Schulz-type pole figure measurements [1], measuring accurate diffraction intensities at different sample orientations (ψ, φ) is vital. In contrast to standard collimators with which significantly smaller beam sizes have to be used, the rotating slit offers the possibility to optimize the irradiated area even for smaller samples without over-irradiation. This results in higher measured intensities (Fig. 7), enabling faster scan times without compromising data quality.

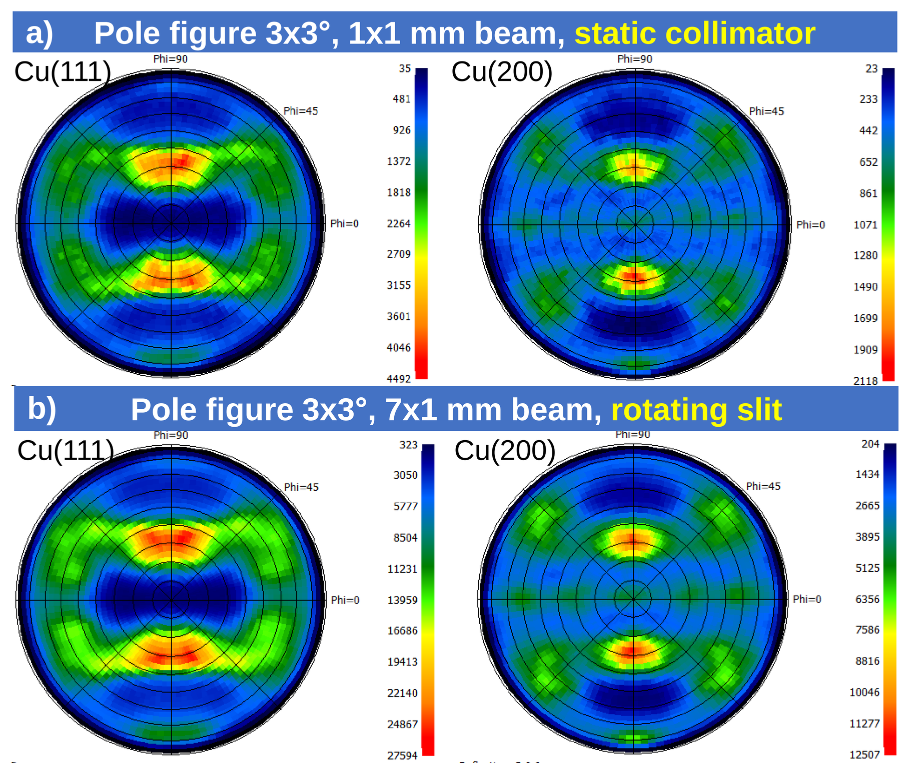

Figure 7: (111) and (200) pole figures measured on rolled Cu with a static collimator (a) and the new rotating slit module (b) with the same measurement time. For both configurations, the beam size was optimized to avoid over-irradiation (1x1 mm vs. 7x1 mm). The data shows a >6-times higher intensity obtained with the new device.

In addition to the shorter measurement times, the possibility to use larger beam dimensions can also improve particle statistics by maximizing the number of crystallites contributing to the overall diffraction signal. This can significantly improve the data quality for coarsely crystalline samples. This effect of improved intensities and particle statistics in measured pole figures and calculated orientation distribution functions (ODF) achievable with the rotating slit in comparison with a microbeam setup is illustrated in Fig. 8.

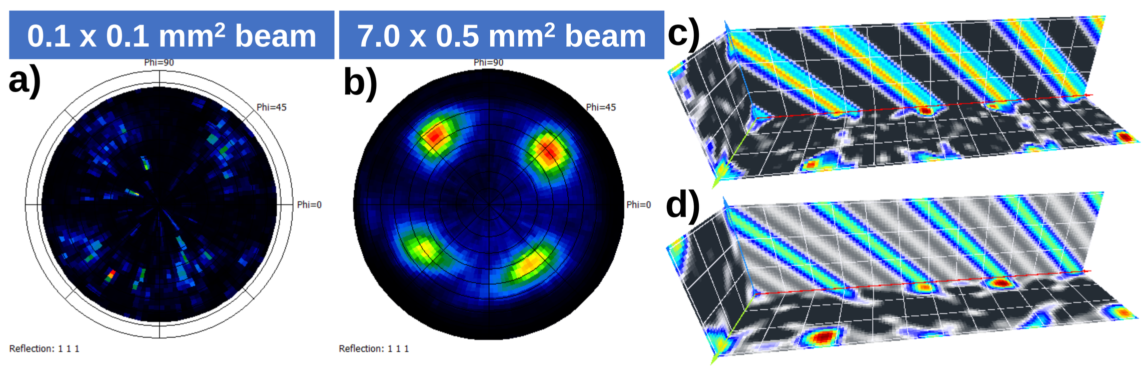

Figure 8-right: Al (111) pole figures measured on a 3D-printed test Al-alloy test coupon (a) with a micro-spot of 0.1x0.1 mm² (with static collimator), and (b) the rotating slit set to 7x0.5 mm². The data shows a clear advantage of maximizing the probed volume to improve particle statistics.

Figure 8-left: ODF representations as 2D sections projected along phi1, PHI, and phi2 (Bunge notation), calculated from the Al-alloy pole figure data obtained with (c) a small spot setup (0.1x0.1 mm²) and (d) with the rotating slit device and an optimized beam size of 7x0.5 mm². ODF analysis performed with the ATEX texture SW [2].

Results – Residual stress analysis on thin films

In contrast to bulk materials, residual stress measurements on thin films typically pose additional challenges due to their finite thickness and strong textures. Therefore, results obtained with the classical single {hkl} method or multiple {hkl} GI-stress method often show artifacts or yield no usable analysis result at all (Fig.9). Therefore, thin films require more advanced approaches.

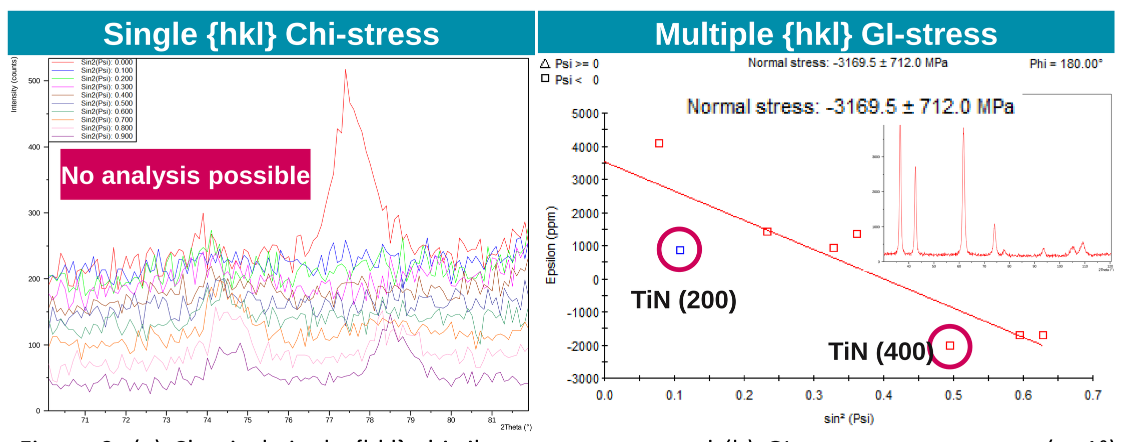

Figure 9: (a) Classical single {hkl} chi-tilt measurement and (b) GI-stress measurement (α=1°) and analysis of a textured, polycrystalline TiN thin film. Due to its thickness (<4 μm) and strong fiber texture the Chi-stress approach did not yield any usable results. From the GI-stress measurement a compressive stress of -3170 MPa could be determined but with a large error of ±712 MPa due to the strong preferential orientation and the {hkl} dependent anisotropic elastic constants (seen in the systematic deviation of the {h00} reflections.

Due to its property of accurately following the tilt of the sample surface, the rotating slit with its benefits can also be used for GID measurements in side-inclination. This method is referred to as ω-φ compensated GI-stress analysis and is a highly useful application for the determination of stress but also texture in thin films [3]. This approach is based on dynamic adjustment of both ω and Φ to keep a constant angle of incidence α of the X-ray beam with respect to the sample surface and correcting the azimuthal direction of the scattering vector φ, while tilting the sample in χ [3,4]. This allows to determine the stress in thin films faster and more accurately (Fig. 10).

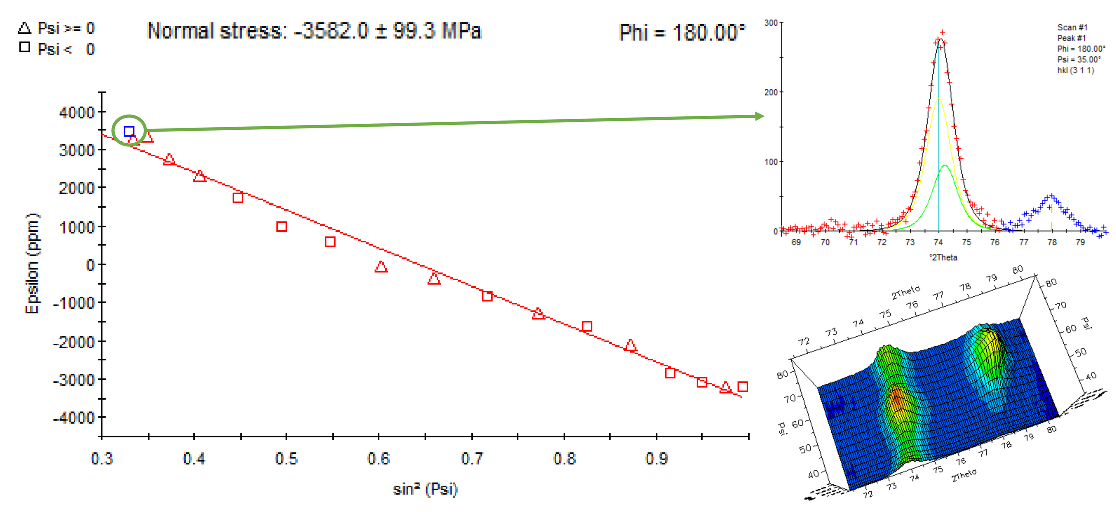

Figure 10: ω-ф compensated GI/chi-stress analysis (α=1°) on the same TiN sample as in Fig. 9, measured on TiN (311), showing a compressive stress of -3.582±99 Mpa, with almost no influence from texture/anisotropy and hence a much smaller error than the classical methods.

Summary

The motorized, rotatable slit/mask collimation device enables optimization of the beam size and its position with respect to the sample surface area during 2-axis measurements involving side inclination along the χ-axis. This makes it the perfect tool for residual stress and texture measurements. It prevents over-irradiation of the sample as well as defocusing-induced peak broadening even with large beam sizes at sample tilts exceeding 75°χ. Therefore, significantly larger beam sizes can be used for these applications, providing higher intensities and shorter scan times (factor of 7 or even higher) can be achieved. Furthermore, for samples exhibiting large crystallites, particle statistics and therefore analytical reliability can be significantly improved.

References

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