



## Application Note XRD 610

# *Single Tilt Method* with 2D Detectors for Residual Stress Determination

- Accurate Stress Results Obtained with Data Collected at Low Diffraction Angles

Understanding the residual stress is essential to property and longevity optimization of products. X-ray diffraction offers a non-destructive window into the atomic scale origin of stress. By directly measuring the variation of atomic spacing along a variety of orientations, the stress condition can be determined. Adjustments to material processing steps can then be performed to induce beneficial compressive stresses.

This application note introduces a new method for stress measurement which removes the need for complex goniometric motion, such as those employed for  $\sin^2\Psi$ , by taking advantage of a large 2D detector and single tilt geometry. Not only does this dramatically simplify the measurement method for conventional materials, but this also opens up the technique to materials such as polymers and coatings.

## Introduction

Measurement of residual stress is challenging in case of weak stress or strain gradients, preferred orientation, anisotropic grain shape, and inhomogeneous phase and microstructure distribution [1]. When the conventional  $\sin^2\Psi$  method is used for stress measurement under these suboptimal sample conditions, nonlinear  $d$  vs.  $\sin^2\Psi$  behavior commonly produces poor results. Additionally, the variation in incident angle commonly applied in the standard  $\sin^2\Psi$  technique results in a variation in both sampling depth and lateral coverage of the X-ray beam, compounding the non-ideal sample conditions mentioned above. Another limitation of the conventional  $\sin^2\Psi$  technique is that diffraction peaks at higher  $2\theta$  angles need to be measured. This results in less sensitivity to sample height errors potentially caused by required goniometric positioning. For thin films, coatings, or polymer materials, high  $2\theta$  peaks may not be accessible or appropriate for stress measurement making it significantly more difficult or even impossible to measure stress with the conventional  $\sin^2\Psi$  method. An alternative method to the conventional iso-inclination  $\sin^2\Psi$  method discussed above is the side-inclination method, where the incident angle is fixed and the sample tilted to the side. This results in a reduction in the incident angle to the sample surface leading to accentuated sensitivity to sample height error.

When using two-dimensional X-ray diffraction, stress measurement is based on a direct relationship between the stress tensor and diffraction cone distortion [2]. The diffraction vectors covered with a correctly designed 2D diffraction system results in sufficient angular coverage such that data collection can be done at a fixed  $\Psi$  angle with only  $\phi$  rotation. For most goniometers utilizing the Eulerian geometry,  $\phi$  rotation will contribute little to no sample height error, translating into a significantly smaller measurement error [3]. Additionally, a single tilt angle brings the advantage of a constant incident angle, and thus, a nearly constant penetration depth for stress evaluation.

## Geometry of the Single Tilt Method

Figure 1 illustrates the diffraction vector distribution for the pattern collected with a conventional (0D/1D) method or area (2D) method. The hemisphere represents all the possible orientation from the origin  $O$  of the sample coordinate  $S_1S_2S_3$ . With the conventional method at  $\Psi=0^\circ$ , the diffraction vector points to the sample normal direction  $N$ . In order to measure stress, the sample has to be tilted at several  $\Psi$  angles, for instance  $0^\circ$ ,  $15^\circ$ ,  $30^\circ$  and  $45^\circ$  as indicated by  $\otimes$ . With the 2D method, the trace of the diffraction vector covers a range as shown by the red curve ( $\Psi = 0^\circ$ ) and the blue curve ( $\Psi = 22.5^\circ$ )

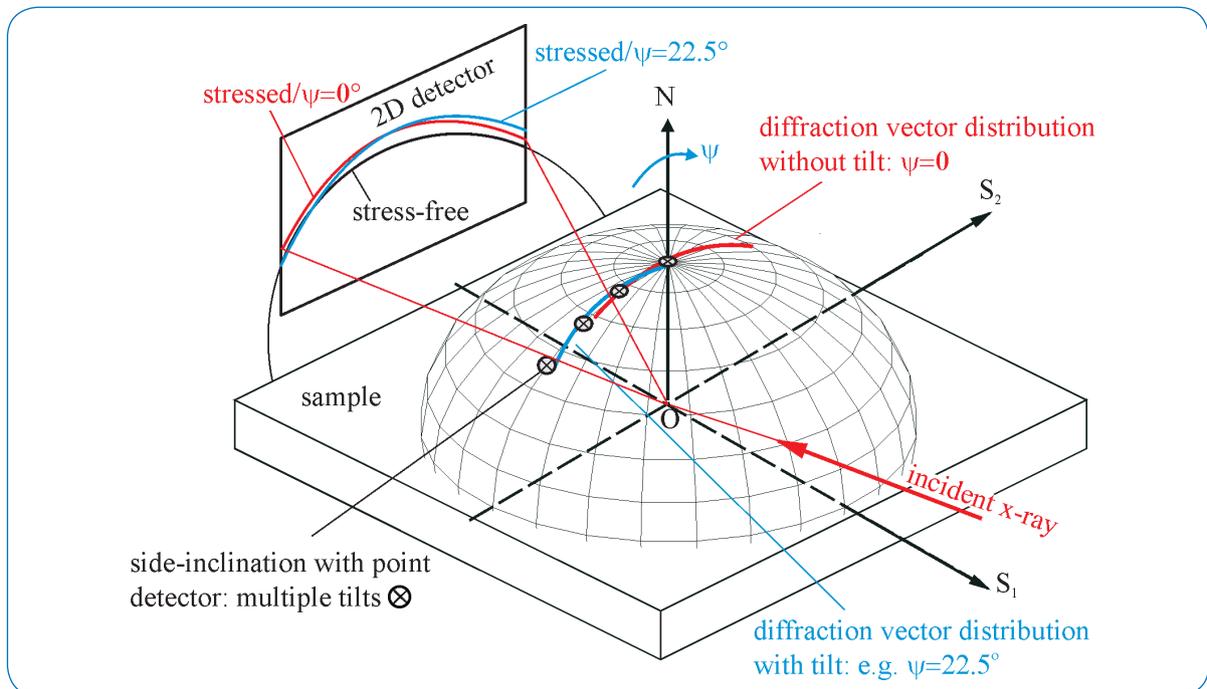


Figure 1 Diffraction vector distribution for 0D / 1D and 2D detectors.

For low  $2\theta$  diffraction rings at the proper detector distance, it is possible to cover sufficient angular range for stress evaluation with a single tilt. The data set for a complete stress tensor must be collected at several  $\phi$  angles, for instance every  $45^\circ$ . For most goniometers with a Eulerian geometry, the  $\phi$  axis is built on an accurate spherical bearing with very small error, while  $\Psi$  rotation is achieved by only a section of a circular track, a design prone to more significant spherical errors. Avoiding  $\phi$  rotation can reduce the sample height variation during data collection, which can result in significant improvements in measurement accuracy.

### Data collection scheme

The diffraction vector distribution corresponding to a data set can be mapped in a data collection strategy scheme to optimize the measurement conditions. Figure 2 illustrates the single tilt scheme generated with DIFFRAC.MEASUREMENT CENTER software for a film of  $\text{Al}_2\text{O}_3(116)$  with  $2\theta=57.5^\circ$ . The red arcs represent the diffraction vector coverage with a 2D detector corresponding to the complete data set.

S1 and S2 are two sample orientations. In this scheme, 8 frames are collected at  $\Psi=22.5^\circ$  and eight  $\phi$  angles with  $45^\circ$  intervals. This scheme produces comprehensive coverage in a symmetric distribution. The resulting data set can be used to calculate the complete biaxial stress tensor including both normal and shear stress components ( $\sigma_{11}, \sigma_{12}, \sigma_{22}, \sigma_{13}, \sigma_{23}$ ).

### Experimental Example

The residual stress in a  $1 \mu\text{m}$  thick  $\text{Al}_2\text{O}_3$  coating is measured with a D8 DISCOVER system equipped with a centric Eulerian cradle and VANTEC-500 2D detector. With  $\text{Cu-K}\alpha$  radiation, the diffraction ring from the (116) planes at  $2\theta=57.5^\circ$  is used for stress evaluation. With 60 seconds per frames, the total data collection time is 8 minutes. The stress calculation is performed with DIFFRAC.LEPTOS V7.9. The counts within each subregion are integrated into a diffraction profile and a Pearson VII function is used to fit the profile to evaluate the  $2\theta$  peak position. Figure 3 shows the stress evaluation results from the data set. The charts above "A" are the fitted data points overlaid on the 2D frames. The charts above "B" are fitted data points in  $\gamma$ - $2\theta$  rectangular coordinates with a magnified  $2\theta$  scale, in which the black line indicates  $2\theta_0$ , the blue cross and line indicates the data points from the profile fitting of each subregion, and the red line represents the calculated diffraction rings from the stress results. The small amount of scattering of the crosses around the red line represents the high quality of the data, reflected in the low standard deviation of the stress results. By clicking on any data point, the integrated profile displays above "C". The measured stress values are given in the region "D" as  $\sigma_{11}=954.7 \text{ MPa}$ ,  $\sigma_{22}=957.9 \text{ MPa}$  and standard deviation  $26.5 \text{ MPa}$  ( $< 3\%$ ).

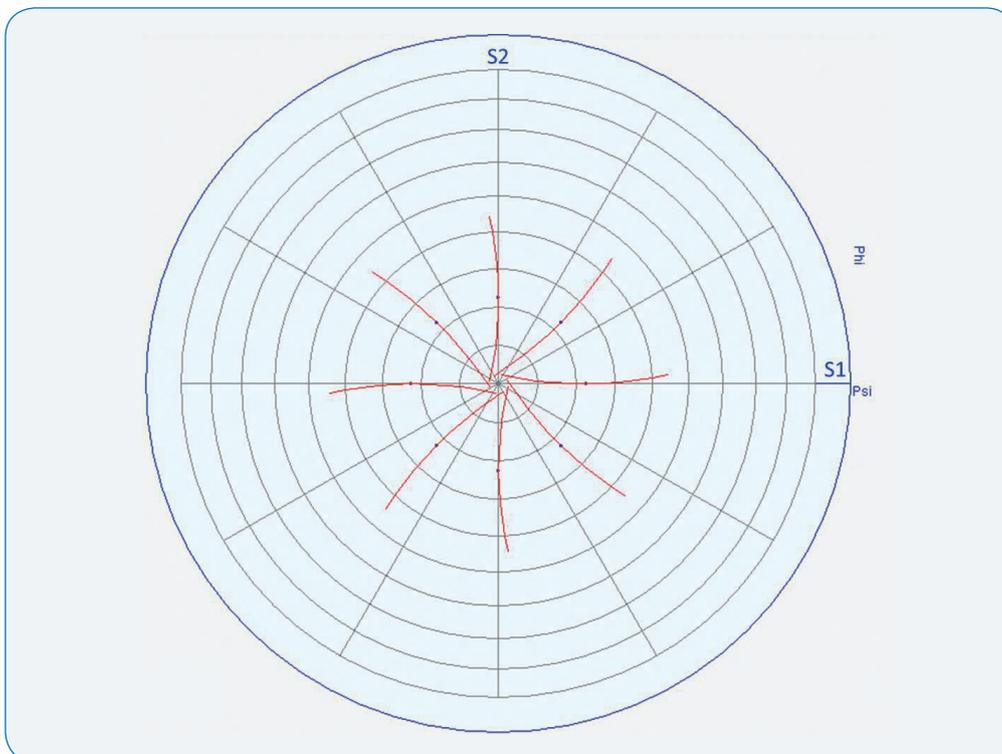


Figure 2 Data collection strategy schemes with single tilt at  $\Psi = 22.5^\circ$  and complete  $\phi$  rotation of  $45^\circ$  steps.  $\text{Al}_2\text{O}_3(116)$ ,  $2\theta=57.5^\circ$ .

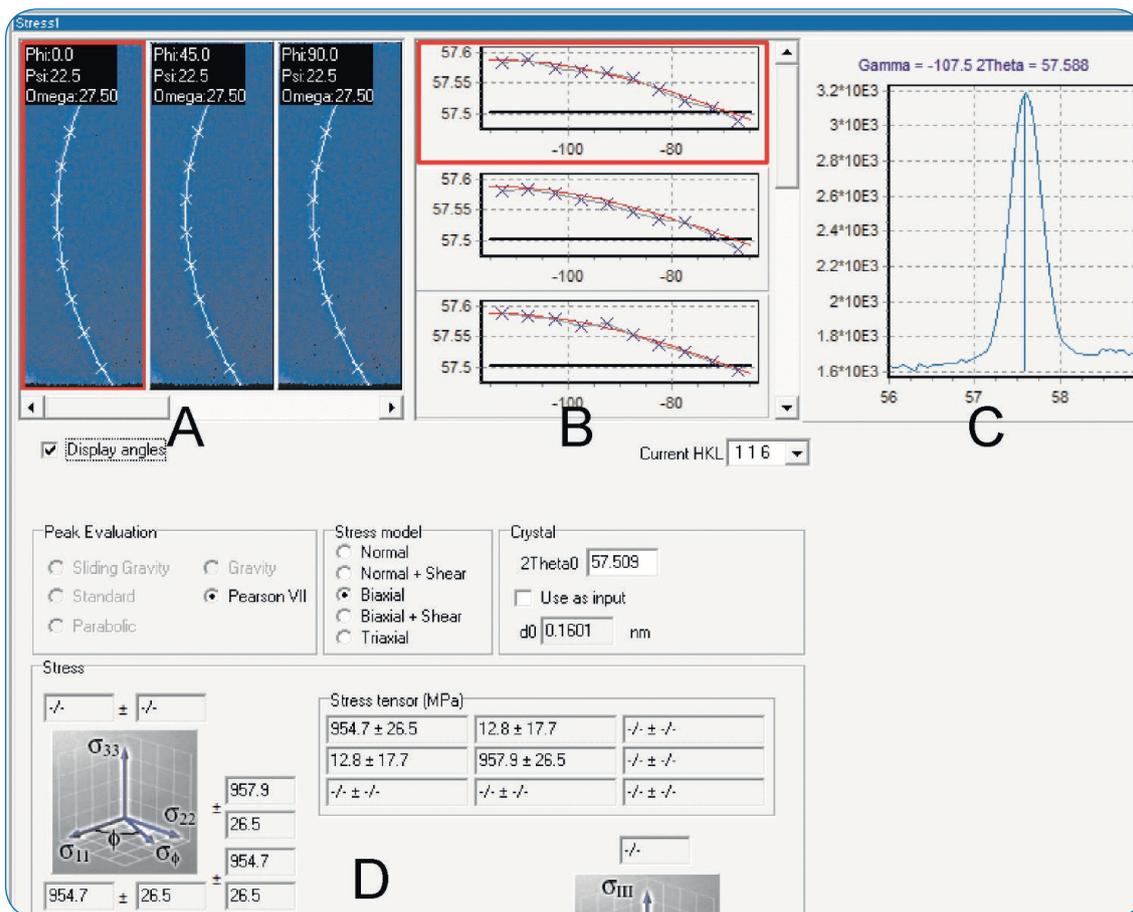


Figure 3 Data evaluation results in DIFFRAC.LEPTOS.

## Conclusions

For materials such as thin films, coatings, or polymers, where the diffraction peaks at high  $2\theta$  angles are not available or appropriate, the single tilt 2D method allows the use of a low  $2\theta$  peak for stress evaluation. With diffraction rings at low  $2\theta$  angle, the diffraction vector distribution can satisfy the angular coverage for stress measurements at a fixed tilt angle ( $\Psi$ ). Without  $\Psi$  change during data collection and with  $\phi$  rotation only, the sample height is accurately maintained resulting in residual stress measurements with high accuracy and high speed.

## Author

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## References

- [1] B. B. He, Measurement of residual stresses in thin films by two-dimensional XRD, *Materials Science Forum*, Vols. 524-525, pp 613-618, (2006).
- [2] B. B. He, *Two-dimensional X-ray Diffraction 2nd Edition*, John Wiley & Sons, (2018).
- [3] "X-ray diffraction device and method to measure stress with 2D detector and single sample tilt", patent pending (filing number US15631533)

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