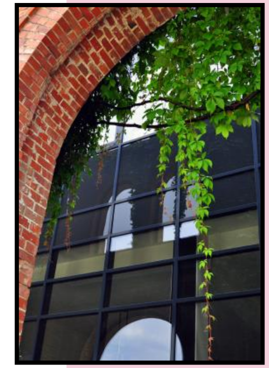
Institute of Materials Engineering
Metallic Materials
Prof. Dr.-Ing. T. Niendorf



Advanced methods near surface X-ray techniques

A. Liehr,

I. Introduction

- Some basic aspects

II. Measurements methods and data evaluation:

- $Sin^2\psi$ method
- Universal plot method (UVP)
- Ω-χ-φ method
- η - method

III. Energy dispersive methods:

- Multiwavelength method and UVP

IV. Transformation methods:

- Initial inverse problem
- Inverse Laplace transformation (ILT)

V. Conclusion

Angle dispersive

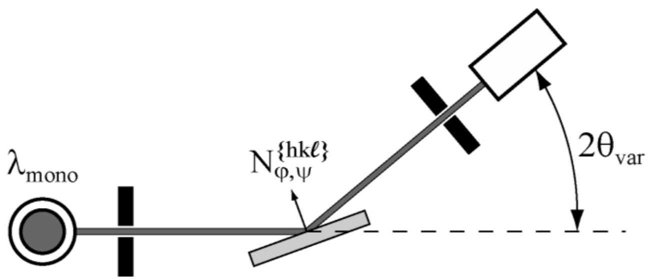
$$n\lambda = 2d^{hkl}\sin\theta$$

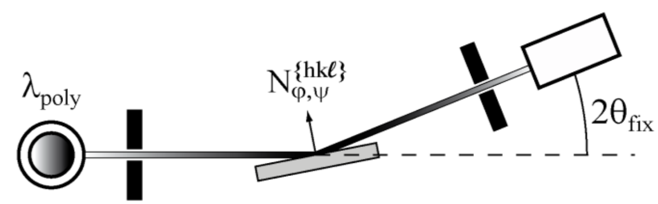
$\downarrow$ const. $\downarrow$? $\downarrow$ exp.

Energy dispersive

$$n\lambda = 2d^{hkl}\sin\theta$$

$\downarrow$ exp. $\downarrow$? $\downarrow$ const.

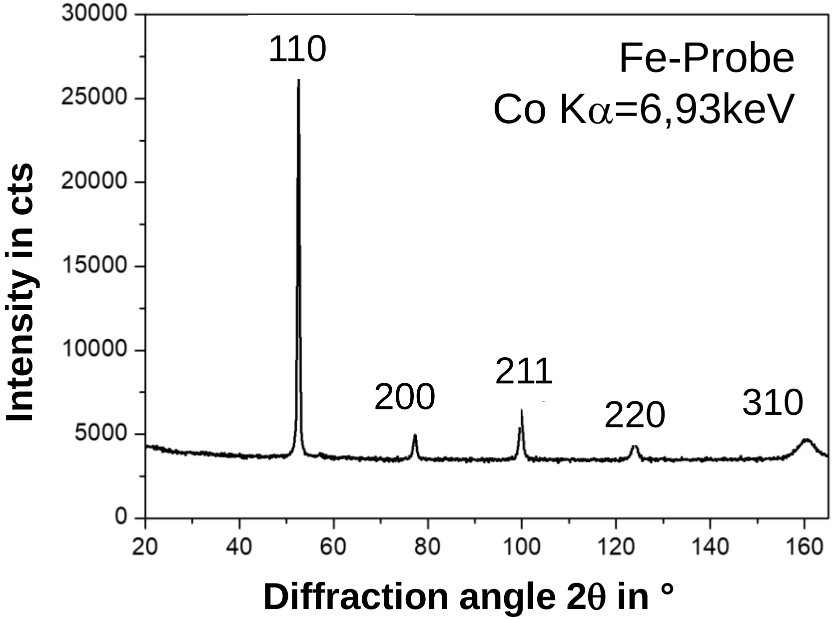


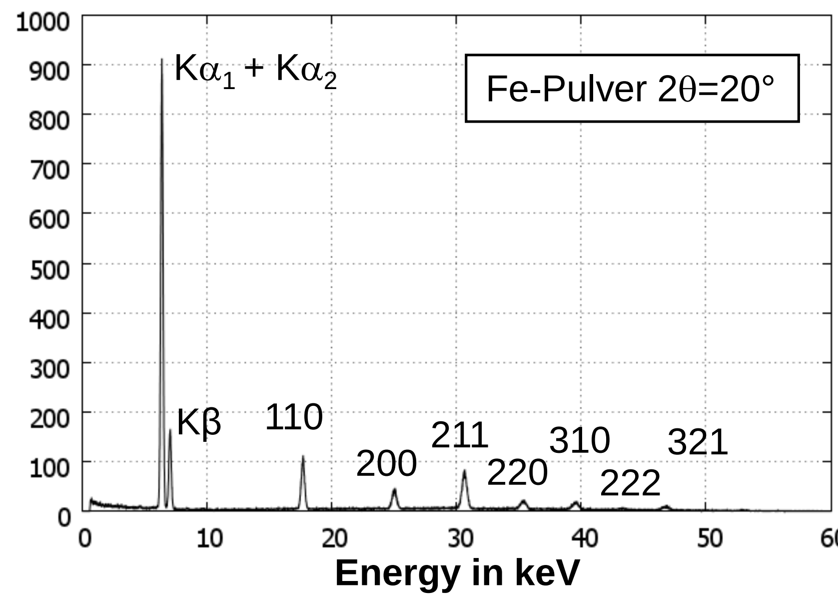


looking for $D^{\{hkl\}}$ value

$$\varepsilon_{\varphi\psi}(hkl) = \frac{d_{\varphi\psi}(hkl)}{d_0(hkl)} - 1 = \frac{E_0(hkl)}{E_{\varphi\psi}(hkl)} - 1$$

Angle dispersive	Energy dispersive
$$n\lambda = 2d^{hkl}\sin\theta$$	$$n\lambda = 2d^{hkl}\sin\theta$$
$n\lambda$ ↓ const., d^{hkl} ↓ ?, $\sin\theta$ ↓ exp.	$n\lambda$ ↓ exp., d^{hkl} ↓ ?, $\sin\theta$ ↓ const.



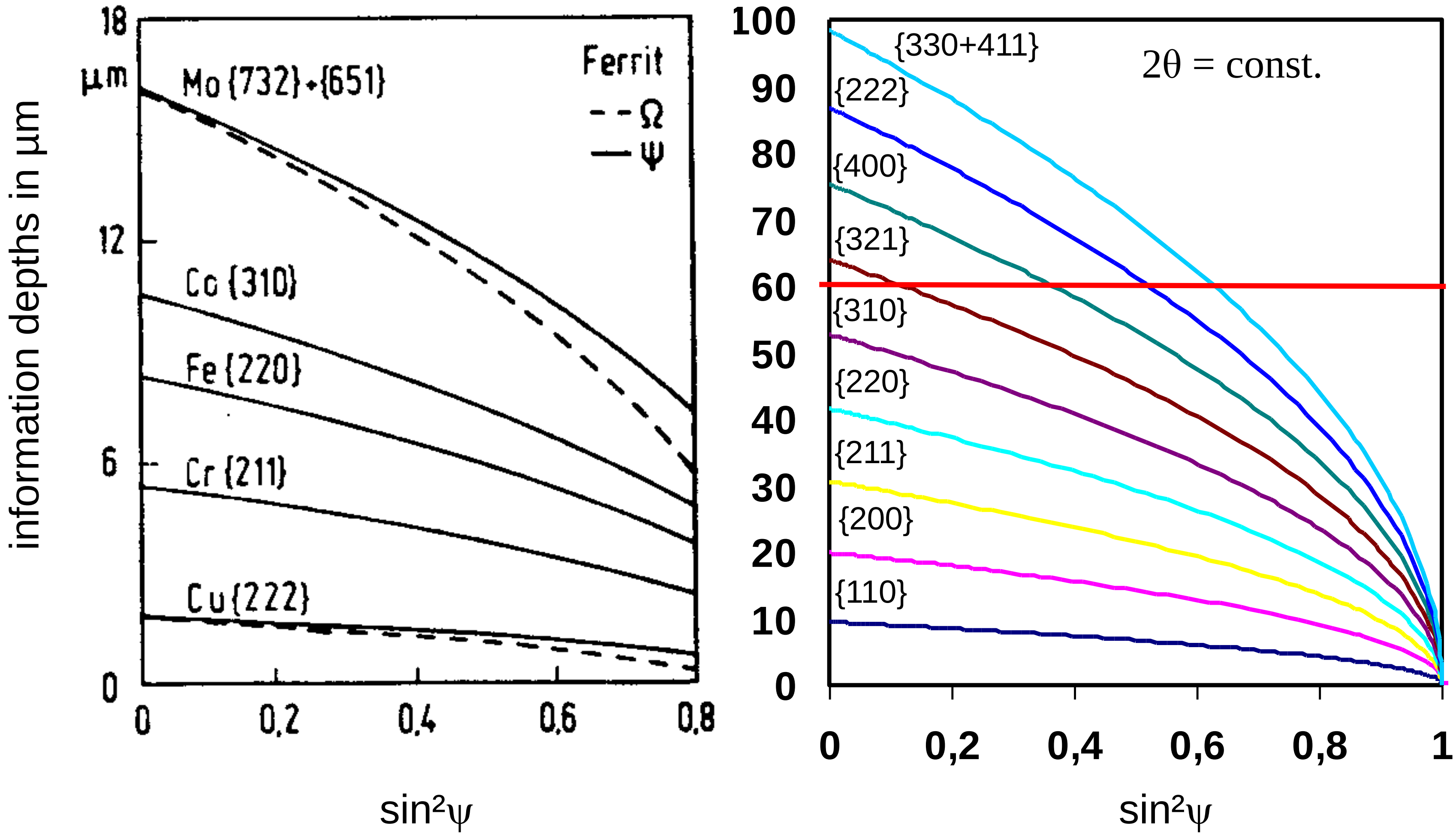


depth τ → **10 µm**

depth τ → **60 µm**

Measurement methods

Penetration depth and sample conditions



Source: (similar to) V. Hauk, Structural and Residual Stress Analysis by Nondestructive Methods

There is a interest in the evaluation and analysis of residual stresses in research and in industry aplications


Materials Science and Engineering, A161 (1993) 41–44

Evaluation of the stress field in a ground steel plate from energy-dispersive X-ray diffraction experiments

H. Ruppersberg and I. Detemple

F. B. Werkstoffwissenschaften, University of the Saarland, W-6600 Saarbrücken (Germany)

(Received July 17, 1992; in revised form September 10, 1992)

Abstract

We show that energy-dispersive X-ray diffraction is a very powerful tool for the investigation of complex stress fields which occur in the surface region of polycrystalline materials after plastic deformation. The interplanar spacings of 11 lattice planes of a ground steel plate were investigated for ψ angles up to 88°, covering a penetration depth of the X-rays from 0.04 to 60 μm. The results were evaluated according to a novel formalism and yield the variation in the residual stress components $\sigma_{11}(z)$ and $\sigma_{22}(z)$ with the depth z below the ground surface. The formalism and the results are critically discussed.

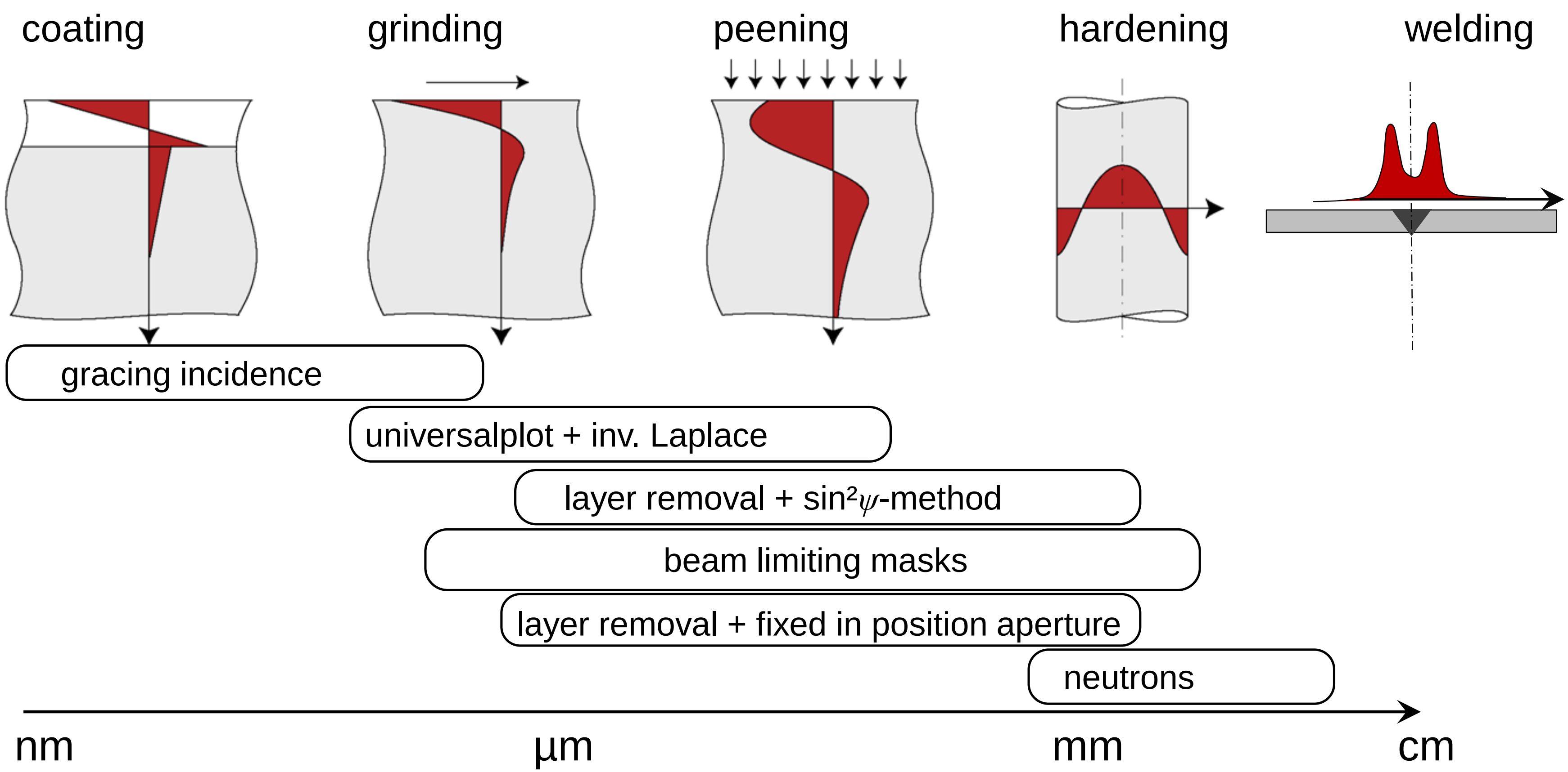
1. Introduction

Actual literature shows many examplesof methods and approaches.

In addition to DIN EN 15305, there are numerous corporate guidelines that regulate the assessment of residual stresses. In some cases also direct production requirements witch shows minimum values in drawings

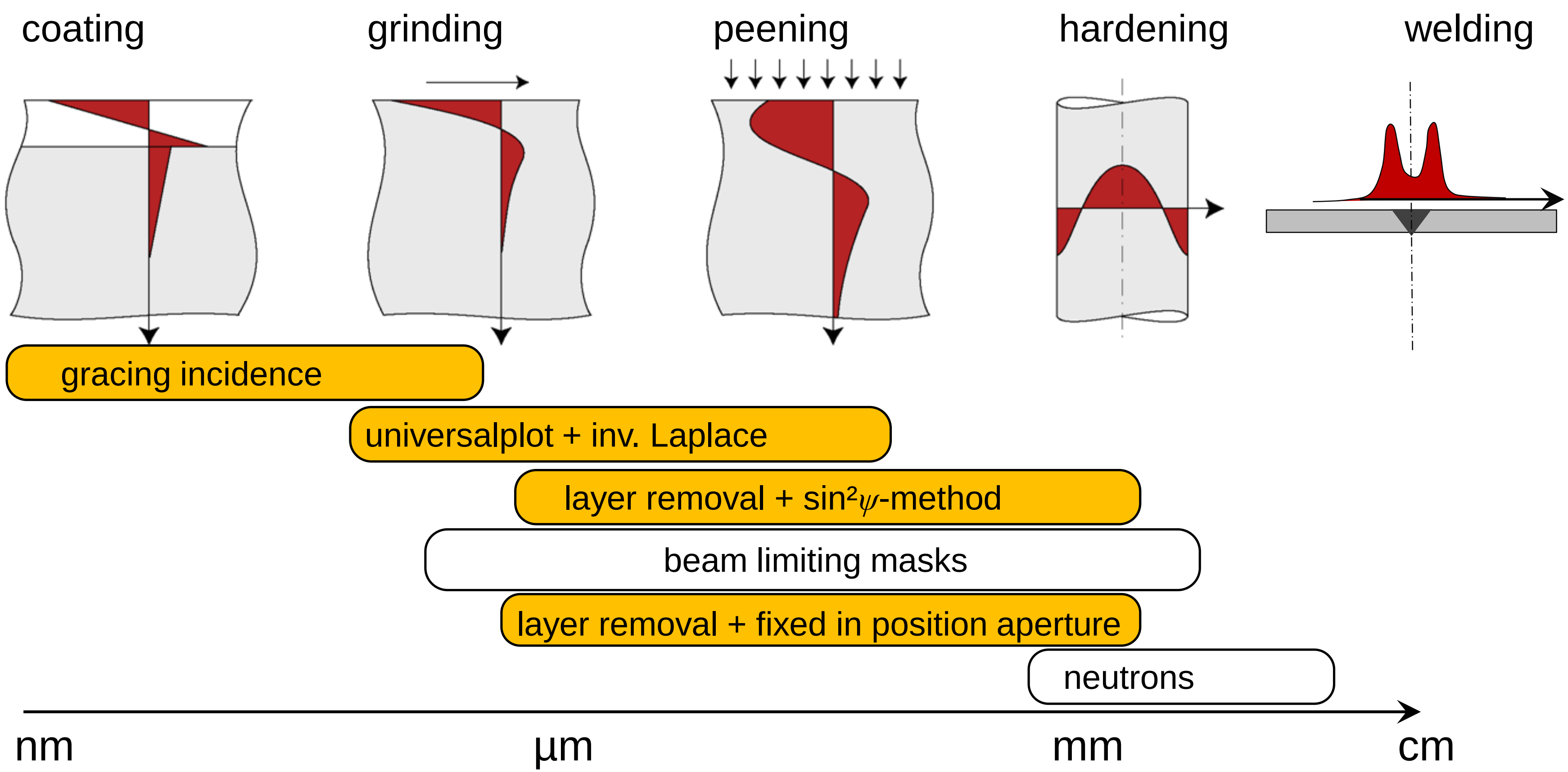
Introduction

Different situation of residual stresses, their depth distribution and the suitable measurement methods



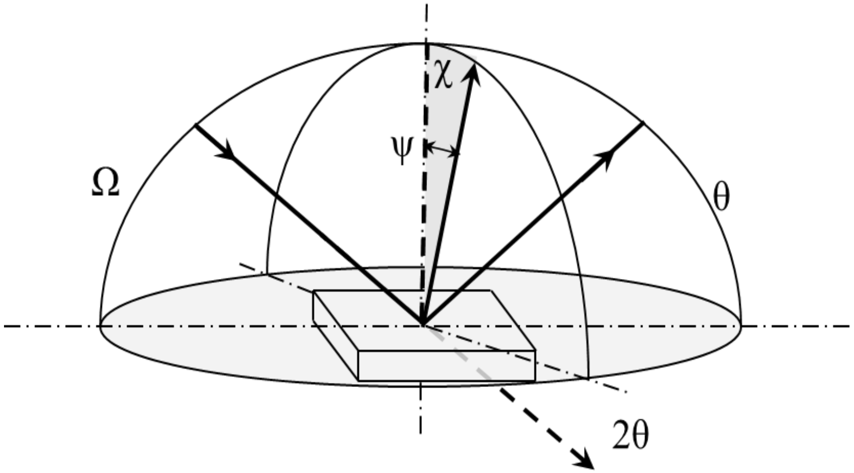
Introduction

Different situation of residual stresses, their depth distribution and the suitable measurement methods



Measurement methods / example

symmetric



universal plot method (UVP)[1]

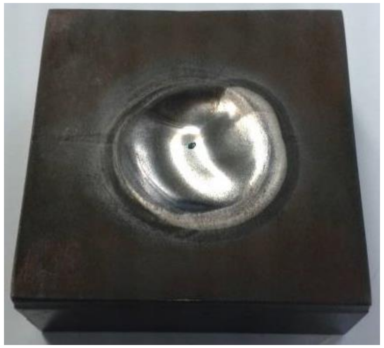
asymmetric

Ω-χ-φ method [2,3]

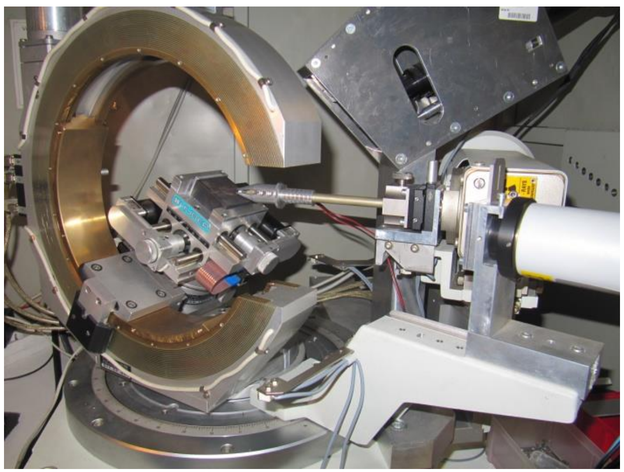
[1] H. Ruppersberg, I. Detemple, J. Krier, Phys. Status Solidi A 116 (1989) 681.
[2] T. Erbacher, A. Wanner, T. Beck, O. Vöhringer, Crystallography (2008), ISSN 0021-8898, S. 377-385
[3] A.Kumar, U.Welzel, E.J.Mittemeijer, Journal of Applied Crystallography, ISSN 0021-8898, S. 633 - 646

Measurement methods

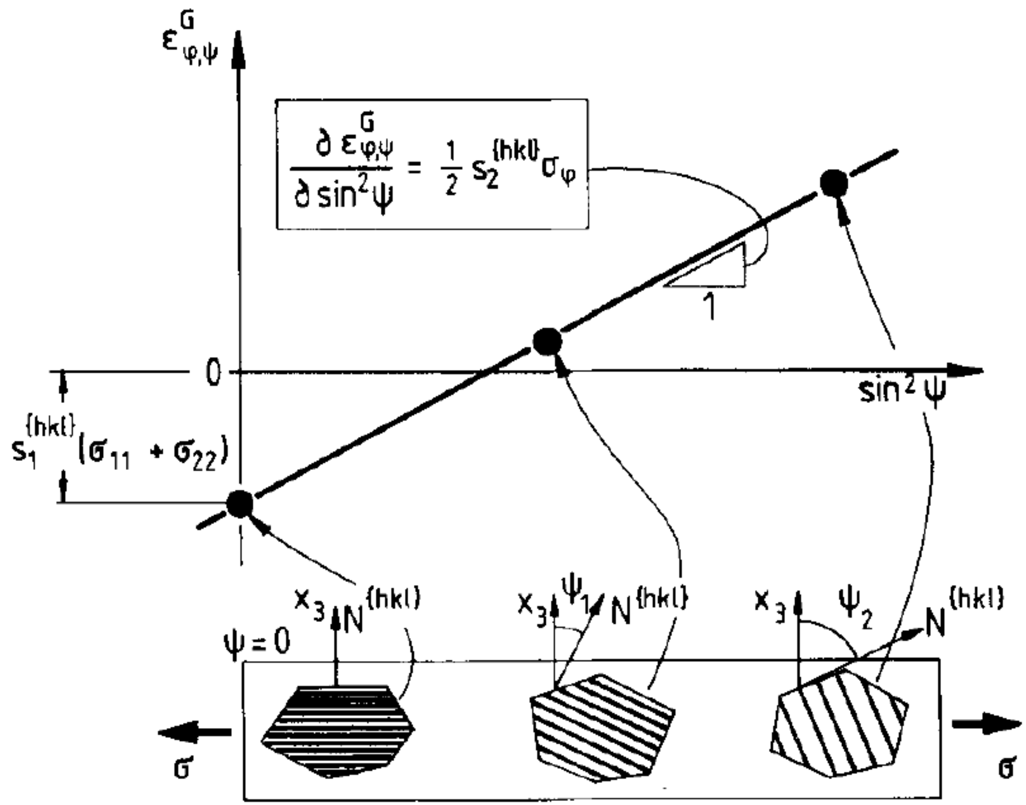
Layer removal + sin²ψ-method



Sample after electro polishing



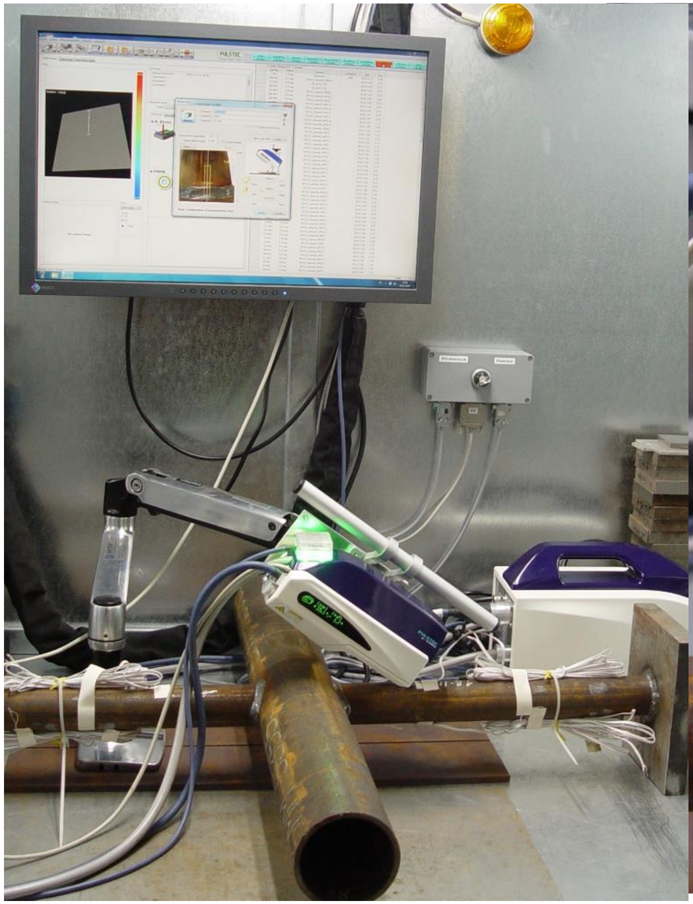
Residual stress meashurement with a conventional ψ diffractometer



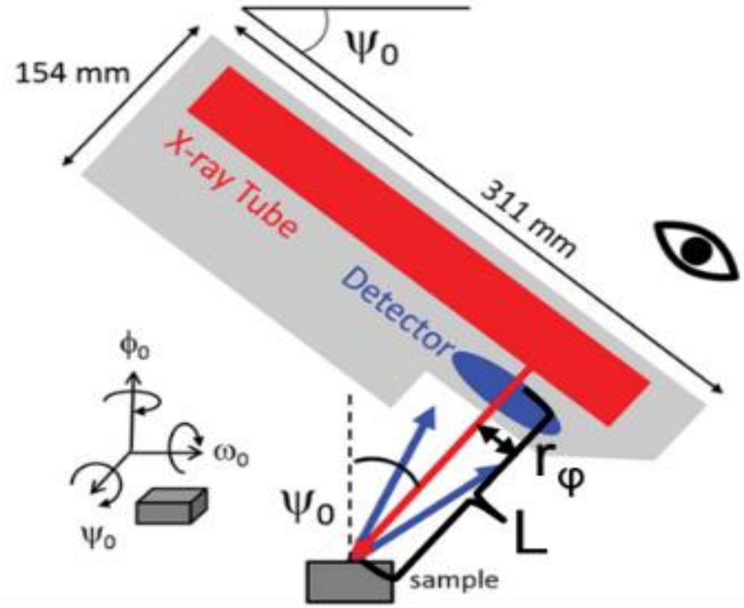
Source: B.Eigenmann und E.Macherauch, Röntgenographische Untersuchung von Spannungszuständen in Werkstoffen - Teil 1

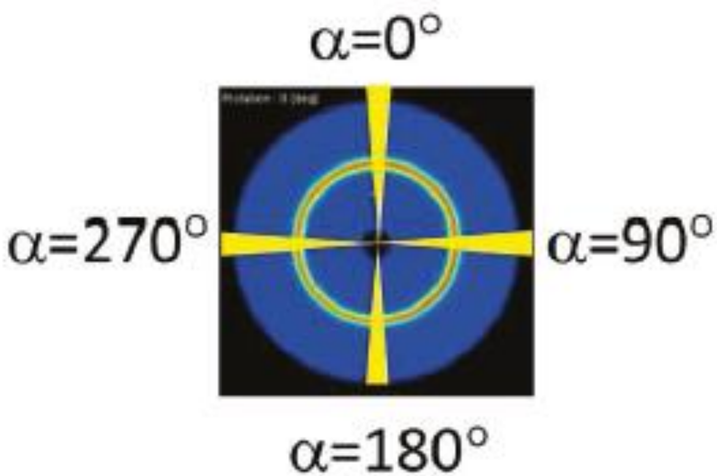

In Benutzung
PLANOLITH
37
00.0

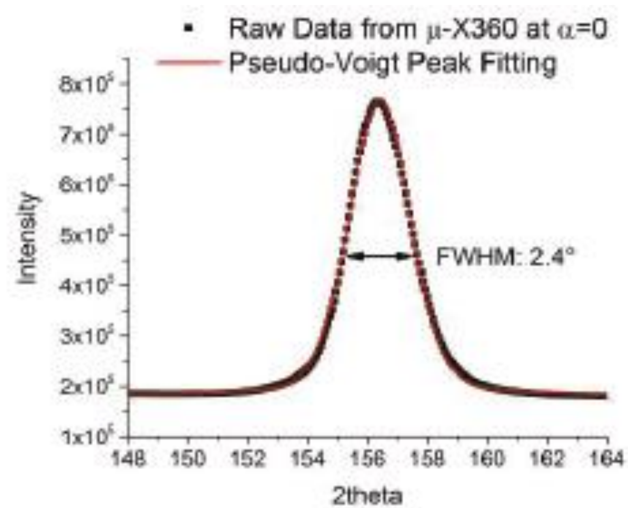
cosα- Method

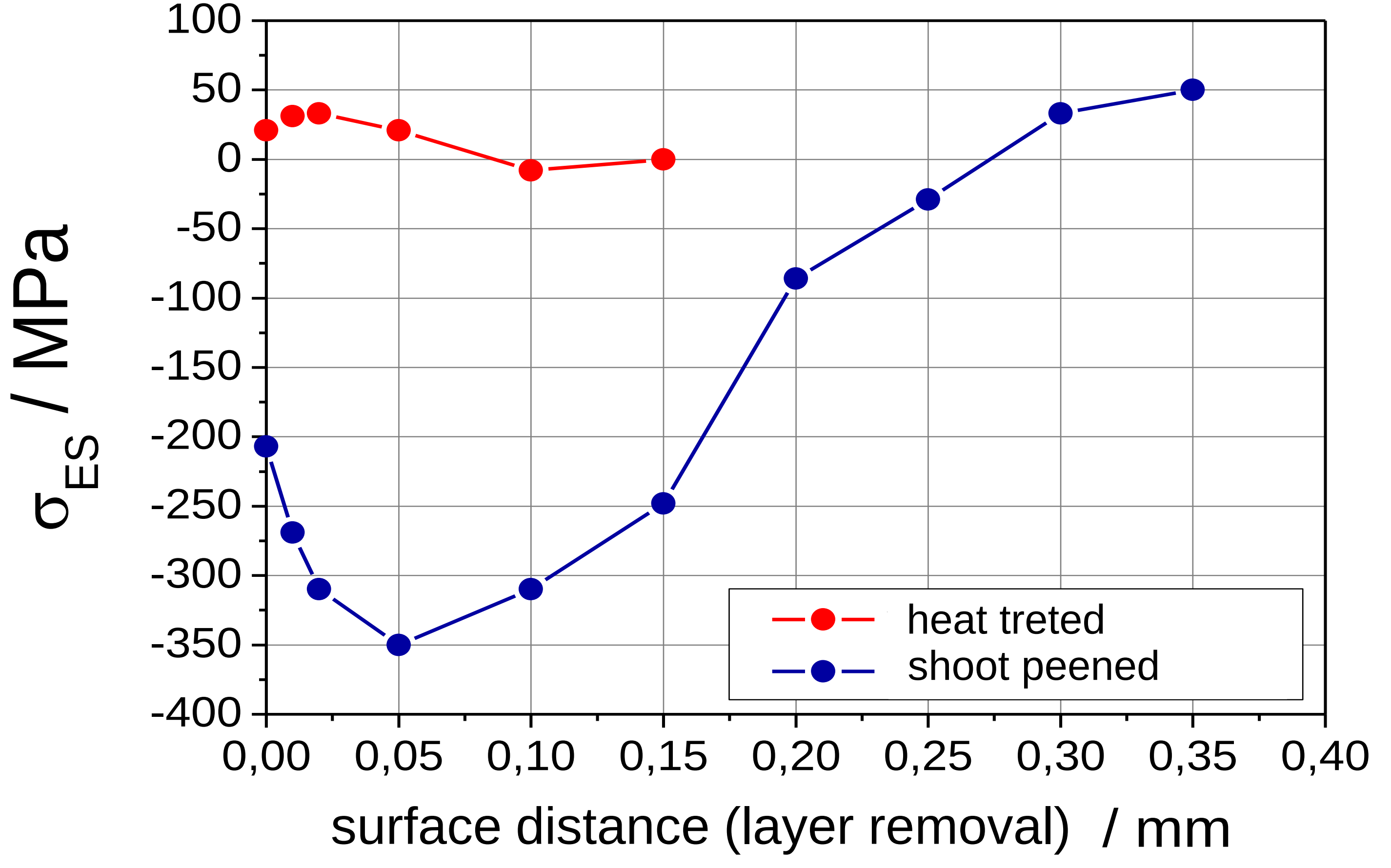


cosα- Method









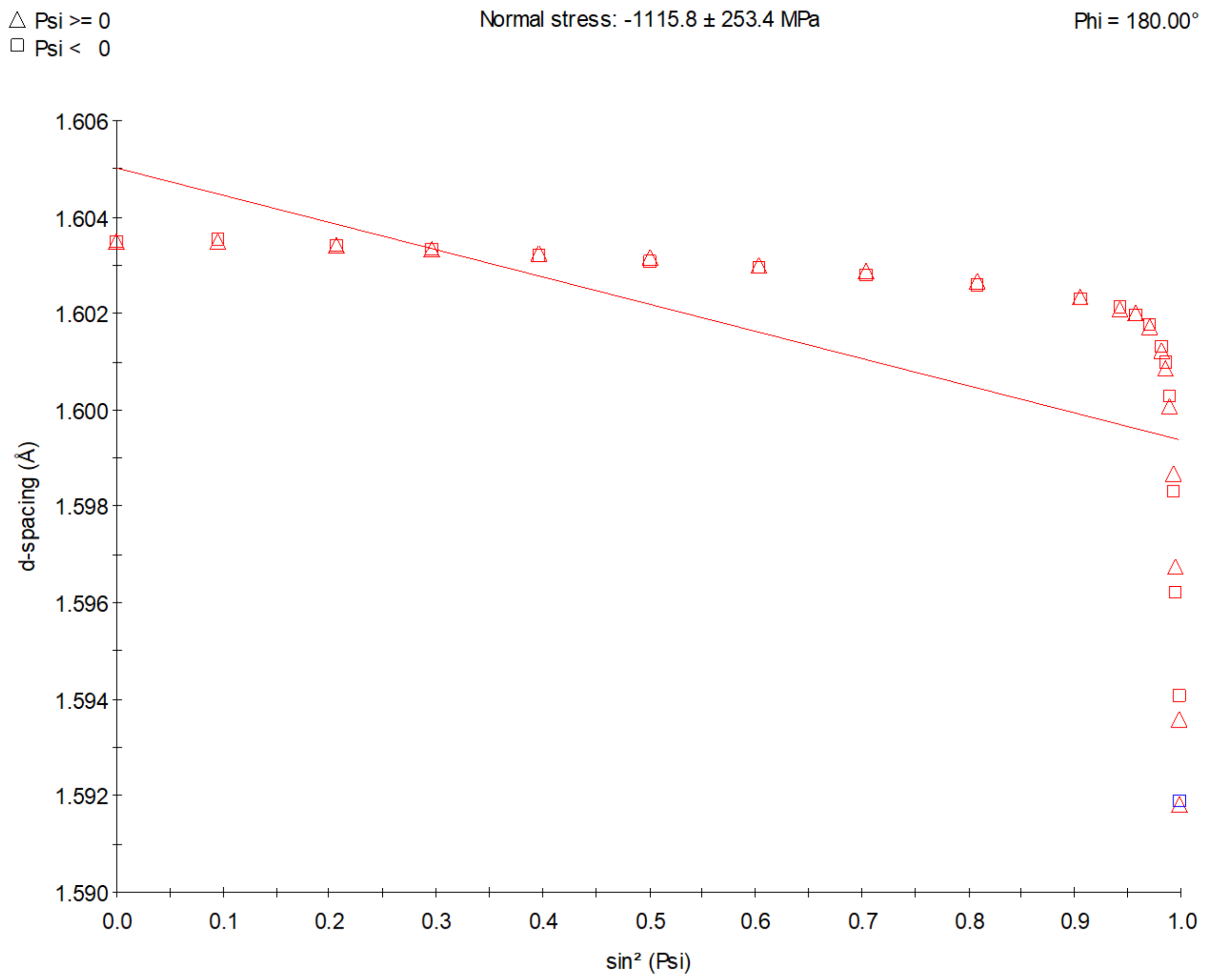
100
50
0
-50
-100
-150
-200
-250
-300
-350
-400
σ_{ES} / MPa
0,00
0,05
0,10
0,15
0,20
0,25
0,30
0,35
0,40
surface distance (layer removal) / mm
heat treted
shoot peened

Measurement methods

Sample: Al_2O_3 ceramic with a grinded surface

Measurement methods: UVP

four circle diffractometer with $Cu_{K\alpha}$ – X-Rays



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Measurement methods: UVP

Calculation and evaluation

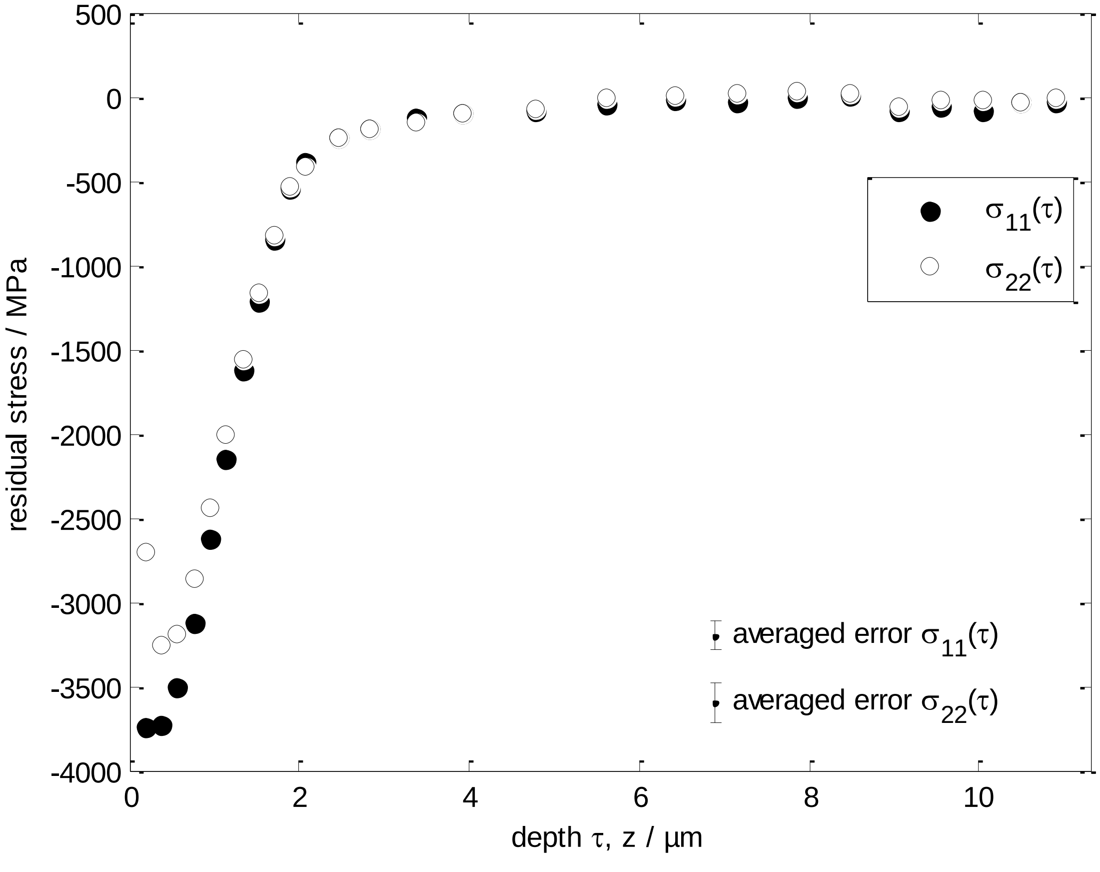
$$f^{+}(\tau) = \frac{\frac{1}{4}[\varepsilon_0\psi(\tau) + \varepsilon_{90}\psi(\tau) + \varepsilon_{180}\psi(\tau) + \varepsilon_{270}\psi(\tau)]}{\left(\frac{1}{2}s_2 \sin^2\psi + 2s_1\right)} = \frac{1}{2}[\sigma_{11}(\tau) + \sigma_{22}(\tau)]$$

$$f^{-}(\tau) = \frac{\frac{1}{4}\{[\varepsilon_0\psi(\tau) + \varepsilon_{90}\psi(\tau)] - [\varepsilon_{180}\psi(\tau) + \varepsilon_{270}\psi(\tau)]\}}{\frac{1}{2}s_2 \sin^2\psi} = \frac{1}{2}[\sigma_{11}(\tau) - \sigma_{22}(\tau)]$$

$$\sigma_{11}(\tau) = f^{+}(\tau) + f^{-}(\tau)$$

$$\sigma_{22}(\tau) = f^{+}(\tau) - f^{-}(\tau)$$

Measurement results: UVP

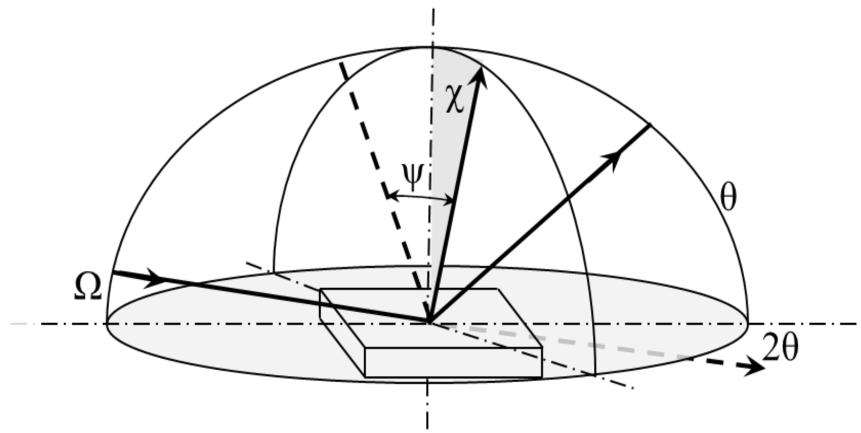


Measurement methods / example

symmetric

universal plot method (UVP)[1]

asymmetric



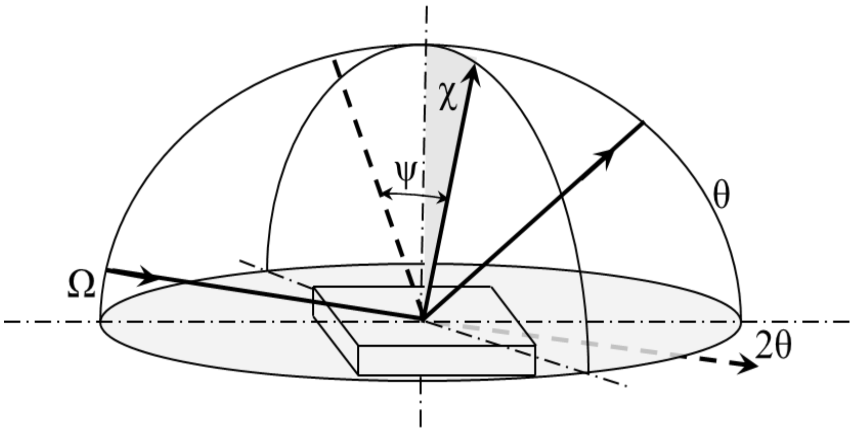
Ω-χ-φ method [2,3]

[1] H. Ruppersberg, I. Detemple, J. Krier, Phys. Status Solidi A 116 (1989) 681.
[2] T. Erbacher, A. Wanner, T. Beck, O. Vöhringer, Crystallography (2008), ISSN 0021-8898, S. 377-385
[3] A.Kumar, U.Welzel, E.J.Mittemeijer, Journal of Applied Crystallography, ISSN 0021-8898, S. 633 - 646

Measurement methods: Ω-χ-φ

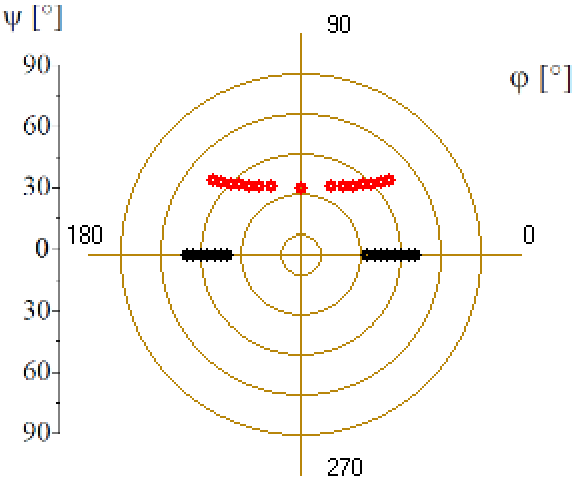
Measurements by constant penetration depth τ

asymmetric



$$\psi = \arccos(\cos\chi\cos(\theta-\Omega))$$

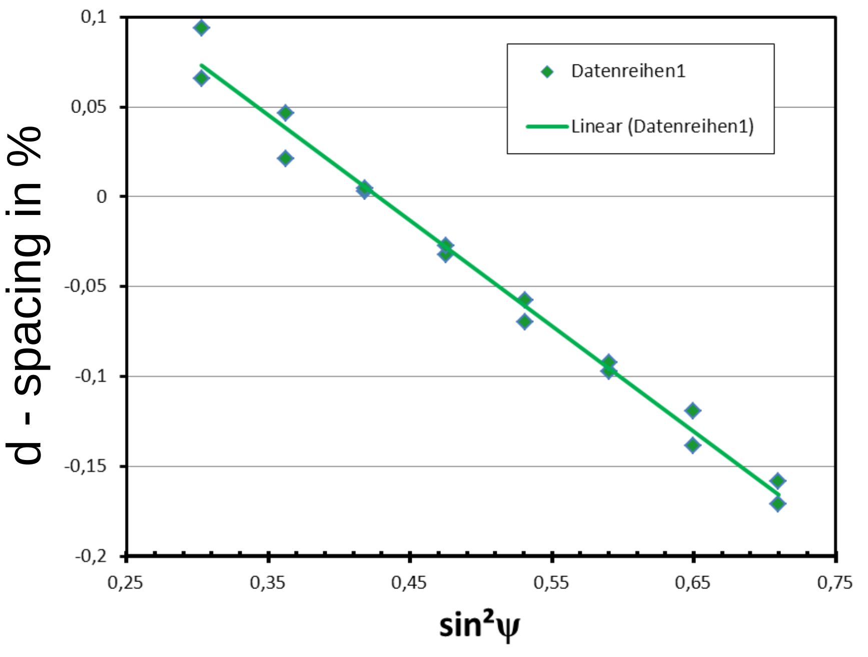
$$\tau = \frac{\cos\chi\sin(2\theta-\Omega)\sin\Omega}{\mu(\lambda)\cdot(\sin\Omega+\sin(2\theta-\Omega))}$$

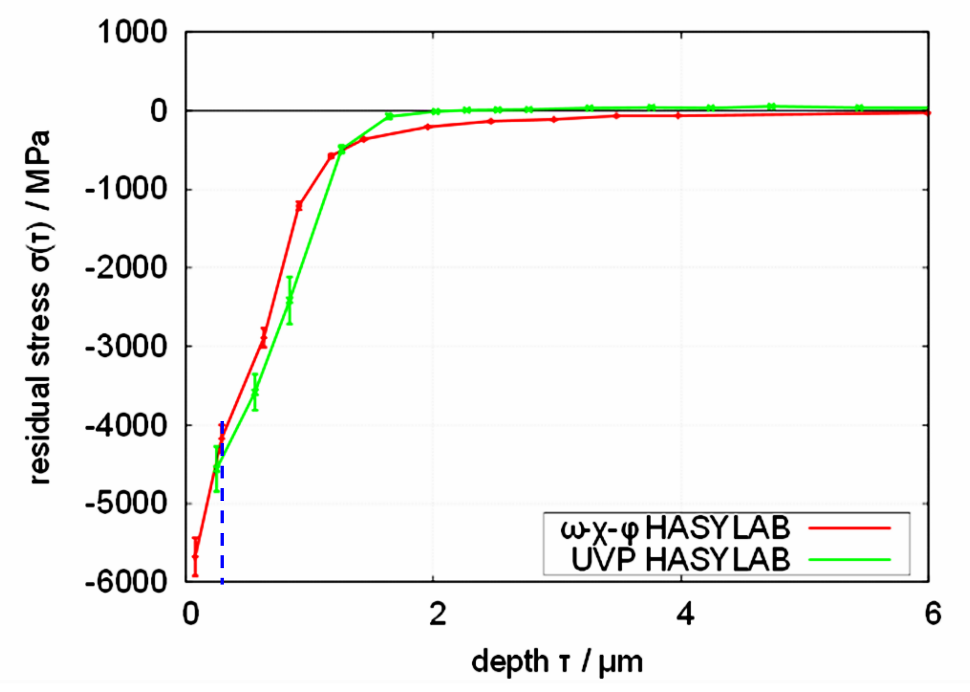


τ=1µm

Measurement results: Ω-χ-φ

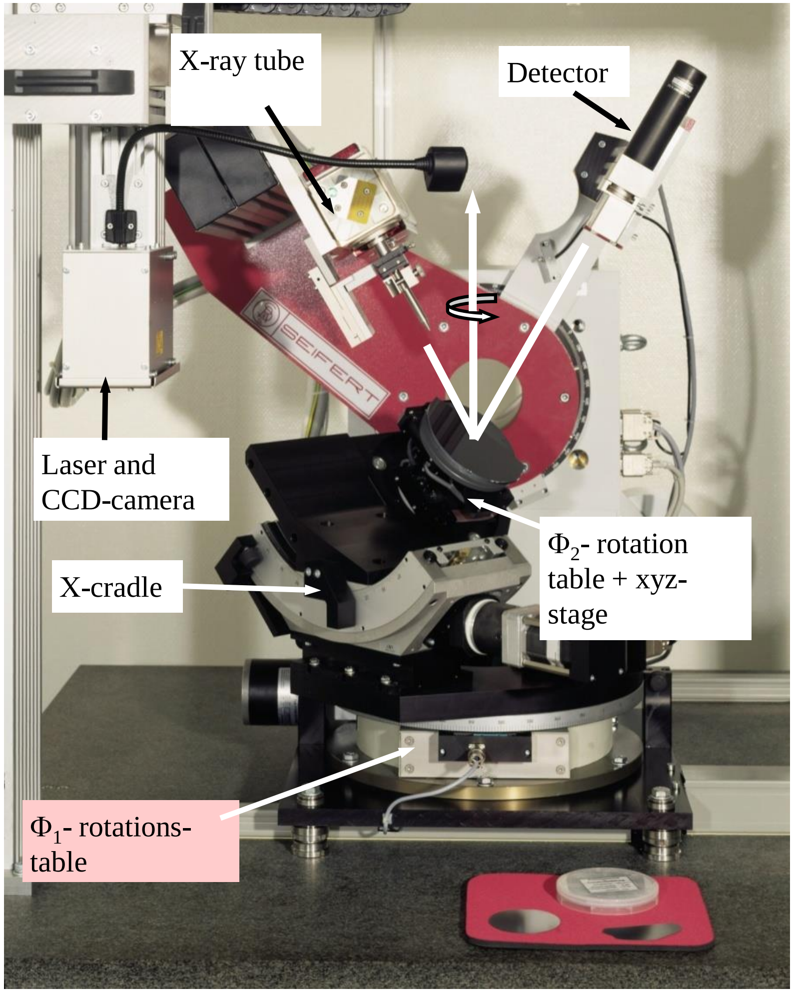
Ω-χ-φ-Methode using Co Kα- X-rays and synchrotron

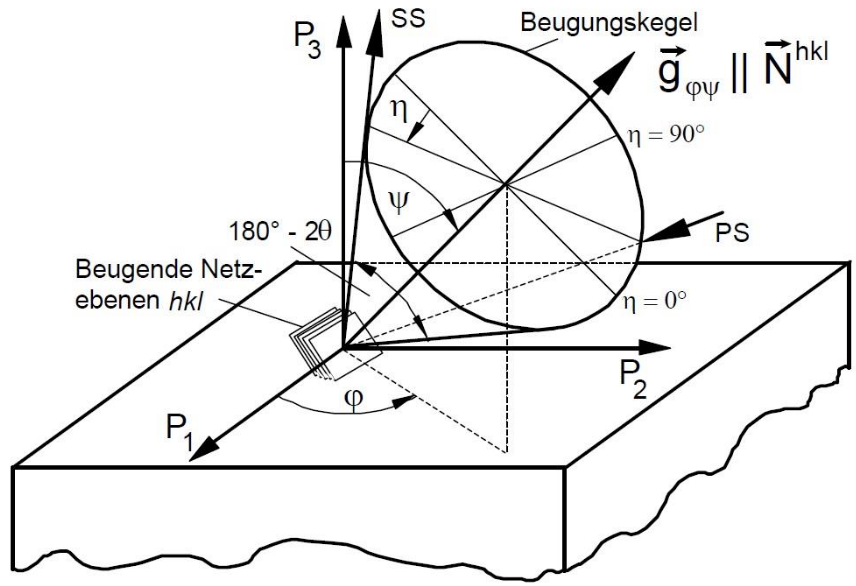




Information depth τ=0,3µm Al_2O_3 116 ceramic sample

Measurement methods: η – method



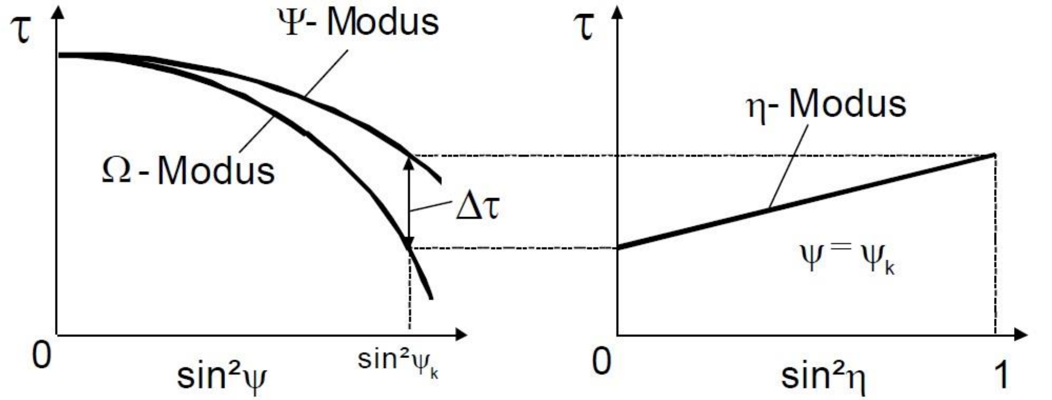


Quelle: C. Genzel

Quelle: C. Genzel. Entwicklung eines Mess- und Auswerteverfahrens zur röntgenographischen Analyse des Eigenspannungszustandes im Oberflächenbereich vielkristalliner Werkstoffe. 1999. Habilitationsschrift Humboldt-Universität zu Berlin.

Measurement methods: η – method

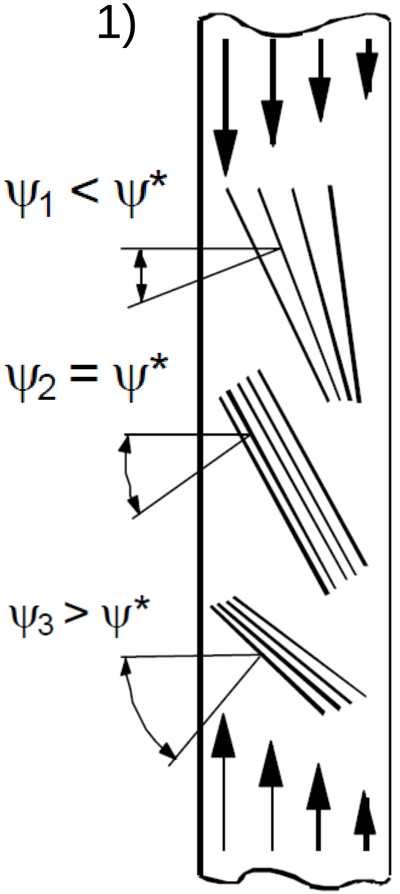
$$\tau = \frac{\sin^2\theta - \sin^2\psi + \cos^2\theta\ \sin^2\psi\ \sin^2\eta}{2\mu \sin\theta\ \cos\psi}$$

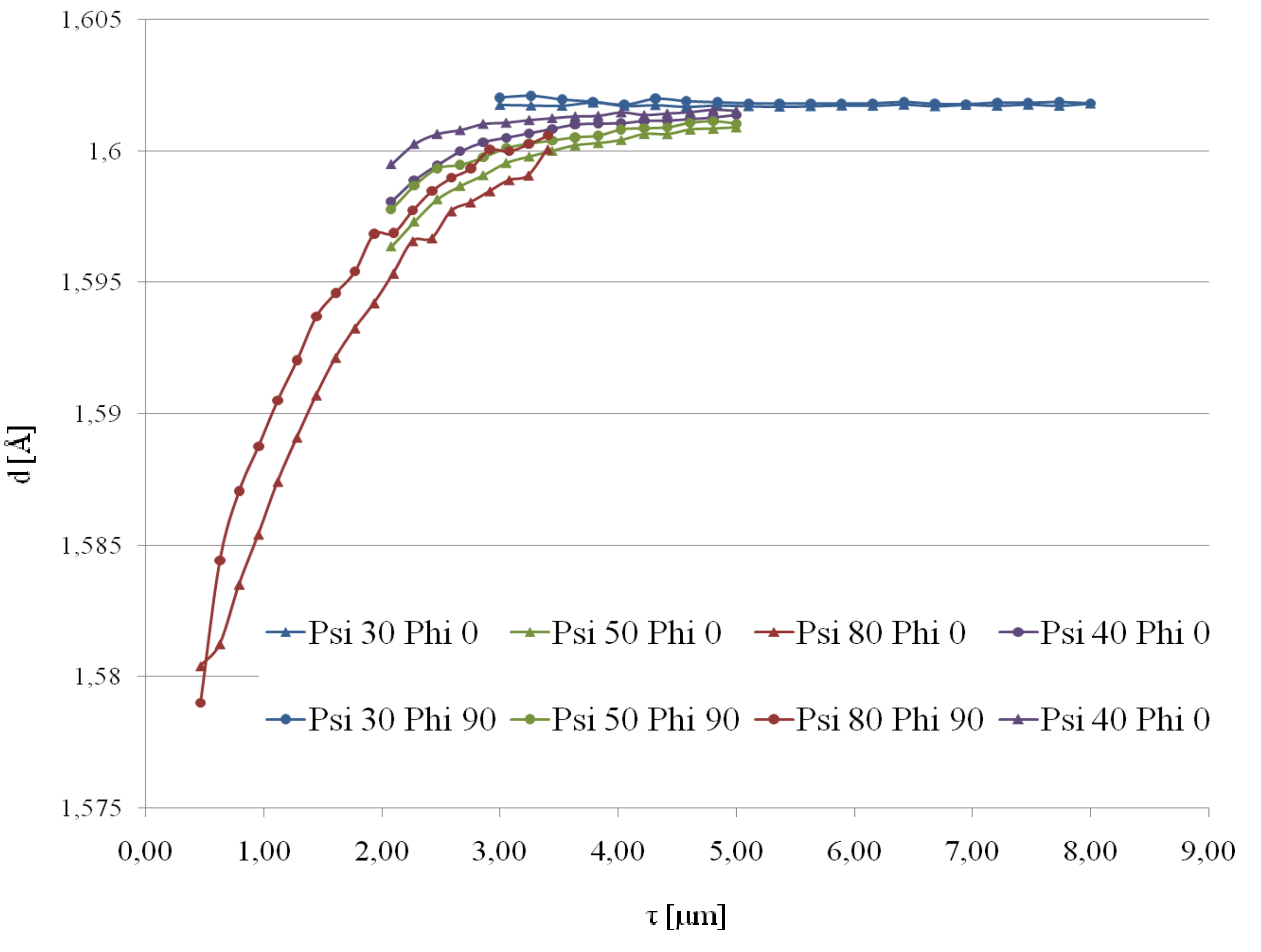


- turning around η with constant ψ bridging the gap $\Delta\tau$ between τ_Ψ and τ_Ω
- $\Delta\tau = \tau_\Psi - \tau_\Omega$ decrease for smaler ψ-angle
- for smaler θ-angle the covered τ-area increases with comparable $\Delta\sin^2\eta$ - steps

Source: C. Genzel. Entwicklung eines Mess- und Auswerteverfahrens zur röntgenographischen Analyse des Eigenspannungszustandes im Oberflächenbereich vielkristalliner Werkstoffe. 1999. Habilitationsschrift Humboldt-Universität zu Berlin.

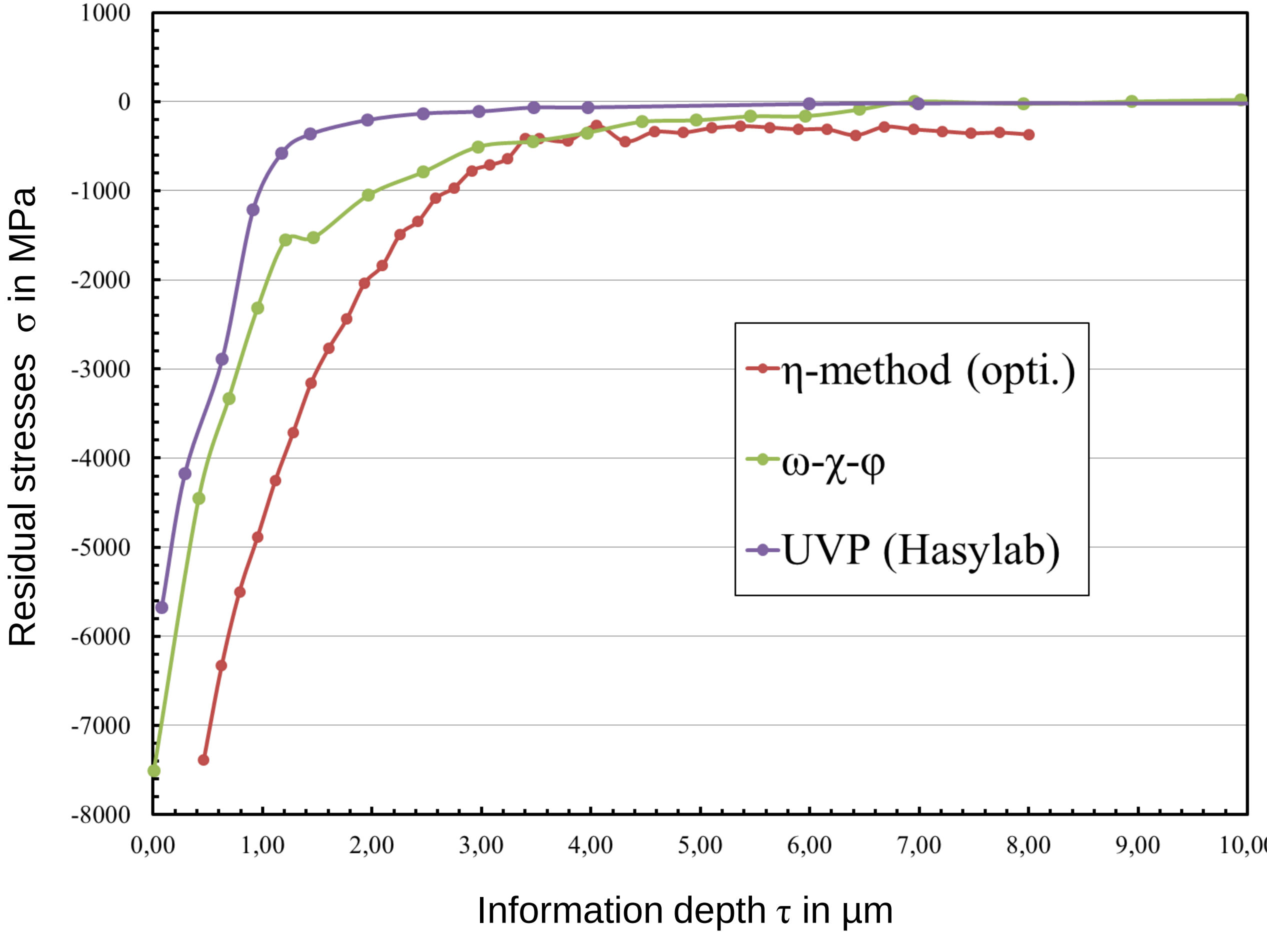
Measurement methods: η – method



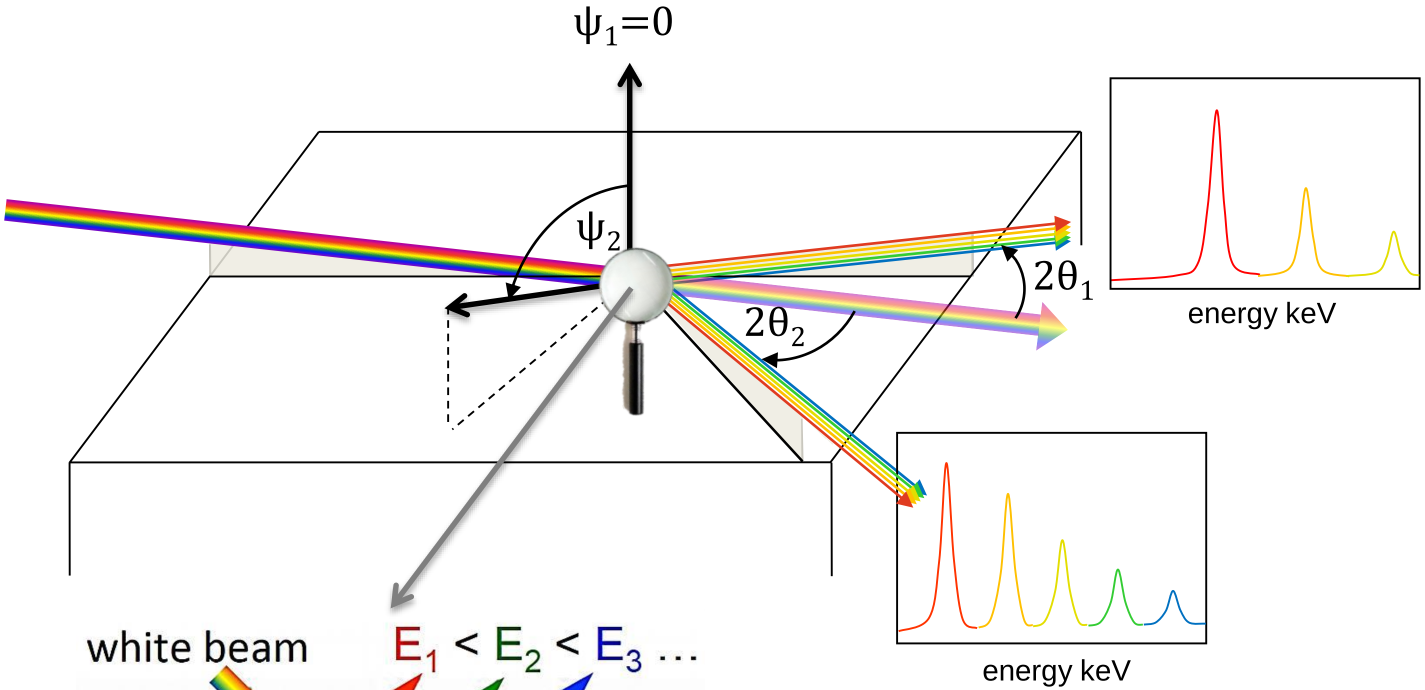


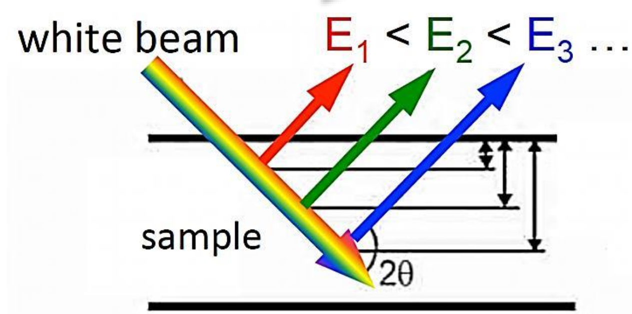
1): C. Genzel. Entwicklung eines Mess- und Auswerteverfahrens zur röntgenographischen Analyse des Eigenspannungszustandes im Oberflächenbereich vielkristalliner Werkstoffe. 1999. Habilitationsschrift Humboldt-Universität zu Berlin.

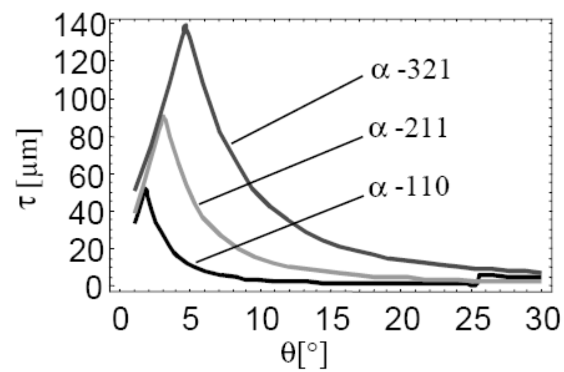
Measurements results (image space)



Energy dispersive two detector mode



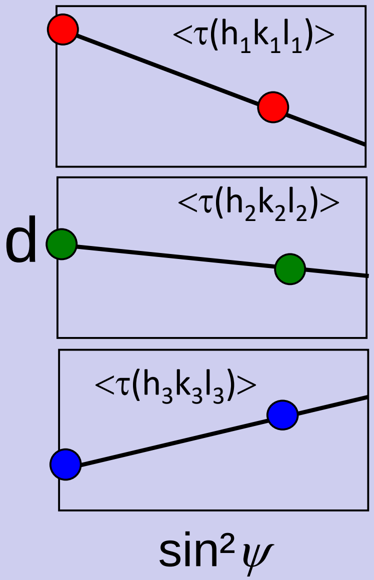




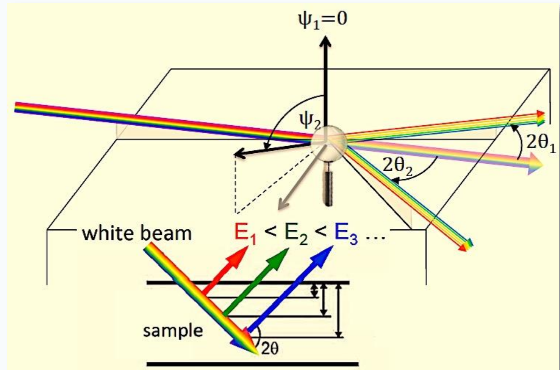
Quelle: C. Stock: Dissertation Berlin

Energy dispersive two detector mode

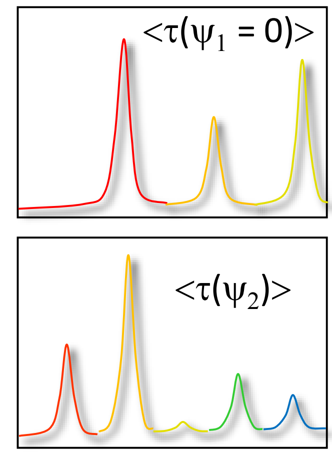
stresses



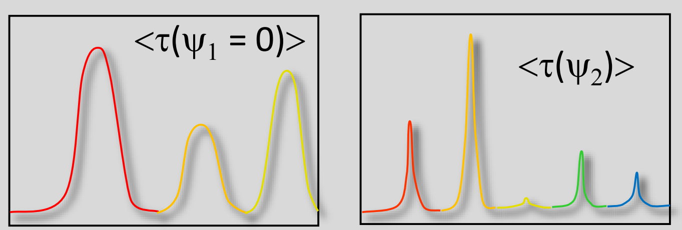
Measurements without sample movement



Phase and texture analysis



Microstructure

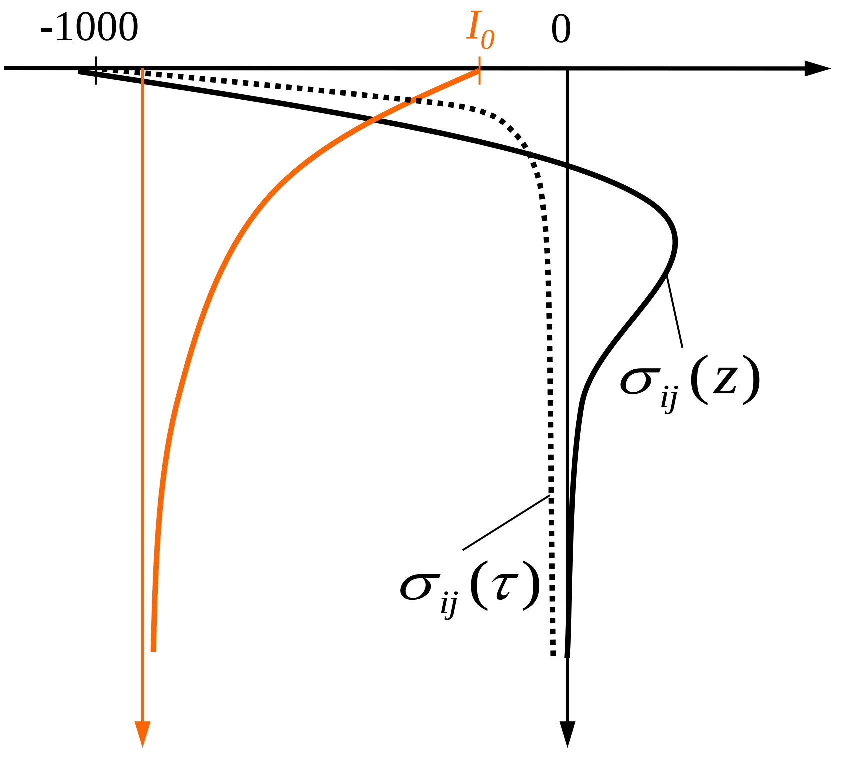


Transformation methods: initial inverse problem

Relation between measured stress in image space and real stress:

Lambert-Beers law: $I = I_0 \cdot e^{-\mu(\lambda)\cdot s}$

$$\sigma(\tau) = \frac{\int_0^D e^{-\frac{z}{\tau}} \cdot \sigma(z)dz}{\int_0^D e^{-\frac{z}{\tau}} dz},$$



where
D - sample thickness,
z - depth in real space,
τ - depth in image space,
$\sigma(z)$ - residual stress in real space,
$\sigma(\tau)$ - residual stress in image space

Graphic: T. Manns: Dissertation Universität Kassel

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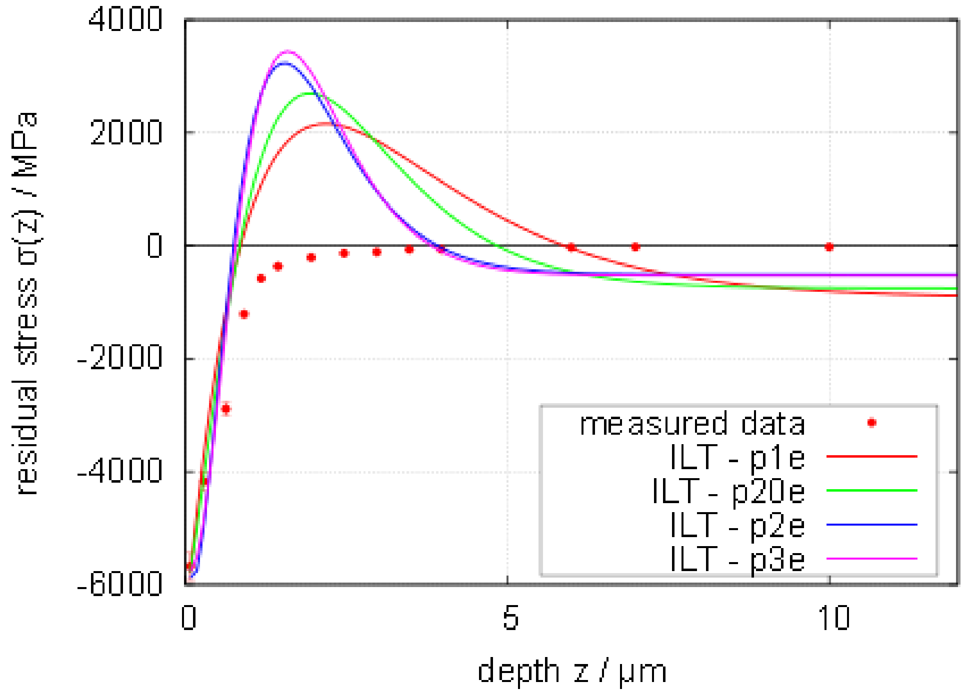
Prof. Dr.-Ing. Niendorf

Transformation methods: Inverse Laplace transformation (ILT)

Initial inverse problem in form of inverse Laplace transformation:

$$\sigma(\tau) = \frac{1}{\tau} L[\sigma(z), \frac{1}{\tau}] = \frac{1}{\tau}\int_0^{\infty} e^{-\frac{z}{\tau}} \sigma(z) dz$$



<u>Problems:</u>

- Complexity of the solution choice.
- No solution error estimation.

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Conclusion

Depth distribution:

- Different measurement methods realize different depth distributions
- Maximum penetration depth in Fe under lab condition ~60µm (synchrotron ~100µm)

Measurement method:

- Measurements with a complete spectrum of an X-ray tube are possible!
- Transformation is necessary to calculate the data in real space

Thank you for your attention

We are grateful to the German Research Foundation (DFG) for the financial support of one of these projects.

We are also grateful to HZB, HZG and DESY for beamtime and to the beamline scientists for valuable assistance during and after our experiments.