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THIRD WORKSHOP ON
THIN FILMS PHYSICS AND TECHNOLOGY
(8 - 24 MARCH 1999)
including
TOPICAL CONFERENCE ON
MICROSTRUCTURE AND SURFACE MORPHOLOGY
EVOLUTION IN THIN FILMS
(24 - 26 MARCH 1999)

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# "X-ray techniques for the characterisation of thin films and layered structures"

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# X-ray techniques for the characterisation of thin films and layered structures

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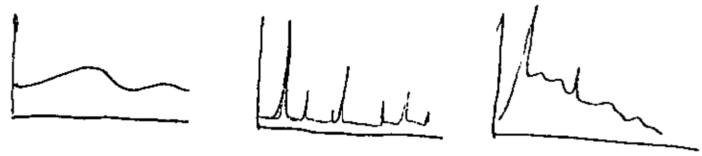
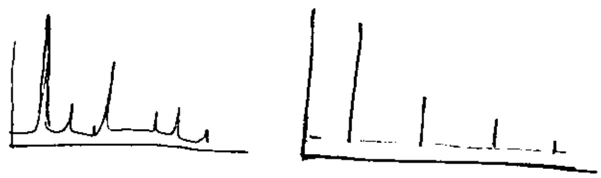
Contents

**1. General classification of structure of thin films**

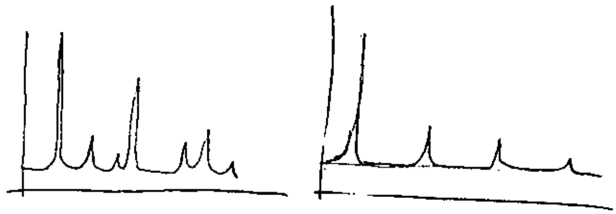
A. <u>Crystalline state</u>: amorphous, crystalline, ordered polymers



Crystalline thin films: polycrystalline, epitaxial

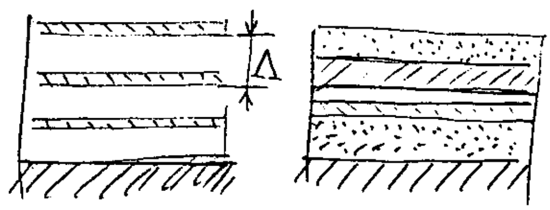


Polycrystalline thin films: with random grain orientation, textured

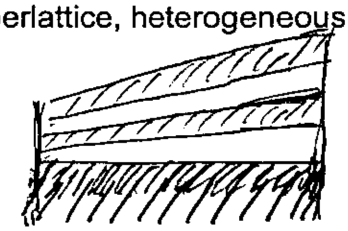


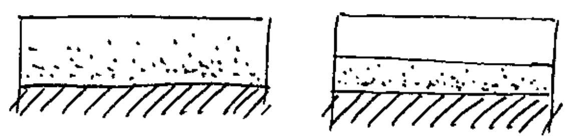
B. <u>Geometrical featuers</u>: single layer, multilayer

Multilayer: superlattice, heterogeneous multilayer (ML)



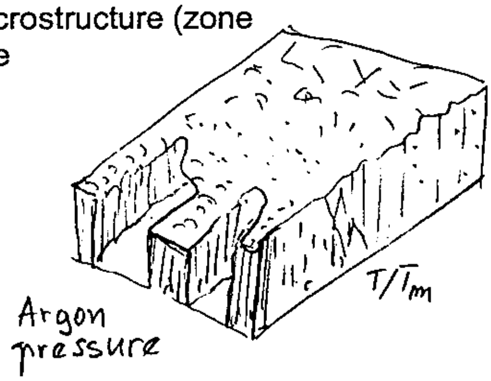

Graded thin films: single layer, superlattice, heterogeneous ML





C. <u>Scale of view</u>: microstructure (zone model), picostructure

Thornton's zone model

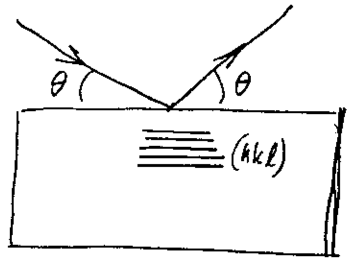



<u>XRD</u>:
macrostress σ
microstrain e
lattice parameter a(hkl)

## 2. Diffraction geometries

<u>Symmetric</u>

Bragg-Brentano optics (B-B)

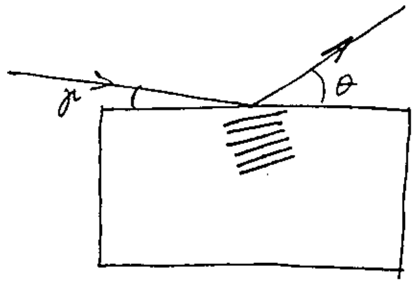



<u>Asymmetric</u>

parallel beam

or

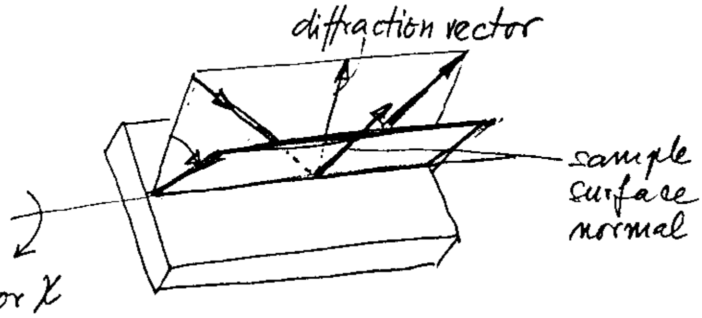
Seemann-Bohlin optics (S-B)




larger irradiated volume
⇒ higher intensity
⇒ better angular resolution

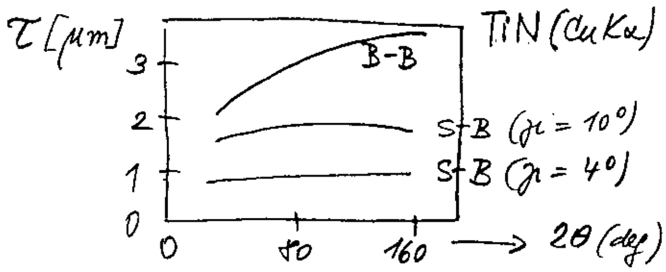
Special diffractometers

(for textur or stress analysis, grazing incidence)




γ or χ ≈ 90°

Sampling depth




S-B: lower sampling depth
⇒ reduction of the substrate reflections

Thickness measurements

(difficult)

↑

TEXTURE

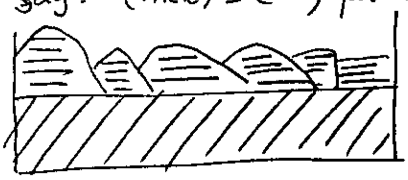
a) from intensity of substrate reflections

b) from intensity of a reflection from the thin film

## 3. Main differences between structure of thin films and powder samples

Say: $(hkl) \equiv (111)$ parallel to the subst.

Unique direction of the film growth ⇒ texture



Adhesion to the substrate ⇒ stress

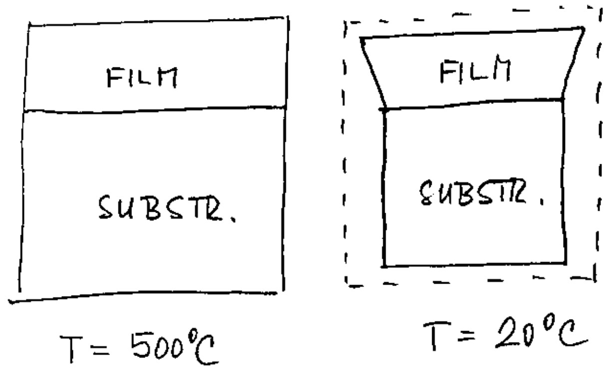
Thermodynamically unstable deposition conditions ⇒ unstable phases, lattice distortions

Bragg's law:

$d$ ... lattice spacing
$\theta$ ... Bragg angle
$\lambda$ ... x-ray wavel.

$$2d\sin\theta = \lambda$$

COMPRESSION OF THE FILM

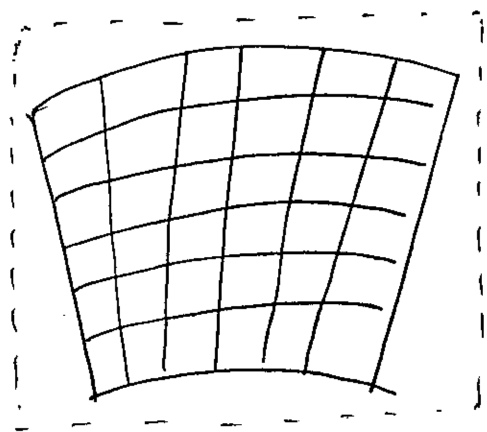



Macroscopic stress ⇒ macrostrain (the same deformation of all grains) ⇒ peak shift.

Residual macrostresses: thermal and intrinsic, with the film in compression or in tension

Microscopic stress ⇒ microstrain (deformations which can vary from grain to grain or inside of grains) ⇒ line broadening

[but line broadening can also be caused by a small grain size)



lattice spacing fluctuations $\left\langle \frac{\Delta d}{d} \right\rangle \Rightarrow \left\langle \frac{\Delta \theta}{\theta} \right\rangle$ line broadening

Residual stress measurements:

„sine square plots"

$$a_\psi = \left(\frac{a_\perp \sigma}{E}\right)\left[(1+\nu)\sin^2\psi - 2\nu\right] + a_0$$

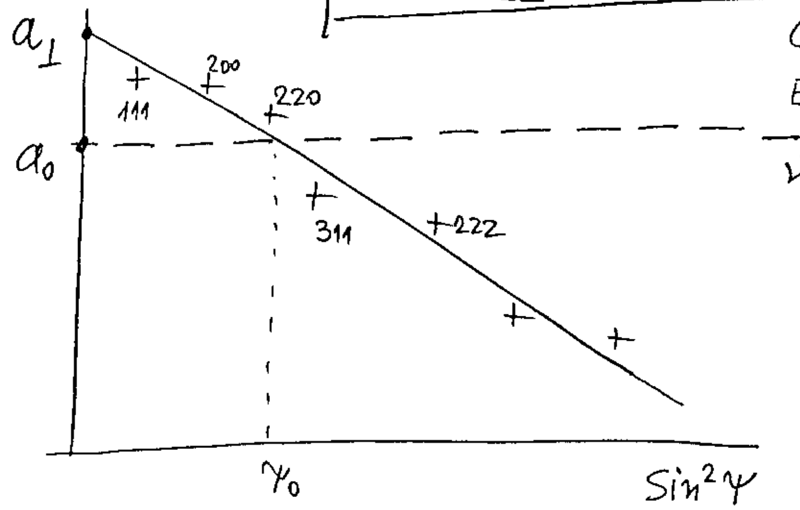
$\sigma$ .. stress [GPa]

E ... Young. modul

$\nu$ .. Poisson's ratio

lattice parameter of a cubic thin film

(S-B) method




Microstrain and grain size estimatioms:
Williamson-Hall plot

(or the Warren-Averbach approach)

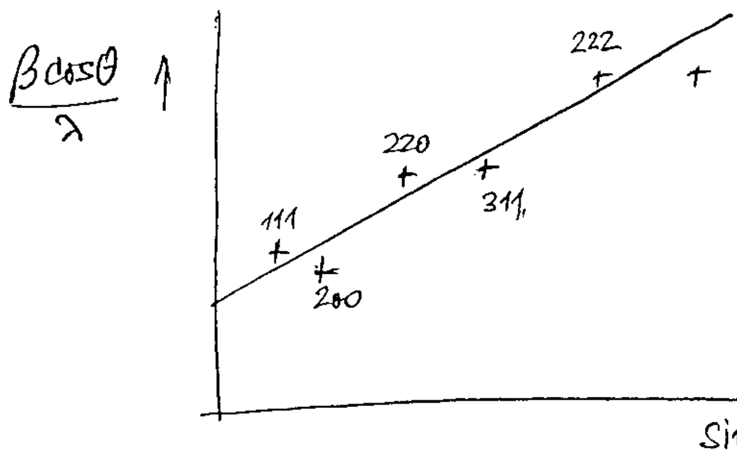
$$\frac{\beta\cos\theta}{\lambda} = \frac{1}{D} + \frac{4e}{\lambda}\sin\theta$$

$e = \left\langle \frac{\Delta d}{d} \right\rangle$ microstrain [%]

$\beta$ ... FWHM

$\theta$ ... Bragg angle

D ... domain size



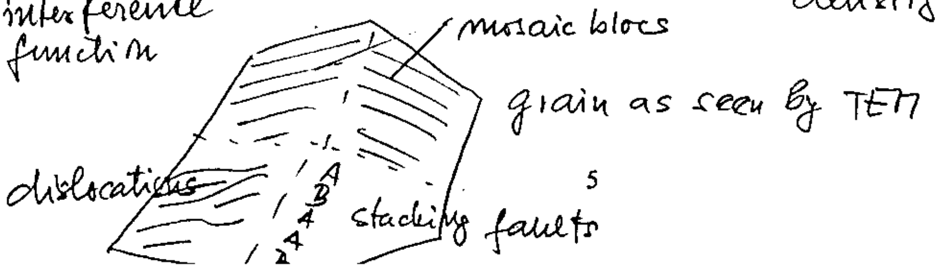

A note to grain size analysis from XRD line broadening

$$\left(\frac{\sin Nx}{x}\right) \Longrightarrow \beta = \frac{\lambda}{D\cos\theta}$$

interference function

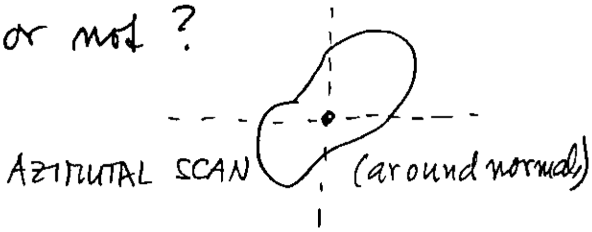
Scherrer's formula

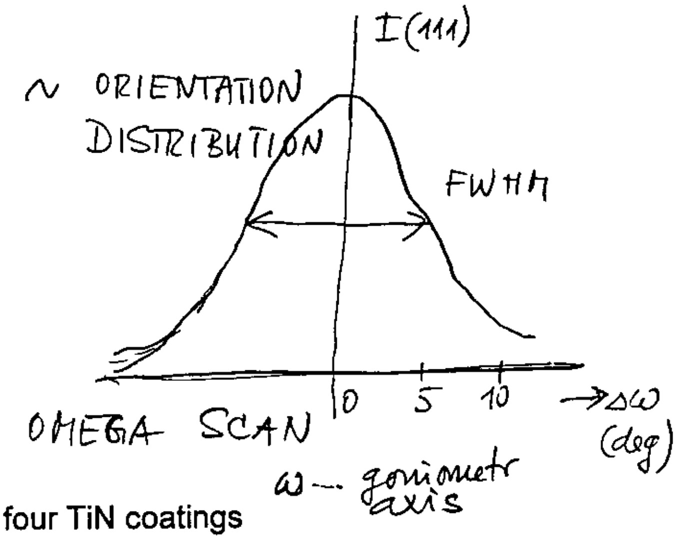
D is an apparent size of coherent blocks (and also reflects density of defects!)



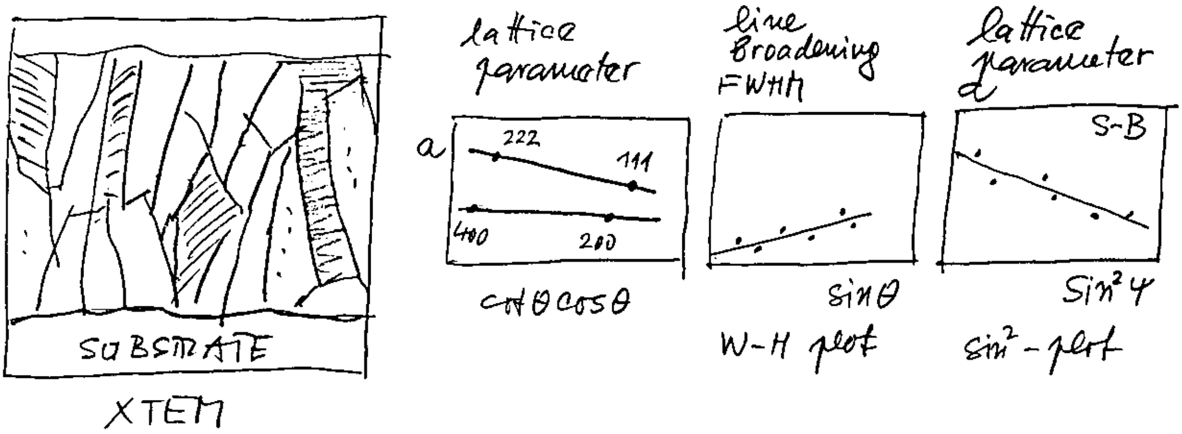
Texture analysis

Is it a fibre texture or not?



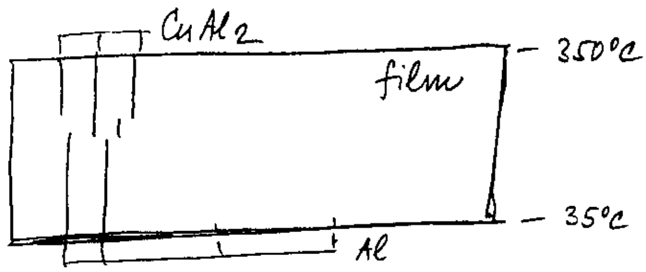

A comparison of micro- and picostructure of four TiN coatings




## 4. Solid state reactions and phase transformations

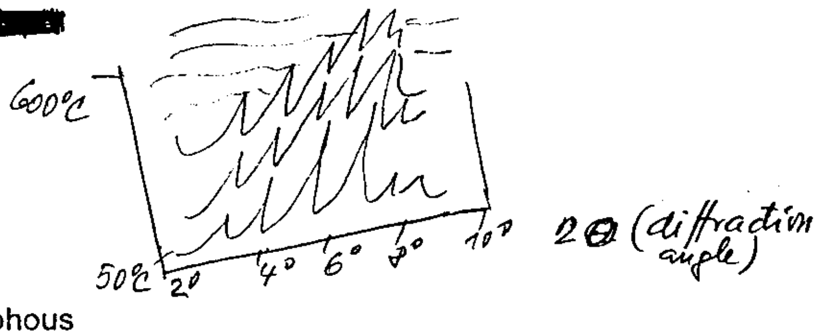
Cu-Al couples, thermal annealing

Cu (150nm)/Al (425nm)

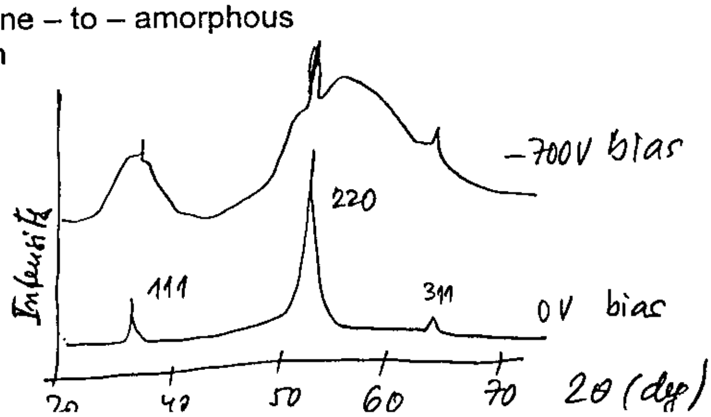



Ta-Si amorphous multilayers, [redacted]
sampling of interdiffusion

Ta(40Å)/Si(70Å)




Silicon, crystalline – to – amorphous
phase transition



## 5. Comparison of XRD with structure imaging techniques

Microscopic techniques (SEM, TEM, STM, AFM, HRTEM) – local information

Problems: destructive, surface structures, structural changes caused by sample preparation

Advantages: grain size, grain morphology, grain packing

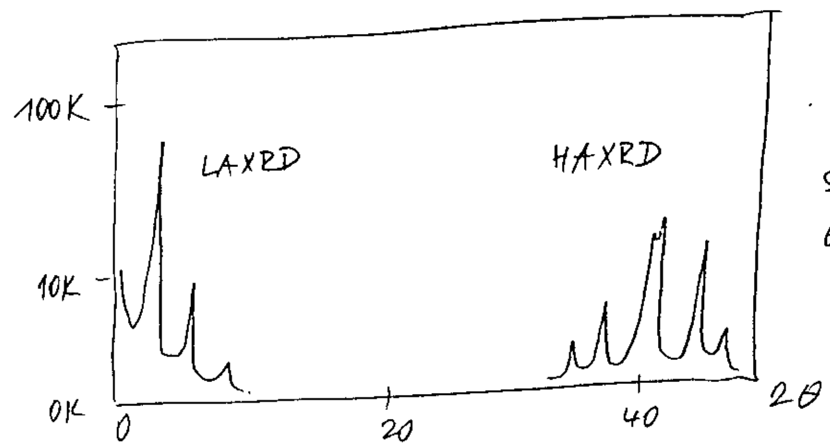
XRD techniques – volume averaged information

Problems: low spatial resolution (interfaces, defects), indirect information

Advantages: nondestructive, fast

## 6. Basic methods of structure analysis of multilayers

General features of XRD patterns of multilayers




Au/Co multilayer

symmetrical θ-2θ scan

Modified „Bragg laws" ⇒ density determination, superlattice periodicity, the average lattice spacing

$$\sin^2\theta_m = \left(\frac{m\lambda}{2\Lambda}\right)^2 + 2\delta_s$$

real part of index of refraction = $1-\delta_s$ ⇒ **density**

$$\frac{2\sin\theta_m}{\lambda} = \frac{1}{\langle d\rangle} + \frac{m}{\Lambda}$$

$\langle d\rangle = \Lambda/(N_A+N_B)$

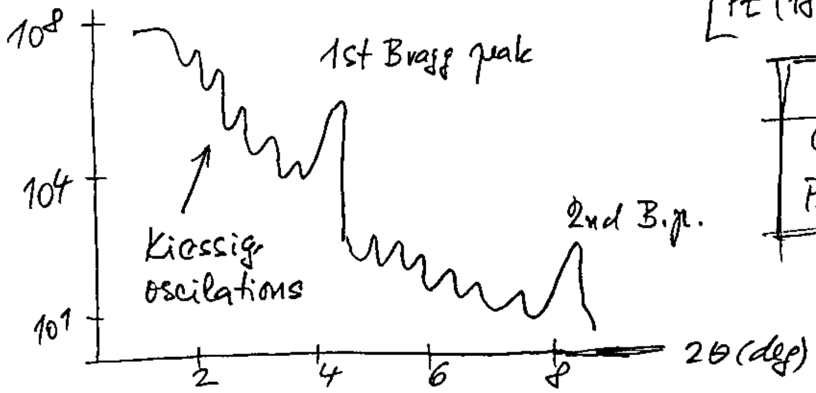
$\lambda$ ... x-ray wavelength

$\Lambda$ ... superlattice periodicity

$m = 0, \pm1, \pm2$ ... order of satellite

Reflectivity curves and their fitting ⇒ density, thickness, roughness

LA XRD

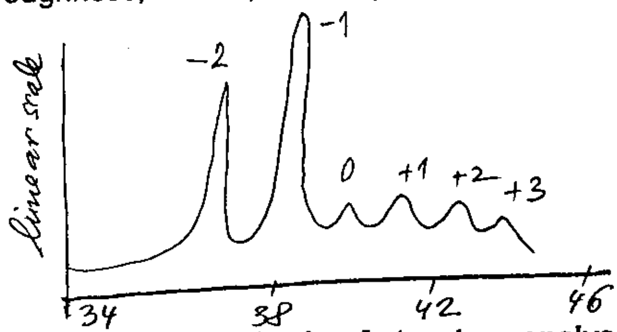


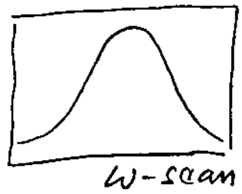

[Pt (18Å)/Co(2Å)] x 15 / GaAs

| | thick (Å) | relat. density | roughness (Å) |
|---|---|---|---|
| Co | 2.1 | 1.32 | 4.1 |
| Pt | 19.8 | 0.88 | 3.4 |

High-angle groups of satellites (for crystalline ML only) ⇒ thickness, roughness, texture, lattice spacings, residual stress, microstrain

HA XRD

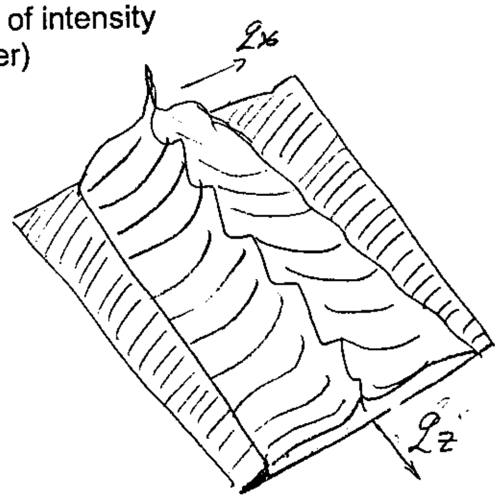



[Ag(40Å)/NiFe(20Å)] × 10

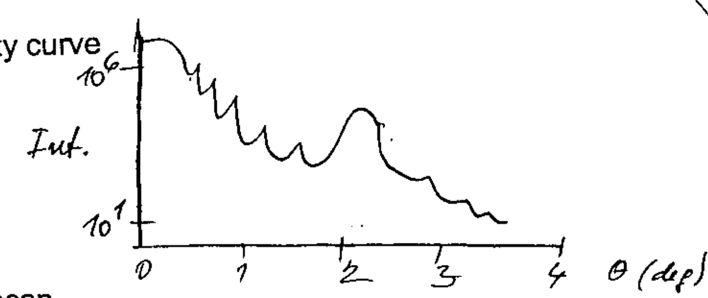
## 7. Advanced methods of structure analysis of multilayers

Diffuse scattering at low angles, area scans for a sampling of intensity distribution in the XZ-plane of the reciprocal space (Au layer)

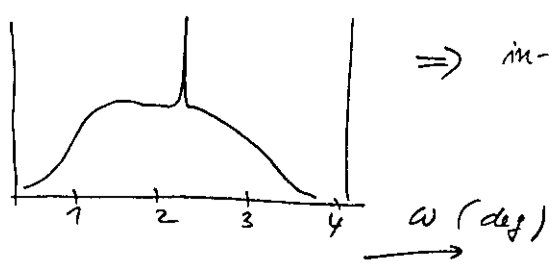



[$Ni_{30}Co_{70}$(5.6Å)/Au(15Å)] x 16 mulitlayer, an example:

- reflectivity curve

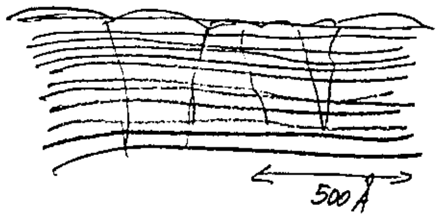



- omega scan

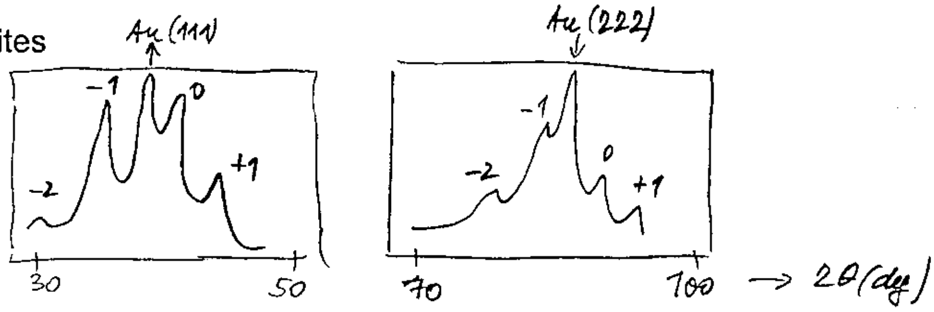



⇒ in-plane correl. length = 390 Å

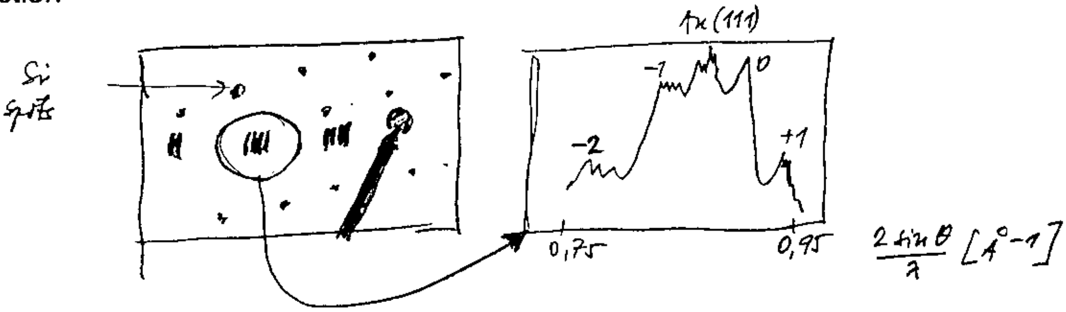
- XTEM




- high-angle XRD, satellites

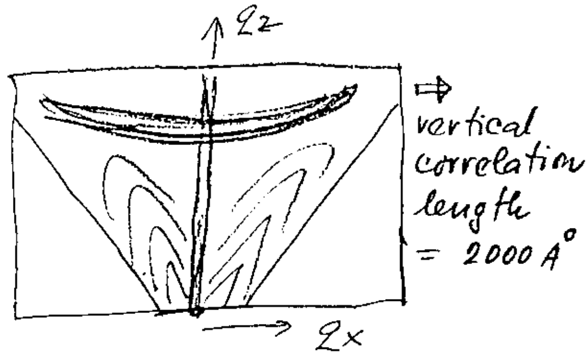



- electron diffraction

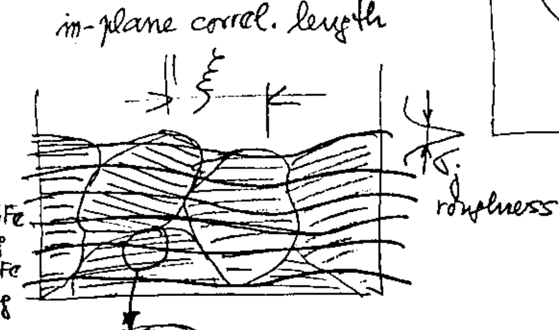



- AFM: surface


- Area scan: vertical correlation of the roughness




- Roughness




- HREM