

# Coherent GISAXS investigation of granular microstructure in thin metal films

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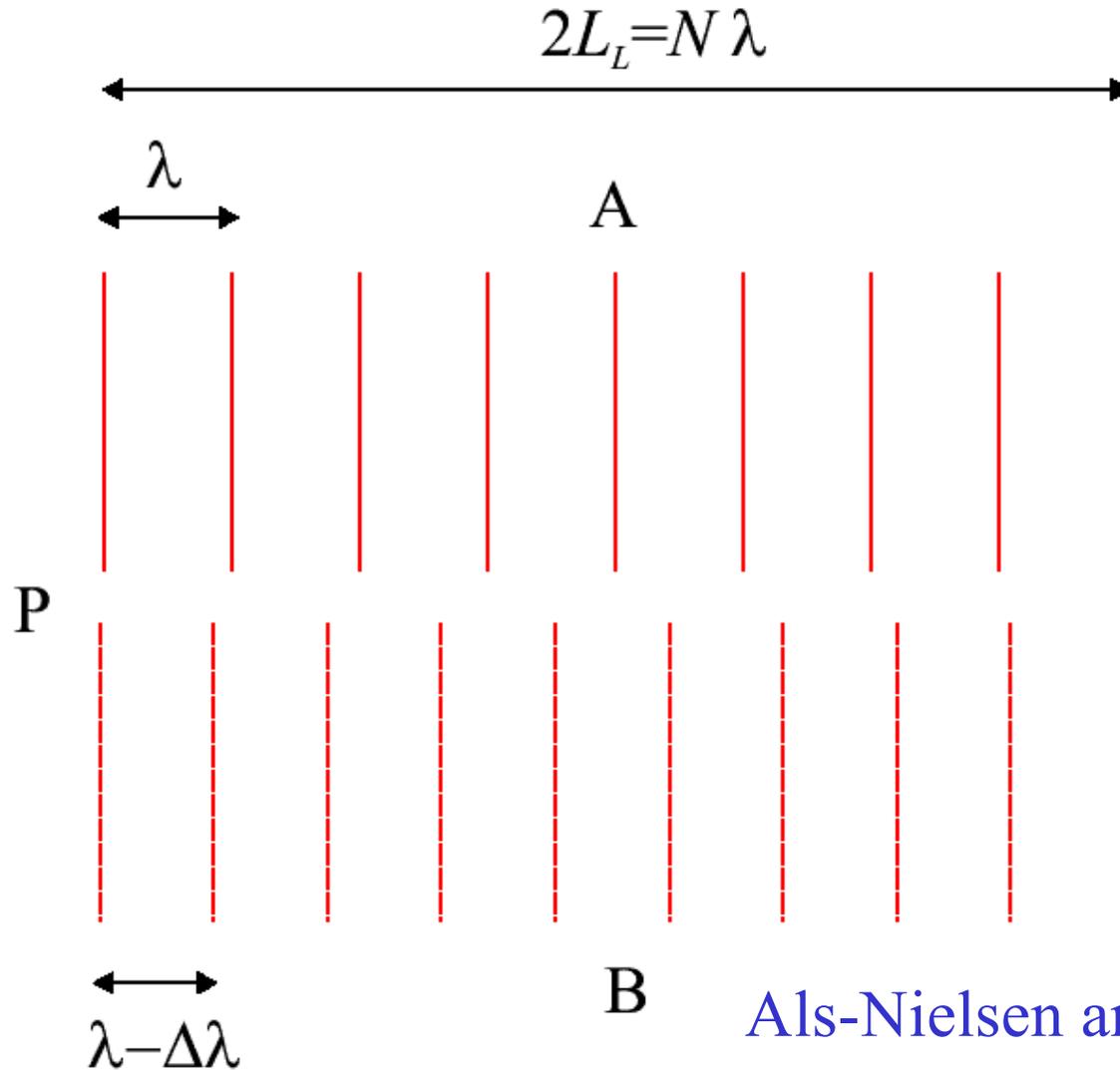
University of Illinois  
APS Sector 34-ID-C

NSLS GISAXS Workshop  
May 17, 2004

# Outline

- Coherence in Diffraction
- CXD Beamline at APS
- Coherent “Yoneda” geometry
- Gold polycrystalline films
- Future prospects for CGISAXS

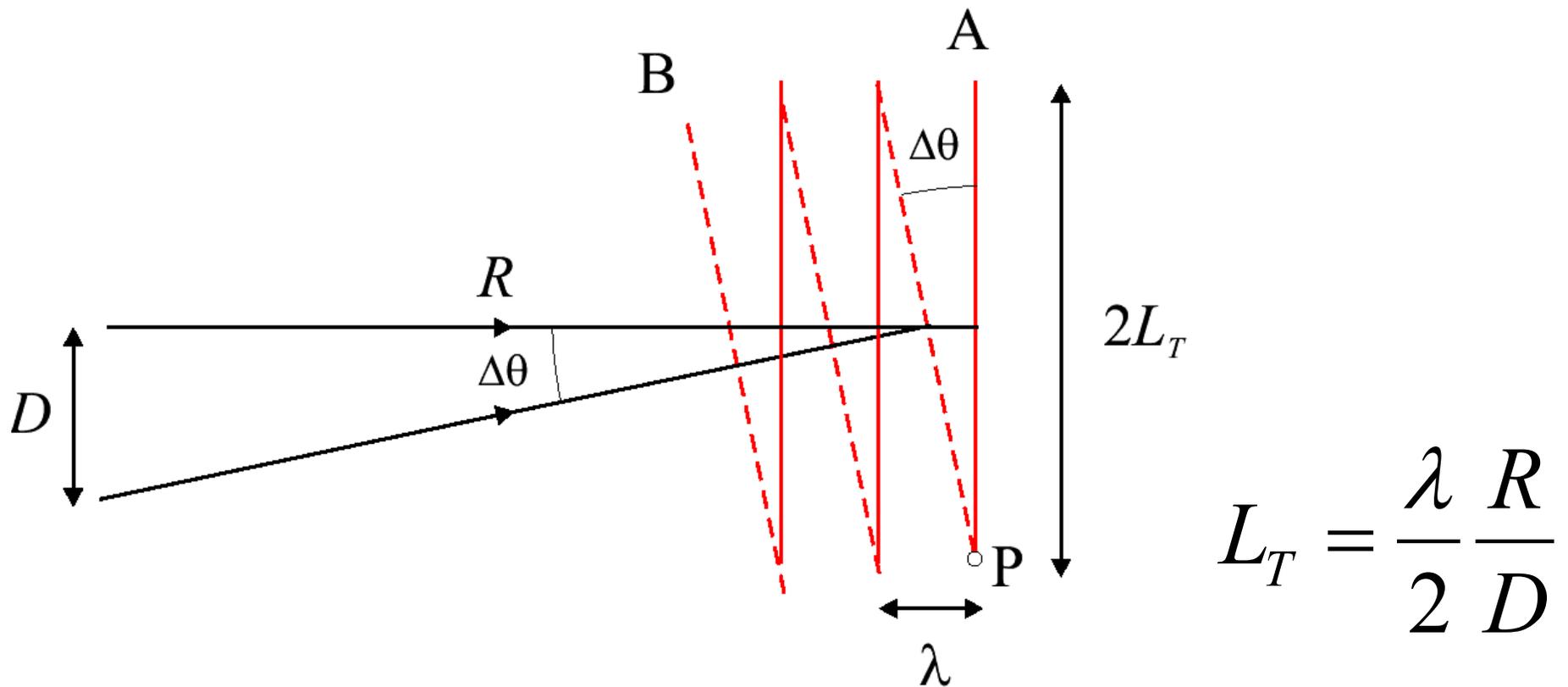
# Longitudinal Coherence



$$L_L = \frac{1}{2} \frac{\lambda^2}{\Delta\lambda}$$

Als-Nielsen and McMorrow (2001)

# Lateral (Transverse) Coherence



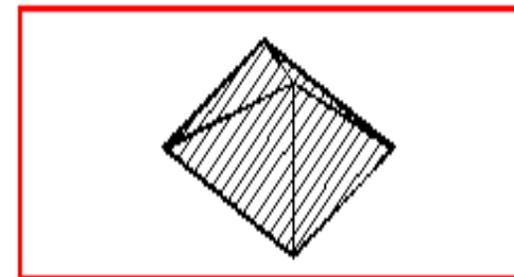
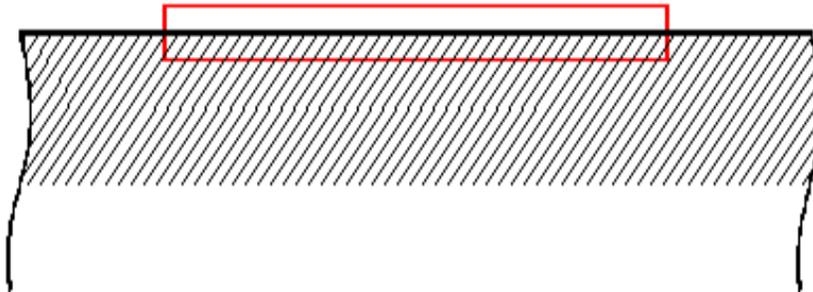
Als-Nielsen and McMorrow (2001)

# Coherence at the APS or ESRF

Typical of 3rd Generation (undulator) Synchrotron Source

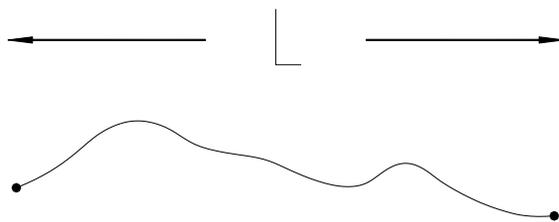
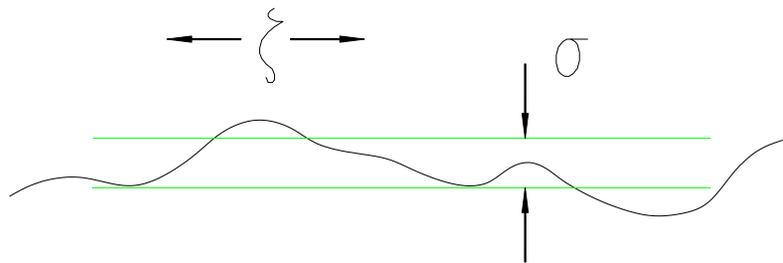
Coherence of	$\xi_{\text{VER}}$	$\xi_{\text{HORIZ}}$	$\xi_{\text{LONG}}$	Flux
Raw Undulator	35 $\mu\text{m}$	9 $\mu\text{m}$	0.004 $\mu\text{m}$	$2 \times 10^{12}$
Si(111) Monochromator	35 $\mu\text{m}$	9 $\mu\text{m}$	1 $\mu\text{m}$	$1 \times 10^{10}$
C(111) Monochromator	35 $\mu\text{m}$	9 $\mu\text{m}$	3 $\mu\text{m}$	$3 \times 10^9$

Coherent region defined by slits

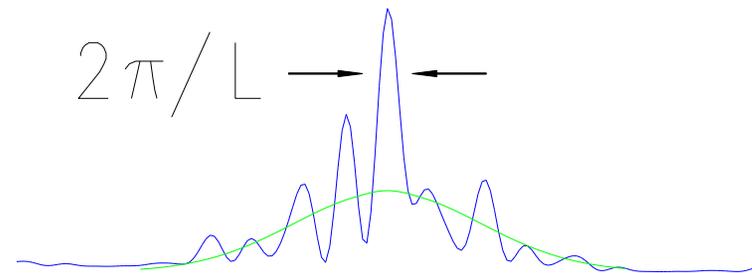
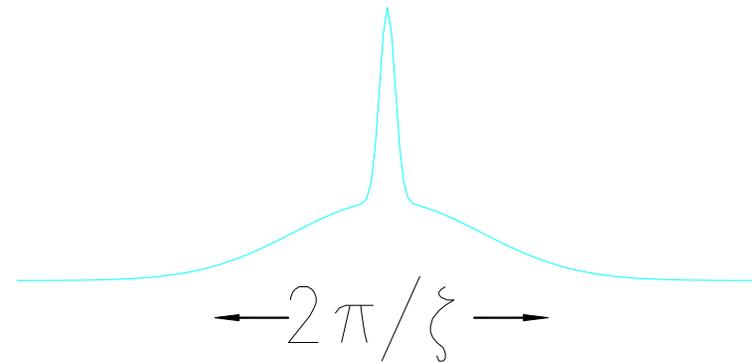


# Diffuse Scattering acquires Structure using CXD

Real Space

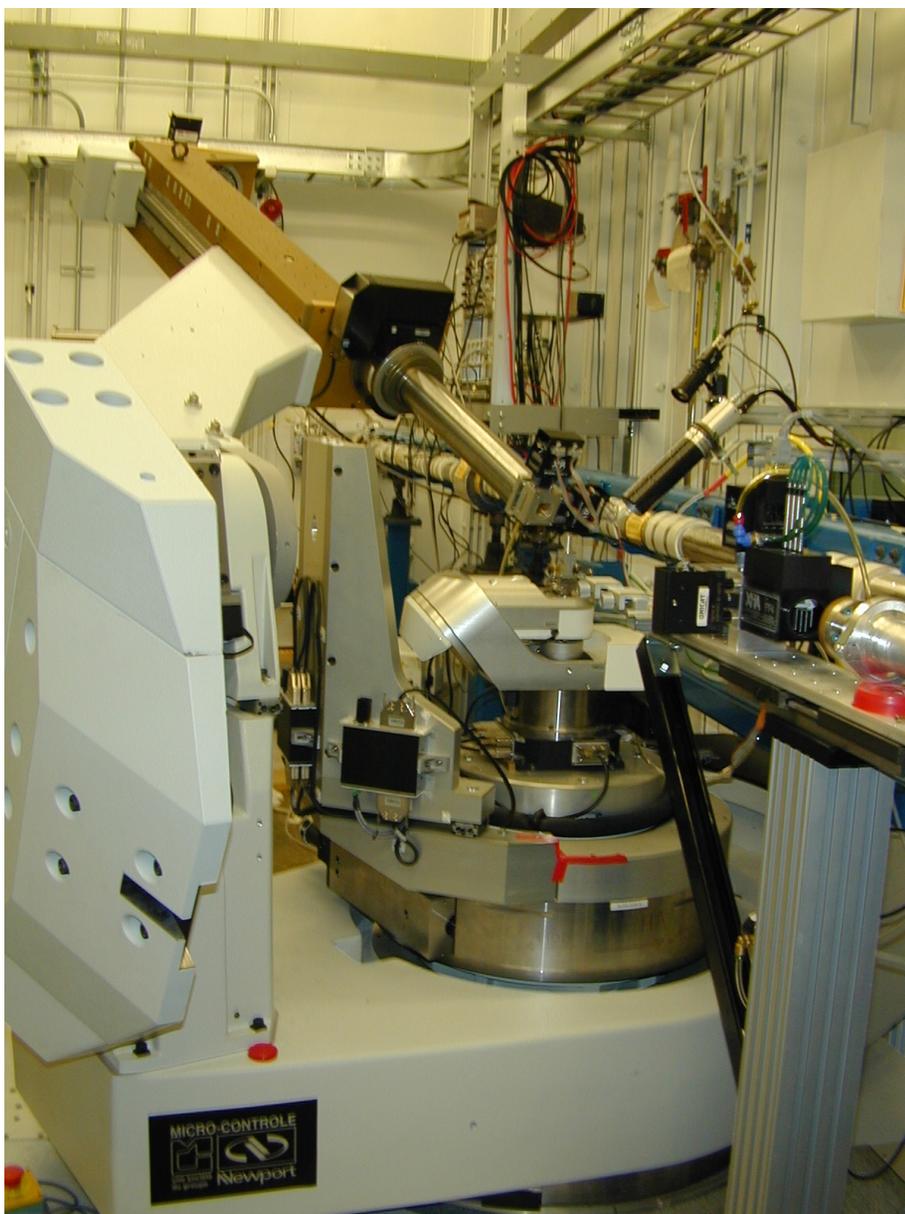


Reciprocal Space

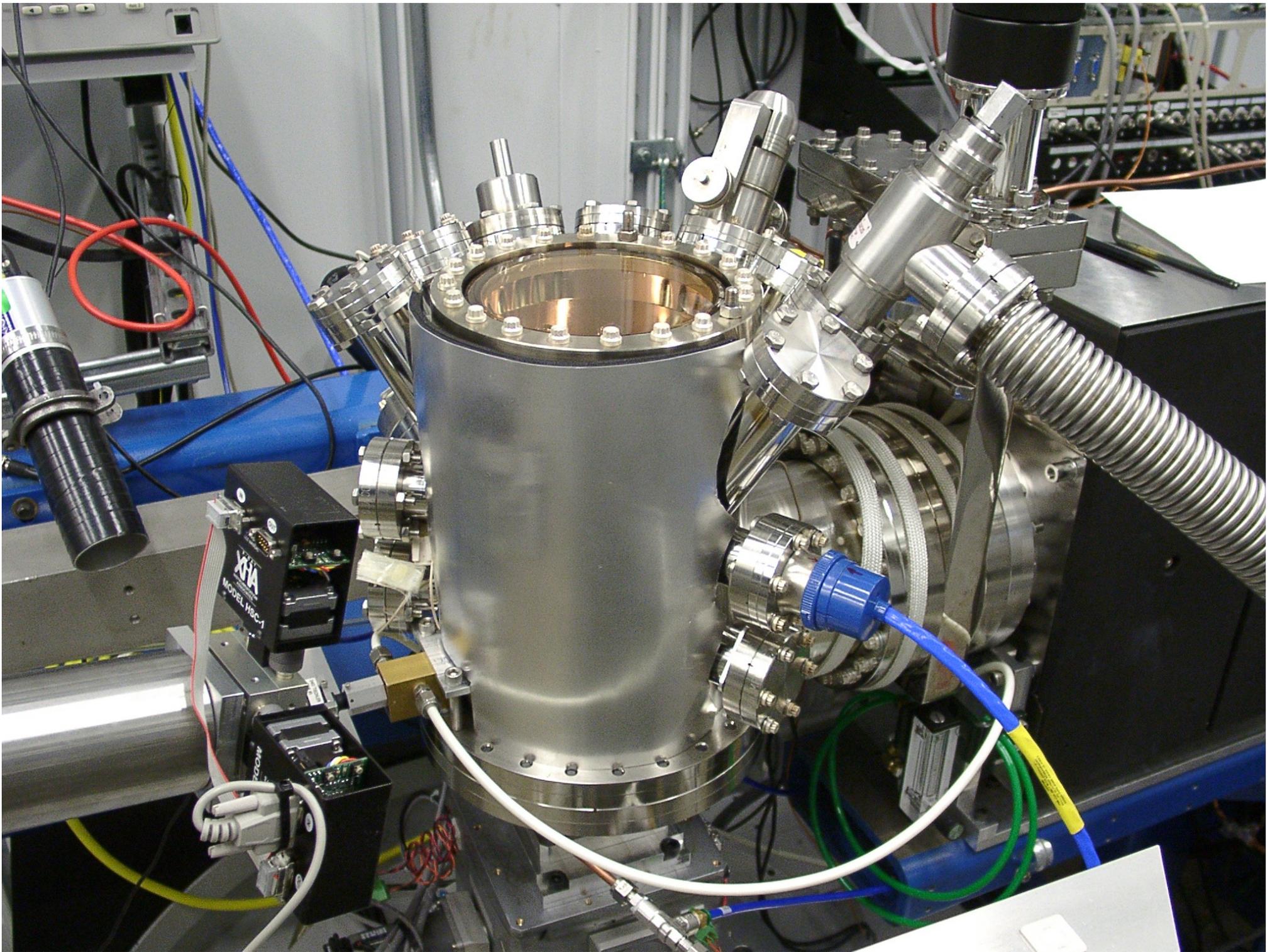


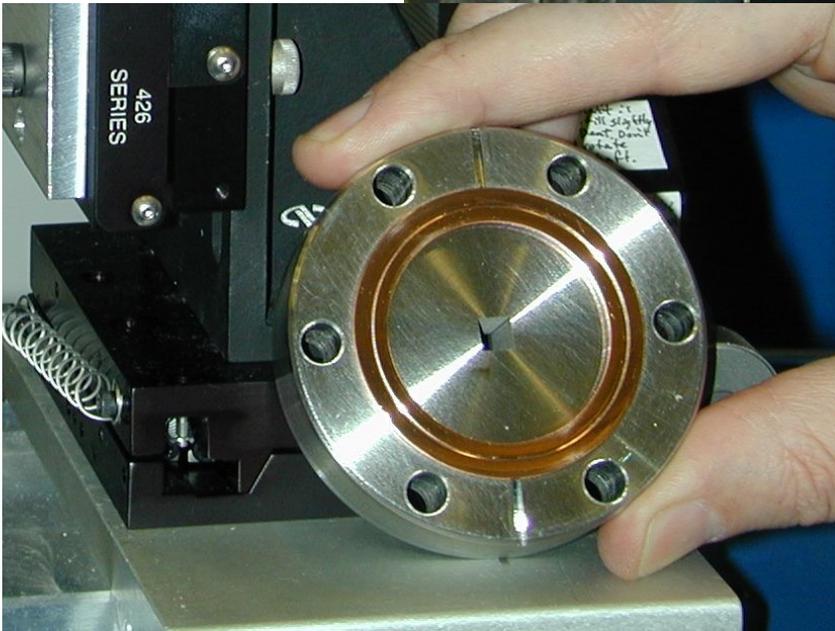
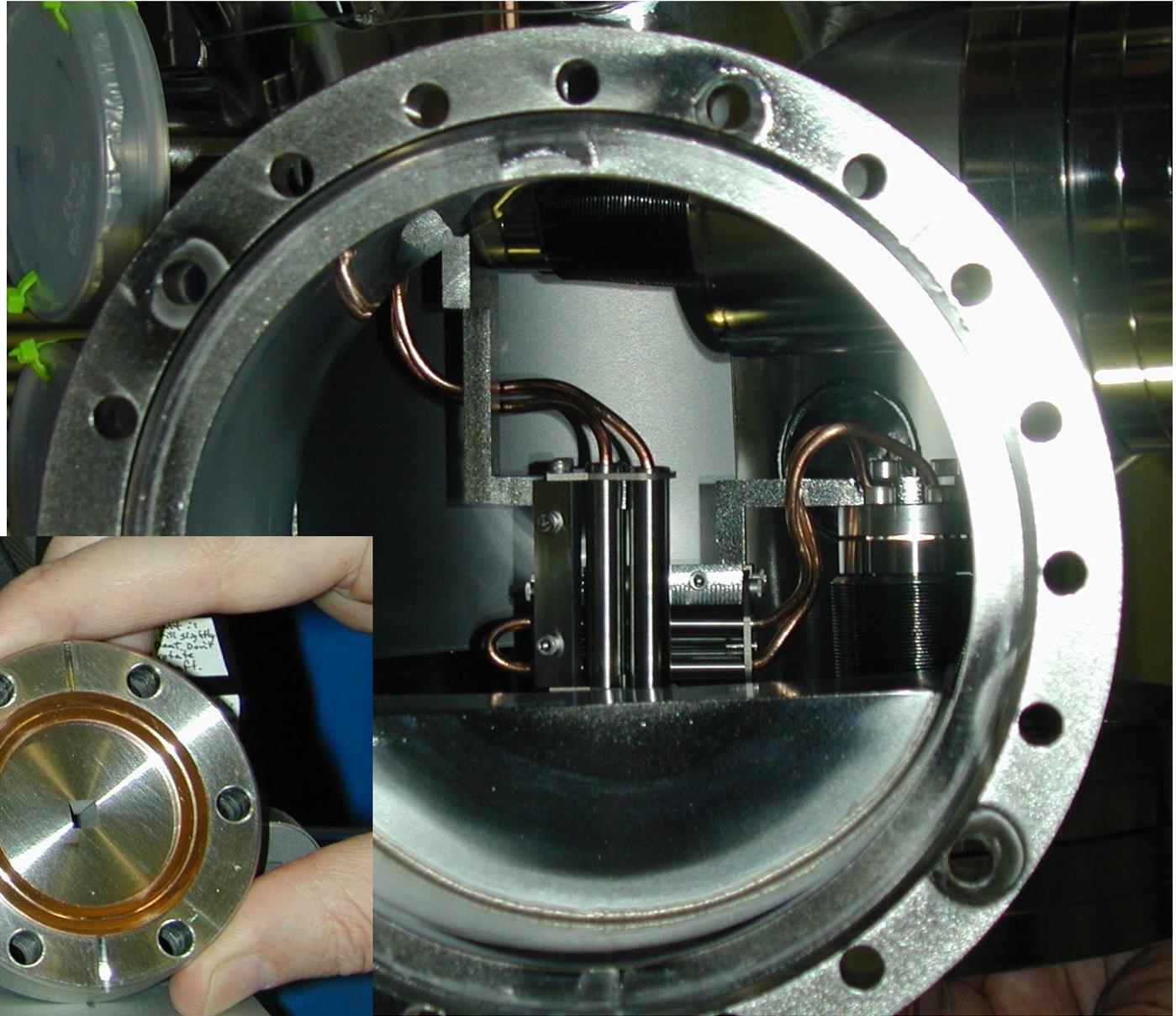
# 34-ID-C Capabilities

- Beam-splitting mirror
  - cuts out half of central cone
- Double Crystal Monochromator
  - Diamond, Si(111) and Si(220)
- Coarse and Fine coherence slits
  - UHV Roller-blade design
- Long-arm Microcontrole Diffractometer



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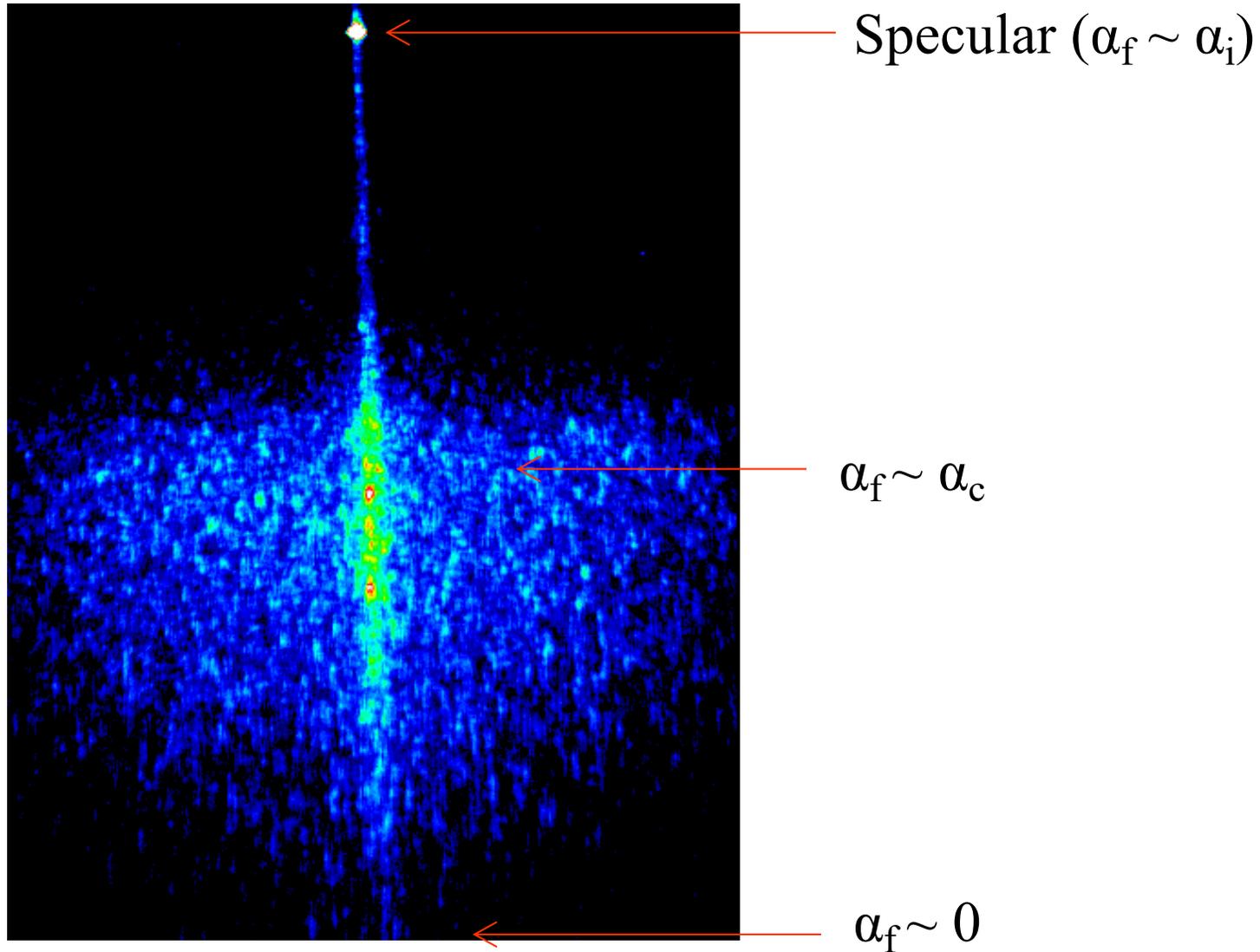




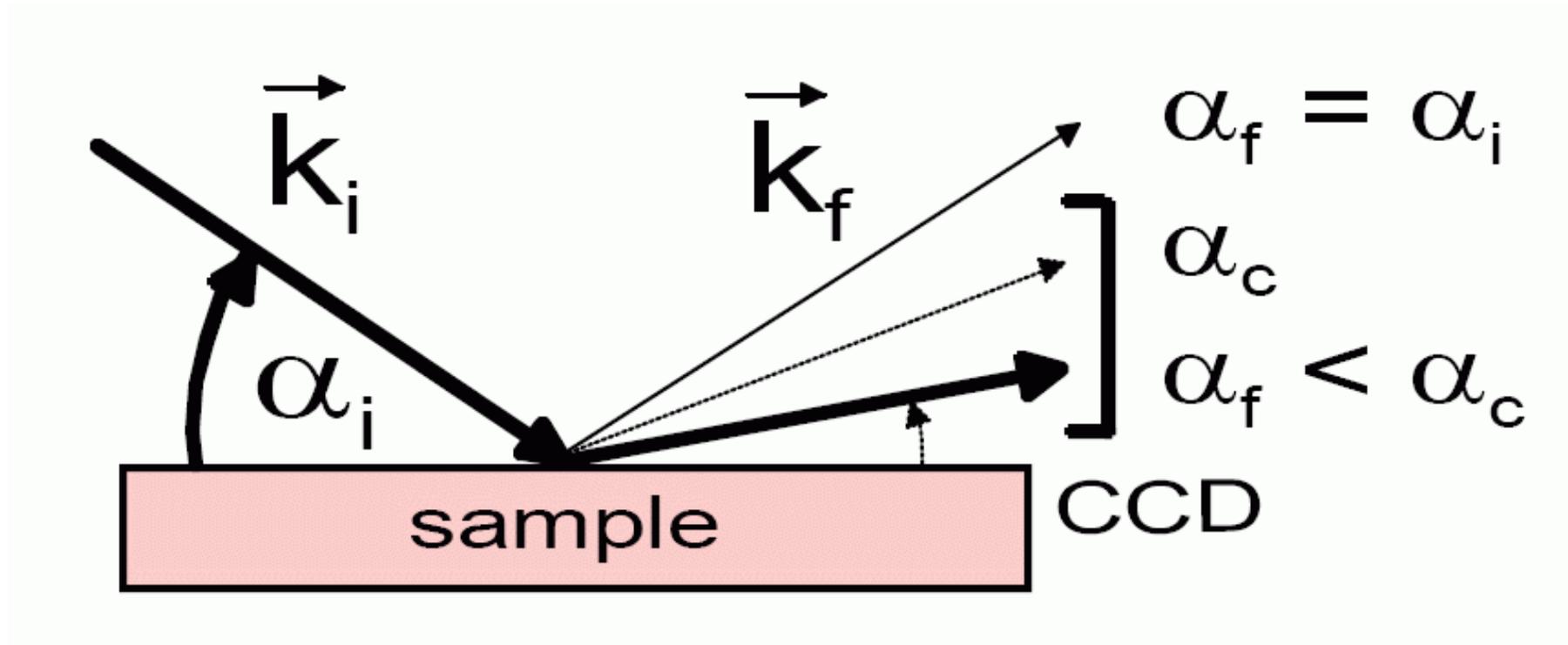
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# Structure in “Yoneda” Peak

Grazing-exit diffraction from a 1000Å Au polycrystalline film

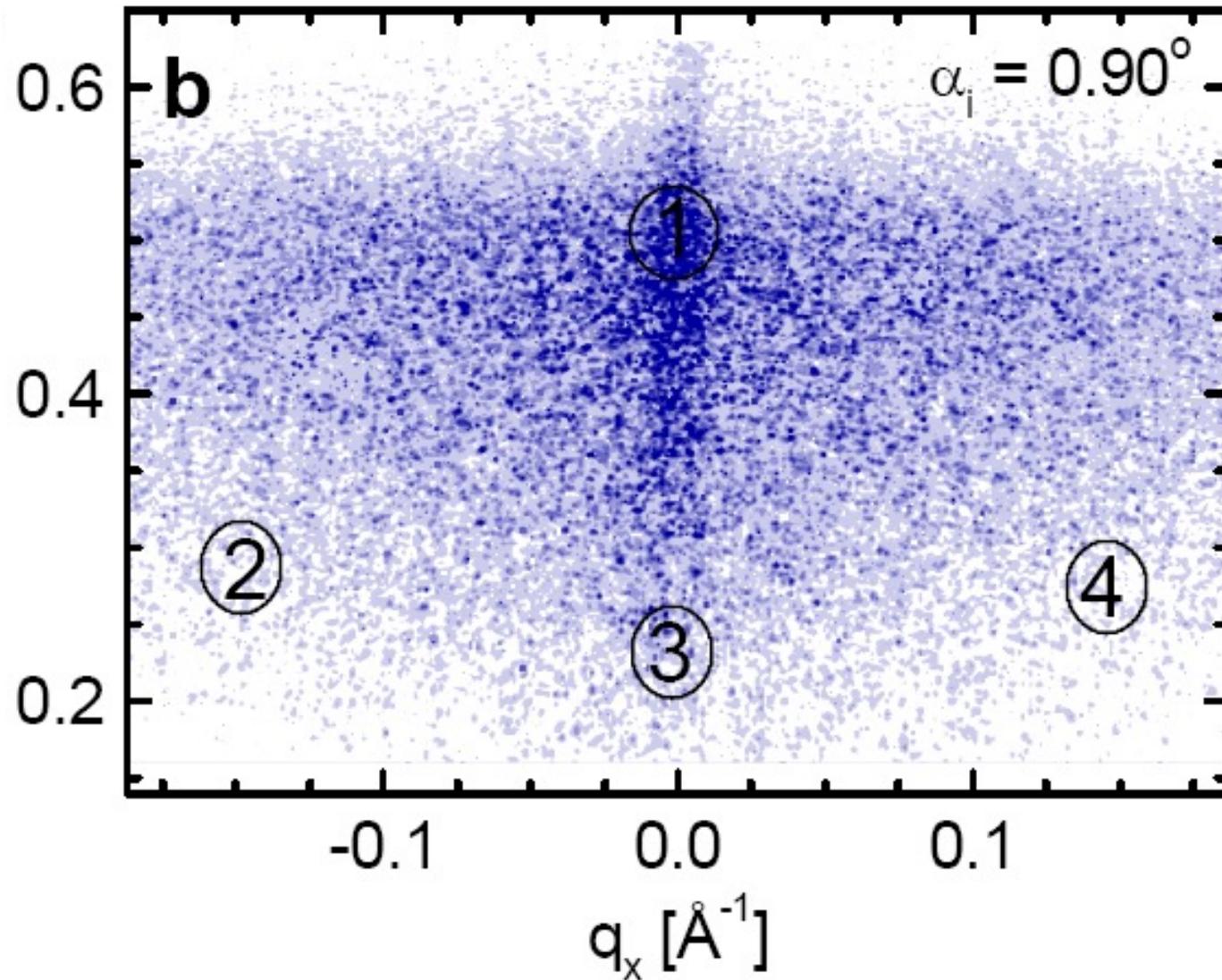


# Coherent “Yoneda” sample geometry

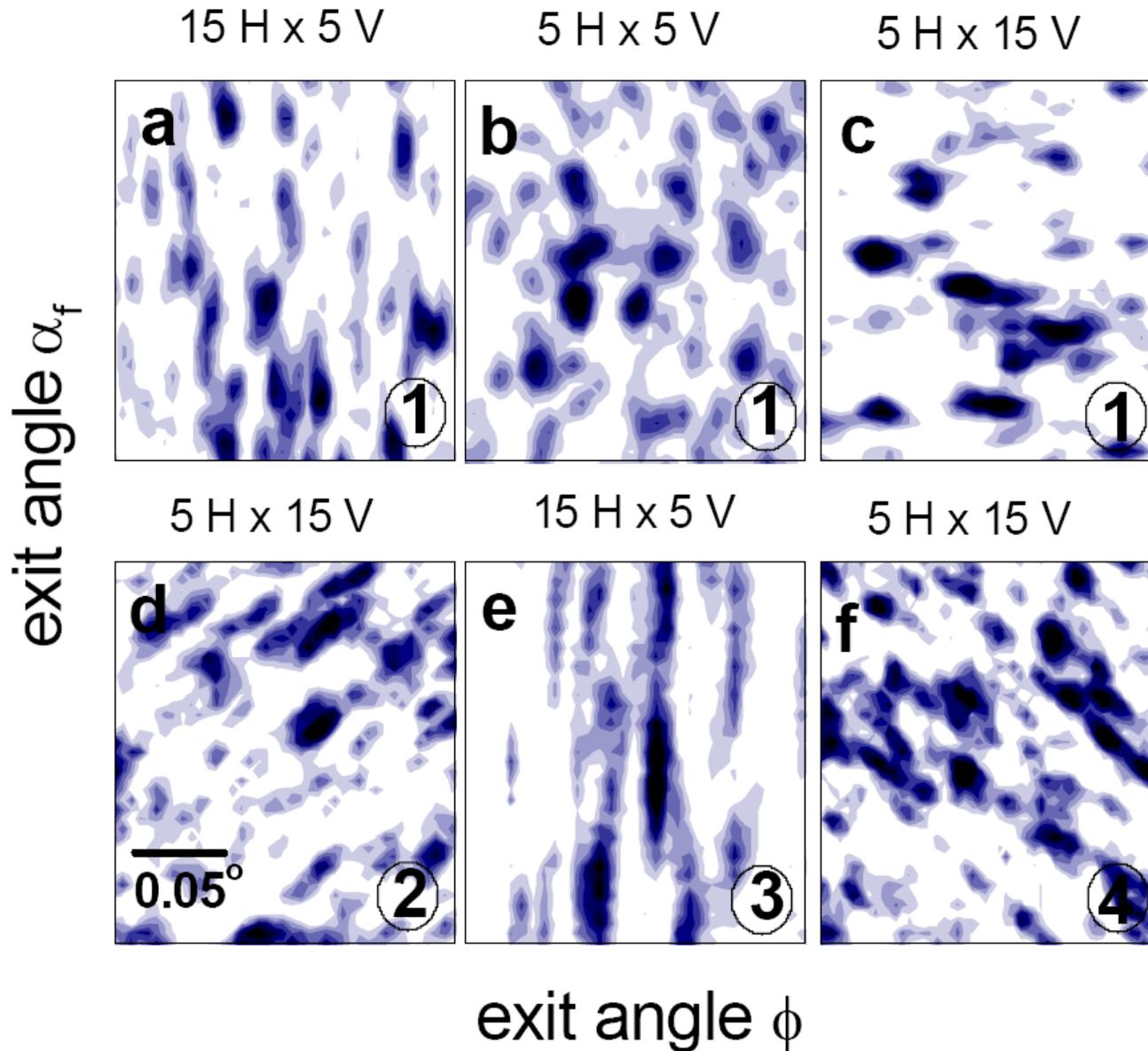


F. Pfeiffer, W. Zhang and I. K. Robinson,  
Applied Physics Letters 84 1847 (2004)

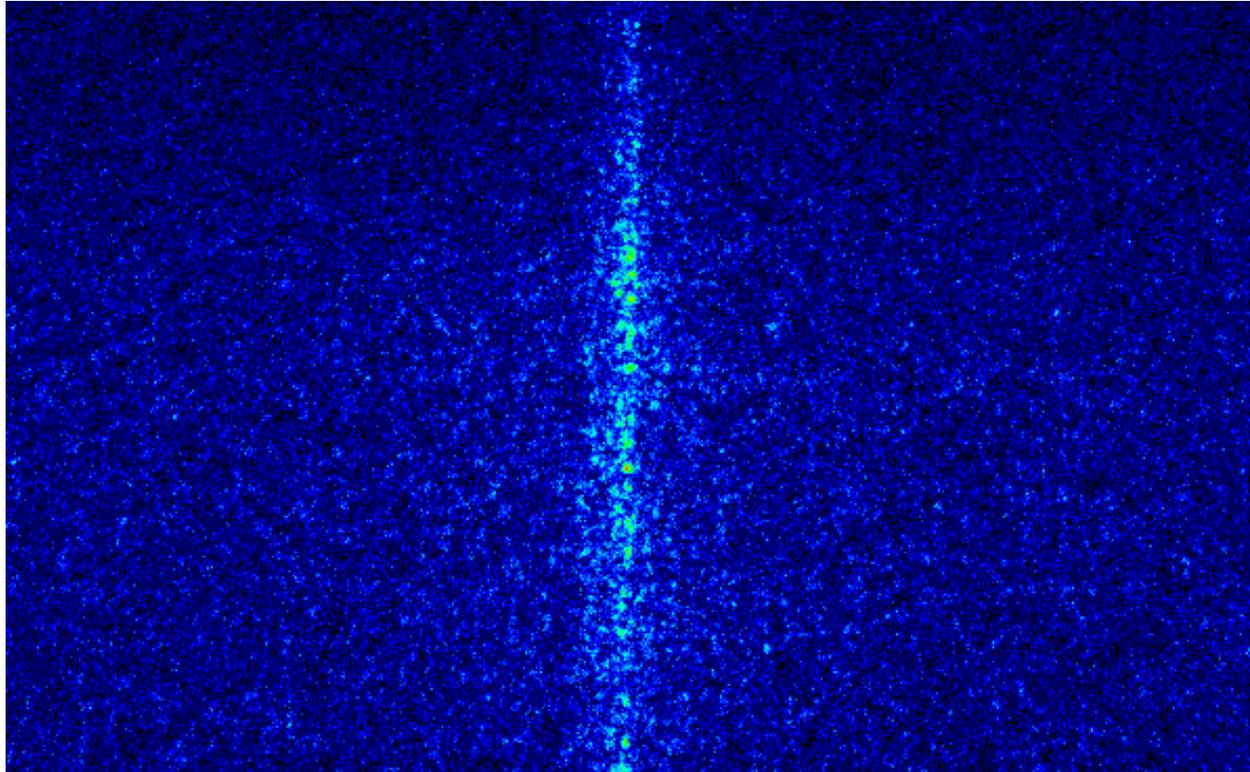
# Anisotropy of speckles



# Variation with entrance slit (microns)

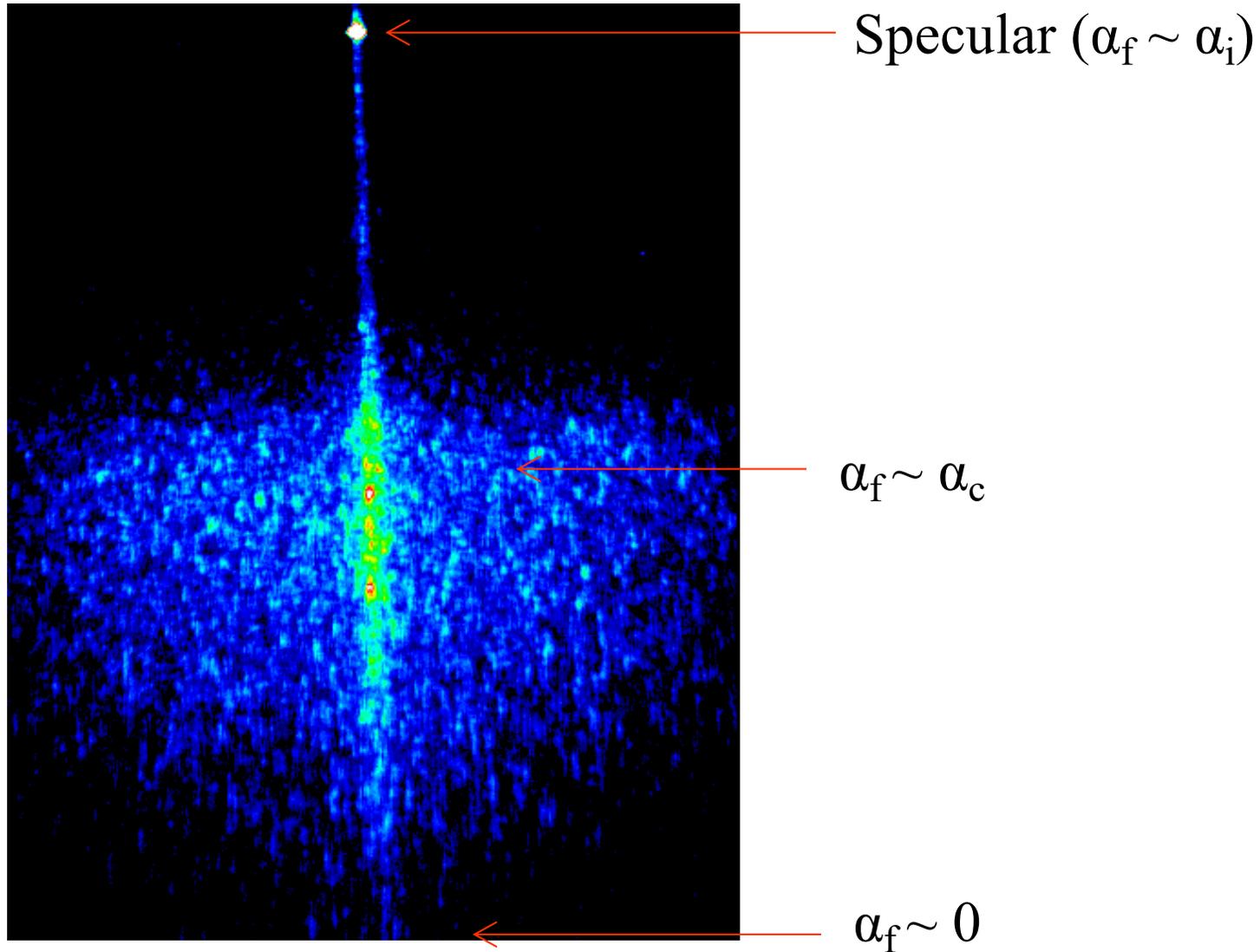


# Angle series, $0.01^\circ$ steps



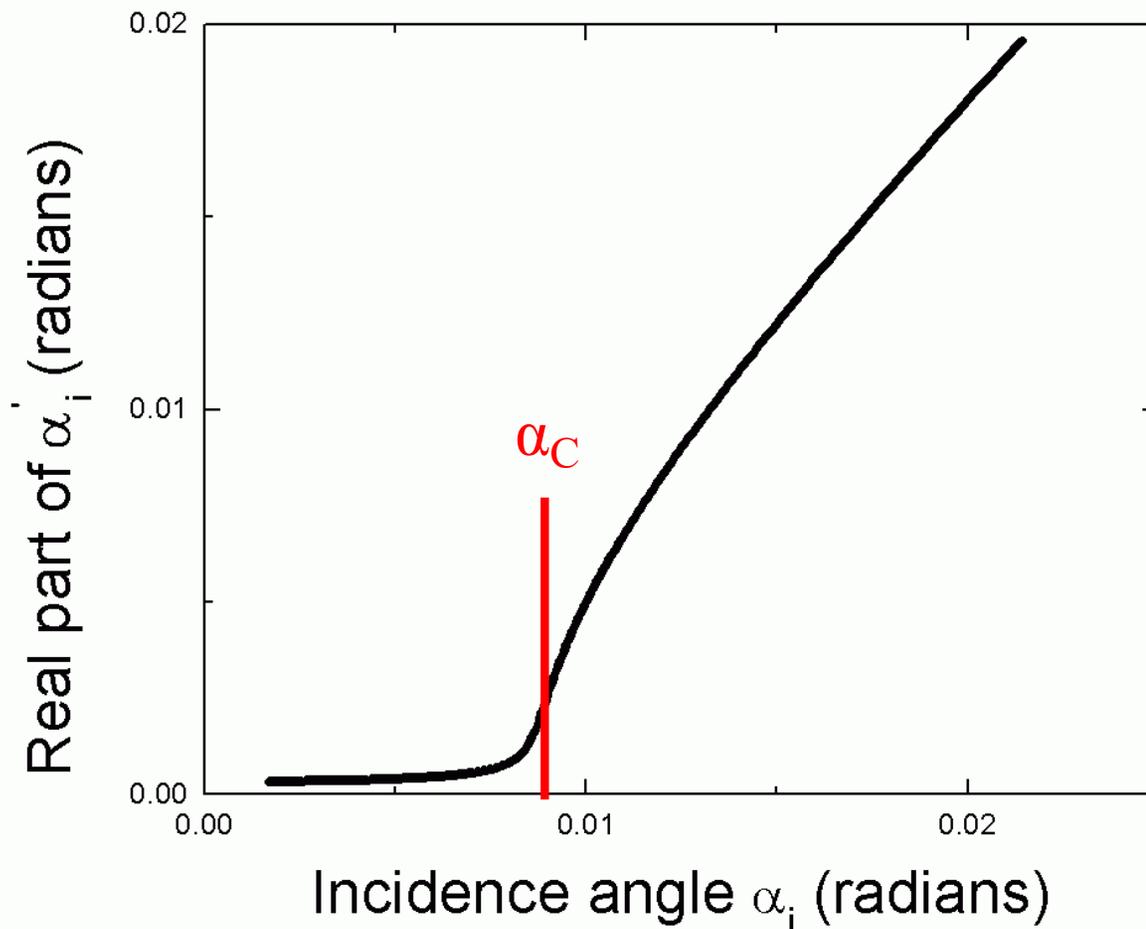
# Structure in “Yoneda” Peak

Grazing-exit diffraction from a 1000Å Au polycrystalline film



# Refraction by Gold Interface

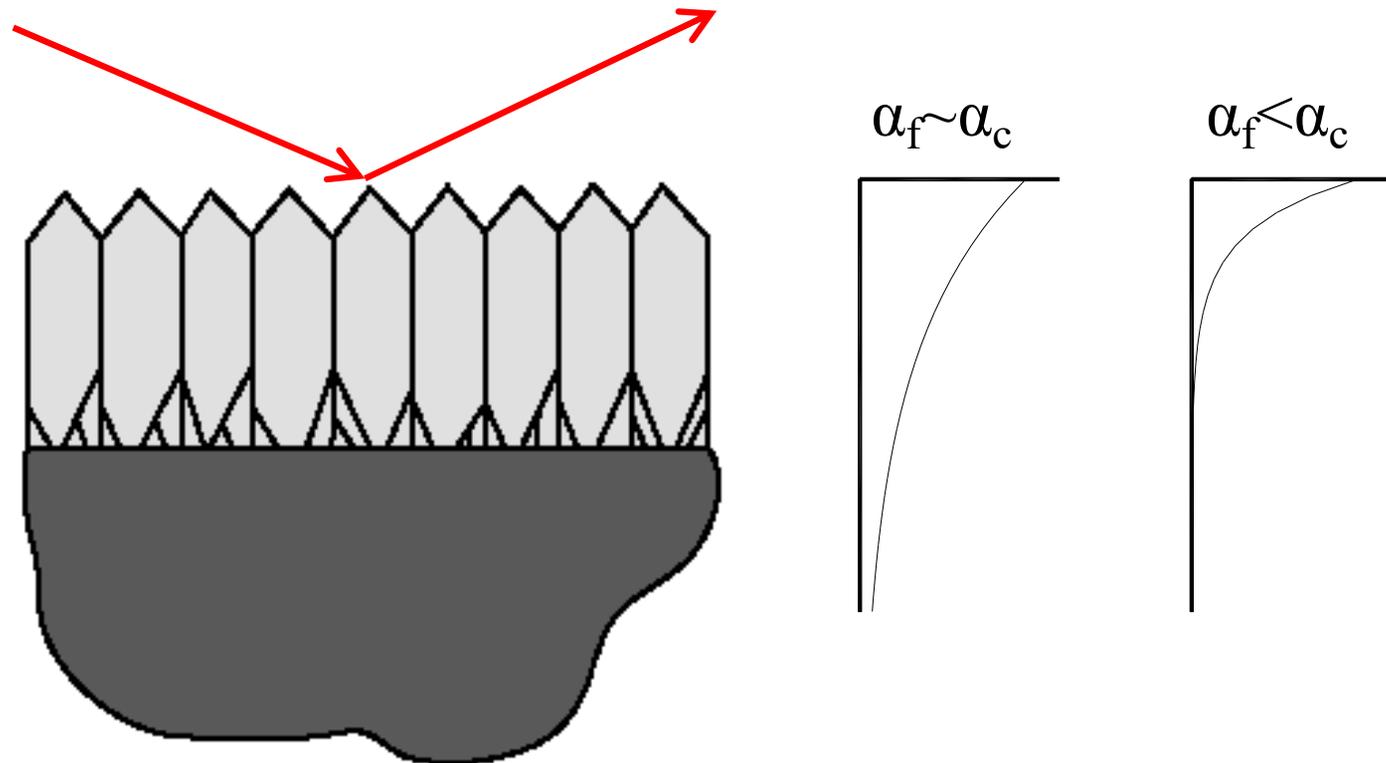
$$\alpha_i' = \sqrt{\alpha_i^2 - 2\delta + 2i\beta}$$



$E=8.92\text{keV}$   
 $\delta=3.76\times 10^{-5}$   
 $\beta=2.9\times 10^{-6}$

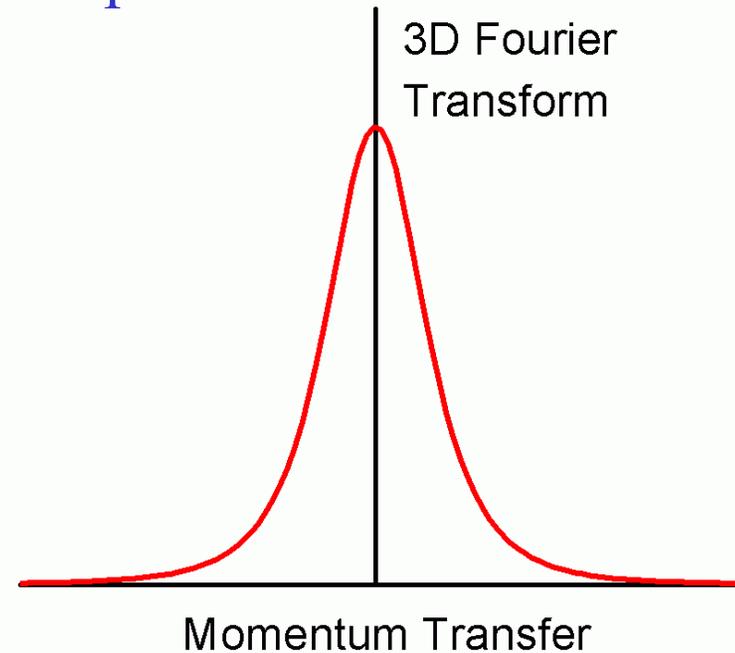
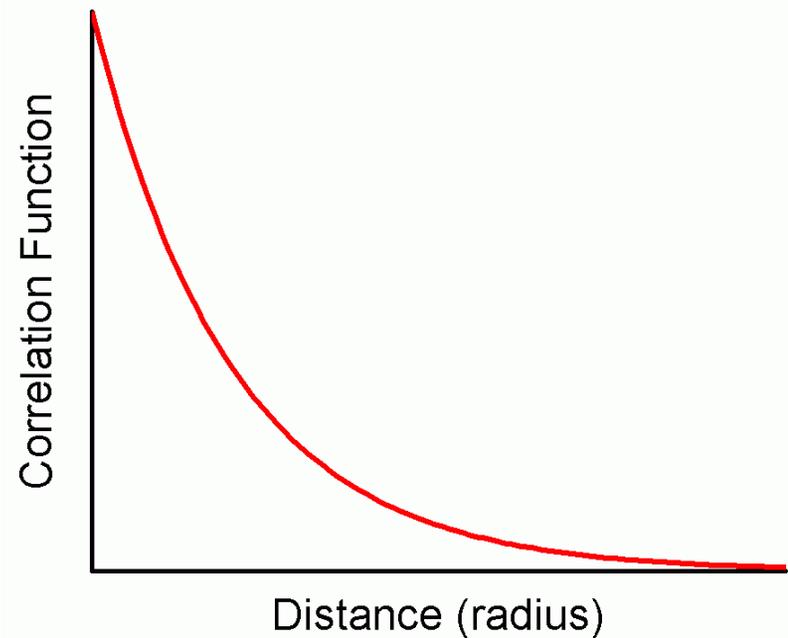
# Competitive Grain Growth

C. V. Thompson, *Ann. Rev. Mat. Sci.* **30** 159 (2000)

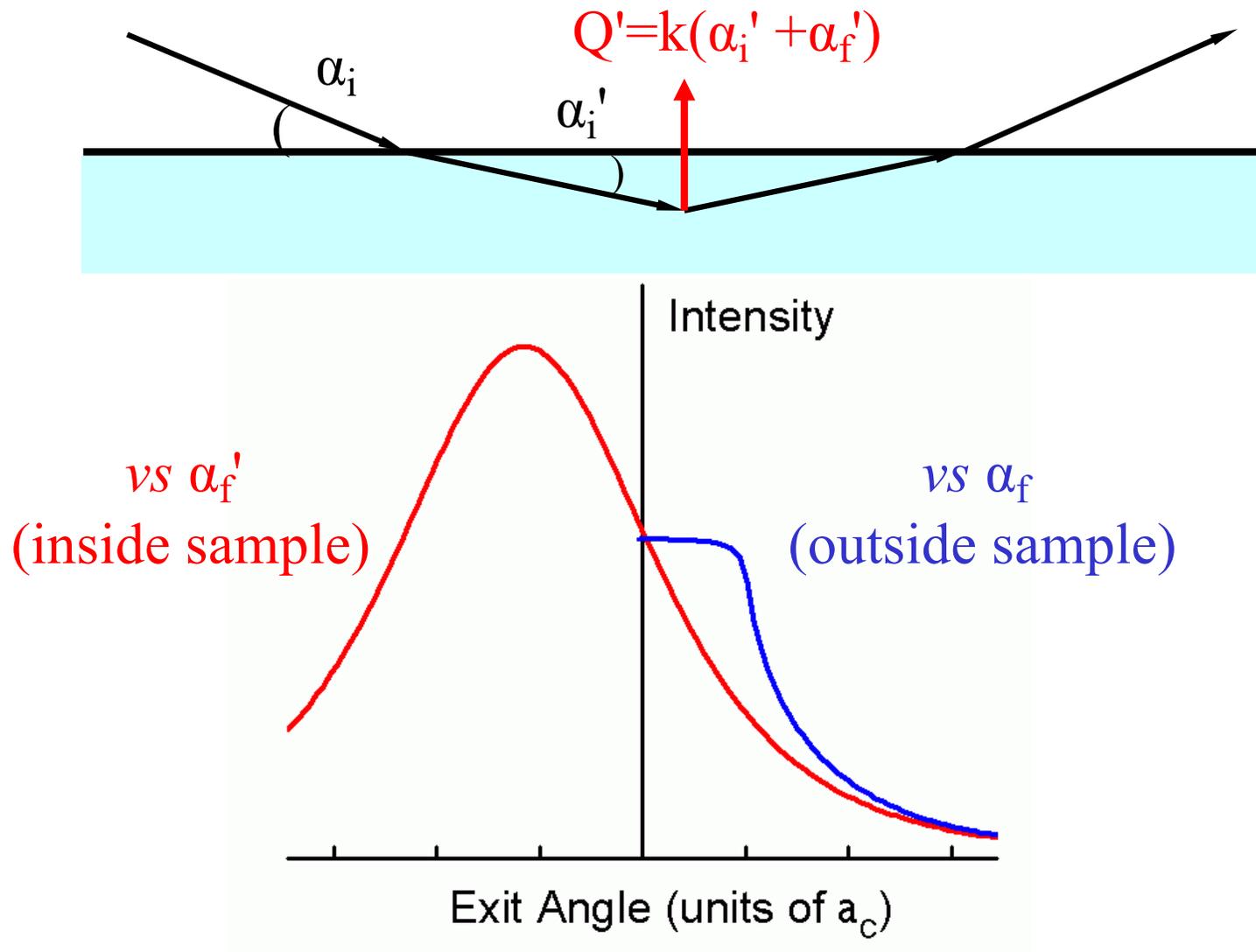


# Model of grain correlations

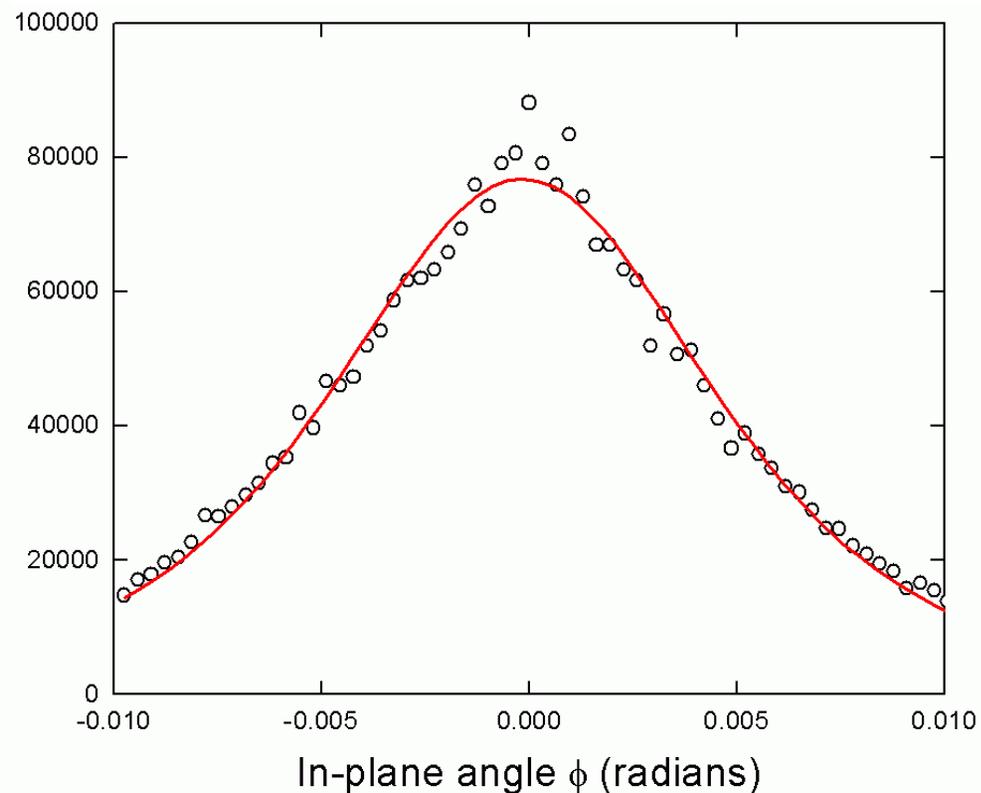
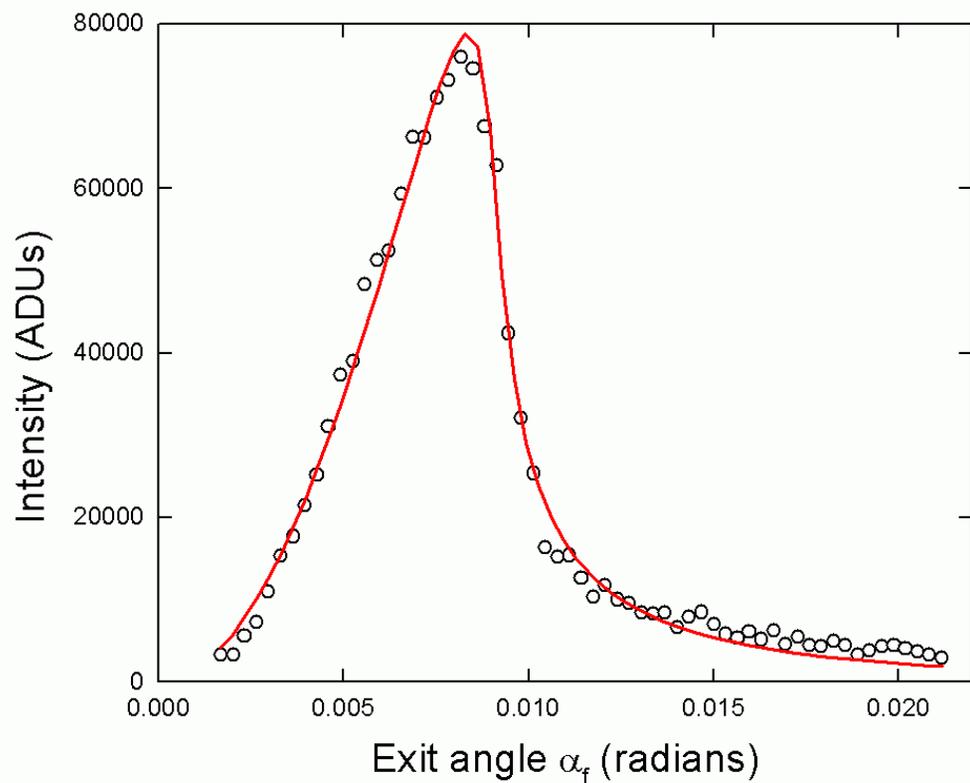
- Density of film assumed uniform, except at location of grain boundaries
- Assume exponential size distribution in 3D
- Expect (Lorentzian)<sup>2</sup> lineshape



# Effect of refraction on lineshape

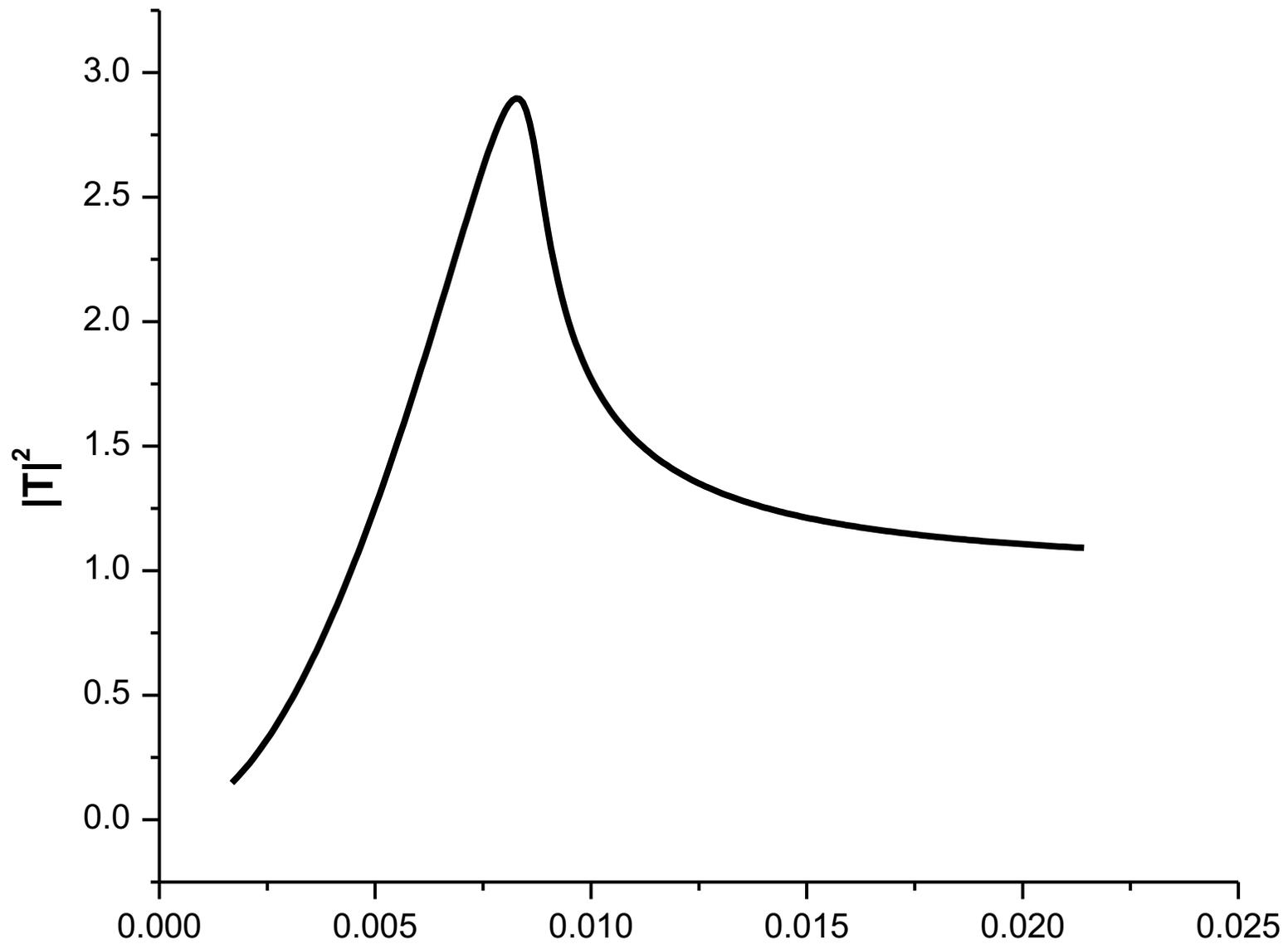


# Fits to (Lorentzian)<sup>2</sup> Profiles



Fit params: center (x,y), amplitude, width=0.0084,  $\alpha_i=0.011$

# Transmission Function



# Transmission Function

- Refractive index

$$n = 1 - \delta + i\beta$$

- Snell's law

$$\cos \alpha = n \cos \alpha'$$

$$\alpha' = \text{Re}(\alpha') + i \text{Im}(\alpha')$$

$$\text{Re}(\alpha') = \sqrt{\frac{1}{2} \sqrt{(\alpha^2 - 2\delta)^2 + 4\beta^2} + \frac{1}{2}(\alpha^2 - 2\delta)}$$

$$\text{Im}(\alpha') = \sqrt{\frac{1}{2} \sqrt{(\alpha^2 - 2\delta)^2 + 4\beta^2} - \frac{1}{2}(\alpha^2 - 2\delta)}$$

- Amplitude Transmittivity

$$T = \frac{\alpha_T}{\alpha_I} = \frac{2\alpha}{\alpha + \alpha'}$$

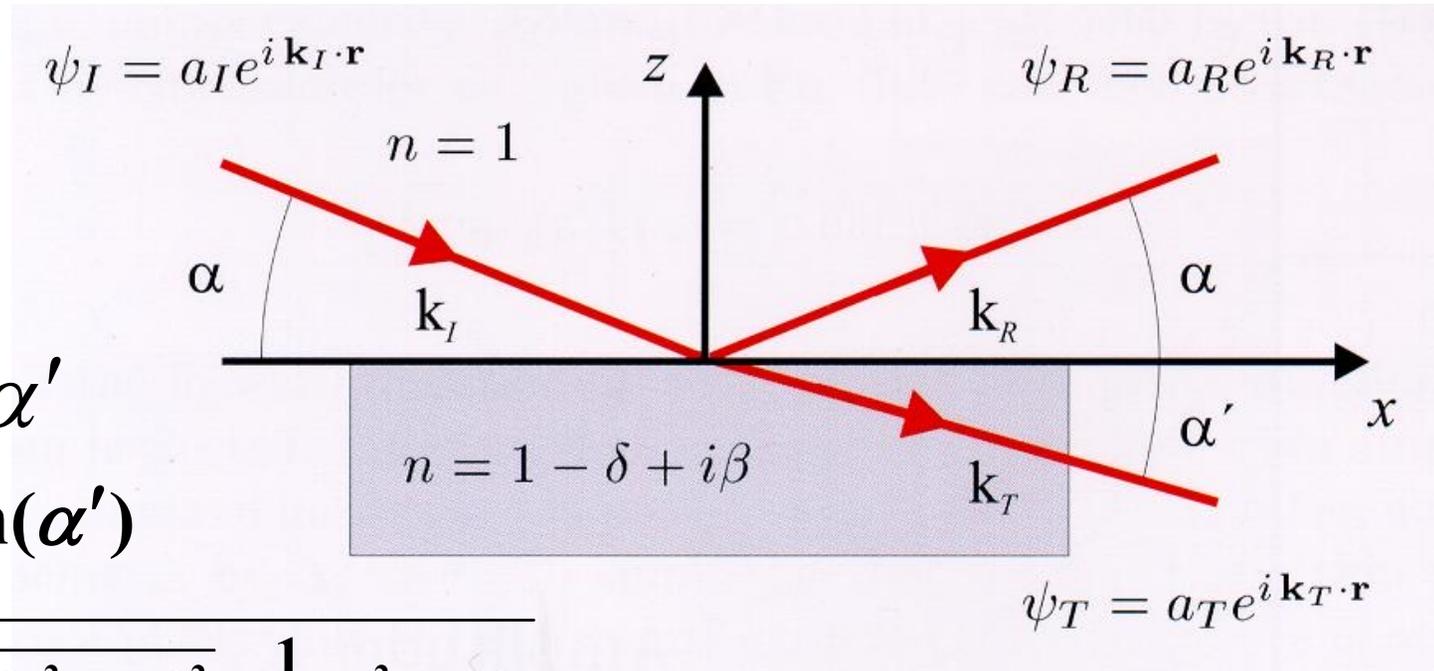
I. K. Robinson | Coherent GISAXS | May 2004

$$T(\vec{k}) = \frac{2\alpha_i}{\alpha_i + \alpha_i} = \text{const}$$

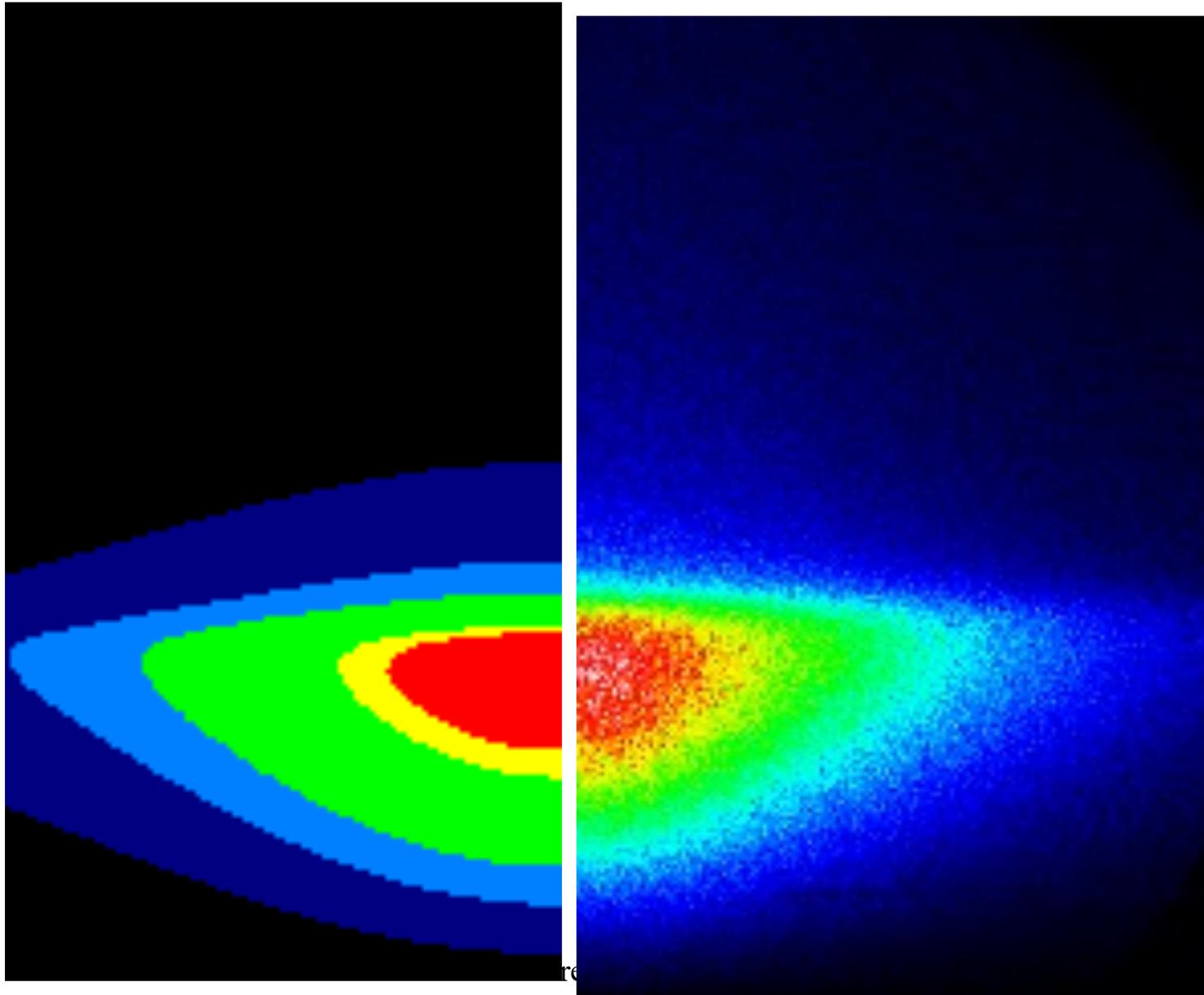
Incidence

$$T(\vec{k}_2) = \frac{2\alpha_f}{\alpha_f + \alpha_f'}$$

Exit



# Simulations of CCD data



- Distorted-wave Born approximation

$$I \sim \left[ \frac{d\sigma}{d\Omega} \right]_{diff} \sim |T(\vec{k}_1)|^2 |T(\vec{k}_2)|^2 S(\vec{q})$$

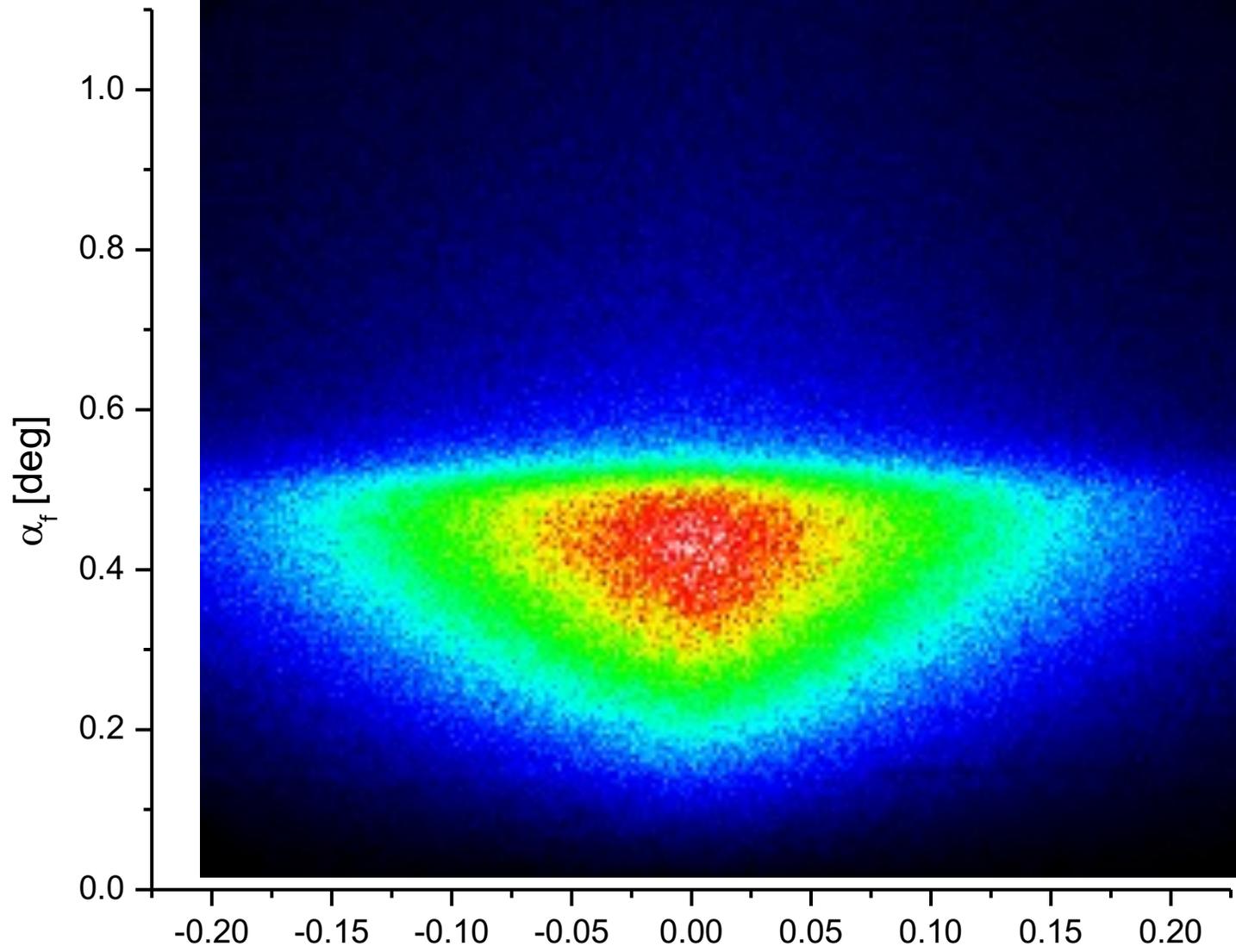
- Assume

$$S(\vec{q}) = \frac{C}{(Q_{2D}^2 + W^2)^P}$$

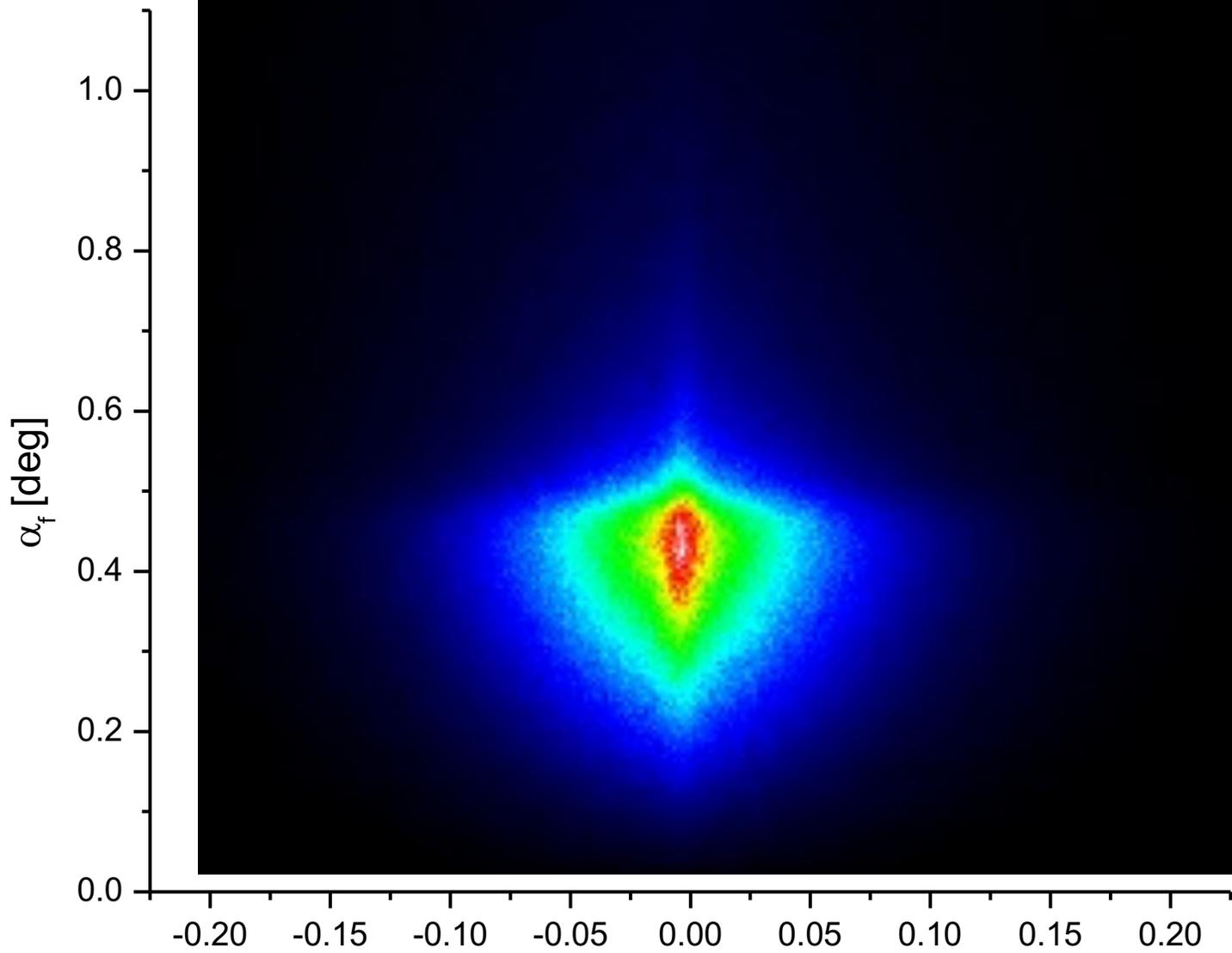
$$= \frac{C}{((\eta Q_y)^2 + Q_z^2 + W^2)^P}$$

$$\text{or } \frac{C}{((\eta\Phi)^2 + (\alpha_i' + \alpha_f')^2 + W^2)^P} \quad \text{with } P=2$$

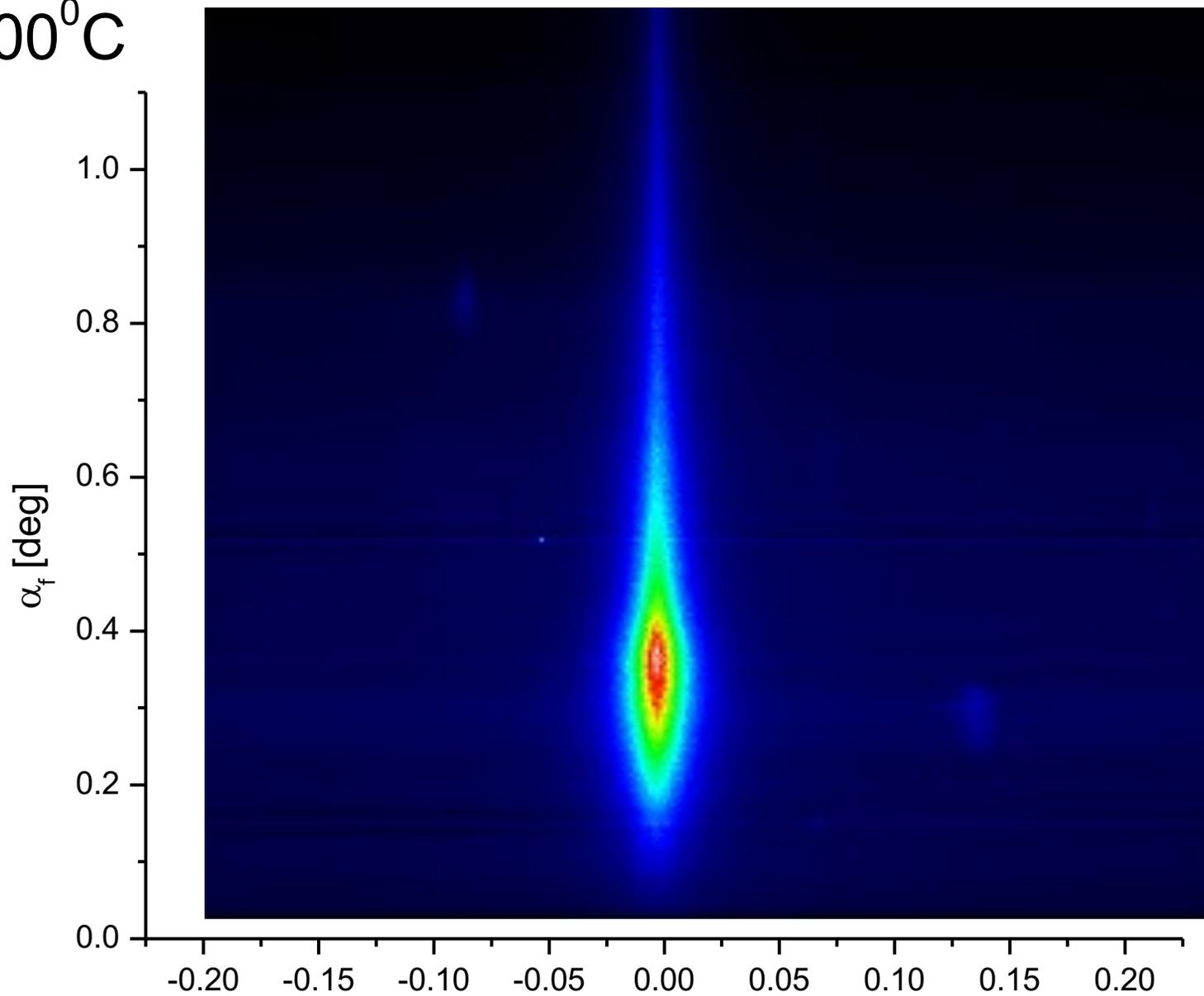
T=60<sup>0</sup>C



T=200°C

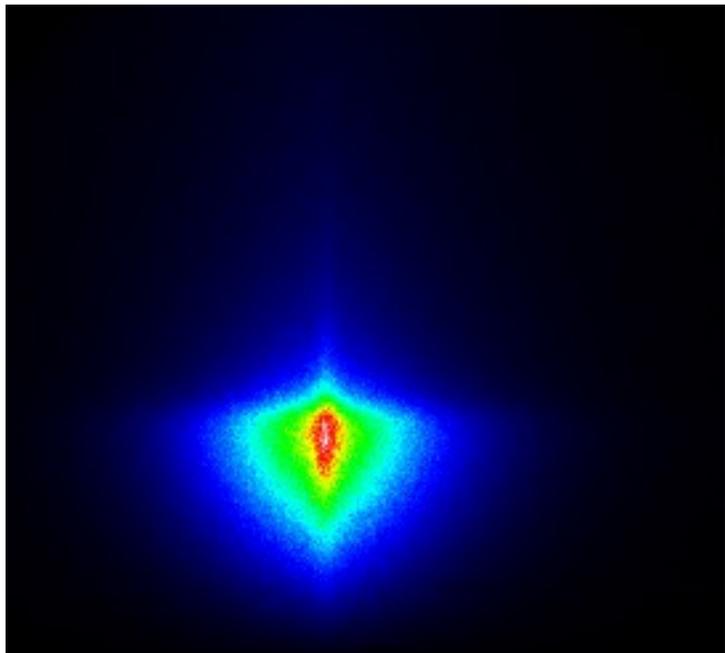


T=500<sup>0</sup>C



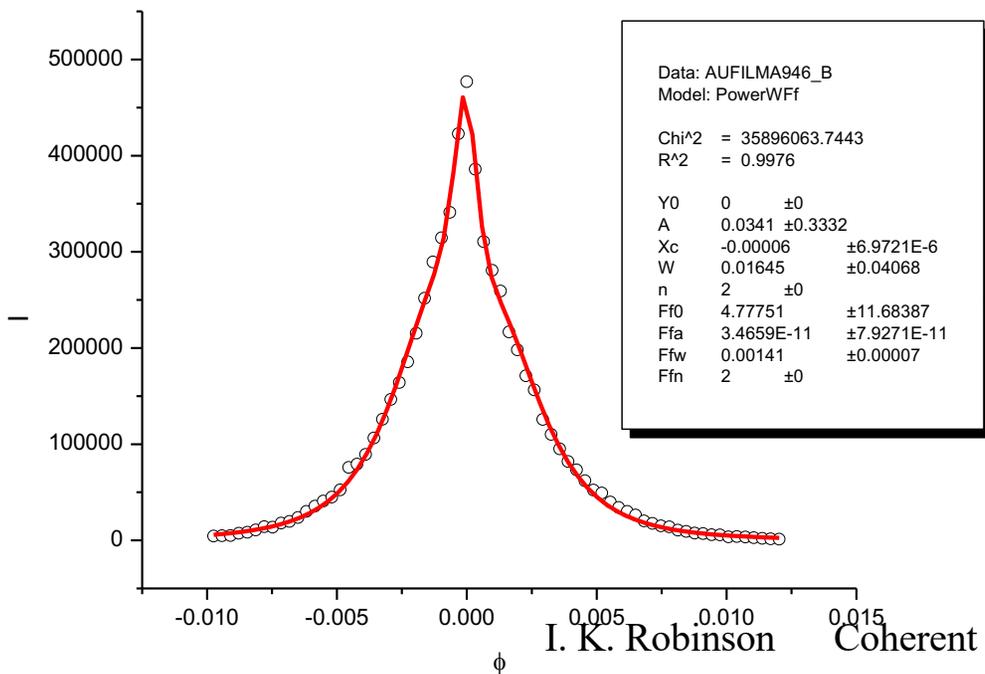
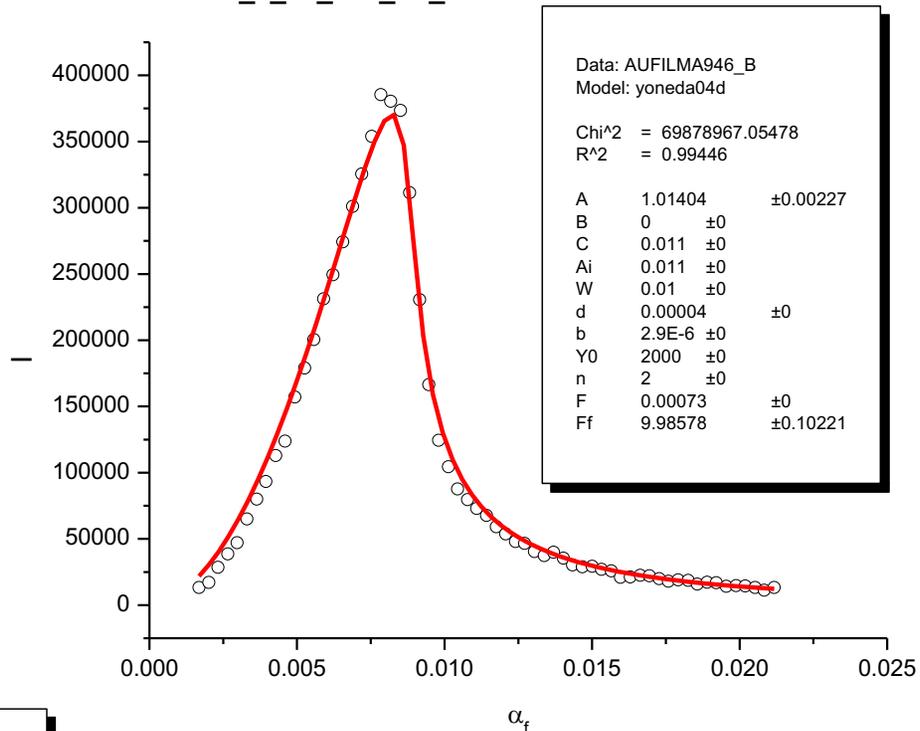
# Conclusions and Future Outlook

- GISAXS explained by refraction +  $|T(\alpha)|$
- CGISAXS sees individual grain distribution
- Surface effects are relatively unimportant
- Invert diffraction pattern to an image
- Study evolution grain by grain
- Other metals, kinds of film
- Island to film transition



aufilm\_a\_94\_610\_nb\_y175

aufilm\_a\_94\_610\_nb\_x113



T=200°C

$\eta \sim 5$  changes with  $\Phi$

W=0.01

- Simulation

