Swanning about in Reciprocal Space

or,

Kenneth, what is the wavevector?

Sean Brennan
Stanford Synchrotron Radiation Laboratory

Principles

The relationship between the reciprocal lattice vector and the wave vector is the key to understanding how to scan

Inverse nature of real and reciprocal space drives the scan parameters

Reciprocal space is a very big space to get lost in

Once you understand the kinds of scans you want and the signal leaving the sample, you can choose a detector for the experiment

Sean Brennan
brennan@stanford.edu

Memorize:

$$q = 4\,\pi \sin\Theta / \lambda \quad \text{(units of 1/Å)}$$

$$E = hc / \lambda$$

where $hc = 12400$ eV-Å

$$q_{max} \sim E \text{ (in keV)}$$

$$s = r\,\Theta$$

Sean Brennan
brennan@stanford.edu

What energy should I use?

- Are you doing anomalous scattering?
  - Typically work 100 and 10 eV below the edge
- Does the sample fluoresce?
  - One solution: use energy less than edge
  - Second: Use either crystal analyzer or solid-state detector to discriminate against fluorescence

Tune photon energy to deal with sample absorption edges

Sean Brennan
brennan@stanford.edu

What energy should I use?

- What r-space resolution do you need?
  - Good r-space resolution = large q-max.
- Do you want to look at multiple reflections in the same zone?
  - Higher orders require higher energy
- Are there sample environment issues (penetration problems)?
  - Higher energies will penetrate more, but flux lower at higher energy
- Not as many photons at high energy (> 20 keV)
- Energy above mirror cutoff -> unfocused experiment

It is amazing how many synchrotron experiments use Cu Ka (8048 eV)

If none of the above matter, I will use 10 keV as a good balance between resolution and flux

Sean Brennan
brennan@stanford.edu

What Slits should I use?

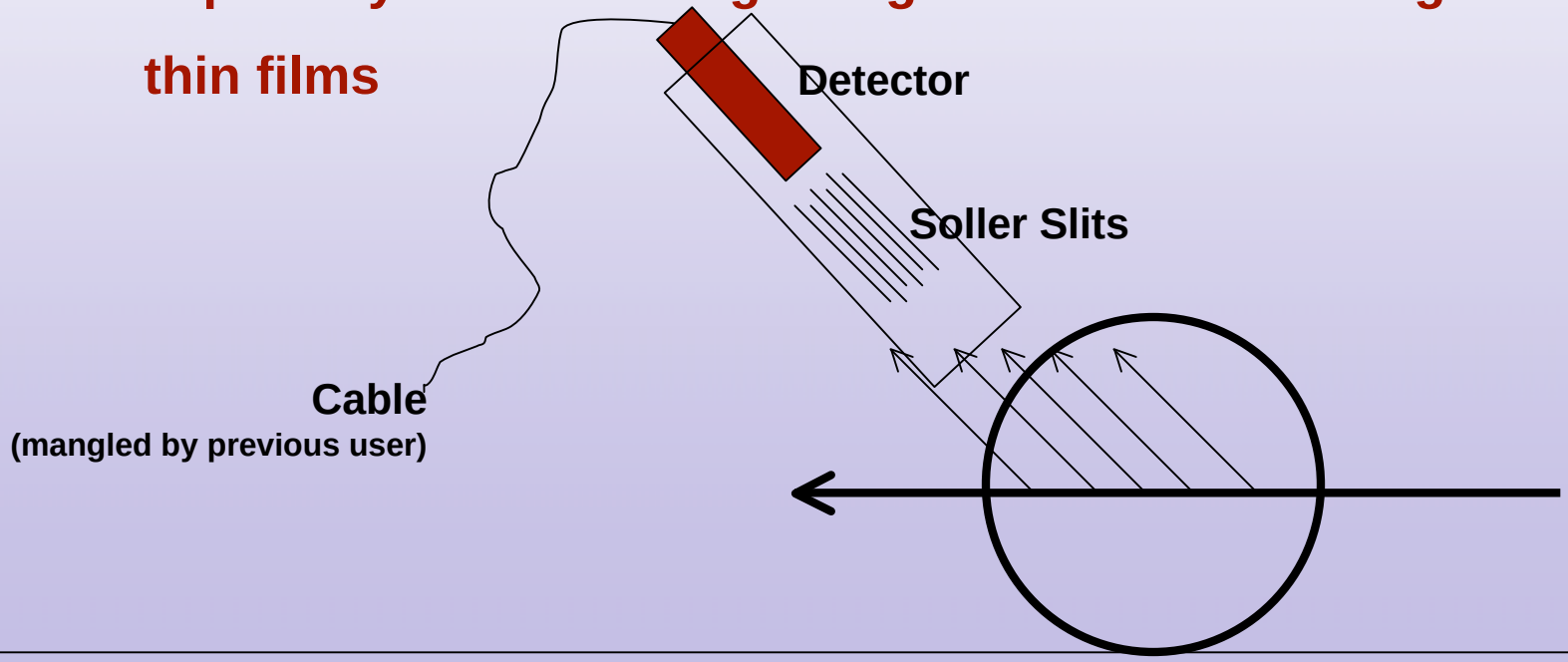
- Incident vertical beam divergence typically 0.1 mrad.
  - 17 mrad~1 degree
  - Beam divergence drives powder diffraction collection strategy
- If information is slowly varying in angle-space, use large slits
  - Amorphous materials
  - Crystal truncation rods
  - Small grain-size materials with large rocking curve widths

Typical HeNe laser pointer has a divergence of 1 mrad

Sean Brennan
brennan@stanford.edu

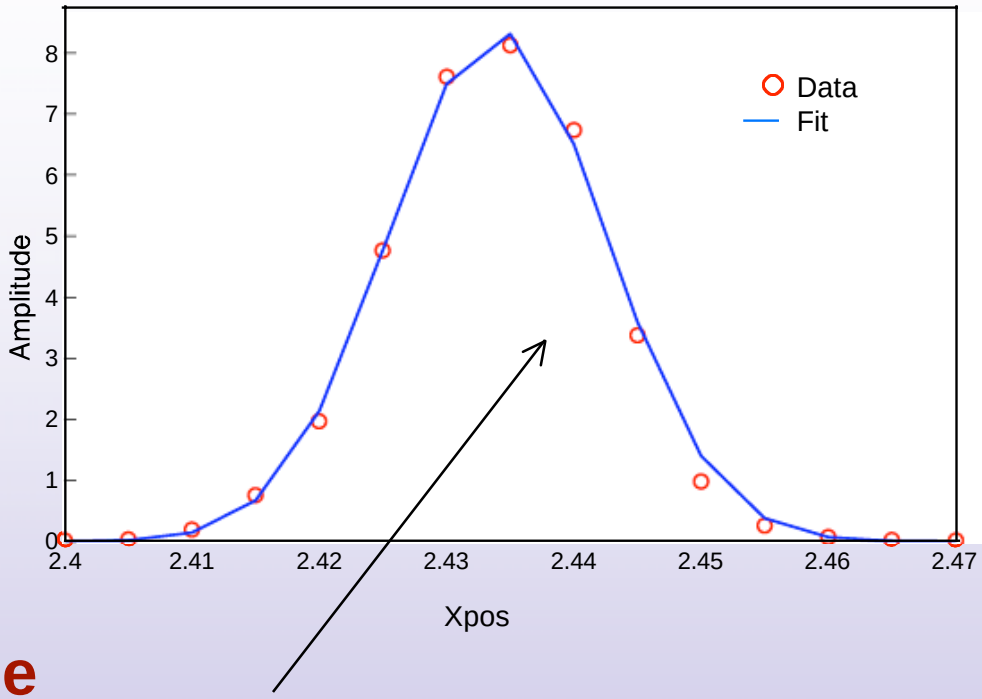
What Slits should I use?

- Analyzer crystal: Very good angular resolution (Ge(111) ~100 microradian acceptance)
- Soller slits: parallel plates to accept same angular value from a large sample- typical angular resolution 1mrad- especially effective for grazing incidence scattering from thin films



Sean Brennan
brennan@stanford.edu

Scan rules of thumb



Not really enough points to effectively determine background, ok for peak position.

- **Scan step size ~ slit size**
- **1mm slit @ 1 m = 1 mrad**
- **0.05 deg steps ~ 1 mrad**
- **~20 points to determine peak position**

Sean Brennan
brennan@stanford.edu

Powder diffraction:

- Reciprocal lattice is spherically averaged
- Match step size to 2theta resolution
- Incident beam divergence too good, need to rotate sample to get good powder average
  - Rock sample (flat plate)
  - Spin sample (capillary sample)
  - Make sure sample is ground sufficiently small

Sean Brennan
brennan@stanford.edu

What is the Orientation Matrix?

- *Data collection program needs to know how the crystal is mounted on the diffractometer to know where a reflection is*
- *Needs to know crystal parameters (a_0,b_0,c_0, α,β,γ)*
- *Needs to know angle settings for two roughly orthogonal reflection (zones, e.g. 100 and 010)*
- *Only needs to know Omega, Chi, Phi for each reflection*
- *Omega is the difference between Theta and 1/2(2Theta)*

Sean Brennan
brennan@stanford.edu

What is Calculation Mode?

- *4-circle diffractometer has too many degrees of freedom. Must set one additional boundary condition.*
- *Simplest B.C. is to fix Theta = 1/2(2Theta), aka "Omega= zero".*
- *For 2-circle diffractometers, still several modes:*
  - *powder, 2circle, nocell*
- *Kappa geometry can also have a symmetric mode (komega)*
- *For real power-users, there is "psi" mode and its siblings.*

Sean Brennan
brennan@stanford.edu

Thin film Scattering:

- Set up orientation matrix so that *h,k* in plane, *l* along plane normal
- Scans at constant *l* are scans at constant incident and take-off angle
- Soller slits critical for good angular resolution

$\sin(\Theta)\sin(\chi) = \sin(\alpha)$ where α is angle of incidence

Sean Brennan
brennan@stanford.edu

Amorphous or Liquid Scattering:

- r-space resolution defined by q_{max}
  - q_{max} of 20 -> $2\pi/20$ ~ 0.6 Å resolution
- q-space resolution defines r_{max}
  - Δq of 0.2 -> r_{max} of 31 Å
- Counting for constant signal better than constant I_0
- Reduction of background critical

Sean Brennan
brennan@stanford.edu

Single Crystal Phase Transitions:

- First-order phase transition occurs throughout entire crystal, so need v. good q-space resolution
- Match Analyzer crystal to monochromator to minimize dispersion
- Scan along principle RLV (*h,k,l*) rather than pure radial scan

Sean Brennan
brennan@stanford.edu

Detectors for SR experiments

- *What is quantum efficiency of detector?*
- *What is energy resolution of detector?*
- *At what count rate does it go non-linear?*
- *How well does it monitor the beam?*

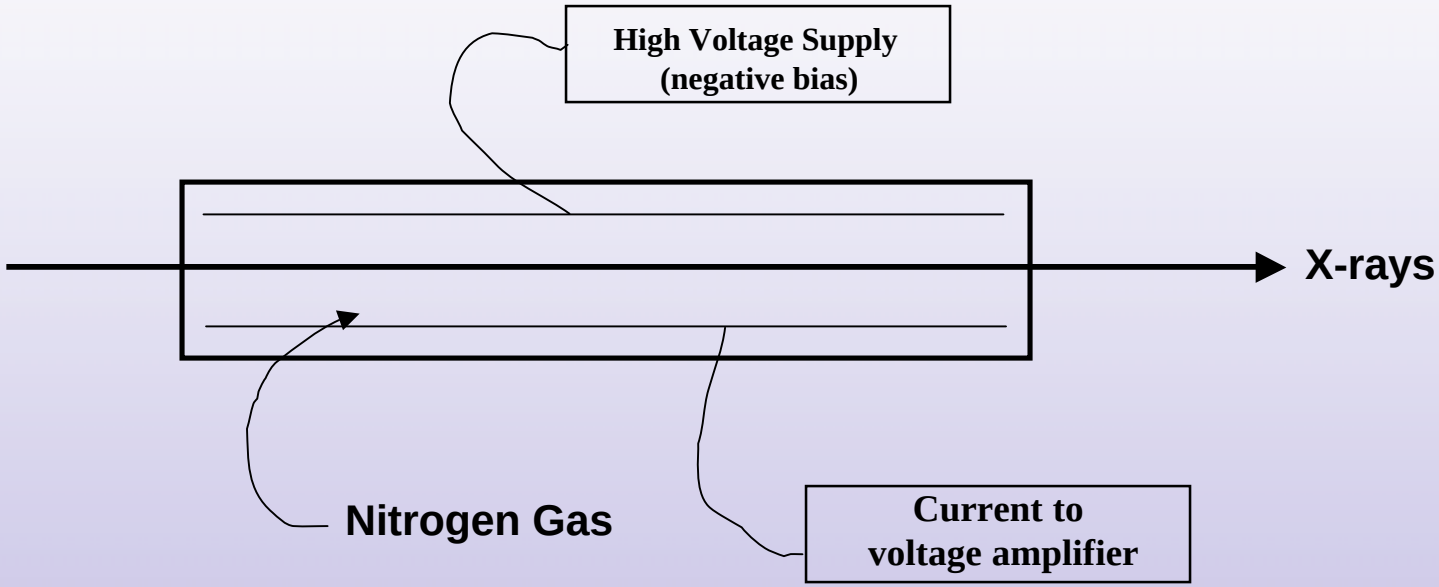
Sean Brennan
brennan@stanford.edu

Ion Chamber

- *Different gases used for different energies*
- *Linearity depends on what gas is used*
- *Is capable of absolute flux measurement (if done correctly)*
- *Must ensure voltage is in plateau region*

Sean Brennan
brennan@stanford.edu

Schematic of an Ion Chamber



Sean Brennan
brennan@stanford.edu

Convert ion chamber current to photon flux:

$$Flux\ [hv/sec] = \frac{I_{abs}\ [coul/sec] * 34.6\ [ev/e-]}{(1-e^{-mu*l})*1.6e\text{-}19[coul/e-]*Energy[ev/hv]}$$

0.1 microamp absorbed current
15 cm chamber length
10 keV photons
0.0044 [1/cm] mu (N_2, 10 keV)

0.1 μA ~ 3.4 x 10^{10} ph/s

Ion trapping (incomplete charge collection) occurs at high fluxes (> 10^{11} ph/sec). Increasing voltage on ion chamber can help, but reducing absorption cross-section of gas is better solution.

Sean Brennan
brennan@stanford.edu

Bicron (NaI detector)

- *Based on conversion of x-ray photon into visible light which is converted to electrons (aka scintillator)*
- *Fast (linear up to ~40 kps)*
  - *Deadtime correction at higher count rates*
- *50% energy resolution*
  - *Ok for separation of harmonic from fundamental*
  - *Not sufficient to remove most fluorescence*
- *NOT happy looking at main beam*
- *Low background (few cps)*

Sean Brennan
brennan@stanford.edu

Solid State Ge detector

- *Good energy resolution (200-300 eV resolution)*
- *Trade energy resolution for count rate*
- *Moderate count rate (linear to ~10 kps)*
- *Good choice if fluorescence background*
- *Not sufficient for elastic-K_b separation*
- *Need regular (2x per day) N_2 filling*
- *Very low background (< 1cps)*

Sean Brennan
brennan@stanford.edu

Vortex (Si drift) detector

- *V. high count rate (linear to 100 kps?)*
- *V. good energy resolution (150 eV)*
- *No need for LN (Peltier-cooled)*
- *Compact, so easy to install on 2theta*
- *Small detector effective area*
  - *Not an issue with slits*
  - *An issue with Soller slits*

Sean Brennan
brennan@stanford.edu

Conclusions

- ***Work with scientific staff before arrival to determine best energy, detector, slits, etc***
- ***Familiarize yourself with SCALC (available on SSRL alpha)***
- ***Think about the information you want:***
  - ***Peak shape?***
  - ***Peak position?***
  - ***Variation of peak with q?***
  - ***How long will you need to count?***
    - ***10 k cts has sqrt(n) < 1% Is that sufficient?***
- ***If you are unfamiliar with the instrument, plan to sit in with another team and ask questions.***

Sean Brennan
brennan@stanford.edu