Data Collection Strategy

7th X-ray Scattering School

Apurva Mehta

U.S. DEPARTMENT OF ENERGY
Office of Science
SSRL
SLAC NATIONAL ACCELERATOR LABORATORY

Outline

- Before .....
- Geometry, detectors, resolution...
- Data collection strategy.

Science

What is the scientific question?

How will probing the structure help?

- Is there an alternative method?
  - TEM
  - EXAFS
  - Neutron diffraction
  - BET porosity measurements
- Can data from a sealed tube diffractometer give at least part of the answer?

If answer to either of the question above is even a hesitant yes.

Do those measurements first

- They will help you do a better and easier synchrotron experiment.
- They will complement your synchrotron data and will simplify data analysis and interpretation.

Science : Structure Determination

SSRL

SLAC

- Structure completely unknown
  - Single Crystal
  - Polycrystalline → Rietveld method
  - Disordered → PDF
- Partially known
  - Site occupancy → full pattern refinement, AXS/REXS
- Interested in noncrystallographic features
  - Particle size → small angle scattering, peak shape analysis
  - Texture → 2D diffraction
  - Strain → 2D diffraction, Q space resolution

Science: Structural Transition

- Impulse driven
  - Phase transition
  - Strain

- Time Dependent
  - Chemical Reaction
  - Relaxation processes

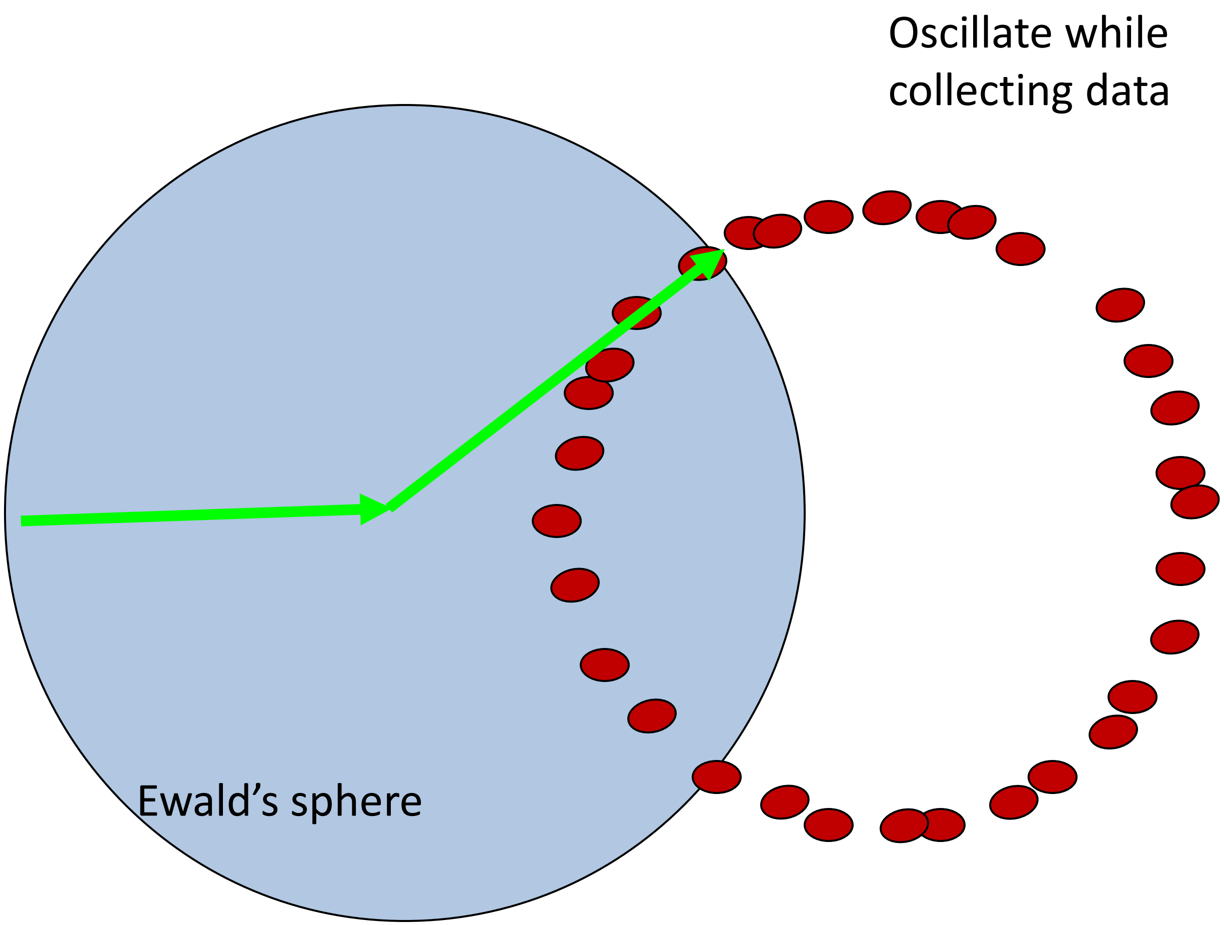
- In-situ rigs and reactors: Vanessa's talk
- Laser pump-probe
- Will the scattering from the reactor windows interfere?

Sample

What does the sample look like?

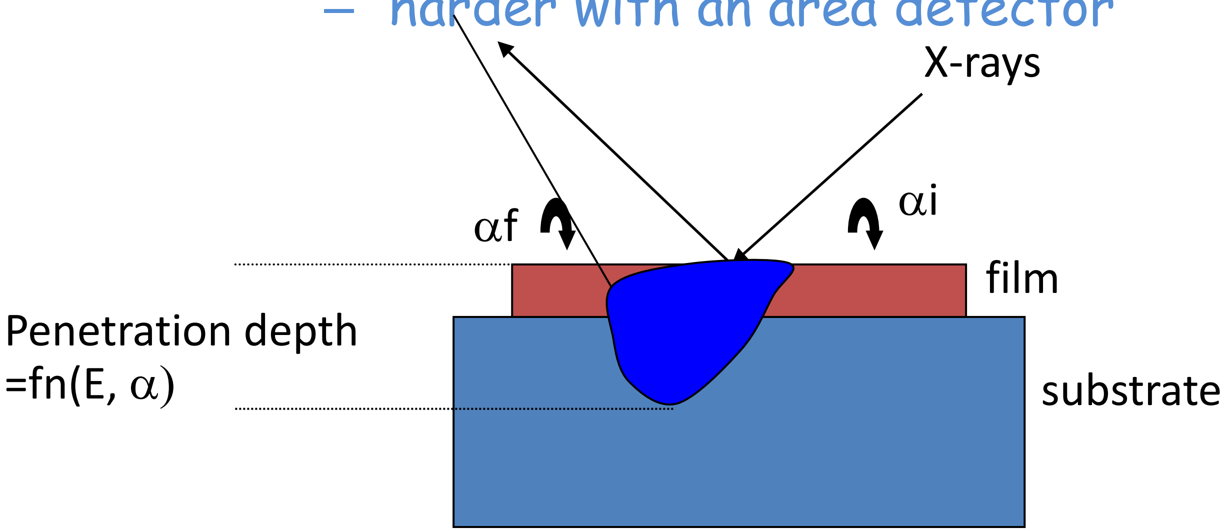
- Bulk
  - powder or solid/pressed pellet
  - Would preferred orientation matter?
    - finely grind AND spin if possible
- Transmission or Reflection:
  - Refl → blocks bottom half of the pattern
  - Refl sample should be infinitely thick if possible
  - Trans samples are ideally ~ μ
- Wet?
- Does it need containment window?

Powder Average and Rocking

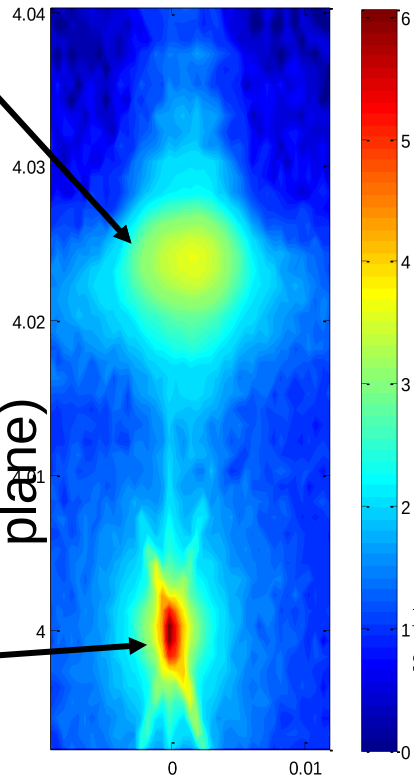


What Does the Sample look like?

- Thin Film
  - Will the scattering from the substrate interfere?
    - Weak - as it is not in diffracting condition (e.g., single crystal substrates)
    - Can it be avoided by placement of the detector?
      - harder with an area detector

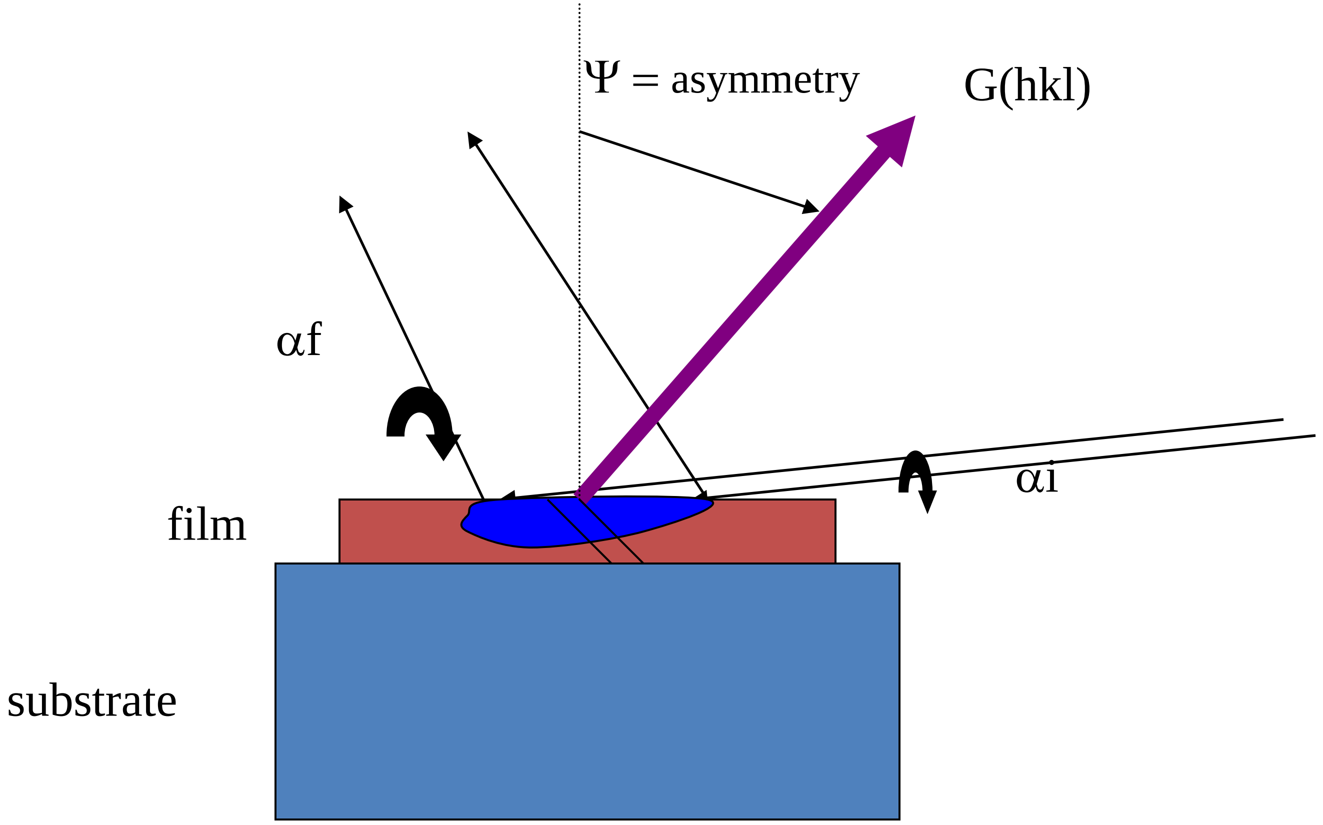


Thin Film


Film peak
Sub. peak
L (out-of-plane)
H (in-plane)
4.04
4.03
4.02
4.01
4
0
0.01
6
5
4
3
2
1
0
intensity on log scale
1
0^

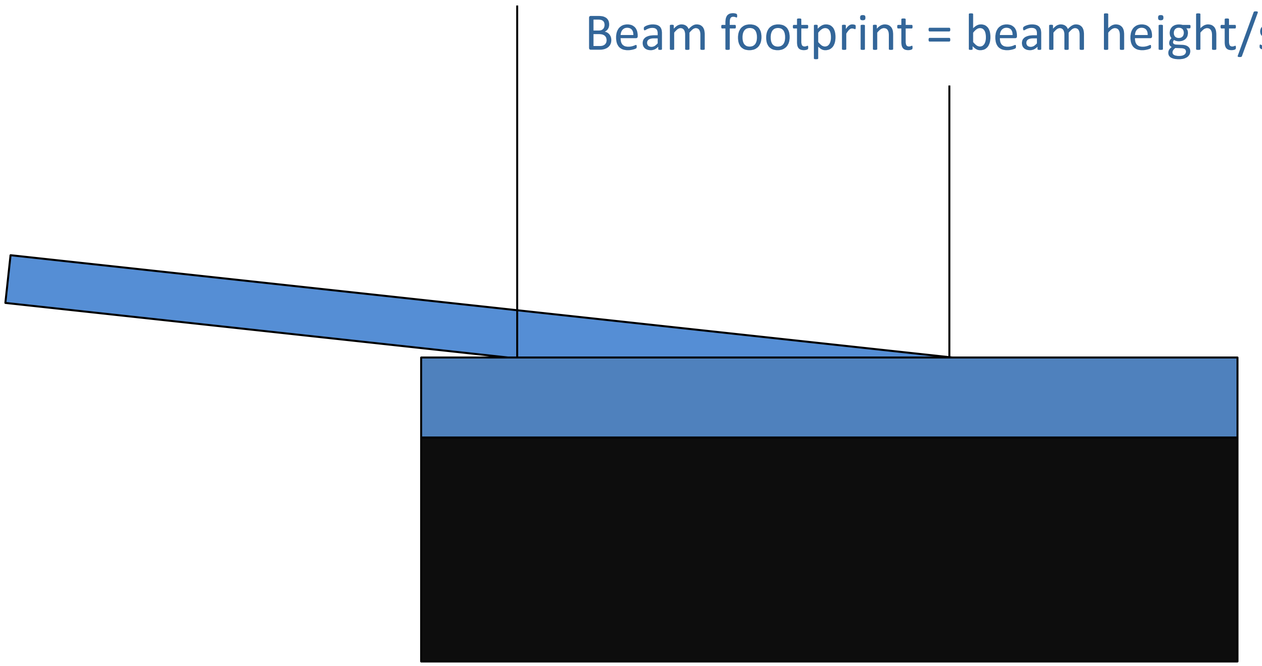
Thin Film: Substrate interferes

Grazing Incidence Geometry



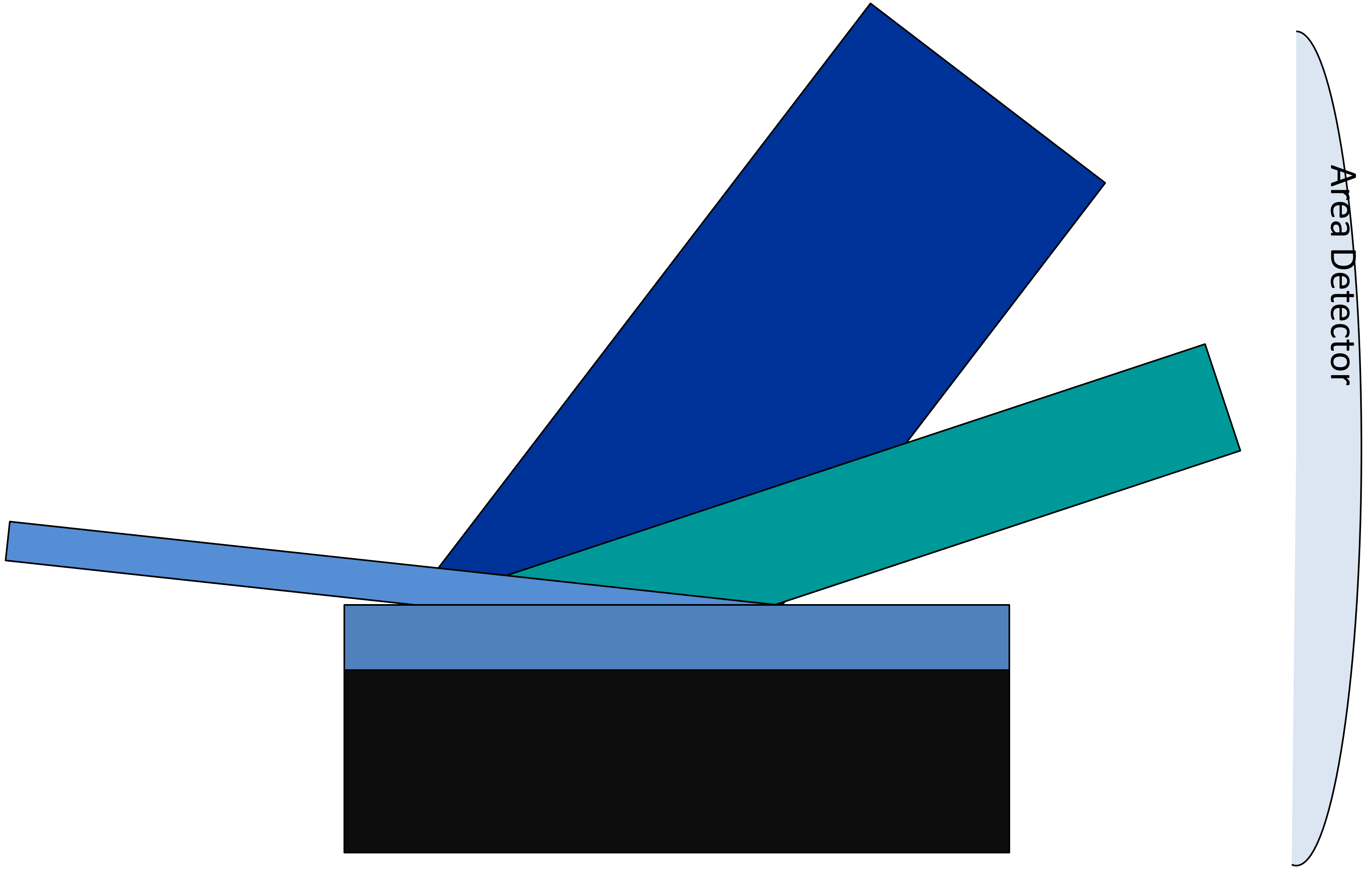
Grazing Incidence : Beam footprint

Beam footprint = beam height/sin(α)



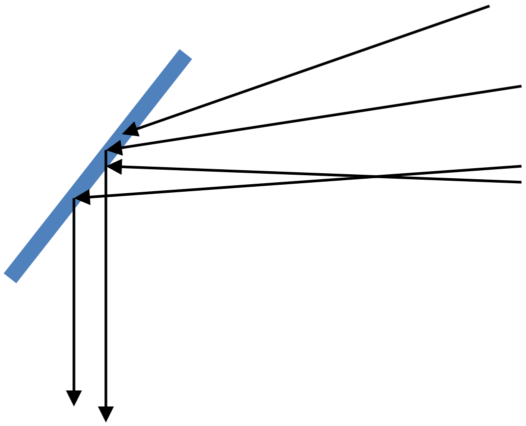
α~ 0.5,
for a 100 micron beam → beam footprint ~10 mm
GIXRD requires long (~ 20 mm) and flat samples

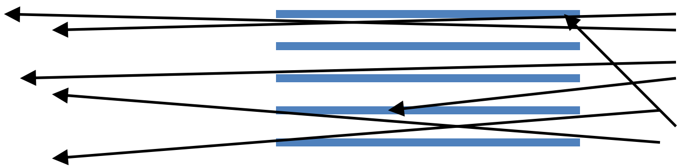
Grazing Incidence: Peak widths



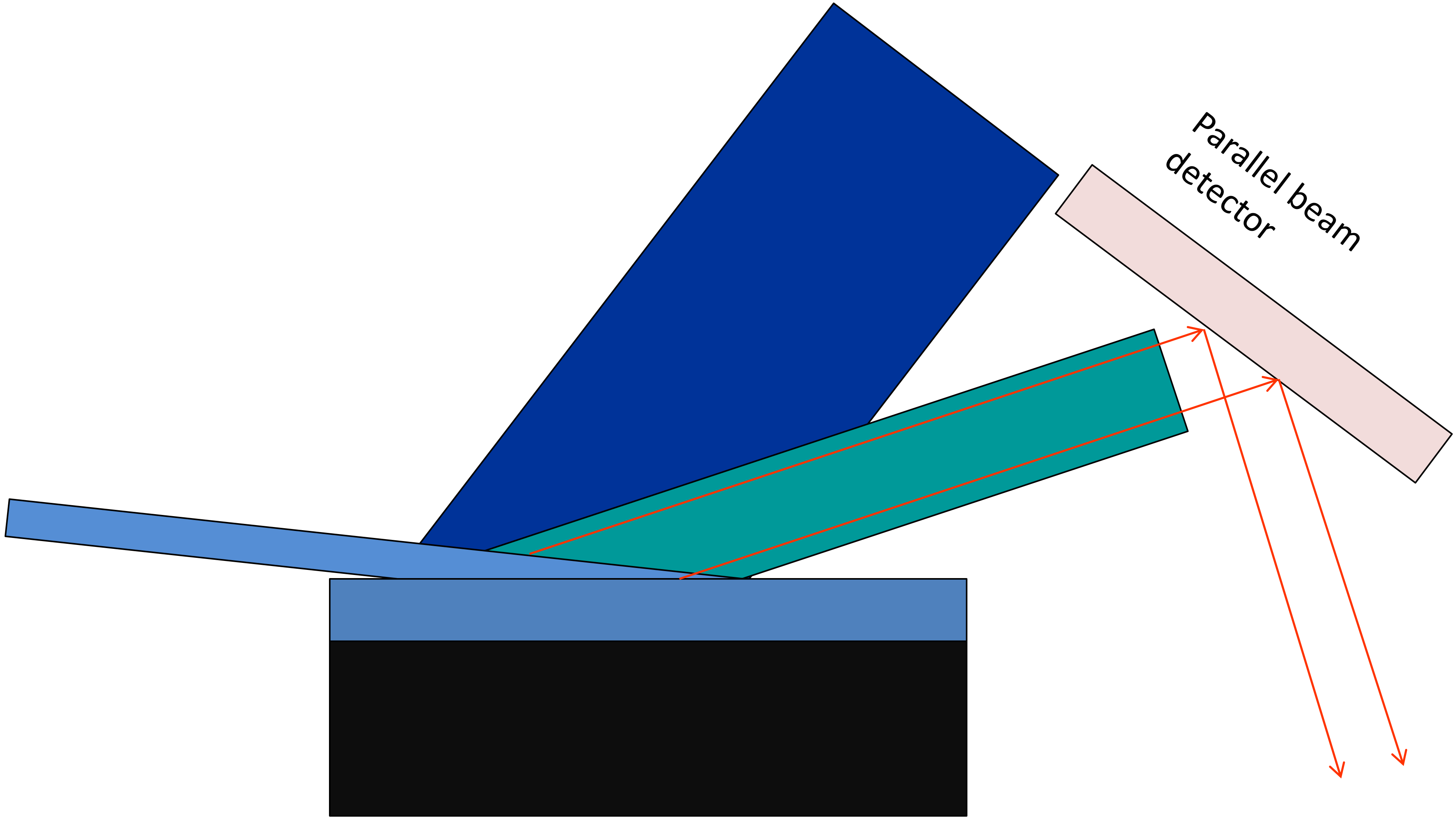
Parallel beam detection

- Crystal Analyzer
  - Perfect crystal (Si or Ge)
- Soller Slits



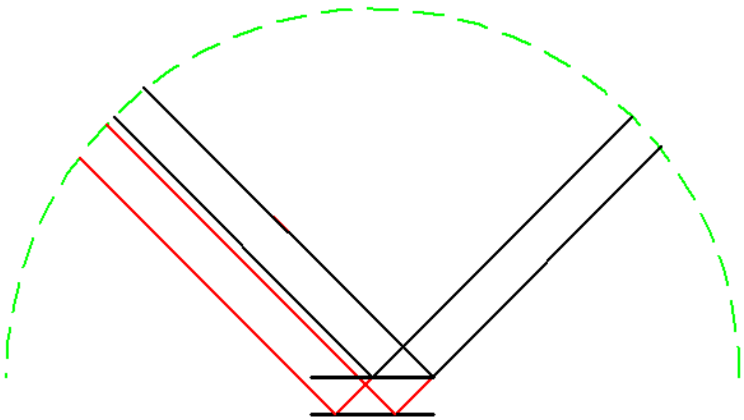


Grazing Incidence: Peak widths

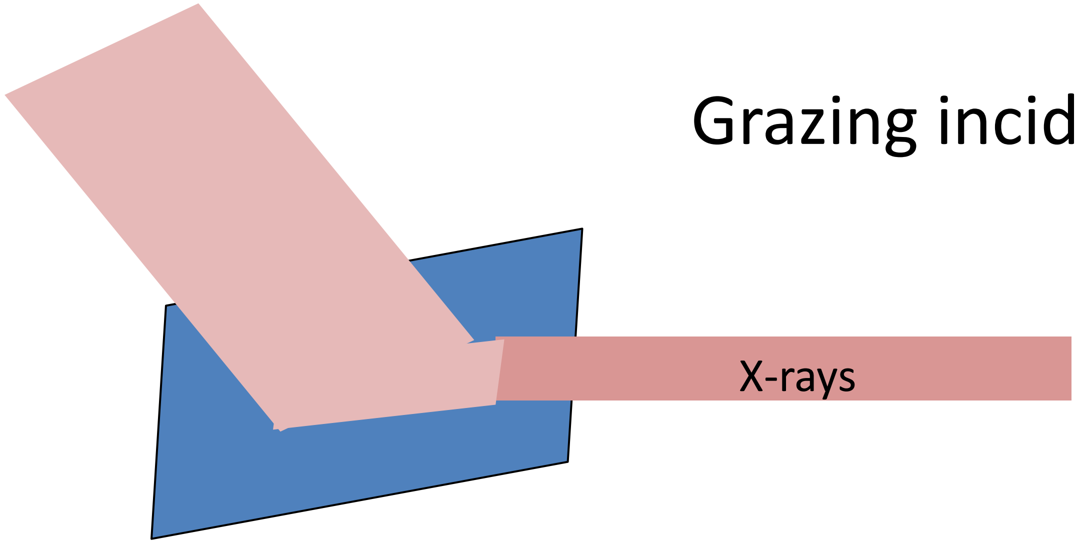


Parallel Beam Detection:

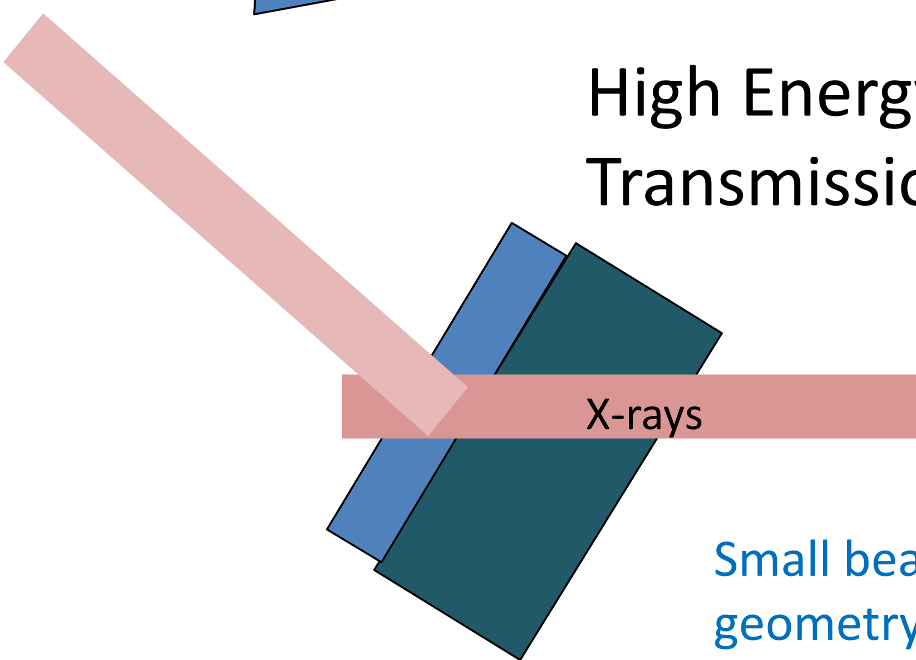
Misalignment Tolerant



Thin Film: inplane peak



Grazing incidence



High Energy Transmission

>16 keV : x-rays can easily penetrate 300 micron Si or Saphire substrate

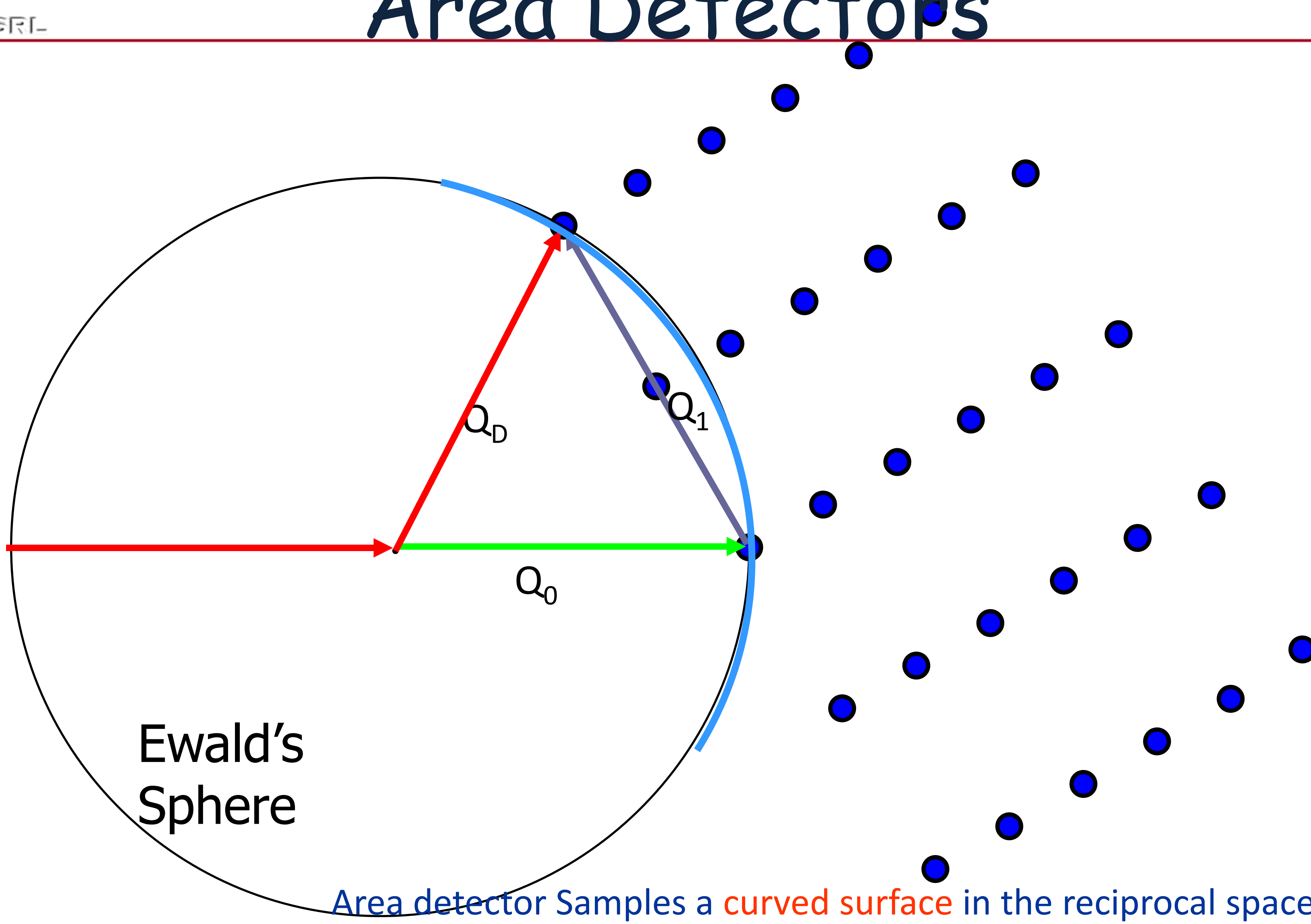
Small beam footprint, easier scattering geometry

Detectors

Detectors

- Point Detectors: data collection sequential
  - Can be used in parallel beam geometry
  - By using attenuators → very large dynamic range
  - low noise, single photon counting
  - Energy Sensitive detectors: E resolution ~ 200 eV
    - Vortex, Ge detector
    - Good when there is need to remove inelastic scattering signal: e.g., fluorescence, compton scattering
  - Energy Insensitive detectors: E resol > 1000 eV
    - photomultiplier, avalanche photodiode

Area Detectors



Area detector Samples a curved surface in the reciprocal space

Detectors

- Area Detectors : parallel data collection.
  - Cannot be easily used in parallel beam geometry
  - Dynamic range usually lower, but improving
  - noise higher, but improving
  - No energy resolution, but can have a low E threshold
  - MAR345, MARCCD, Pilatus

Detectors

Can you use

- Area detector for lower resolution, quick measurements?

- High resolution point detector for details?

Optimal scan range and resolution

Instrumental Resolution & peak width

$$W_m^2 = W_s^2 + W_i^2$$

Measured sample instrumental

Ideally $W_i < 0.5\ W_s$

But not too small as improvement in instrumental resolution comes at the cost of intensity

OK for nanocrystals

Area Detector

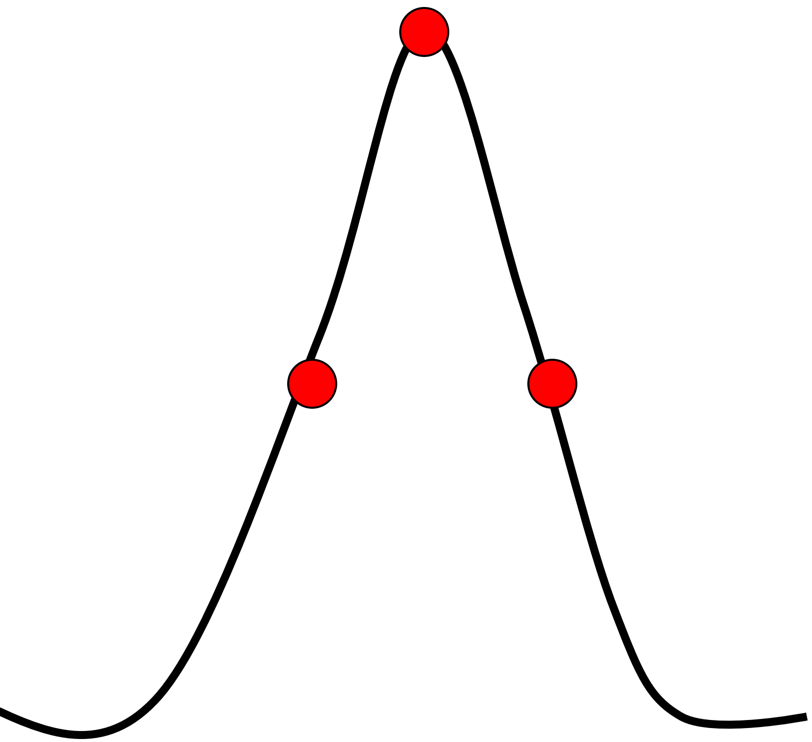
1mRad Soller Slits

Crystal Analyzer

Increased Resolution

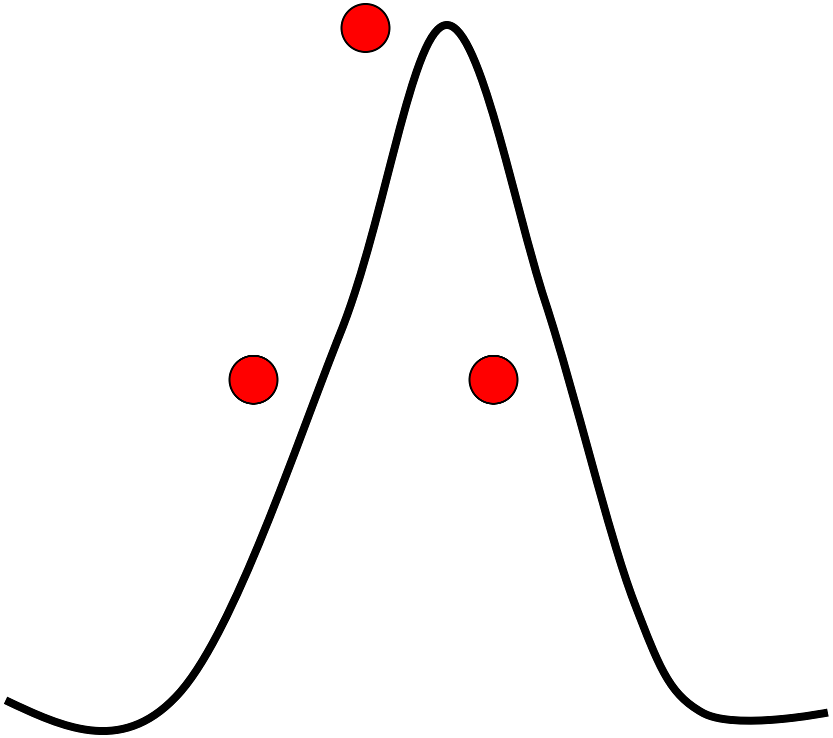
Optimal Scan

Step Size



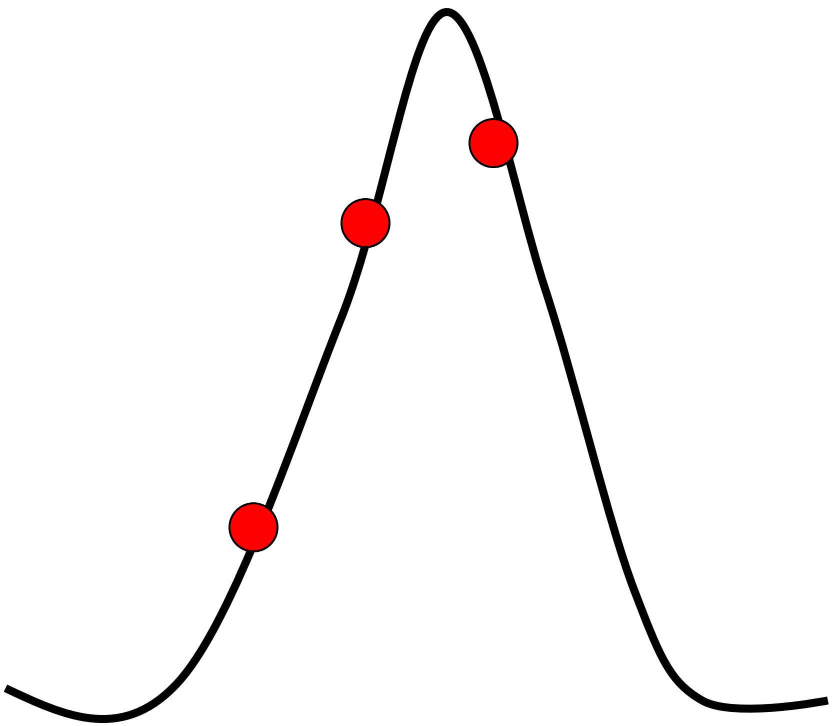
Optimal Scan

Step Size



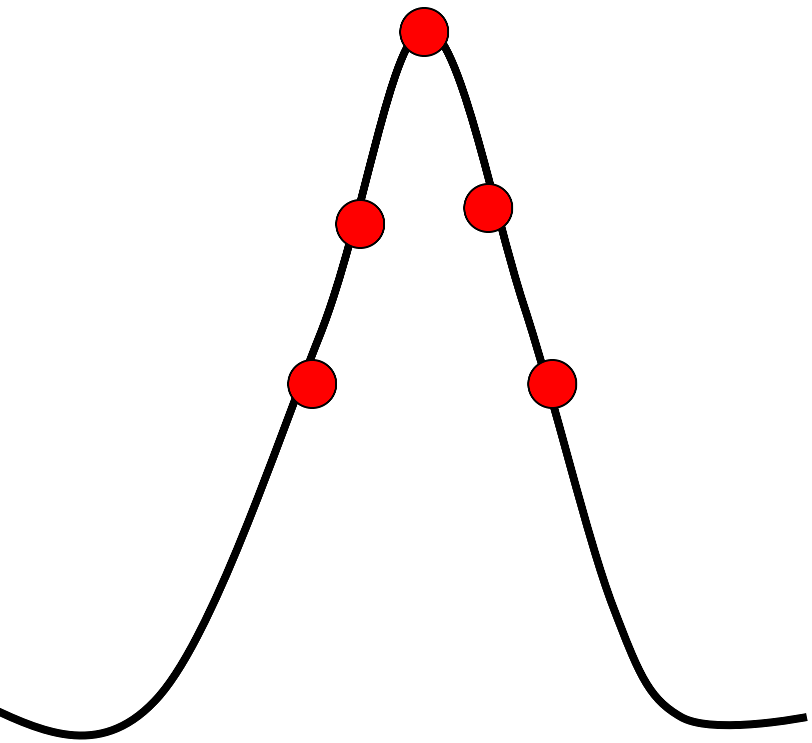
Optimal Scan

Step Size



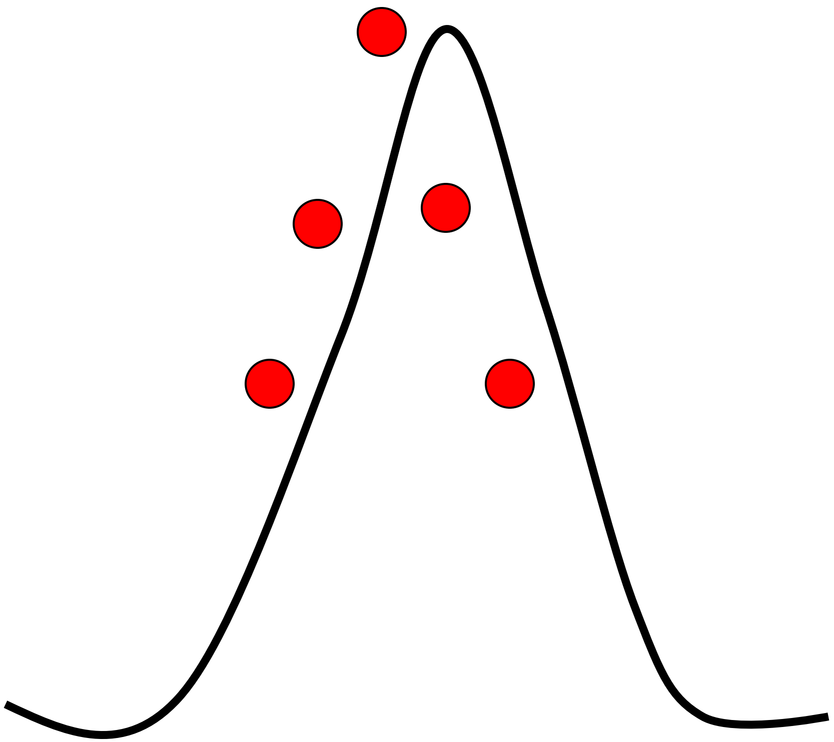
Optimal Scan

Step Size



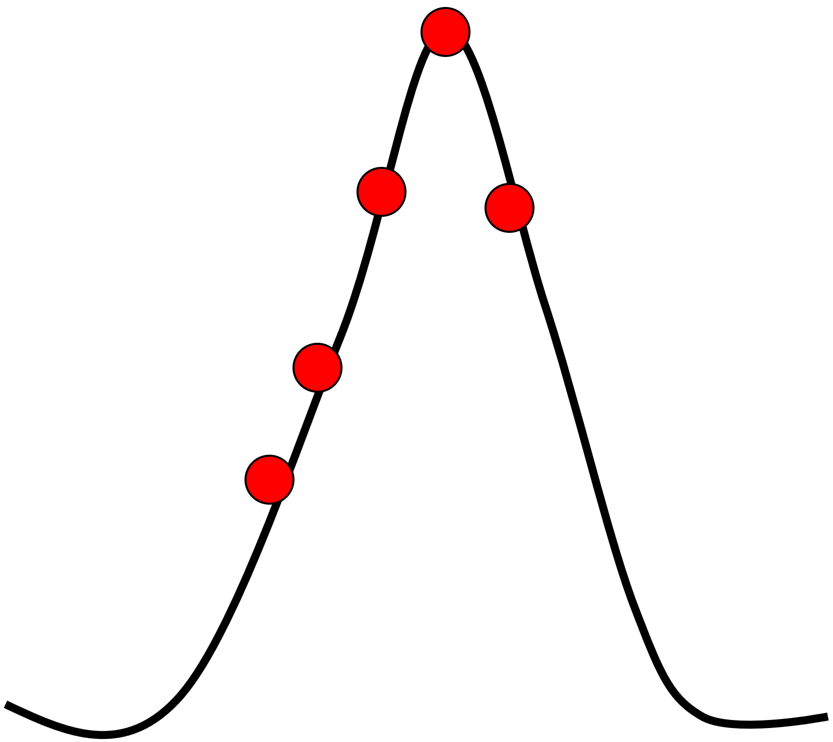
Optimal Scan

Step Size



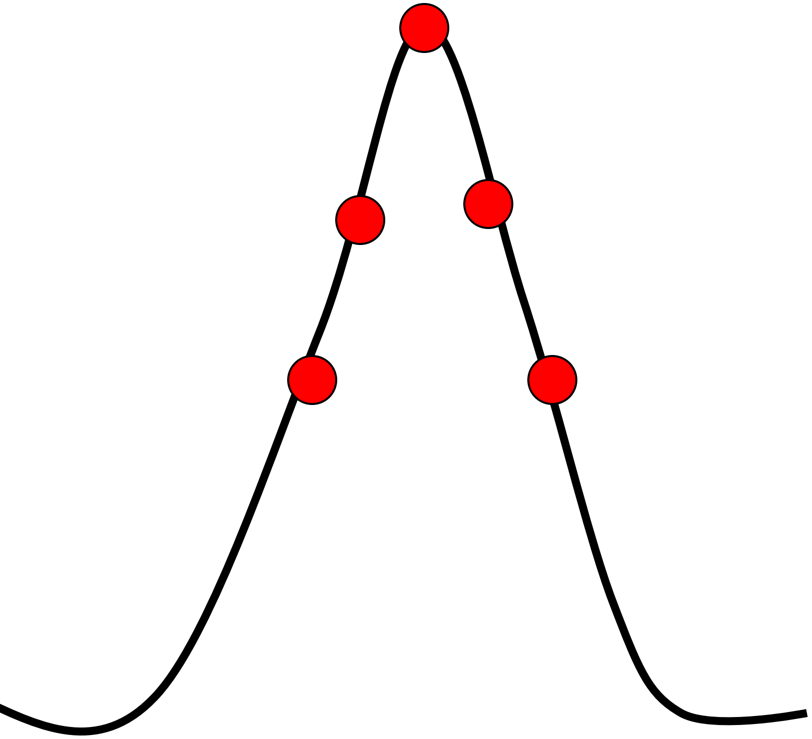
Optimal Scan

Step Size



Optimal Scan

Step Size



Minimum of 5 points in FWHM

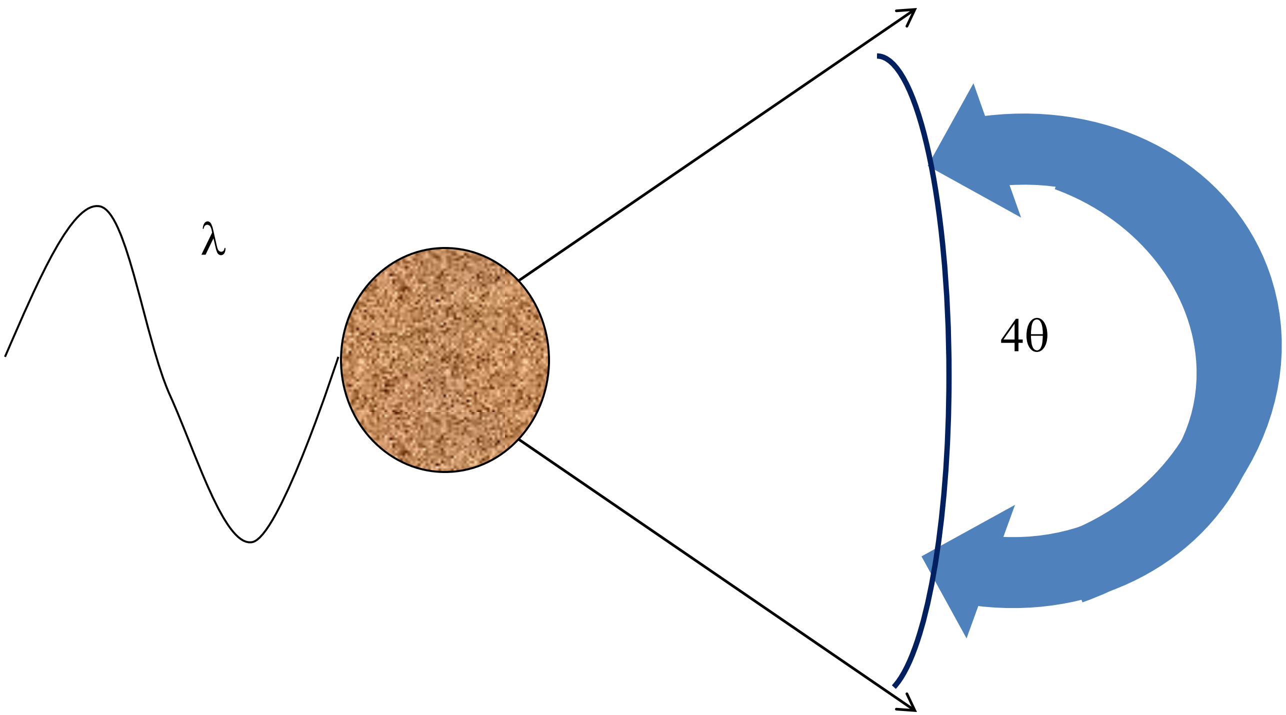
But probably not more than 10

Keep the dwell time so that Each Sweep is not too long
~ 2 -6 hr.

If better statistic reqd
Repeat sweeps and add.

- Averages out some of the time dependent drifts
- If unexpected beam dumps still have usable data
- Allows better control on statistics

Diffraction Pattern



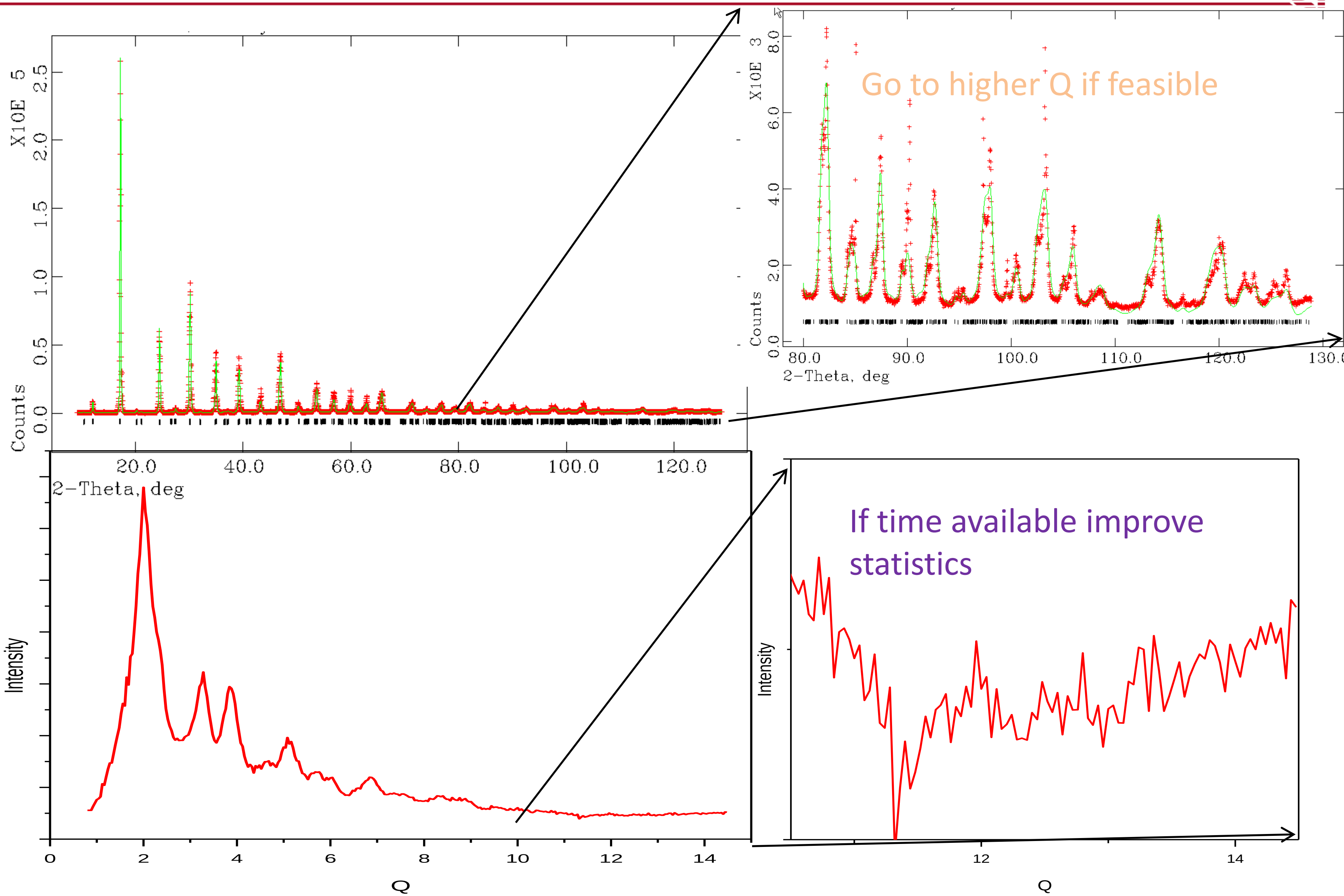
$Q = 4\pi \sin(\theta)\, E/hc$

Diffraction Pattern:

Contains all the contrast relevant information at the resolution of

$\lambda/2\sin(\theta) \sim 2\pi/Q_{max}$

Best use of time



Data Collection Strategy

- Before the experiment
  - Q Range
  - Q resolution
  - X-ray Energy
    - Fluo bkg, Resonance scattering
  - Diffractometer and sample geometry -
    - 2 circle, 4 circle, GI, transmission
  - Detectors
- During Data collection
  - Scan parameters (step size, dwell time.)
  - Beam damage?
    - Dehydrate, photo- redox, reacts with the environment

Questons?