Texture in Electronic Applications

Introduction and Overview

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NIST, 10th October, 2000

Registrants last week

- 13 Industry
- 13 National Labs
- 13 University

Administrative items

- Overview
  - Materials: piezoelectrics, ferroelectrics, dielectrics, magnetics, metallization, etc
  - Material forms: thin film (polycrystalline, epitaxial), bulk
  - Producing/controlling texture: deposition methods and parameters; substrate effects; processing; seeding methods
  - Measuring texture: techniques; accuracy; reciprocal space view
  - Effects of texture: intrinsic; extrinsic; morphological
- Quote
  - “NIST is a user facility with which industry should become better acquainted.”

Importance of texture

- **Texture becomes important when materials properties are anisotropic** (i.e. almost always!)
  - intrinsic (tensor) property, e.g. switching behavior in ferroelectrics.
  - extrinsic - typically linked with morphological texture or interface properties, e.g. grain boundary leakage current in dielectrics, fracture toughness

- **In some cases the link between the texture of a material and its properties and application has been clearly demonstrated**

- **In others, texture has been observed (and may change during processing); however no link between texture and important properties has been demonstrated**
  - is texture just "along for the ride"?
  - are we lacking the necessary insights to understand why it matters?

Measurement Techniques

- X-ray diffraction
  - synchrotron, rotating anode, fixed tube
  - 4 circle goniometer, 2 circle (powder) diffractometer
  - conventional detectors, area detectors
  - Rietveld analysis of powder patterns
  - different correction methods for defocussing, absorption, etc

- Electron diffraction
  - EBSD: micro texture; grain size restrictions
  - TEM: highest resolution; specimen preparation !!!!

Measurement Techniques (cont)

- Neutron diffraction (posters)
  - very good for bulk but not thin films

- Other methods
  - stereology
    - morphological texture
    - relate to crystallographic texture
    - grain size restrictions
  - optical methods: birefringence, SHG, etc
  - Raman
  - AFM
  - any technique where response is function of orientation/texture

Issues

- new techniques
- effective use of existing methods
- need to understand techniques to understand results
- thin film standards
  - certified texture
  - certified thickness
  - need both

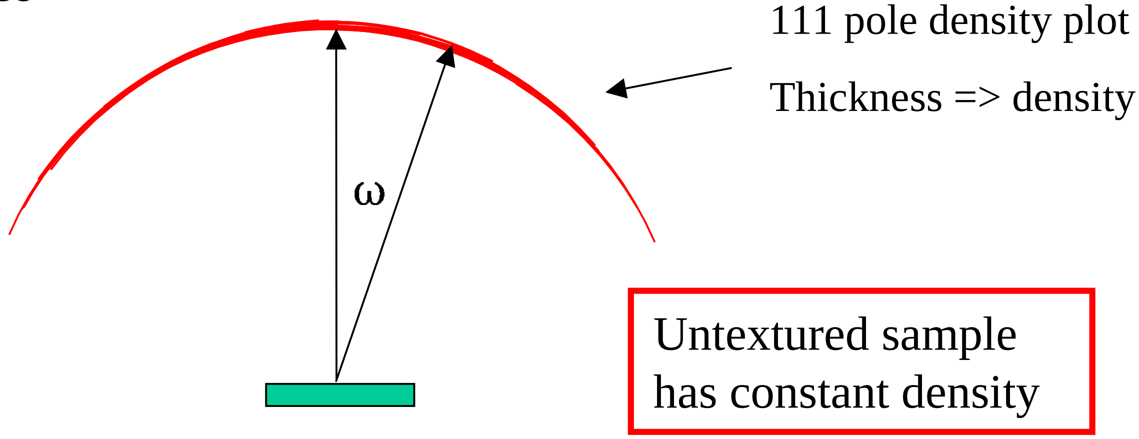
Applications and challenges

- performance enhancement by texture optimization
- texture as a monitoring tool
- measurement of random volume fraction
- the best technique for the job
- check techniques against each other
- fibre texture measurements using powder diffractometer
- accuracy of measurements
- effects of slit sizes

reciprocal space analysis

Measurement of texture

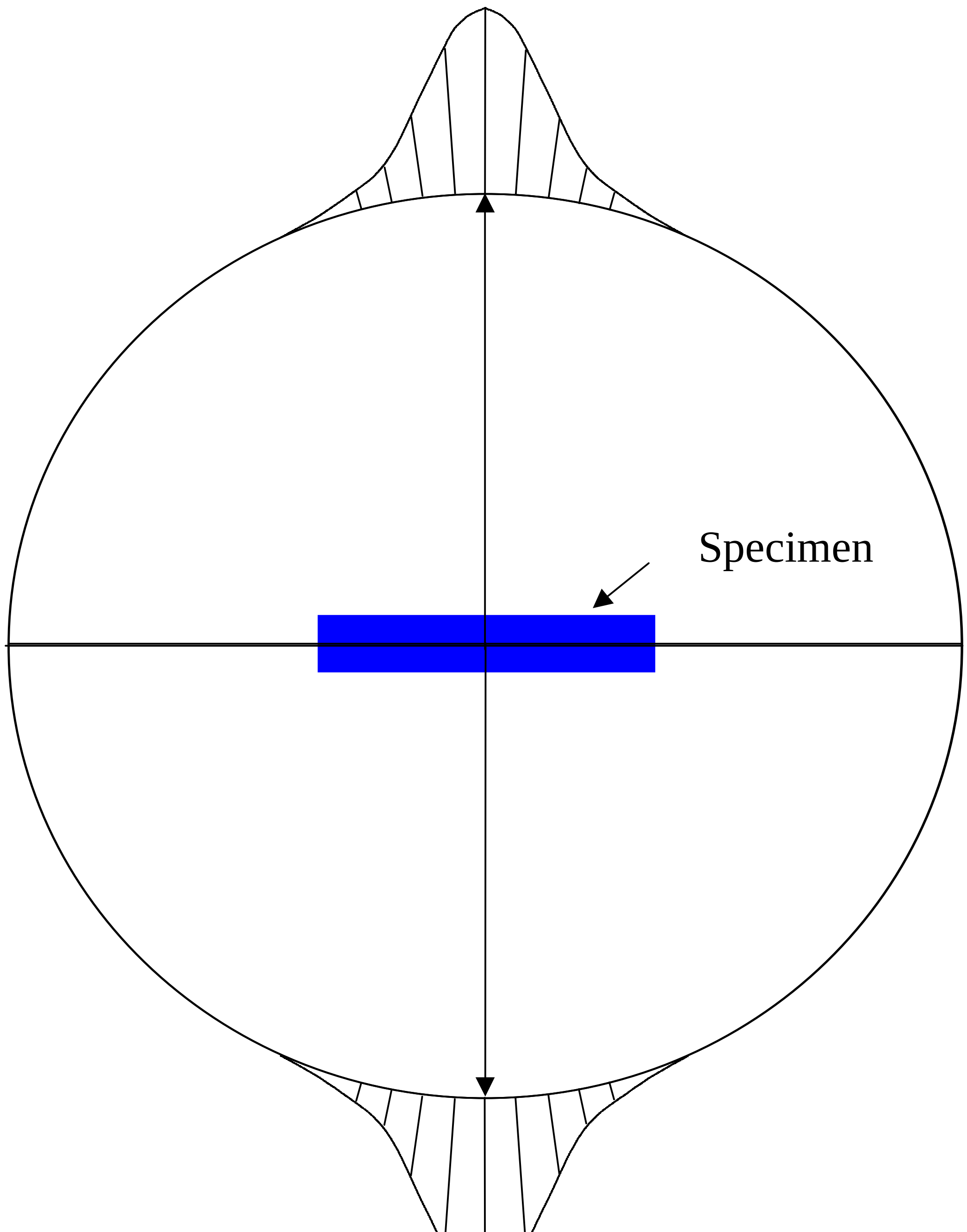
- Texture is function of relative orientation of crystal axes of grains in material with respect to specimen orientation
- Example: specimen with (111) preferred orientation normal to surface



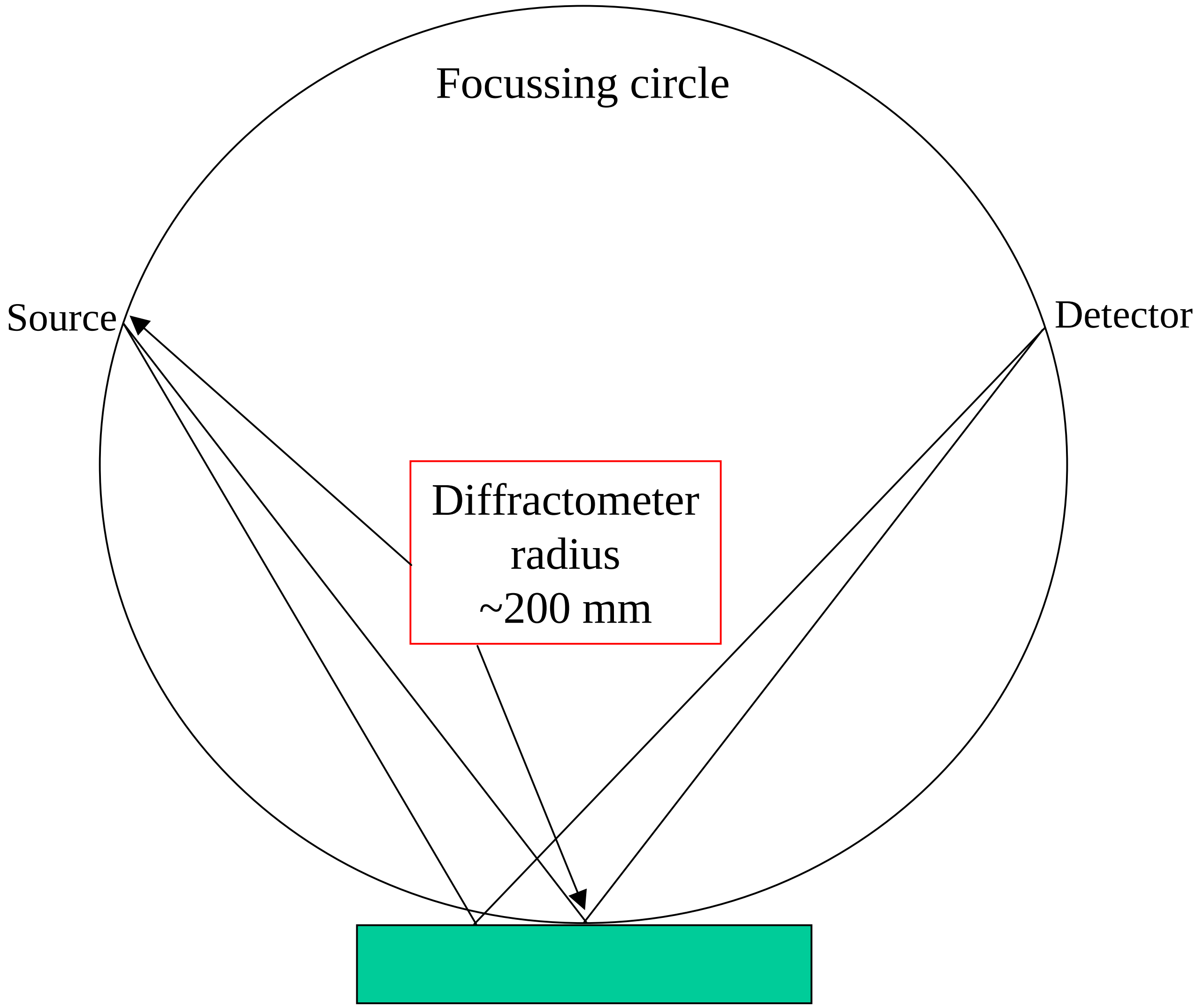
$$\text{Texture}(\omega) = \frac{\text{Volume fraction of sample at orientation } \omega}{\text{Volume fraction of untextured sample at } \omega}$$

- Diffracted x-ray intensity ∝ diffracting volume
- Diffracted x-ray intensity(ω) *could* give texture(ω)
- But there are problems:
  - defocussing
  - absorption
  - variation in irradiated area

Schematic reciprocal space of textured specimen



Defocussing



Parafocussing optics

Scattering angle varies on flat specimen

Angle changes very small for 10 - 20 mm specimen

Tilt procuces much larger angle changes

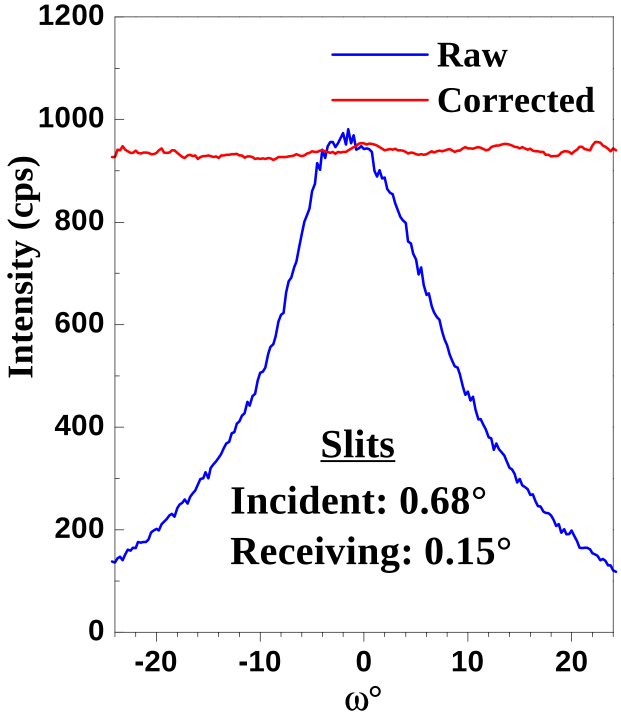
Measurement technique has been developed

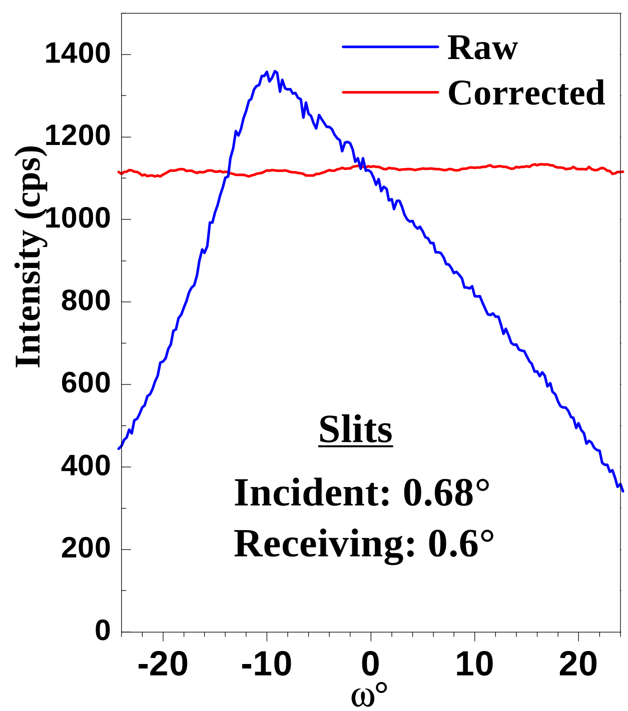
- TexturePlus
- software available:

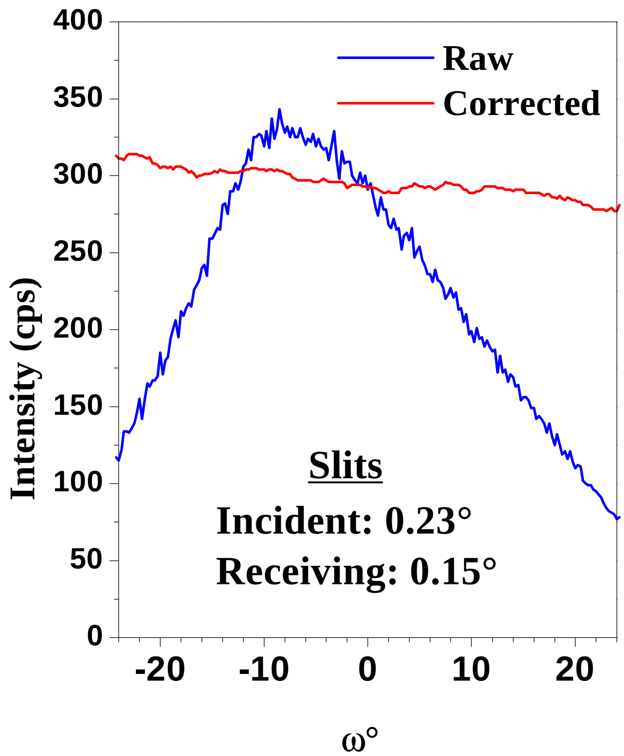
http://www.ceramics.nist.gov/webbook/TexturePlus/texture.htm

- uses 2 circle diffractometer
- only rotates about one axis for each scan so best for fibre texture
- only can scan up to Bragg angle of peak so not good for weakly textured specimens
- as large an irradiated area as machine can do
- therefore high intensity
- good for thin films
- relatively fast

Verification of correction factor using untextured alumina powder (SRM676)





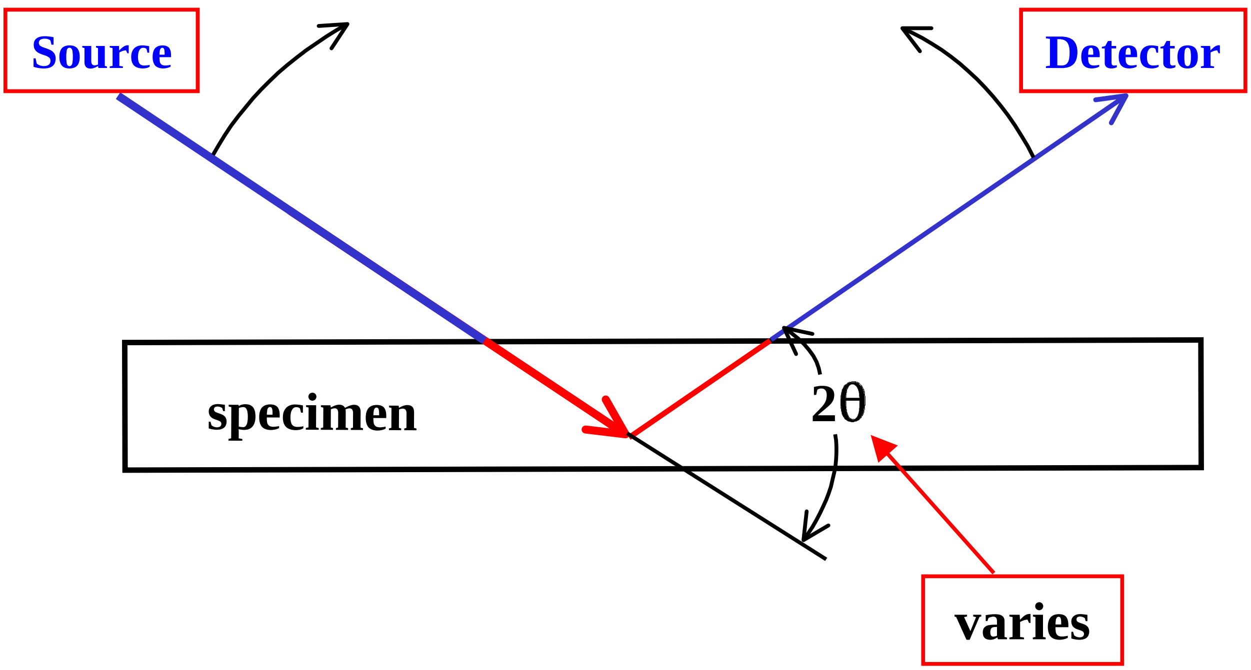


Peak: $30\bar{3}0$
Data collected with different slit arrangements.

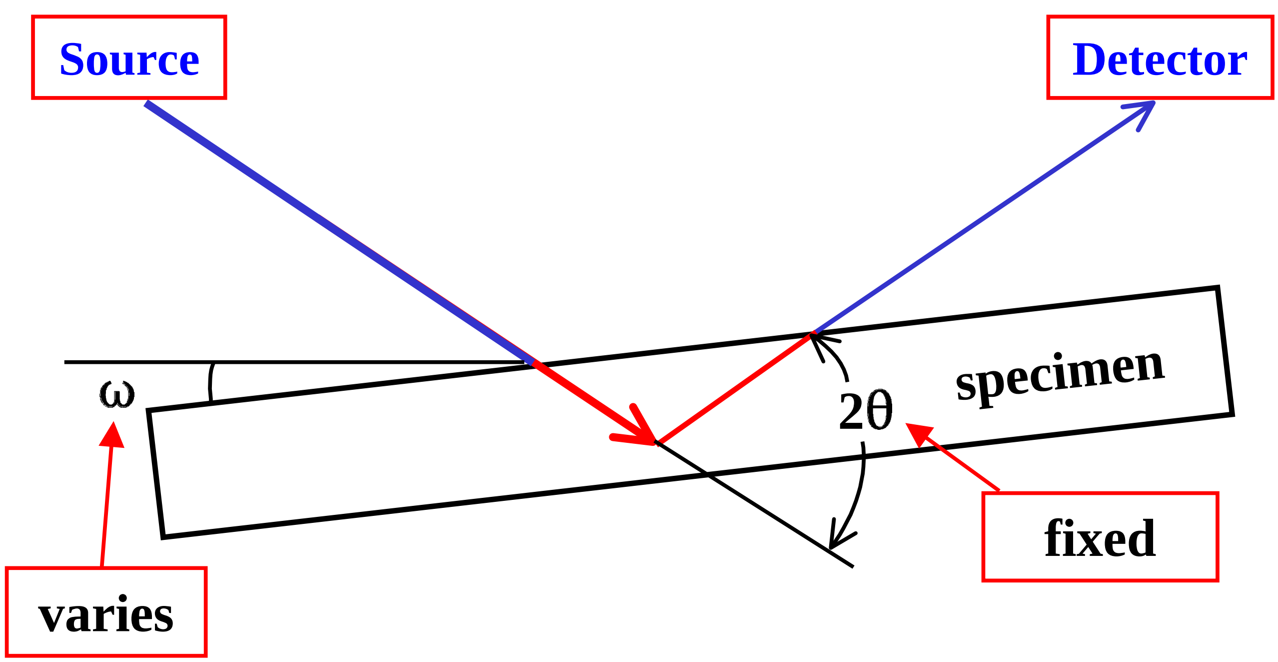
Raw data: peaked
Corrected curves: flat
- **raw data can mislead**
- **corrections work**

Corrections large at high |ω|
- **up to 5x**

θ-2θ scan

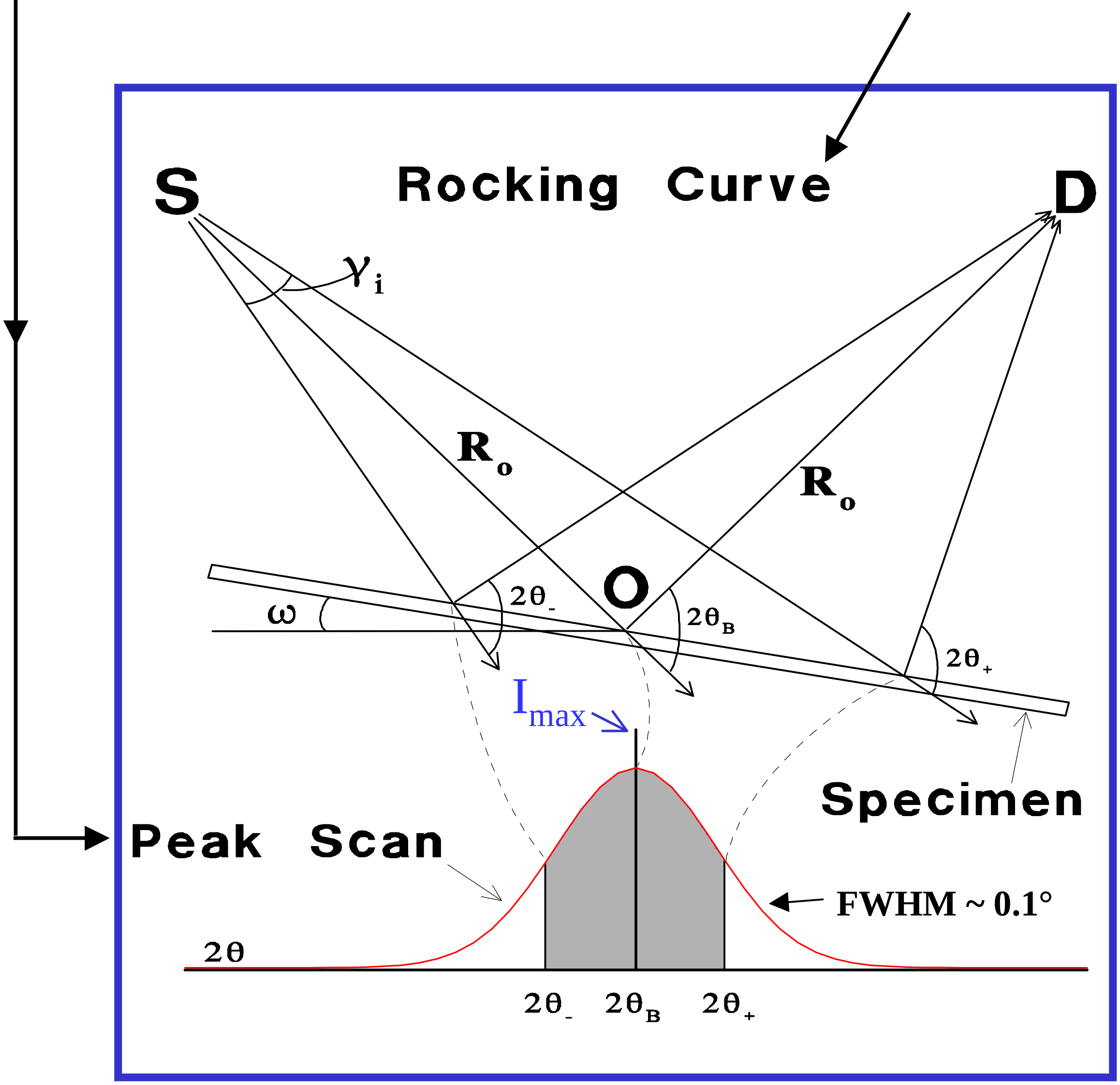


ω scan



Defocussing correction

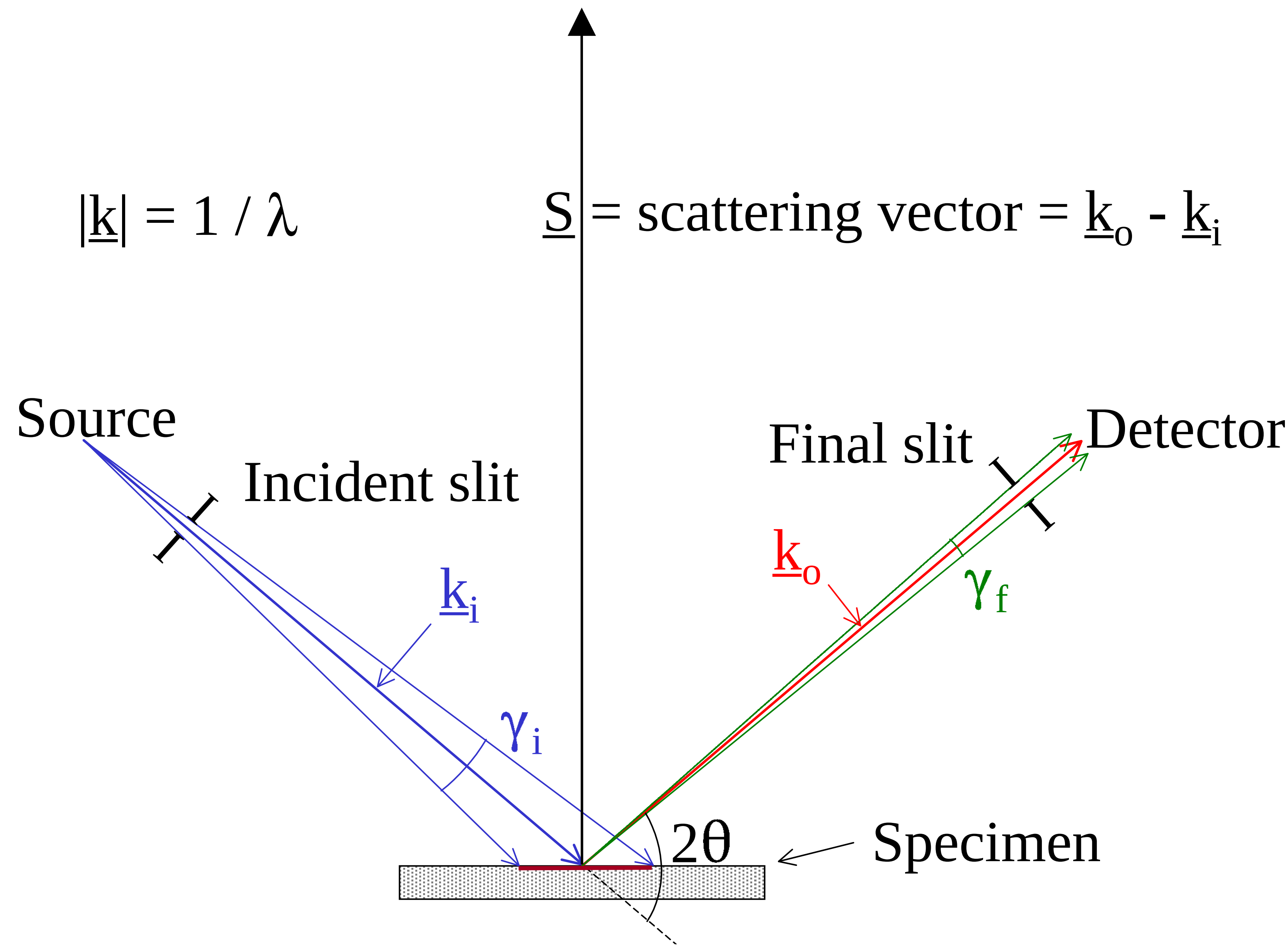
Scan 1: θ-2θ scan   Scan 2: ω scan



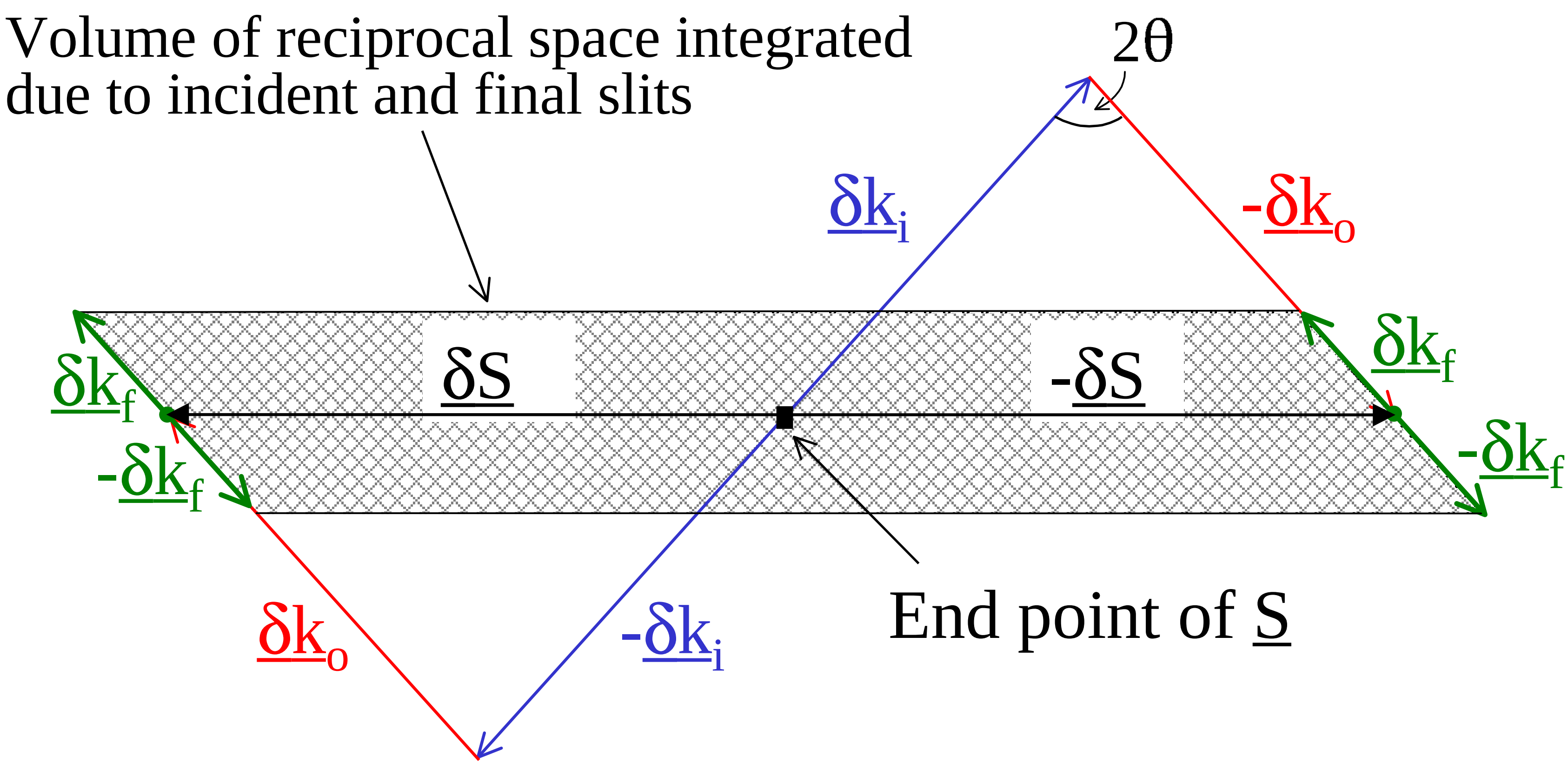
Calculate intensity from untextured specimen

$$I_{rand}(\omega) \propto \int_{-\gamma_i/2}^{\gamma_i/2} I_{pk}(2\theta(\omega,\gamma))\, d\gamma$$

Divergent x-ray source geometry



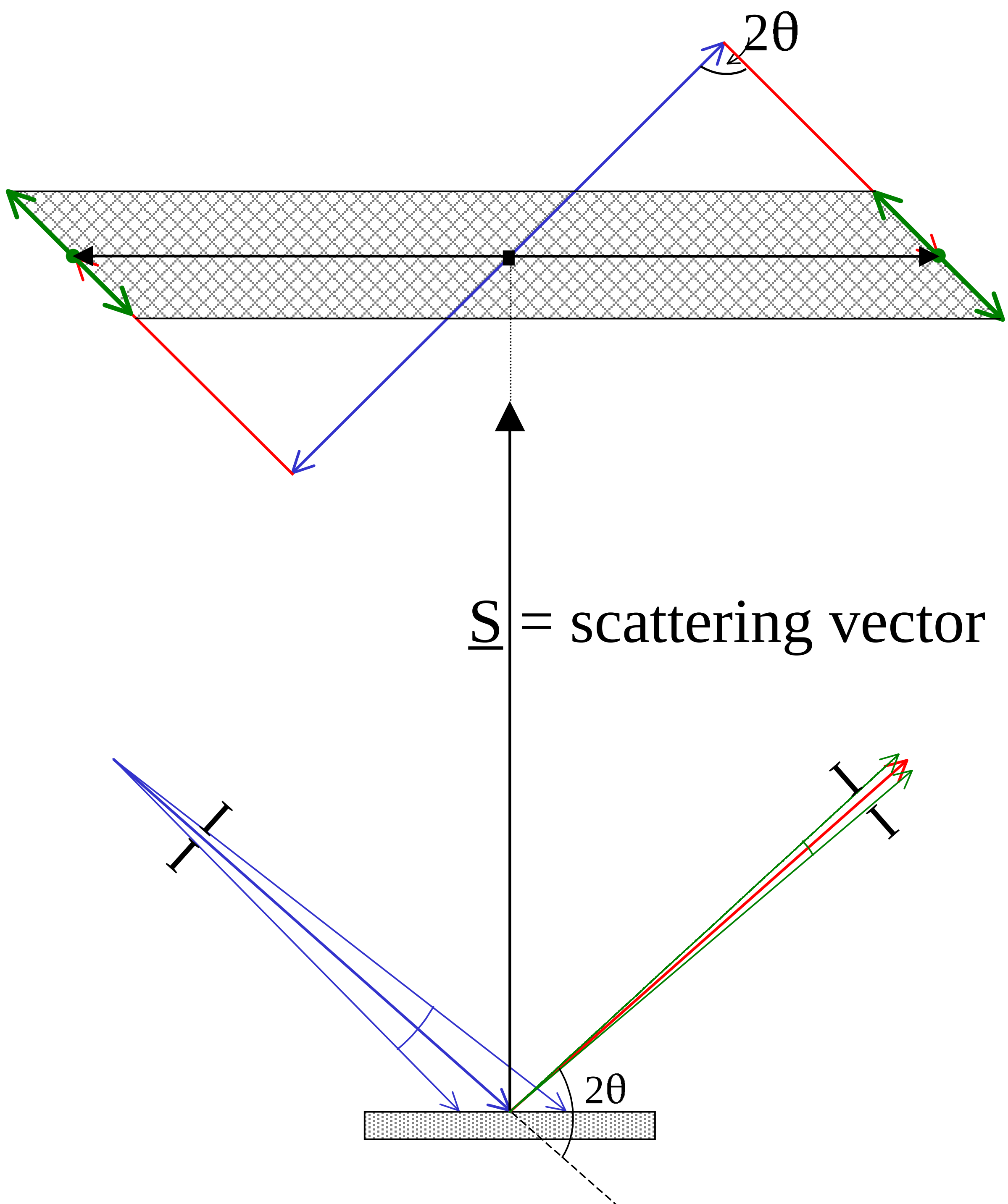
Reciprocal space schematic

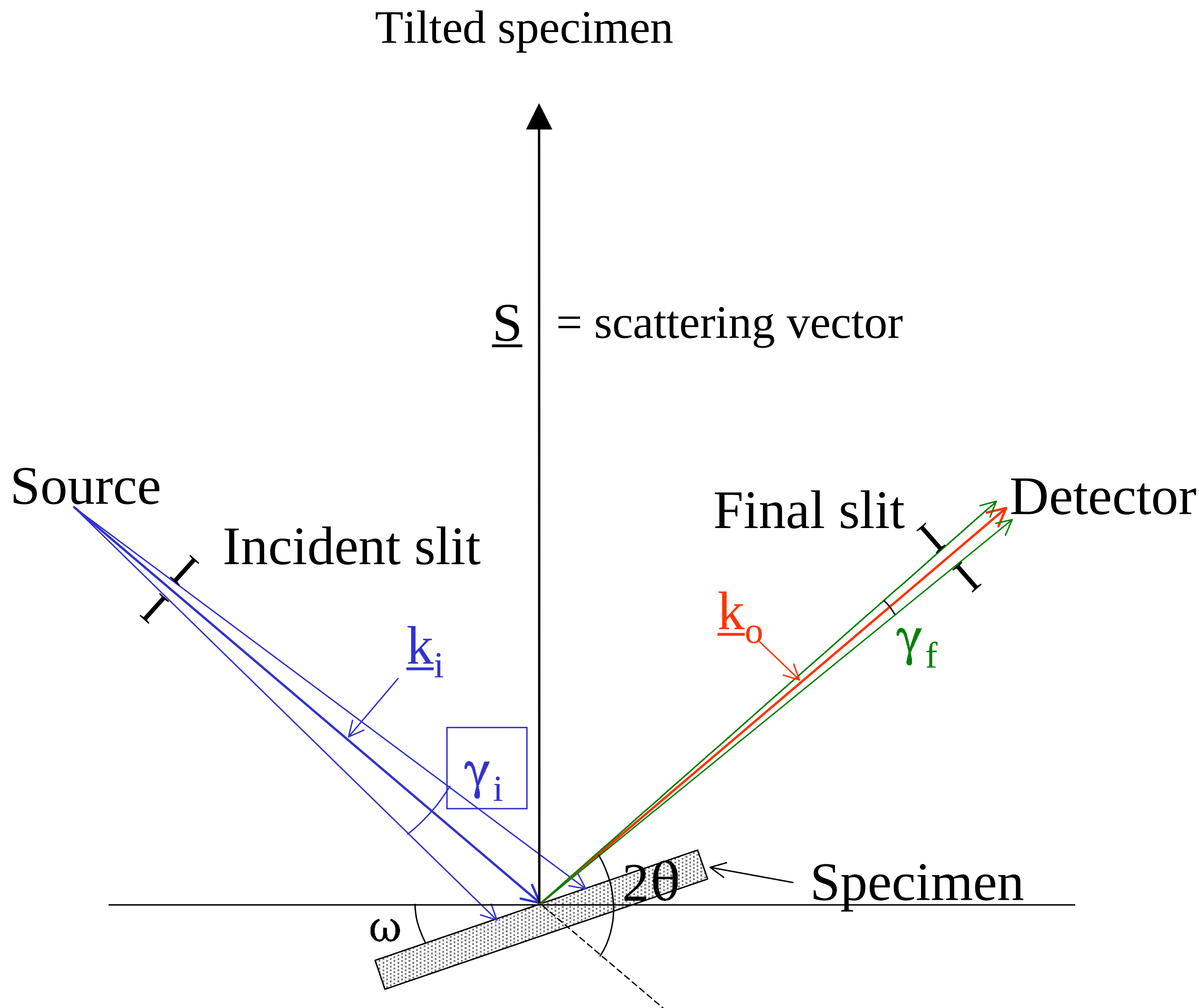


Width (2δS):

- ~ texture measurement resolution
- determined mainly by γ
- simple measurement with sapphire confirmed this

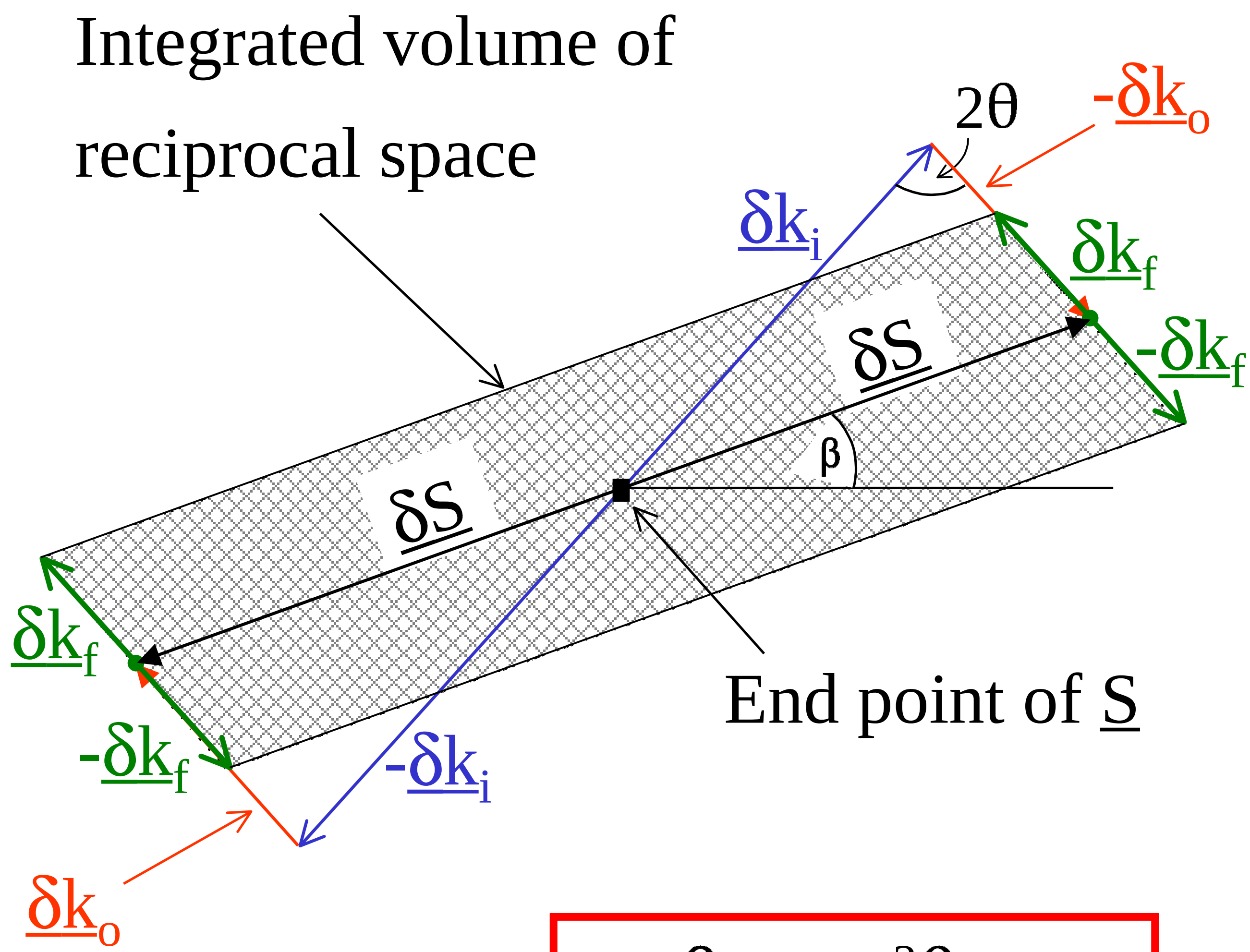
Top magnified ~ 100x relative to S





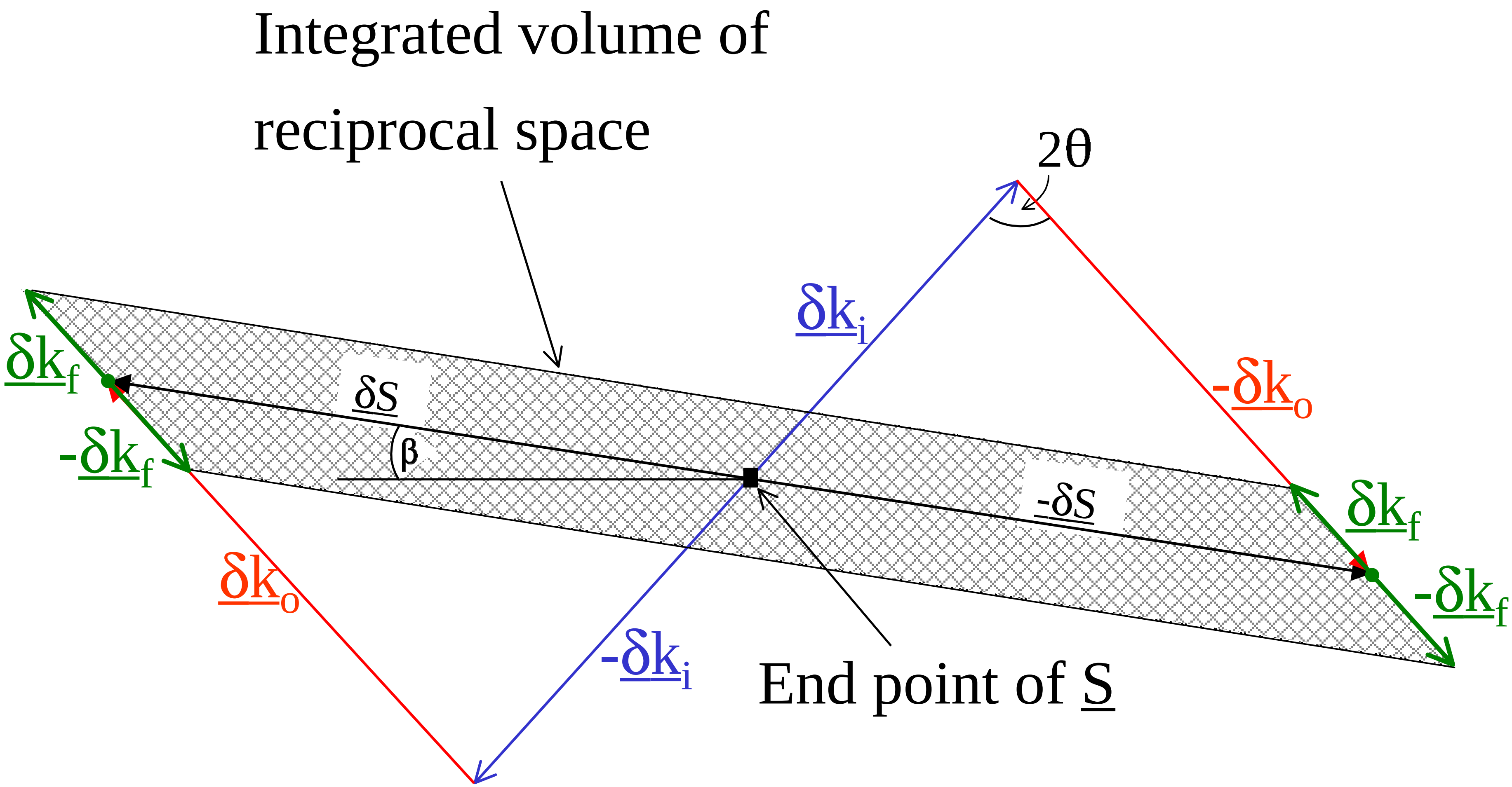
Tilted specimen
S = scattering vector
Source
Incident slit
k_i
γ_i
Final slit
Detector
k_o
γ_f
2θ
Specimen
ω

For $\omega > 0$:



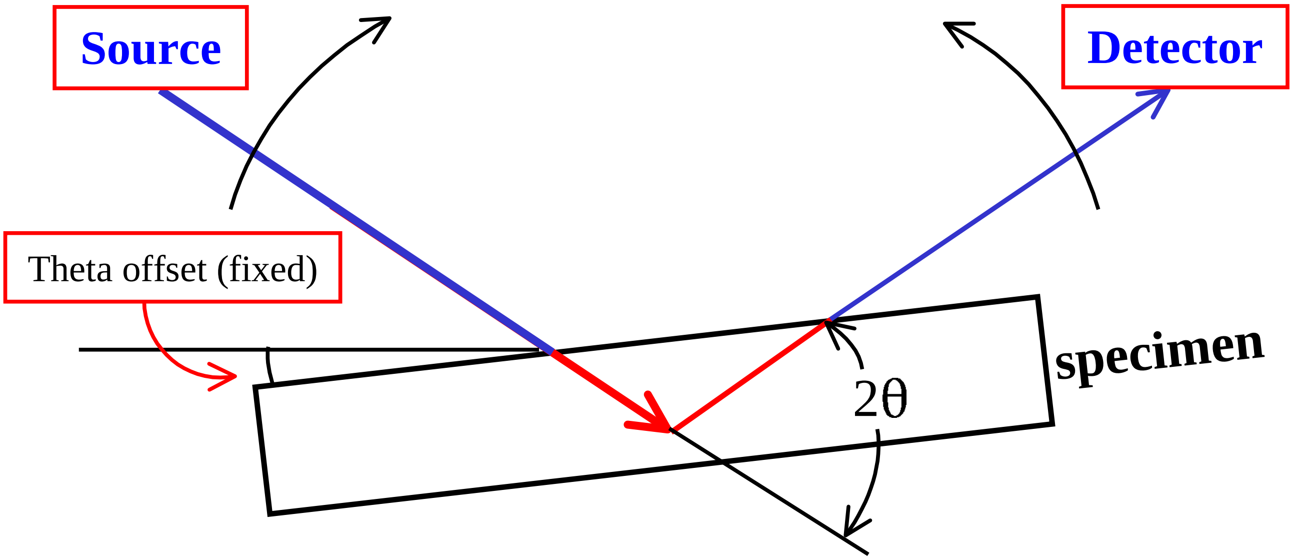
$$\tan\beta = \cos^2\theta \tan\omega$$

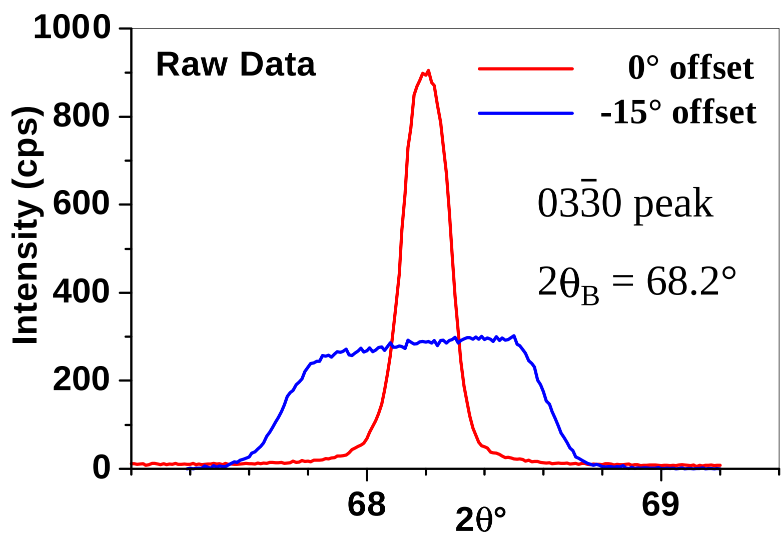
For $\omega < 0$:

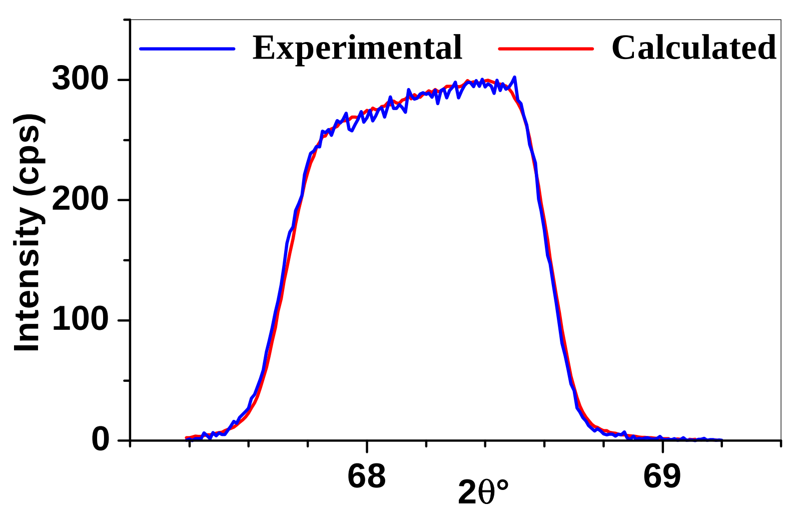


$$\tan \beta = \cos^2\theta \tan \omega$$

Theta-offset scans on **SRM676** alumina powder







"Isometric form of SRM676 grains effectively eliminates preferred orientation effects"

Powder pattern and rocking curve data for textured alumina

Specimen from Desi Kovar (University of Texas)

11 powder peak intensities divided by intensities from JCPDS 10-173

Note: analysis by Rietveld methods is more rigorous

Very approximate result: ~ 80% basal plane texture ~20% random