

Are your X-ray powder diffraction patterns any good?



Quality of powder diffraction data

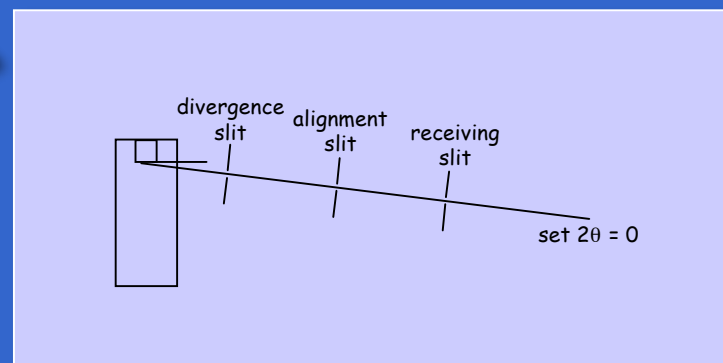
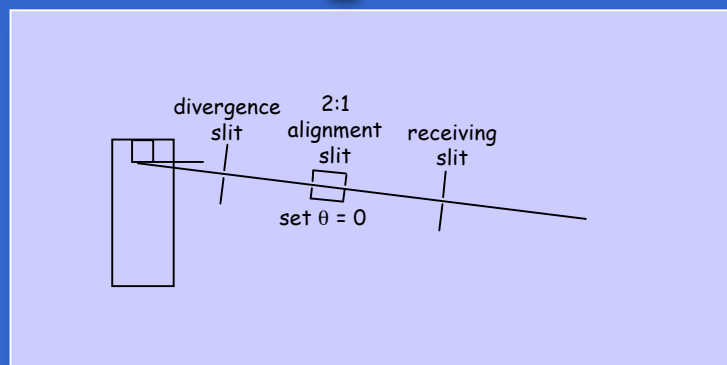
1. Is diffractometer properly aligned & operating correctly?
2. Is the specimen properly prepared?
3. Are proper scan variables used?

Diffractometer alignment

Alignment steps

Align instrument to see focal spot
Set take-off angle

Zero alignment
2:1 alignment



Quality of powder diffraction data

Diffractometer alignment

Alignment checks

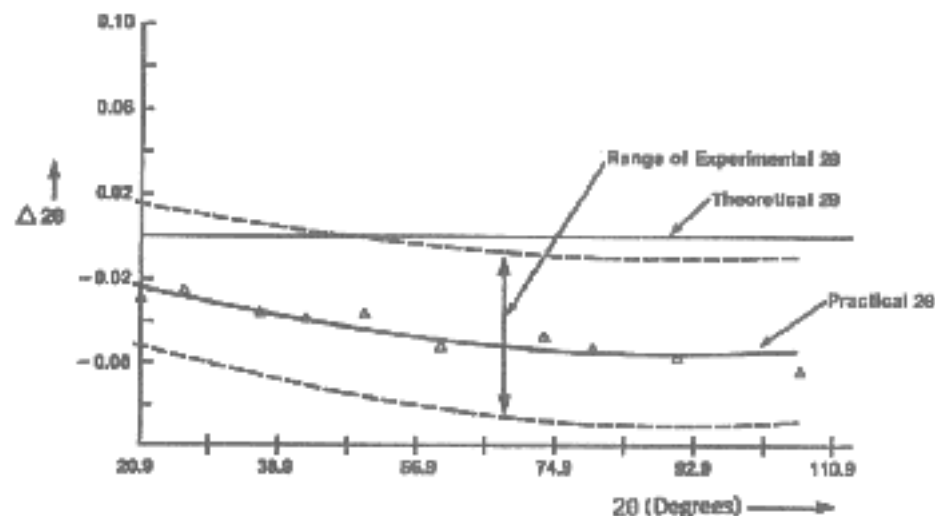
Run calibration pattern - use suitable standard

ex: NIST SRM 640c (Si), others

Compare measured 2θ values with NIST or standard values

Look for:

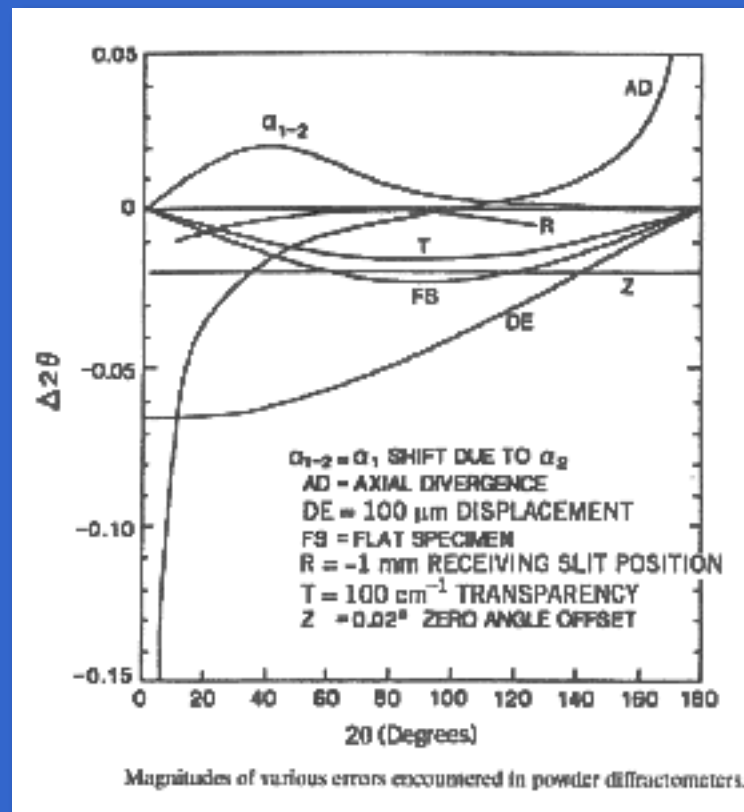
intensity loss
angle shifts
resolution loss



A calibration curve for α -quartz, showing theoretical, practical, and experimental curves.

Powder diffractometer

Alignment checks



Quality of powder diffraction data

Diffractometer alignment

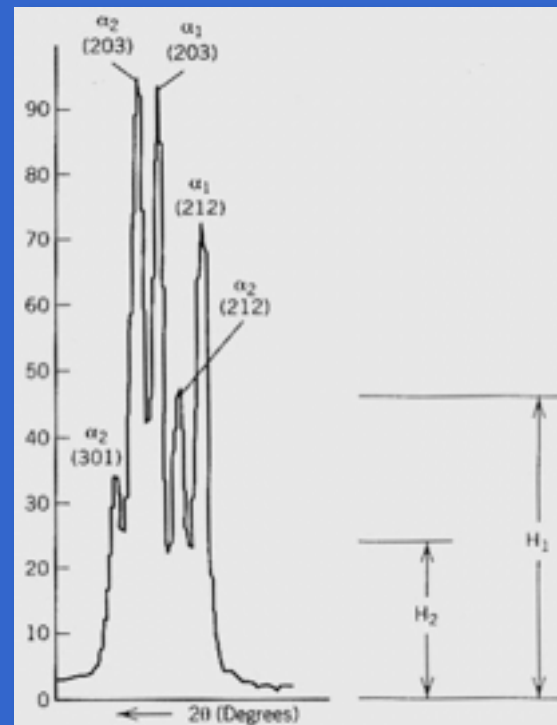
Alignment check

"five fingers of quartz" (SiO_2)

Measure:

position & intensity
of $(203)\alpha_1$

H_1/H_2



Quality of powder diffraction data

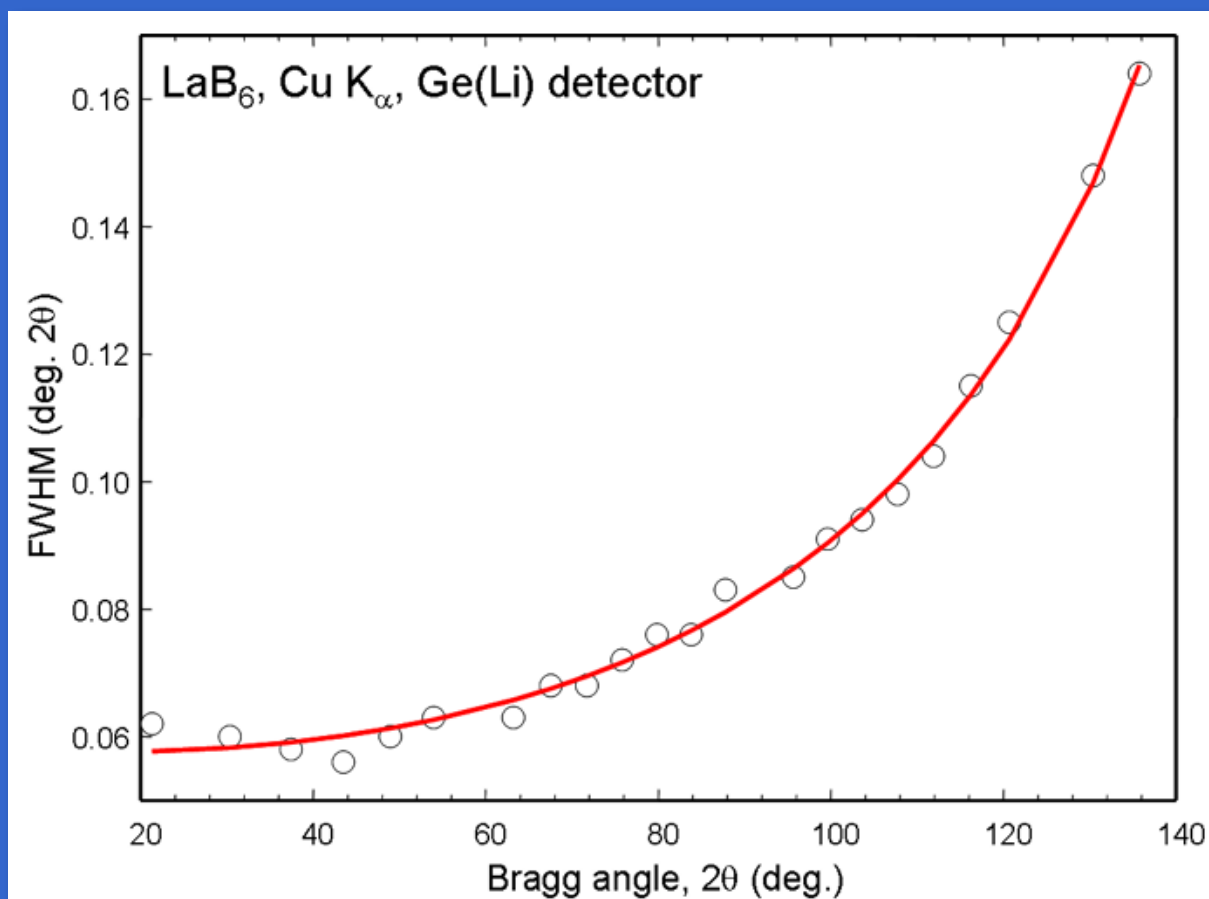
Alignment checks

Run calibration pattern - use suitable standard

Use NIST SRM
660 standard
(LaB_6)

Measure FWHM
values

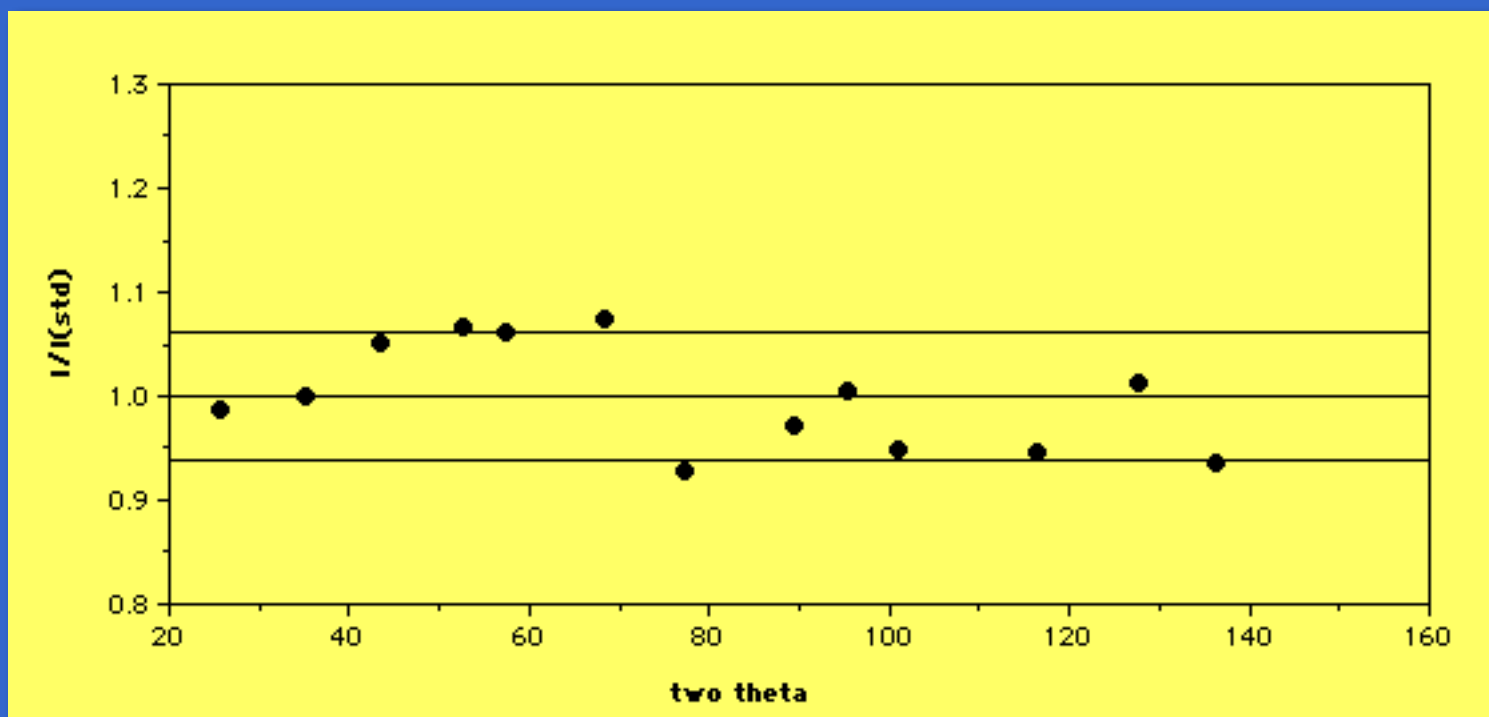
Compare measured
FWHM values with
NIST values



Quality of powder diffraction data

Alignment checks

Run calibration pattern - use suitable standard

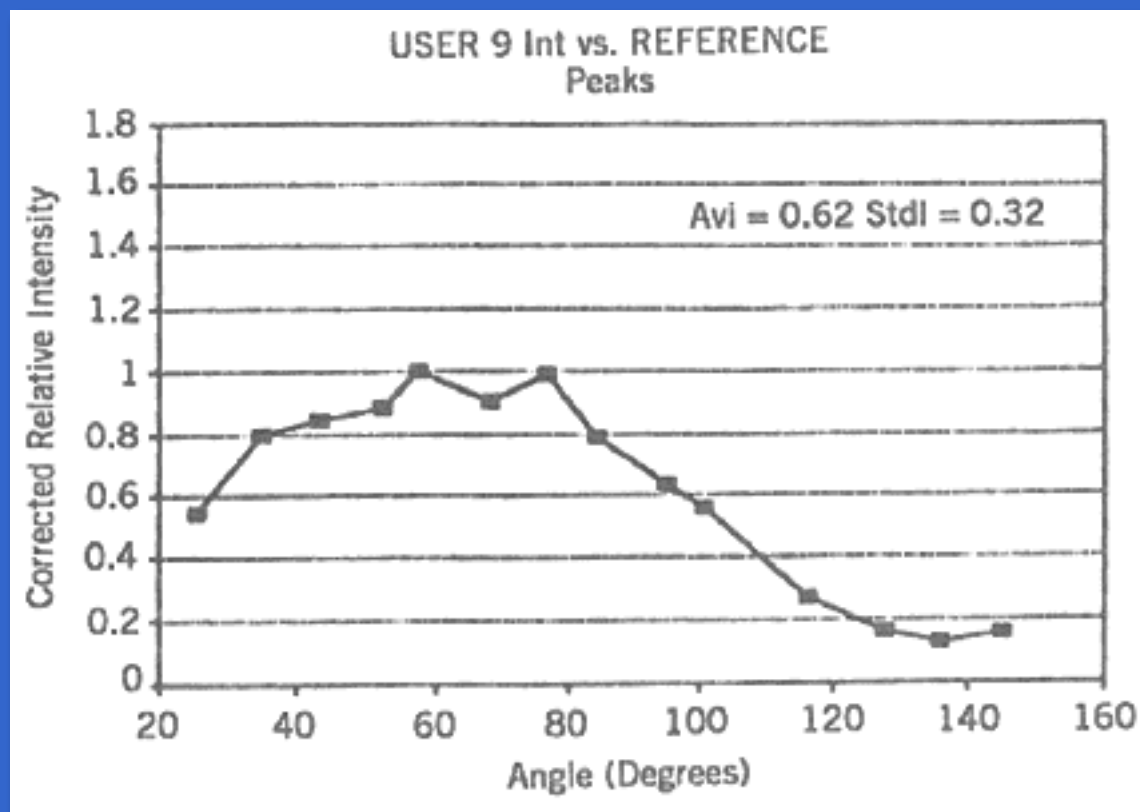


NIST SRM 1976 (Al_2O_3)

Quality of powder diffraction data

Alignment checks

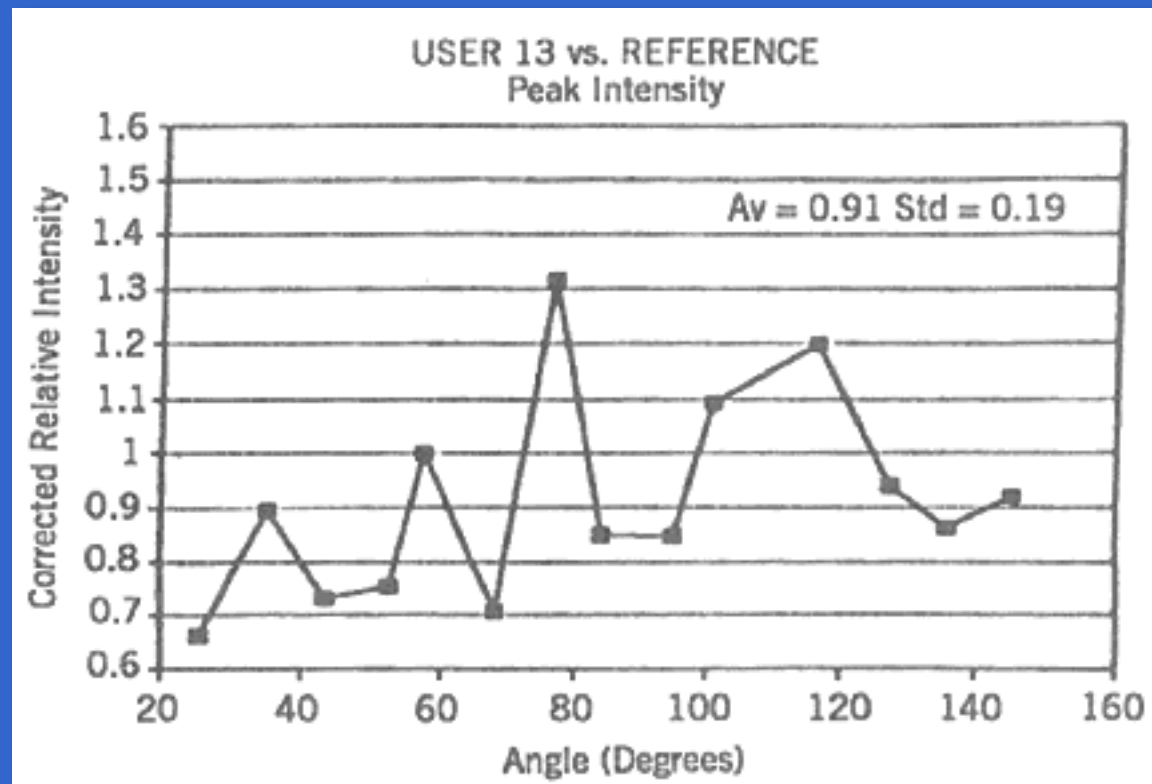
Run calibration pattern - use suitable standard



Powder diffractometer

Alignment checks

Run calibration pattern - use suitable standard



Quality of powder diffraction data

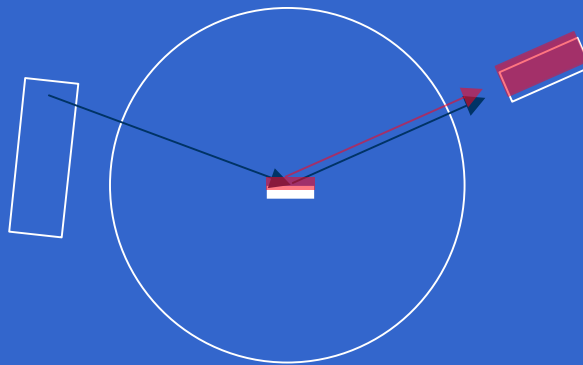
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2. Is the specimen properly prepared?
3. Are proper scan variables used?

Quality of powder diffraction data

Is the specimen properly prepared?

Requirements

sample surface must be on diffractometer rotation axis



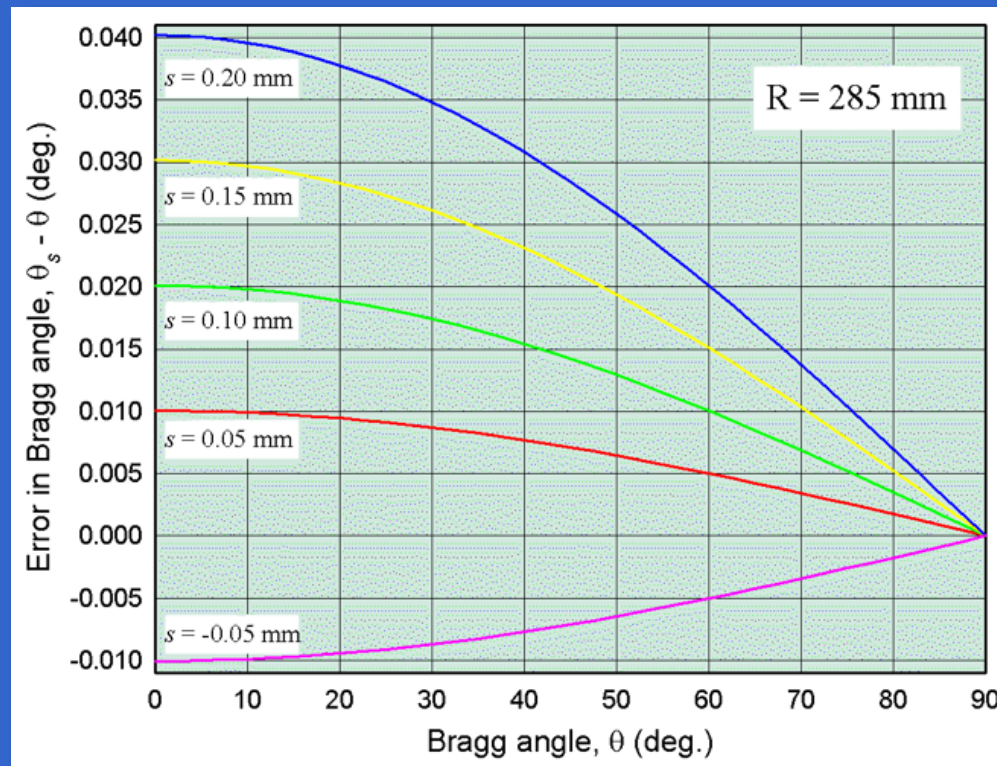
Reflection from off-axis specimen at wrong 2θ angle

Quality of powder diffraction data

Is the specimen properly prepared?

Requirements

sample surface must be on diffractometer rotation axis

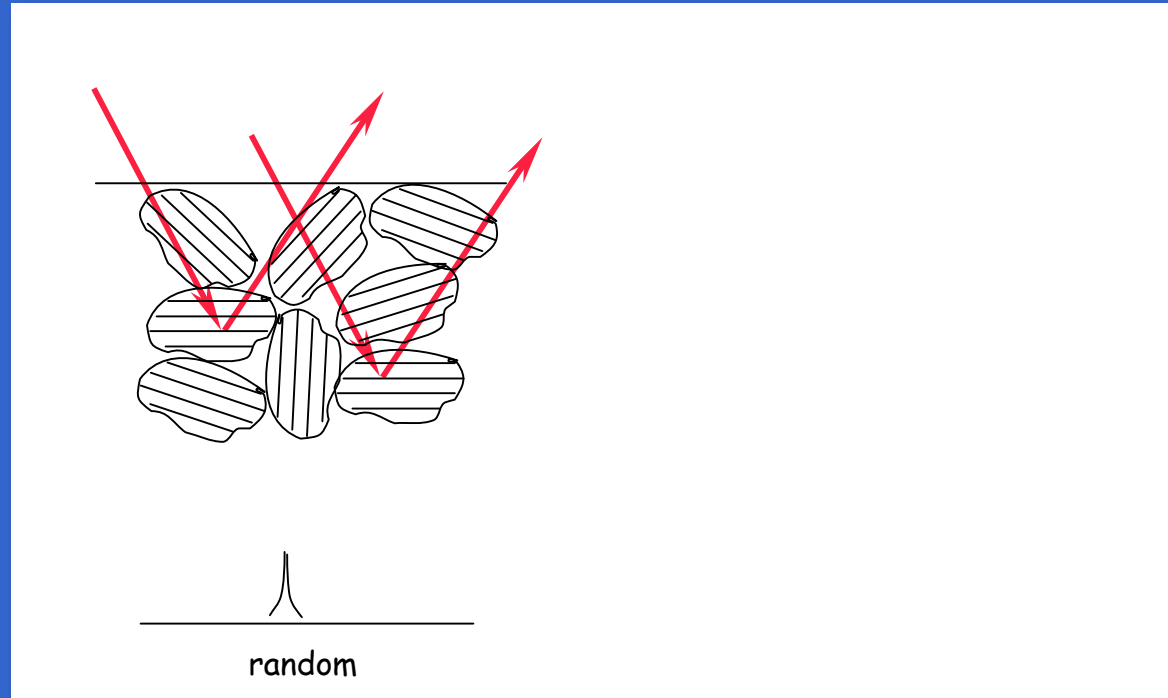


Quality of powder diffraction data

Is the specimen properly prepared?

Requirements

random particle orientation - no "preferred orientation"

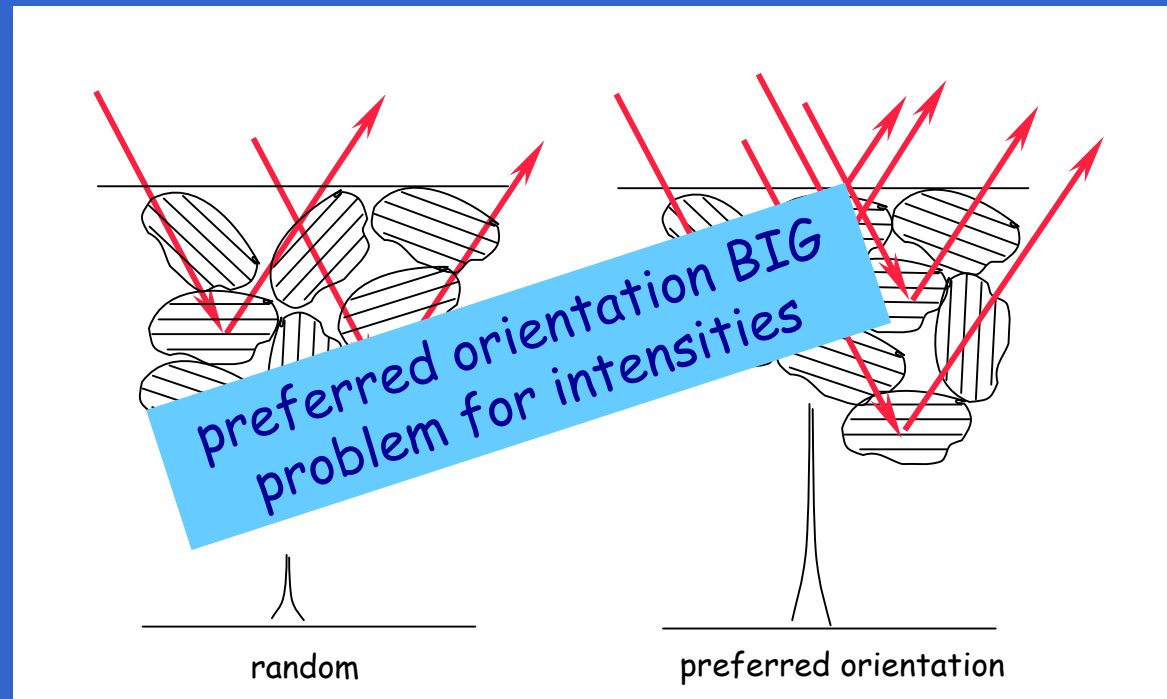


Quality of powder diffraction data

Is the specimen properly prepared?

Requirements

random particle orientation - no "preferred orientation"



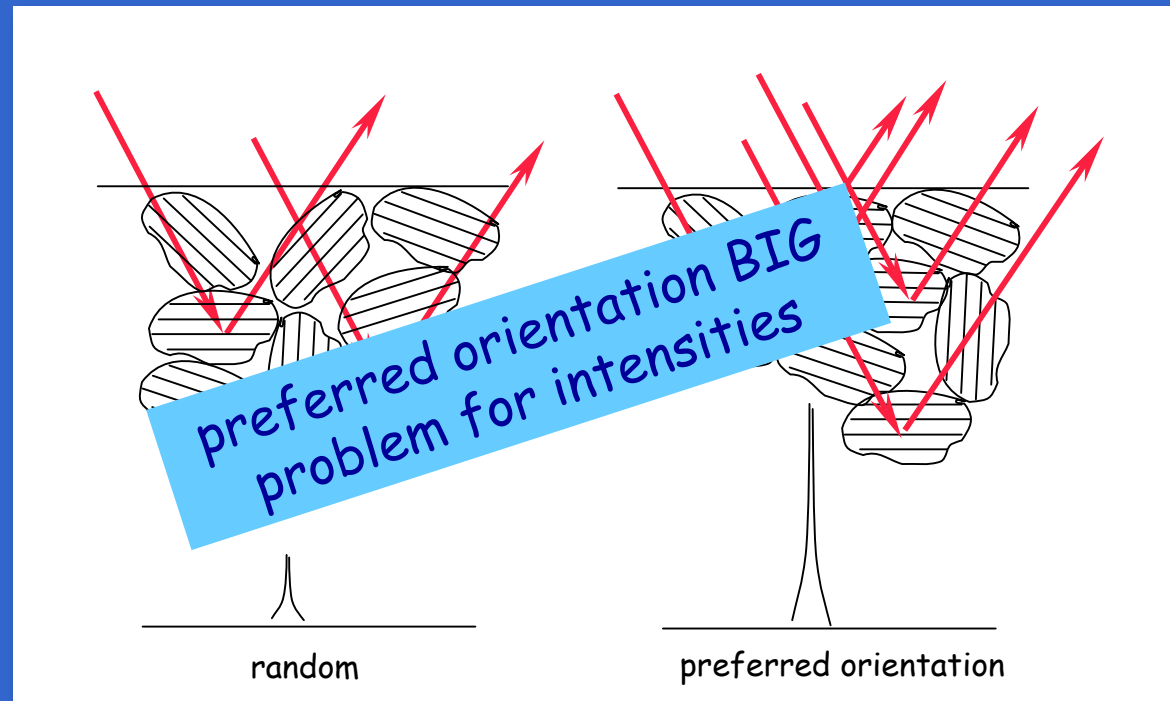
Note: only particles with planes parallel to specimen surface will reflect

Quality of powder diffraction data

Is the specimen properly prepared?

Requirements

random particle orientation - no "preferred orientation"



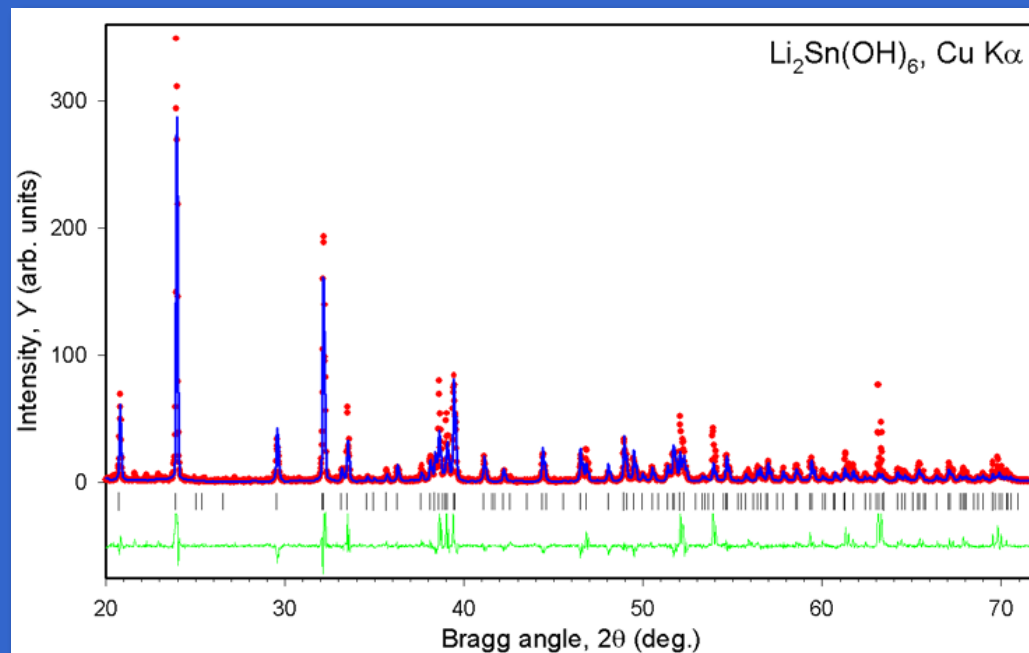
Reflection intensities can be significantly changed

Quality of powder diffraction data

Is the specimen properly prepared?

Requirements

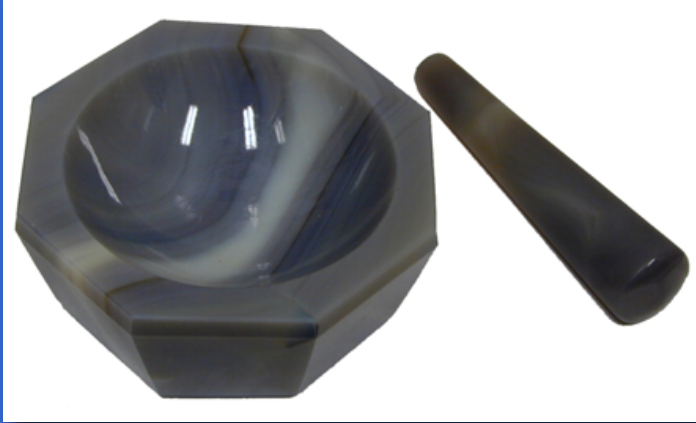
>10⁶ randomly oriented particles to avoid preferred orientation & to get correct intensities



Measured intensities for coarse powder not correct

Quality of powder diffraction data

Is the specimen properly prepared?



1-10 microns particle
size preferred

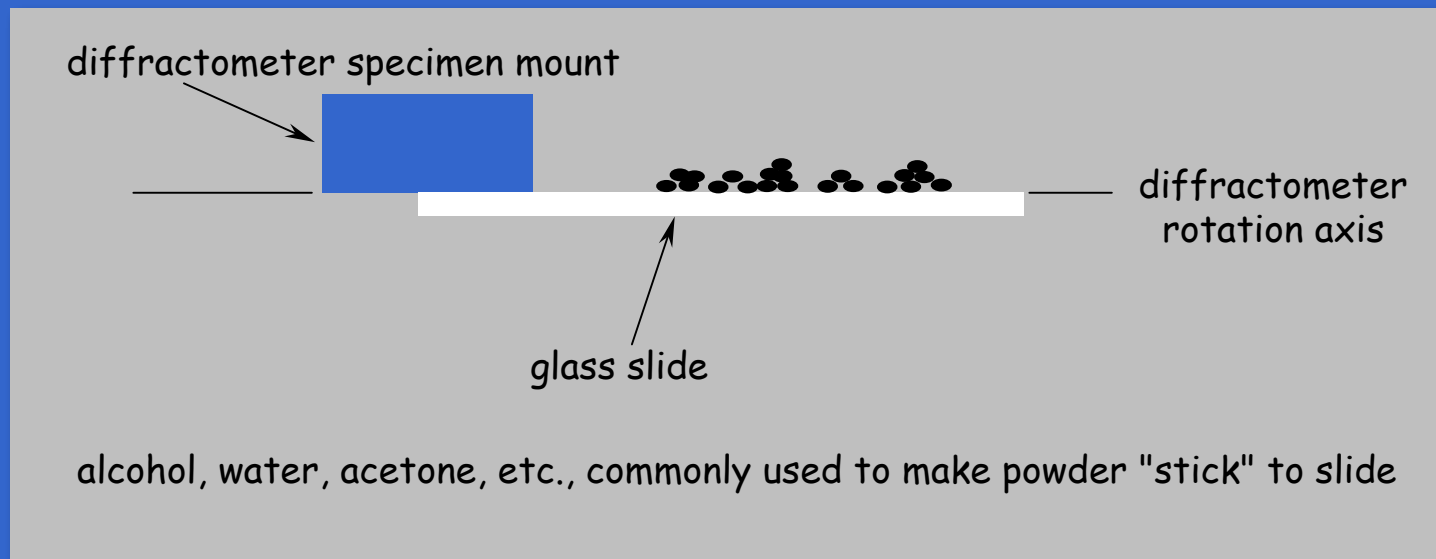
Common techniques - powders

Slide prep
Cavity front fill
Cavity back fill
Cavity side drift

Quality of powder diffraction data

Common prep techniques - powders

Slide prep



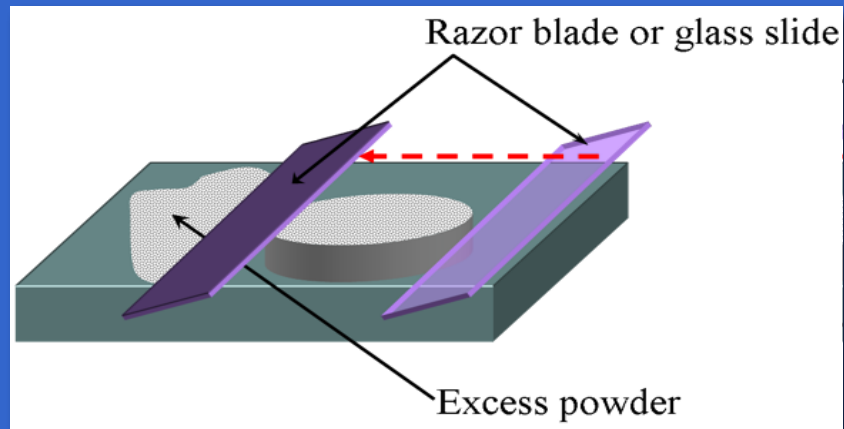
Problems -

- specimen surface not on rotation axis
- number of particles too small
- strong tendency for preferred orientation
- scattering from glass

Quality of powder diffraction data

Common prep techniques - powders

Cavity front fill



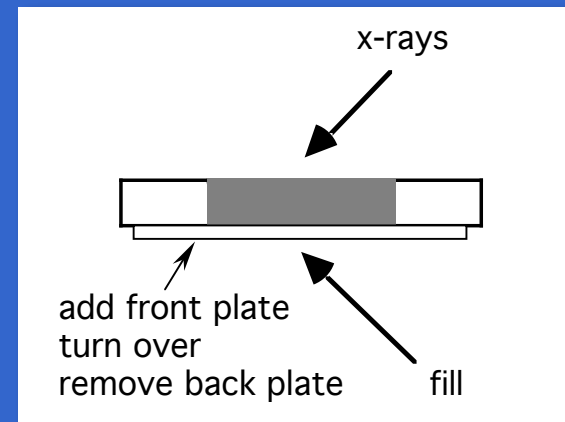
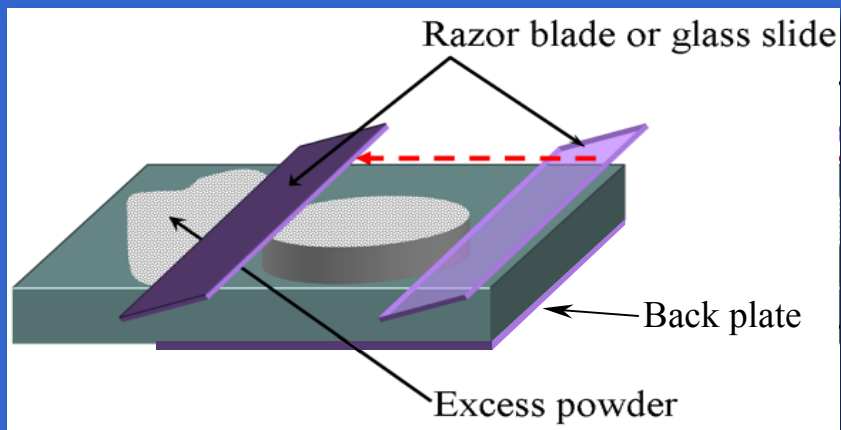
Problems -

- tendency for preferred orientation
- scattering from specimen holder at low 2θ angles

Quality of powder diffraction data

Common prep techniques - powders

Cavity back fill



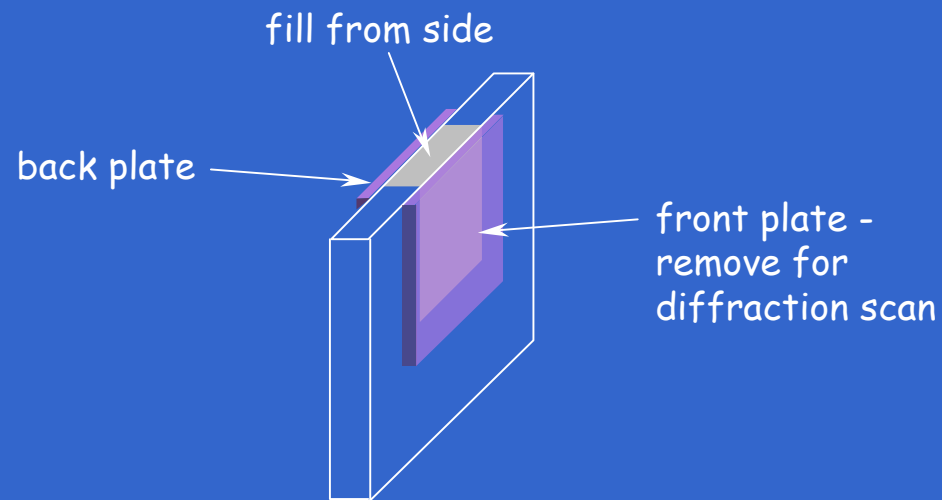
Problems -

slight tendency for preferred orientation
scattering from specimen holder at low 2θ angles

Quality of powder diffraction data

Common prep techniques - powders

Side drift



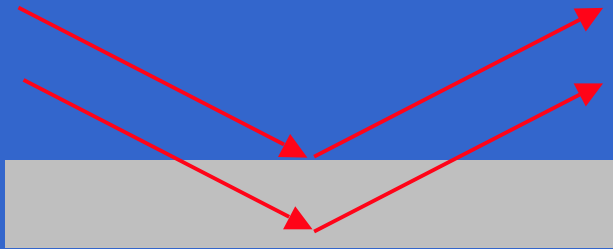
Problem -
tedious sample prep

Quality of powder diffraction data

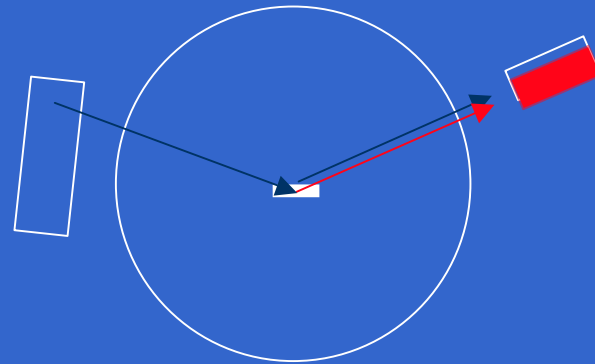
Is the specimen properly prepared?

Note on specimens with low mass absorption coefficients

sample surface must be on diffractometer rotation axis



Because of low absorption, reflection comes from deep within specimen



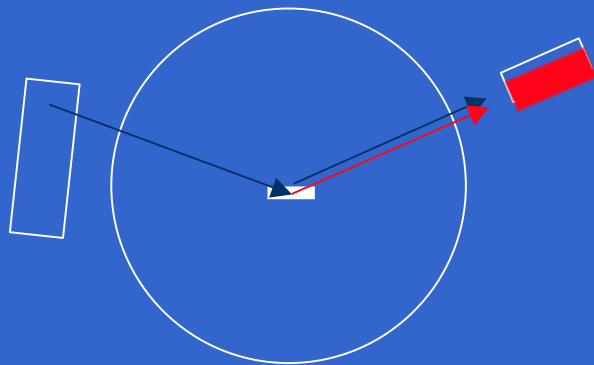
Solution: make specimen very thin

Quality of powder diffraction data

Is the specimen properly prepared?

Note on specimens with low mass absorption coefficients

sample surface must be on diffractometer rotation axis



Solution: make specimen
very thin



Problems:

number of particles
small - low intensities
& non-random

scattering from
specimen holder

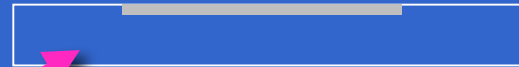
Quality of powder diffraction data

Is the specimen properly prepared?

Note on specimens with low mass absorption coefficients

Problem:

scattering from
specimen holder



Use single crystal with surface ground 'off-reflection' -
'zero background plate' - quartz or silicon

Get from The Gem Dugout, State College PA (USA)

<http://www.thegemdugout.com/>

Quality of powder diffraction data

For extensive survey of specimen prep techniques, see

Preparation of Specimens of Specimens for X-ray Fluorescence and X-ray Diffraction Analysis, V. E. Buhrke, R. Jenkins, and D. K. Smith, eds., Wiley-VCH (February 1998)

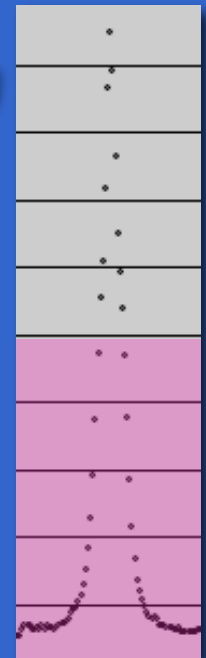
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Need to choose:

X-radiation ← probably $\text{CuK}\alpha$ - changing tube
 scan range ← varies with reasons for
 slit sizes ← measurement, time-consuming
 step size ← complicated, but important
 step dwell time ← scan done by repeatedly
 stepping for an angle, then
 counting at that angle

need $10(N+2) = \sqrt{N}$ counted points
 across top of peak



Quality of powder diffraction data

Addendum - quote from Jenkins & Snyder, *Introduction to X-ray Powder Diffractometry*

"While the automated system (X-ray diffractometer) clearly offers many advantages (over older, manual systems), it is also clear that the automation tends to divorce the user from the actual processes involved in collection and analysis of data. Unless the user is prepared to make a conscious and often time-consuming effort to understand the procedures being employed, he or she can be easily misled by such misconceptions."