

Texture Analysis with Area Detectors: The Basics

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Outline

- **ACKNOWLEDGEMENTS and REFERENCES**
- **DEFINITIONS**
- **BASICS**
- **INSTRUMENTATION**
- **PRACTICAL ASPECTS**
- **THINGS TO BE AWARE OF**
- **TEXTURE ANALYSIS**
- **EXAMPLES**



Acknowledgements and References

Acknowledgements:

Mark Rodriguez: Sandia National Laboratories, Albuquerque, New Mexico 87185

Colleen Frazer: Phillips Lumileds Lighting Co., San Jose, California 85131

References:

Excellent overviews on texture and ODF.

- **Crystallographic Texture in Ceramics and Metals, Mark D. Vaudin, J. Res. Natl. Inst. Stand. Technol., V. 106, pp 1063-1069 (2001)**
- **[http:// www.ccp14.ac.uk/ccp/ccp14/ftp-mirror/nist-texture/Texture%20ReadMe.pdf](http://www.ccp14.ac.uk/ccp/ccp14/ftp-mirror/nist-texture/Texture%20ReadMe.pdf)**
- **<http://aluminium.matter.org.uk/content/html/eng/default.asp?catid=100&pageid=1039432491>**
- **<http://www.ccp14.ac.uk/ccp/ccp14/ftp-mirror/nist-texture/KRIS%20KOZACZEK.PDF>**
- **<http://neon.mems.cmu.edu/rollett/27750.old.Spg05/Intro.Xray.PFs.ppt>**
- **Intensity Correction in Texture Measurements of Polycrystalline Thin Films By X-ray Diffraction, Y.S. Liu et al, Tex and Microstructures, V35, No.3/4, Sept/Dec 2003, pp 283-290**
- **Absorption Corrections for X-ray Texture Measurements of Any Shape Sample, T. Montesi et al, Tex and Microstructures, Vols. 14-18, 1991, pp 567-572.**



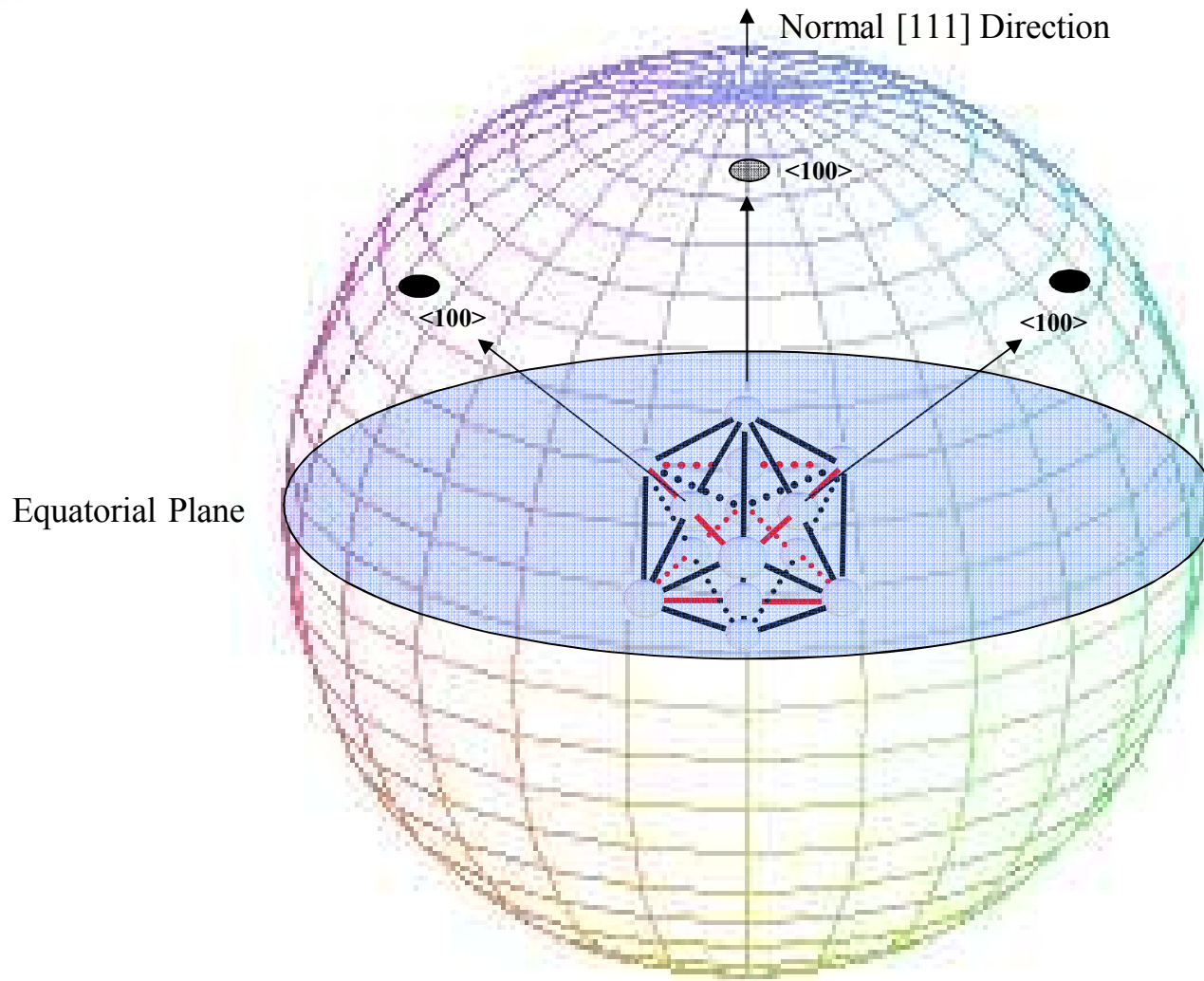
Definitions

Texture (Preferred Orientation): The alignment of certain crystallographic planes; either induced or naturally occurring, in a particular direction from a reference plane.

Pole Figures: A two-dimensional representation that shows the variation of pole density with pole orientation for selected set of crystallographic planes with a specified orientation relative to the sample.

<http://aluminium.matter.org.uk/content/html/eng/0210-0010-swf.htm>

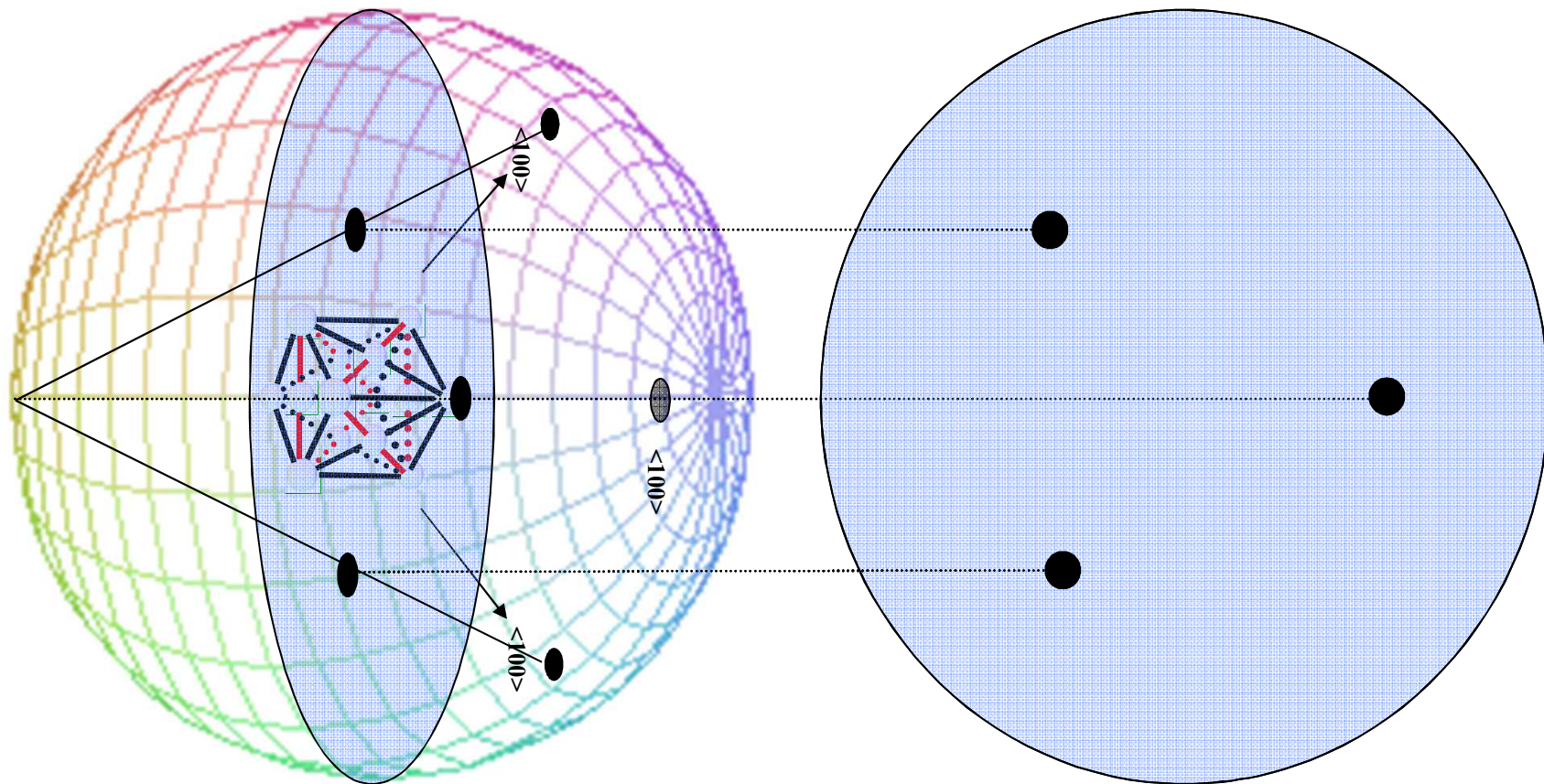
Biaxial $\langle 100 \rangle$ Texture



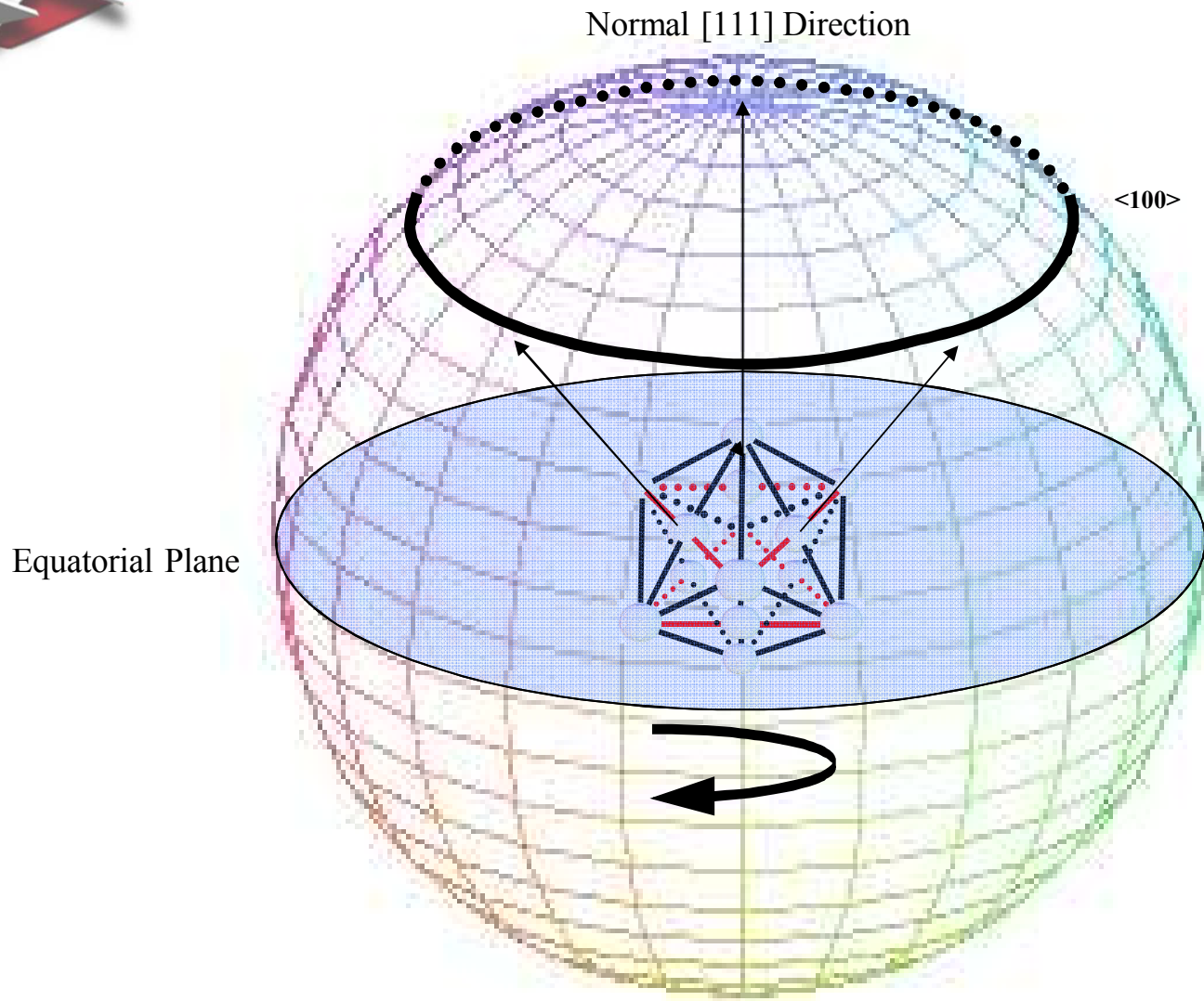
A preferred in-plane and out-of-plane grain orientation.

Biaxial $\langle 100 \rangle$ pole figure

stereographic projection

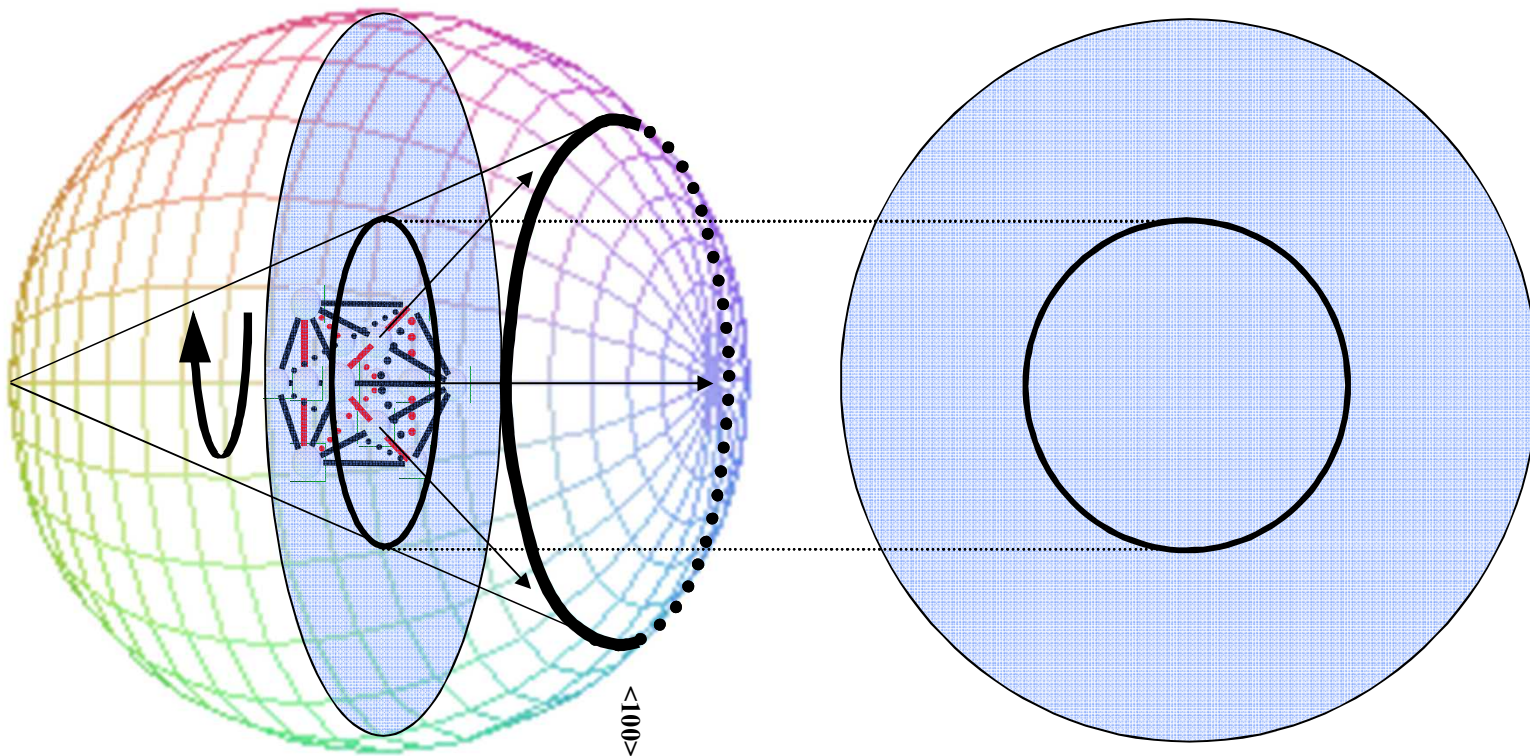


Fiber $\langle 100 \rangle$ Texture



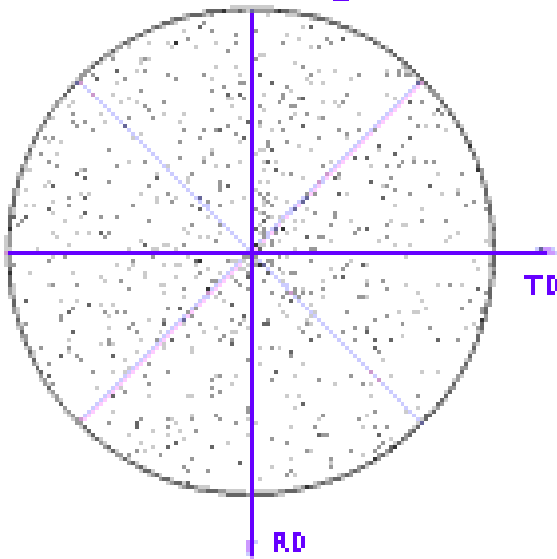
Circular symmetry about some sample or crystal axis

Fiber $\langle 100 \rangle$ pole figure (ideal) stereographic projection



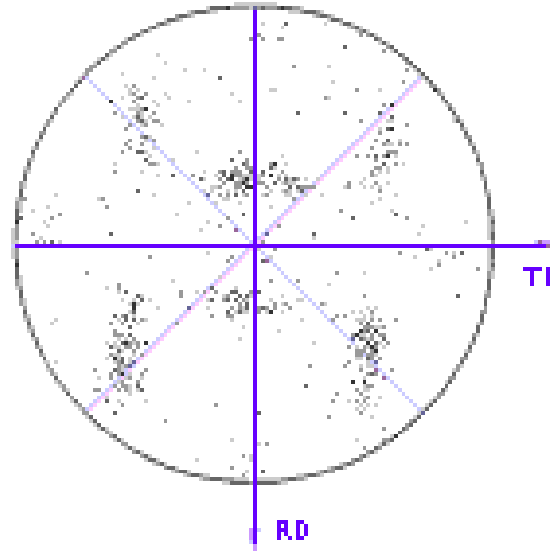
For **polycrystalline** materials, a set of poles can be plotted for each individual grain to produce a pole figure.

$\langle 100 \rangle$ Pole Figure



Pole figure showing **random** texture

$\langle 100 \rangle$ Pole Figure



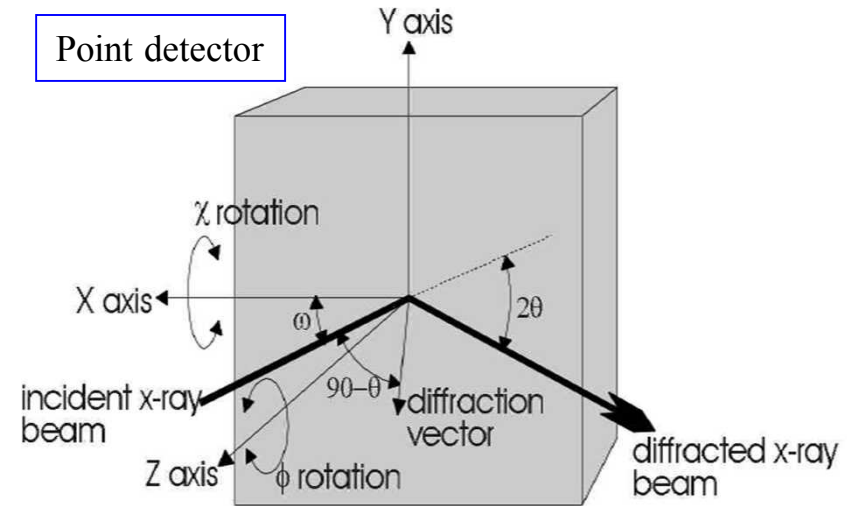
Pole figure showing **preferred** texture

http://aluminium.matter.org.uk/content/media/flash/metallurgy/anisotropy/pole_figure_dynamic.swf?targetFrame=100pole

Texture Measurements by Diffraction Methods

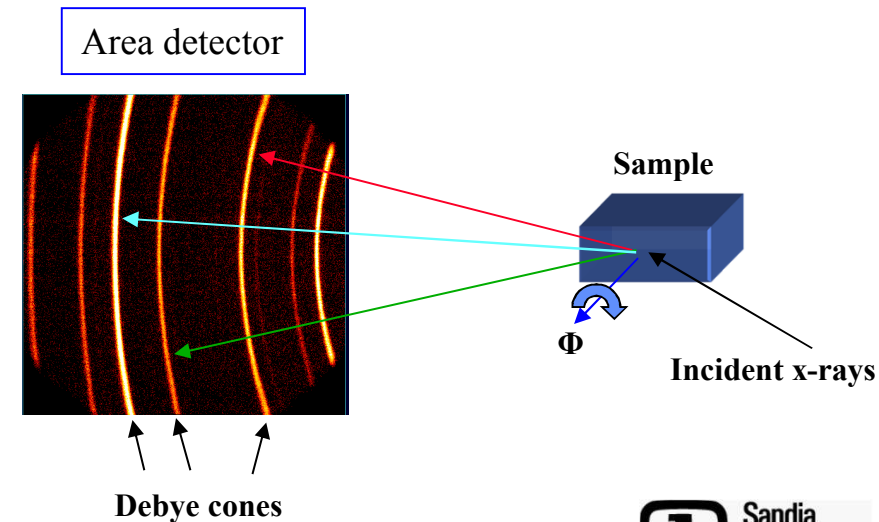
Fixed diffraction vector method

Need at least two independent rotations (e.g. Φ and χ) but usually texture goniometers allow three sample rotations (Φ , ω and χ).

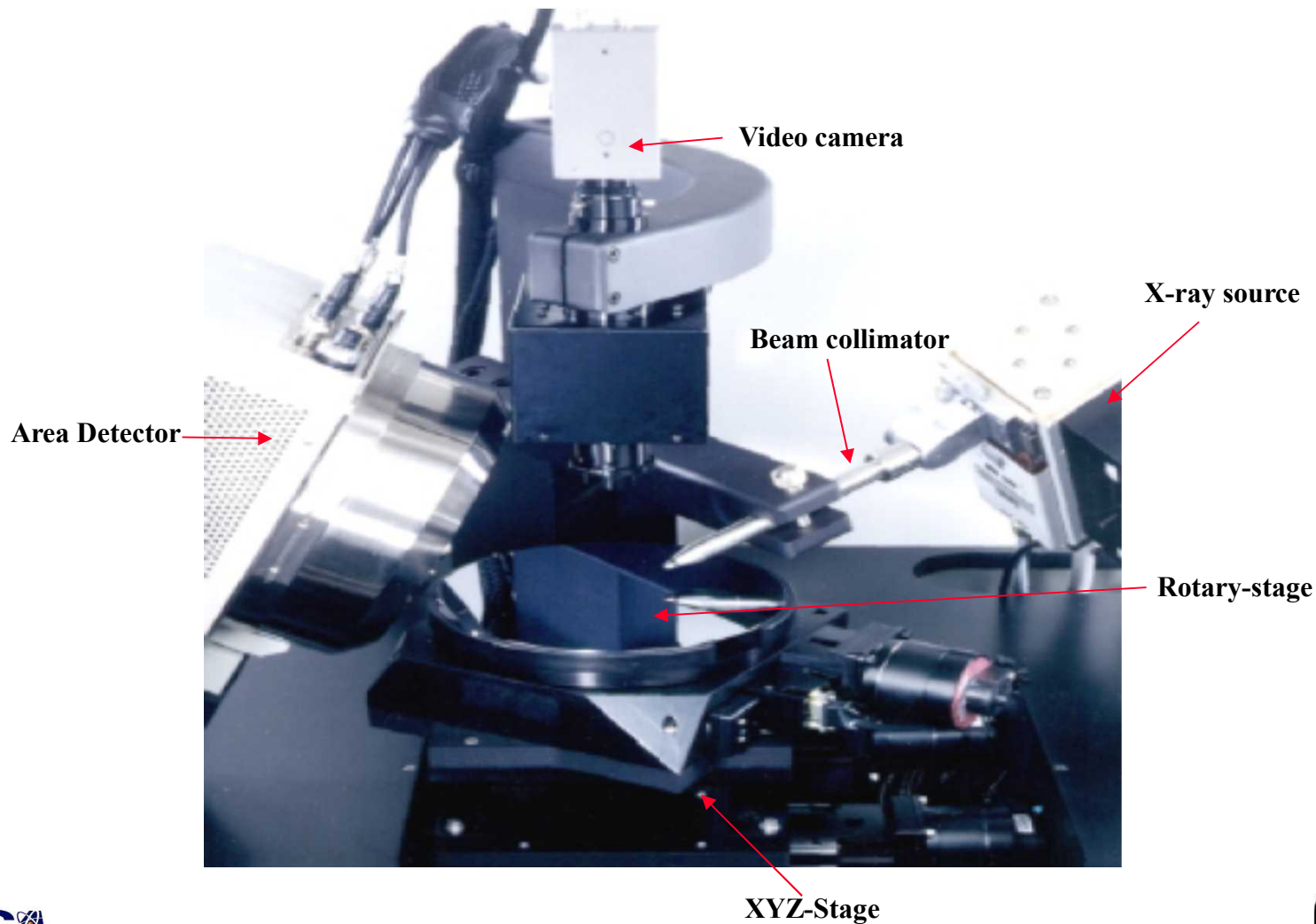


Variable diffraction vector method

Diffraction vector varies along Debye cone and for different Debye cones. Only one sample rotation angle is needed (Φ). **Basic advantage is speed and more diffraction information.**



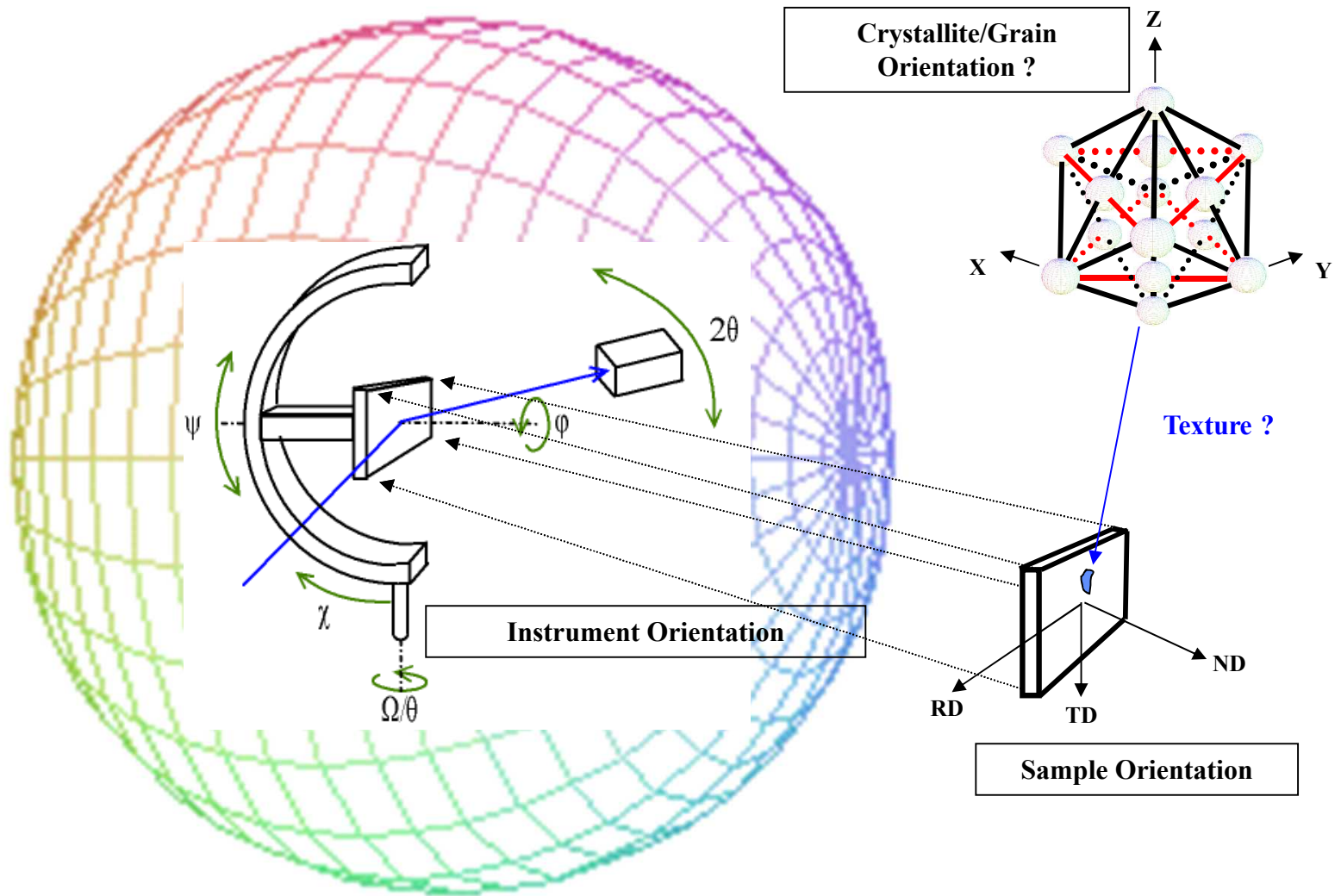
Texture (XYZ –Stage) Mapping Diffractometer



Texture (1/4 Cradle XYZ stage) Diffractometer



Orientation relationships used in texture analysis





Practical Aspects

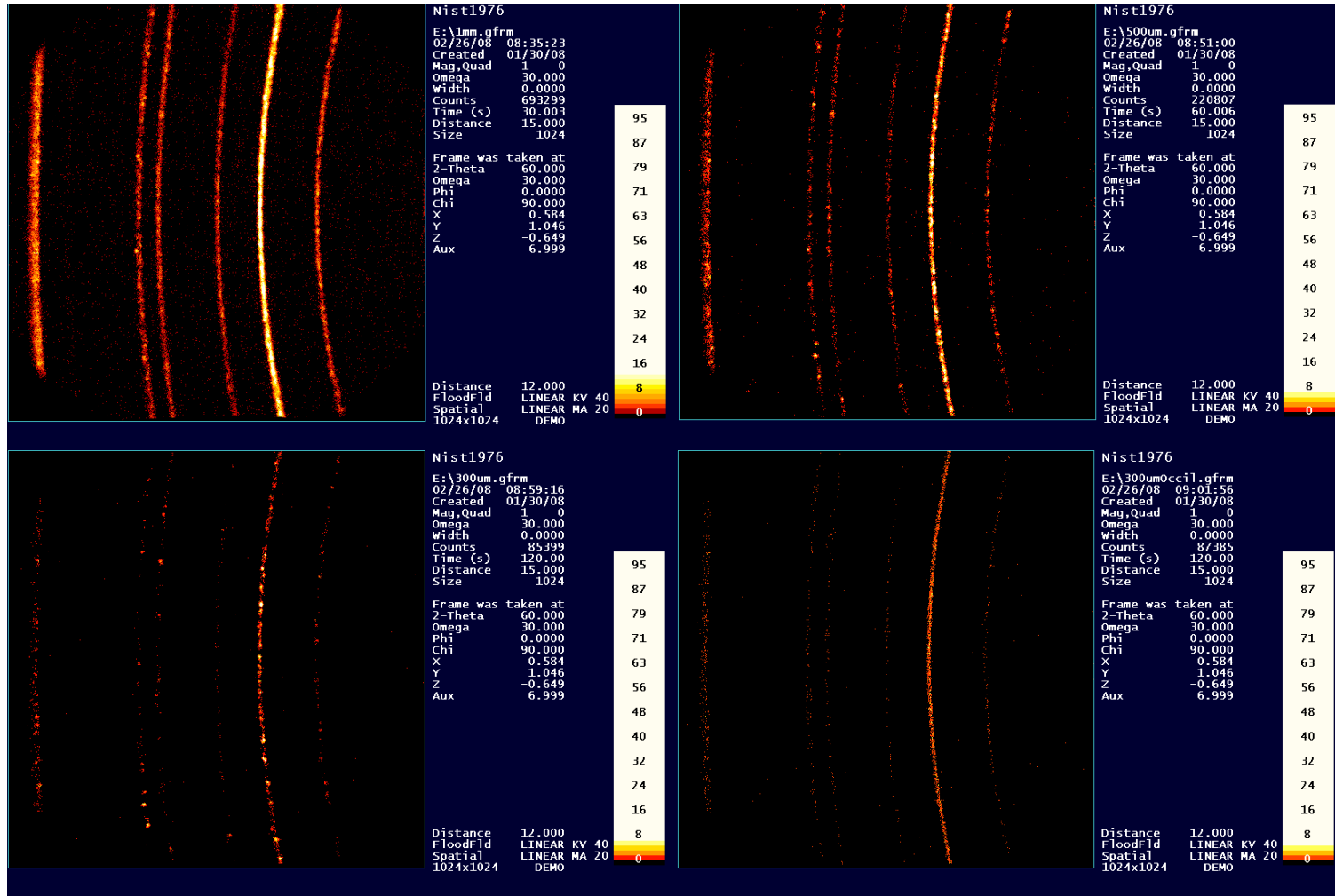
- Typical to measure three PFs for the 3 lowest values of Miller indices.
- Why?
 - A single PF does not uniquely determine orientation(s), texture components because only the plane normal is measured, but not directions in the plane (2 out of 3 parameters). **Area detector however can measure both.**
 - Multiple PFs required for calculation of Orientation Distribution
- X-ray foot print should be the same if not smaller than your analysis area.
 - Beware of sample shadowing at higher χ angles
- Make sure you know your sample orientation, if not make a mark.
- Handy tools
 - PDF for looking up hkl 2theta angles.
 - Have a means; i.e. program or tables, for determining interfacial angles.



Thing to be aware of:

X-ray spot size and oscillation effects

NIST 1976 Standard

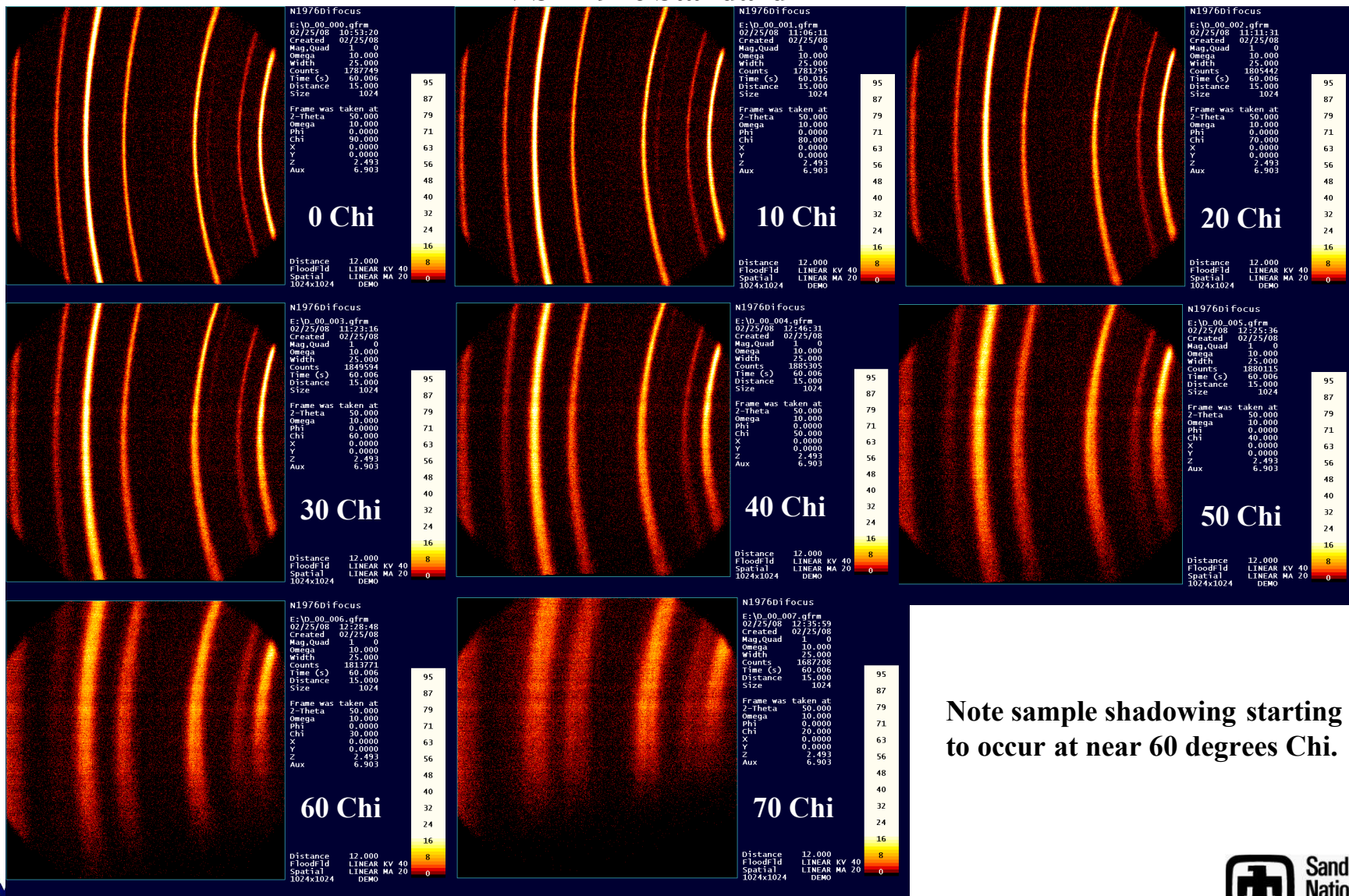




Thing to be aware of:

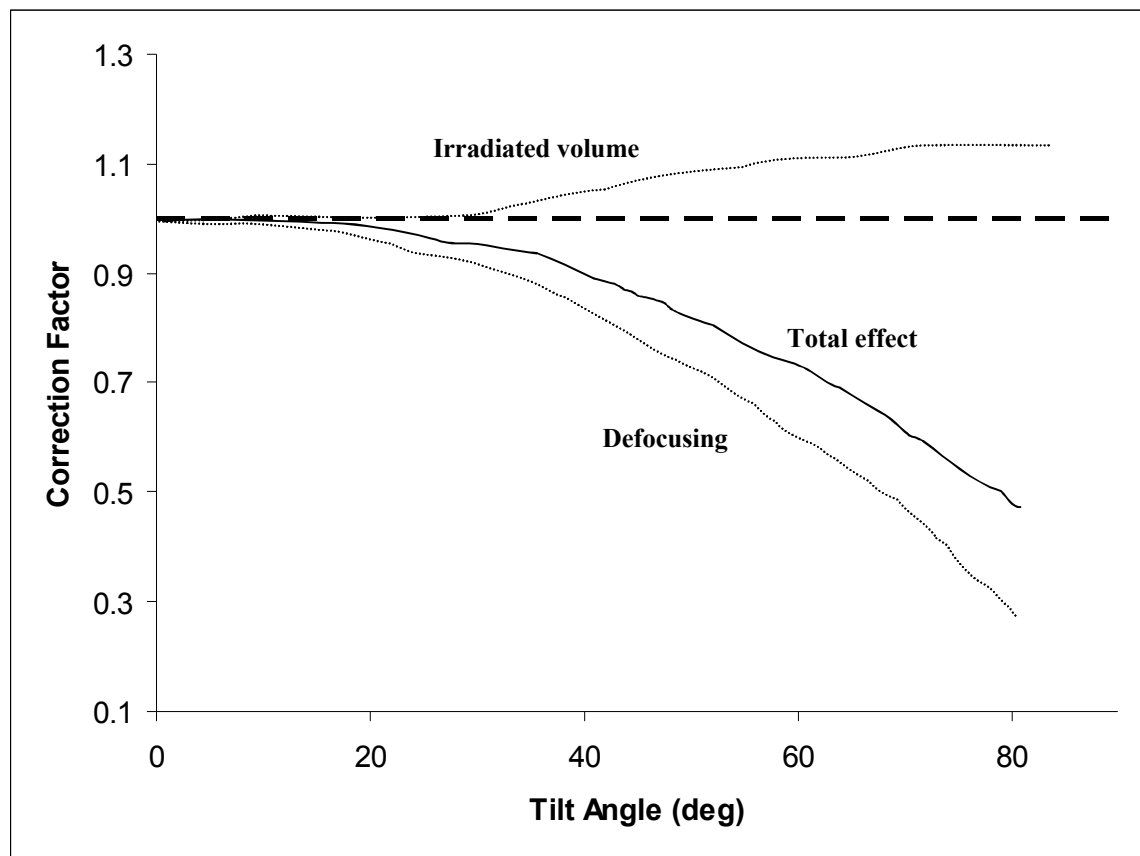
Chi Defocusing (1mm pinhole) effects

NIST 1976 Standard



Effect of irradiated volume and defocusing on intensity

(110)NiTi – diffraction 2Theta=42.45 deg Cu-K α radiation



Thing to be aware of:

Defocussing

- The combination of the $\theta-2\theta$ setting and the tilt of the specimen face out of the focusing plane spreads out the beam on the specimen surface.
- Above a certain spread, not all the diffracted beam enters the detector.
- Therefore, at large tilt angles, the intensity decreases *for purely geometrical reasons*.
- **This loss of intensity must be compensated for, using the *defocussing correction*.**

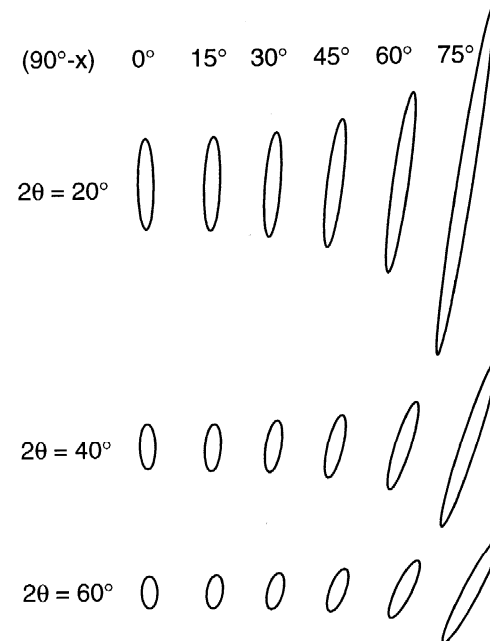


Fig. 13. Change in shape and orientation of the irradiated spot on the sample surface for different sample inclinations as a function of tilt angle α and Bragg angle 2θ . The incident beam is cylindrical with 2 mm diameter.

Thing to be aware of:

Defocusing Correction

- Defocusing correction more important with decreasing 2θ and narrower receiving slit.

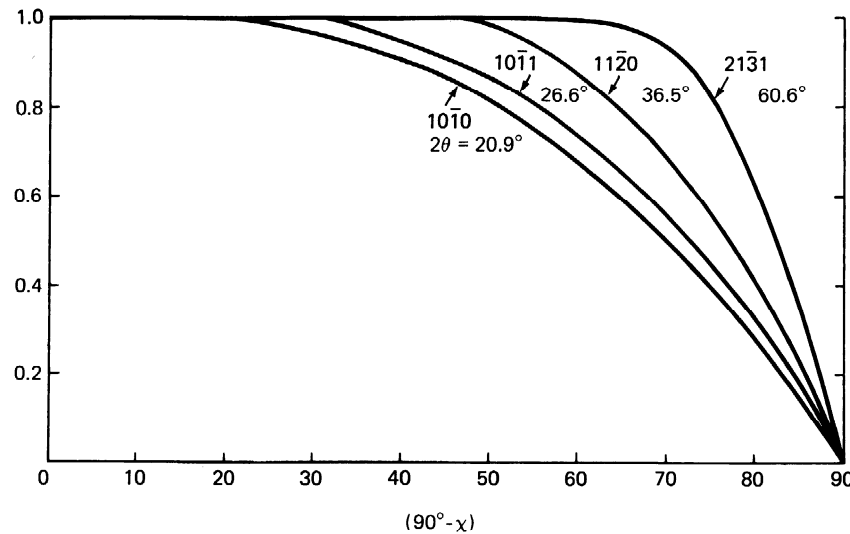
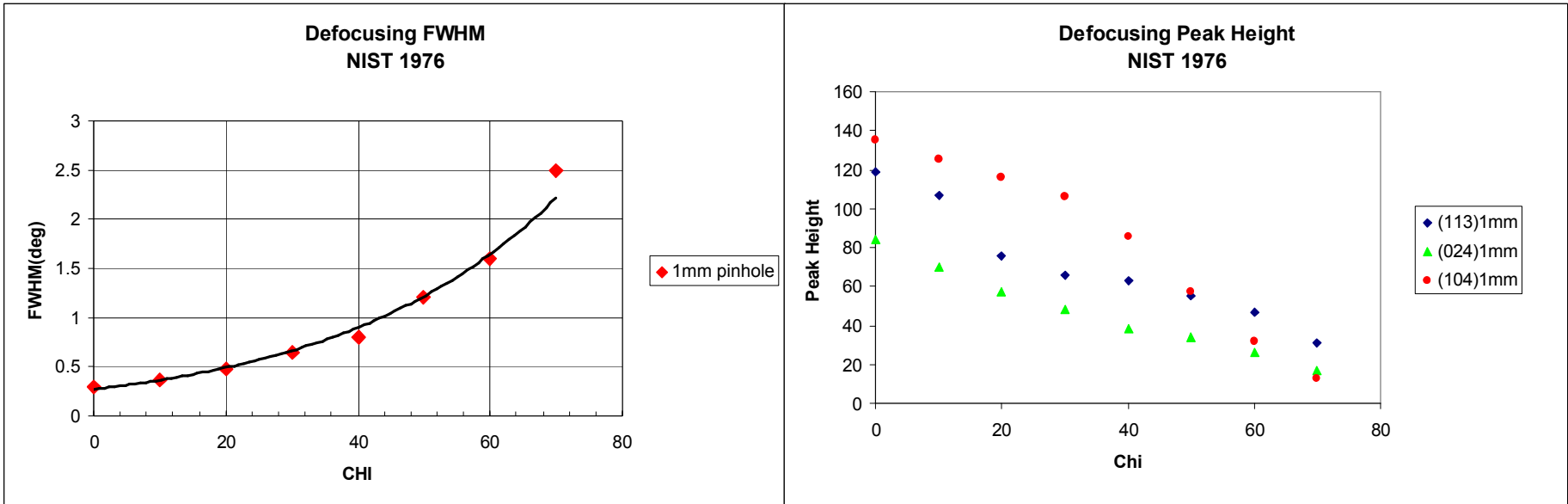


Fig. 24 Intensity correction for x-ray pole-figure determinations in reflection geometry. Selected reflections of quartz. Cu $K\alpha$ radiation (from Baker *et al.*, 1969).

Thing to be aware of:

Defocusing affects FWHM and Peak Height

NIST 1976 Standard (Corundum Plate)



Why do the observed peak heights vary for different hkl's?



Orientation Distribution Function

The full 3-dimensional representation of crystallographic texture is given by the orientation distribution function (ODF) which can be achieved through evaluation of a set of pole figures or diffraction spectra. Subsequently, all pole figures can be derived from the ODF.

The ODF is defined as the volume fraction of grains oriented along a certain direction g .

$$\text{ODF}(g) = 1/V \, dV(g)/dg$$

the direction g is normally identified using three Euler angles. The orientation distribution function, ODF, cannot be measured directly by any technique. Traditionally both X-ray diffraction and EBSD may collect pole figures. Different methodologies exist to obtain the ODF from the pole figures or data in general. They can be classified at first based on how they represent the ODF. Some use to represent the ODF as a function, sum of functions or expand it in series of harmonic functions. Others, known as discrete methods, divide the ODF space in cells and focus on determine the value of the ODF in each cell.

<http://aluminium.matter.org.uk/content/html/eng/default.asp?catid=100&pageid=1049375768>



Corrections to Measured Data

- Random texture [=uniform dispersion of orientations] means *same* intensity in all directions.
- Background count must be subtracted.
- X-ray beam becomes defocused at large tilt angles ($> \sim 60^\circ$); measured intensity from random sample *decreases* towards edge of PF.
- Defocusing correction required to *increase* the intensity towards the edge of the PF.



Intensity Distributions Are Not Pole Figures

- **Intensity to pole density conversions include:**
 - Absorption corrections (necessary for tilts >70 degrees)
 - Defocusing corrections
 - Background corrections
 - Variable diffracting volume (especially for thin films)
- **Corrections in pole figure space:**
 - Sample misalignment
 - Parallax errors (detector space)
 - Interpolation in pole figure space
 - Pole figure normalization
- **Goniometer misalignment errors are generally not correctable**
(e.g. peak shifts with tilt, etc.)
- **Individual pole figures and sets of pole figures must be self-consistent**

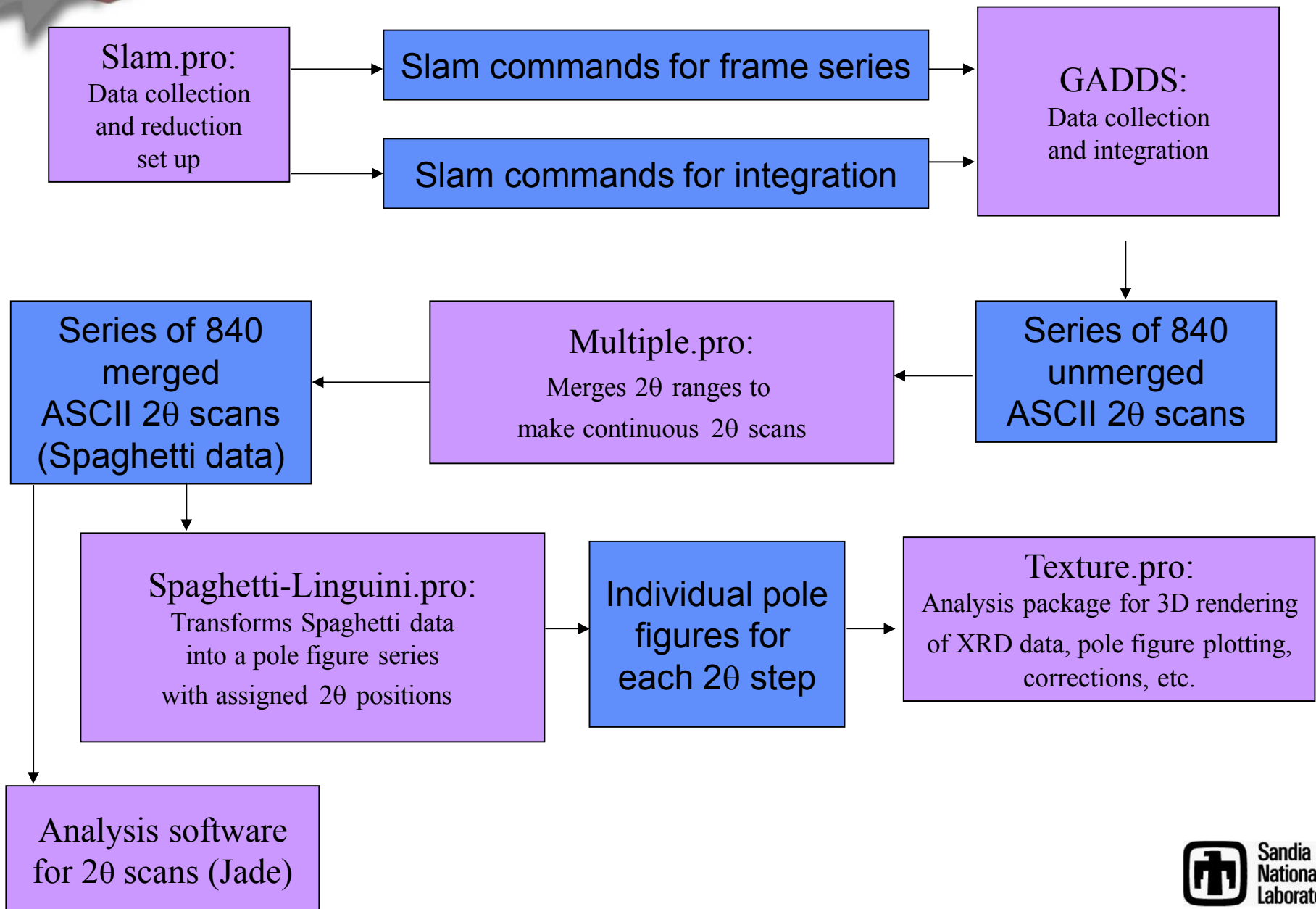


Normalization

- Normalization is the operation that ensures that “random” is equivalent to an intensity of one.
- This is achieved by integrating the un-normalized intensity, $f'(\theta, \psi)$, over the full area of the pole figure, and dividing each value by the result, taking account of the solid area. Thus, the normalized intensity, $f(\theta, \psi)$, must satisfy the following equation, where the 2π accounts for the area of a hemisphere:

$$\frac{1}{2\pi} \int f(\Theta, \psi) \sin \Theta d\Theta d\psi = 1$$

Area Detector Data Collection Scheme and Analysis Flow



Slam file generator to set up runs for data collection

Run Description:

Title/Sample Name: 08122

Working Directory: C:\mark\08122

If phrase, use double quotes.

Run Parameters:

Phi range: from 0 to 360 in 6 degree increments

Scan Width 17 degrees

Scan Time 10.0 seconds

Mode: Scan

Axis: 2. Omega

Omega is scanned in order to simulate a $\theta / 2\theta$ (symmetric) geometry

Run and Integration Angles

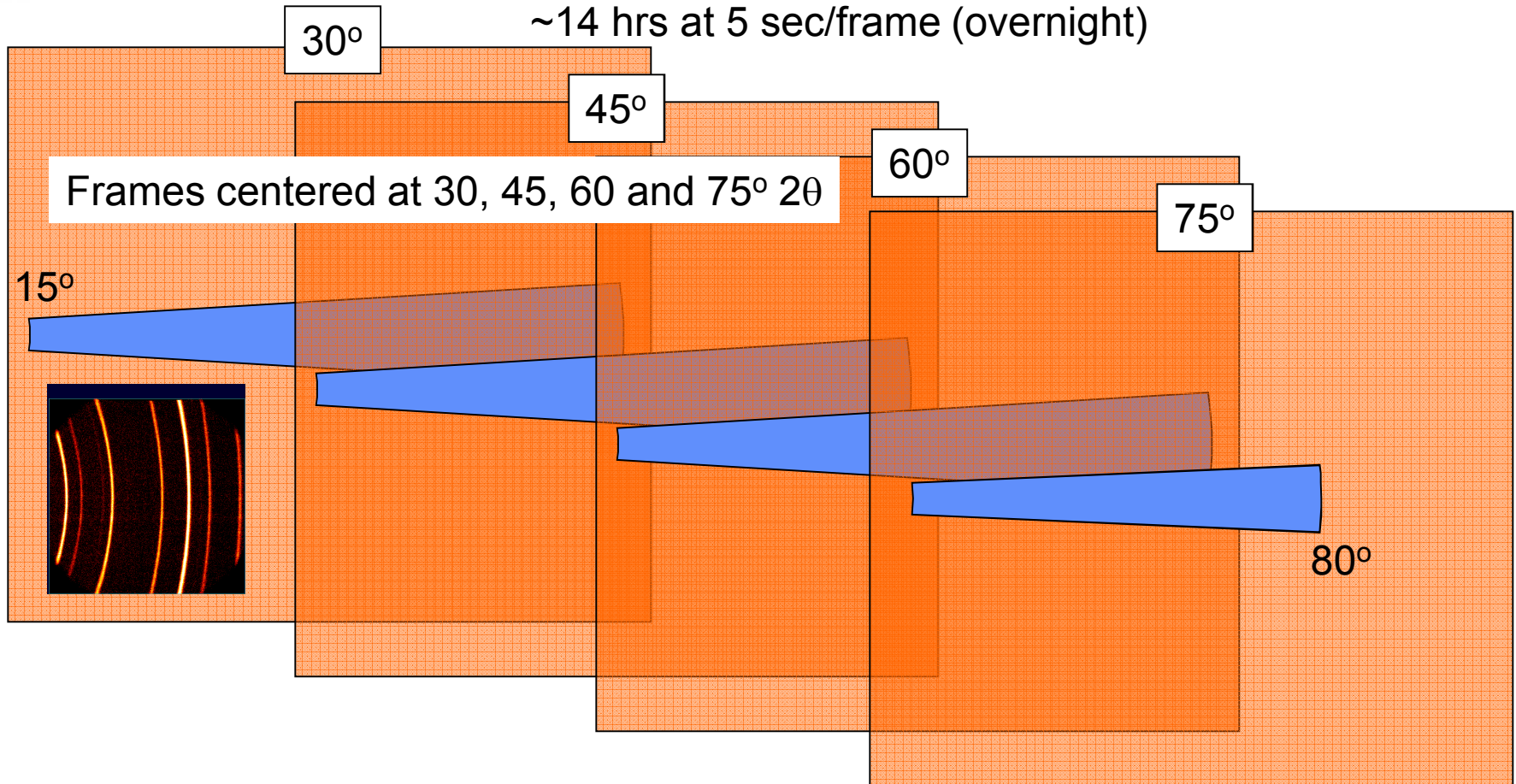
2theta	Omega	Integ Int Low	Integ Int High	Chi Low	Chi High
30.0000	6.00000	15.0000	45.0000	-93.0000	-87.0000
45.0000	13.5000	30.0000	60.0000	-93.0000	-87.0000
60.0000	21.0000	45.0000	75.0000	-93.0000	-87.0000
75.0000	28.5000	70.0000	80.0000	-93.0000	-87.0000

output_integration.slm

output_scan.slm
Type: SLM File
Date Modified: 6/13/2008 9:54 AM
Size: 792 KB

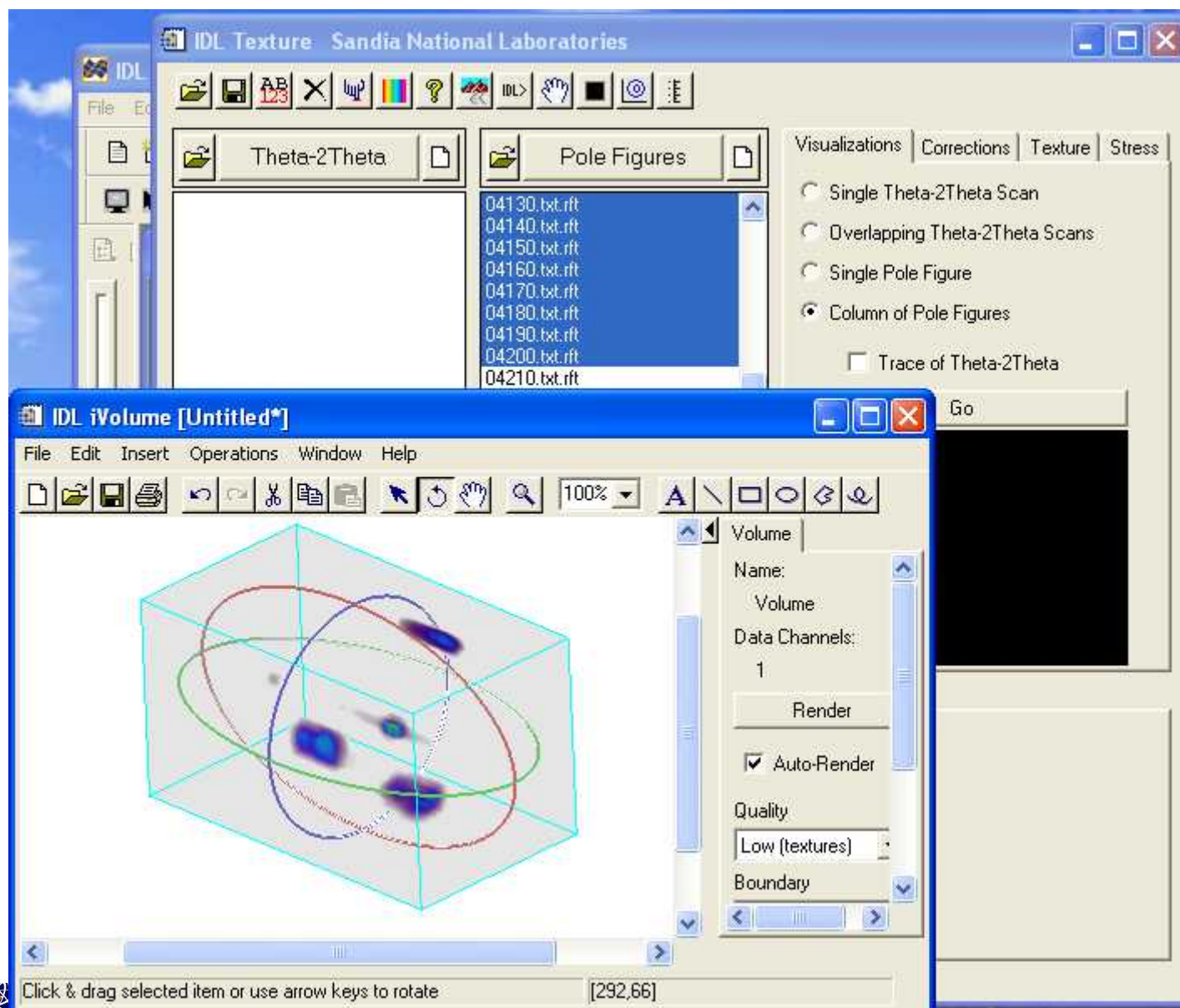
Analysis scheme

4 frame series, each with 14 chi tilts (0 -78 chi in 6° steps)
and 36 phi rotations (0-354 in 6° steps) = 3360 frames
~14 hrs at 5 sec/frame (overnight)

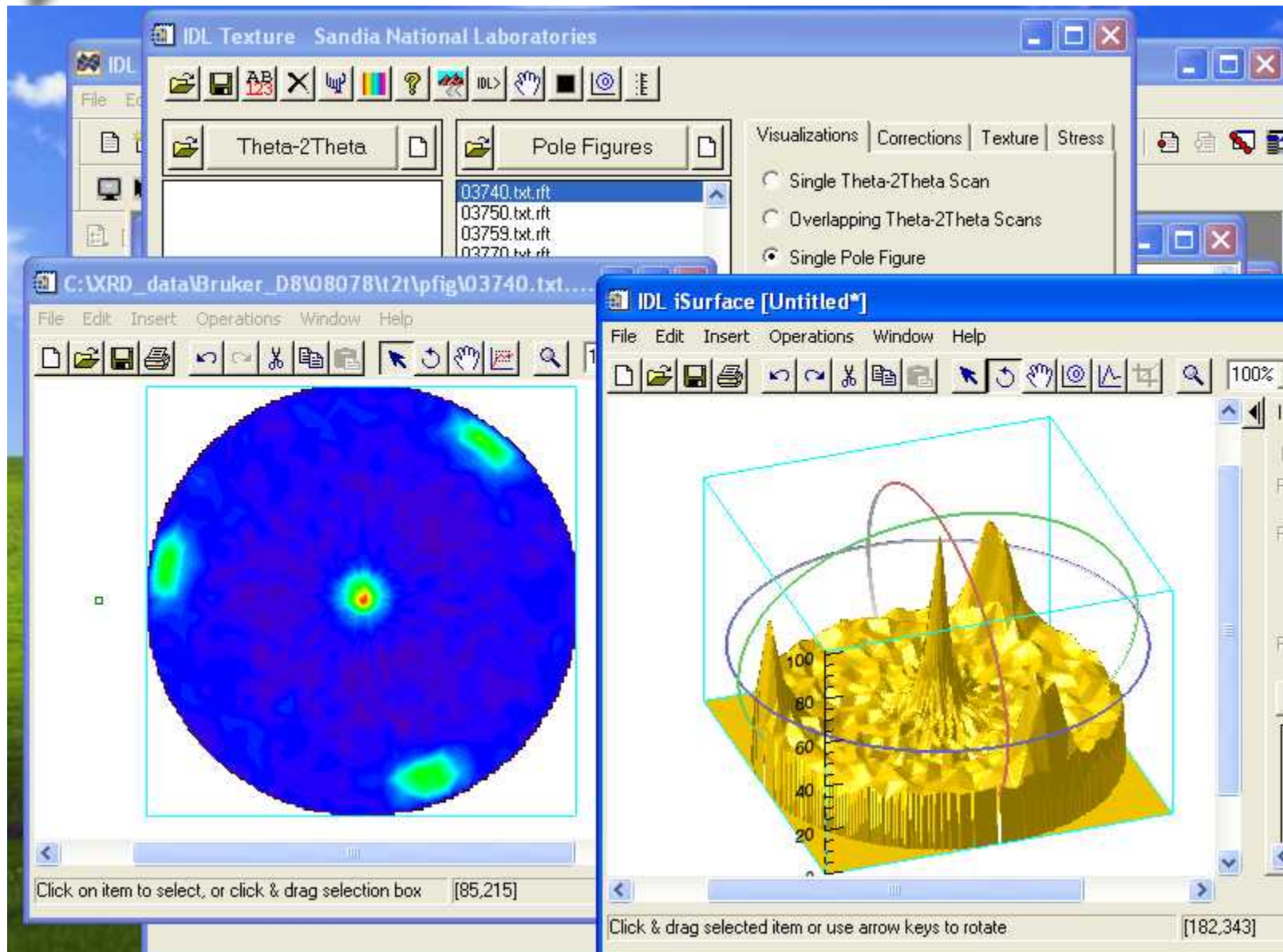


Ranges integrated $\pm 3^\circ$ in χ over 30° in 2θ to generate four 2θ ranges for each χ/ϕ setting. After merging of files this generates 780 2θ scans each with a designated χ/ϕ

Pole figure visualization software



Individual pole figures can be generated as 3D or contour plots





notes

780 scans each with 0.1° step size = 507000 intensity values distributed over $15 - 80^\circ 2\theta$, $0 - 78^\circ \chi$, $0 - 354^\circ \phi$

0.05 step size = 1,014,000 data points

Spaghetti-Linguini takes ~15 minutes to generate pole figure data files

3D rendering of pole figure series data must be done in small ranges of 2θ (e.g. 10° increments)

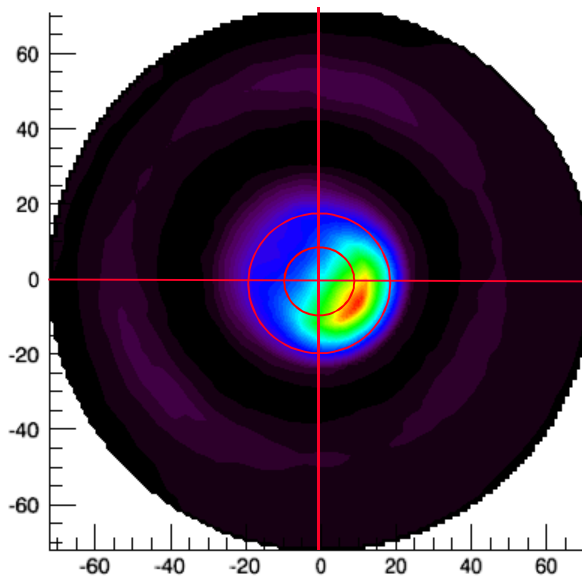
Same type of data collection can be done with point detector but may require longer scan time

Data useful for phase ID, texture, macrostrain, etc.

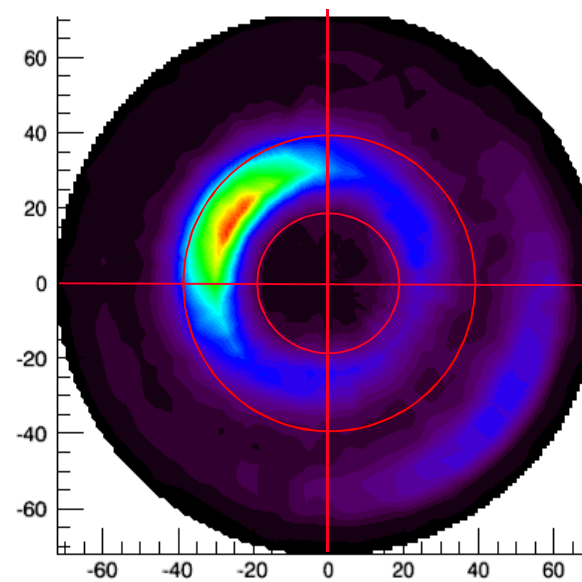
Sputtered Mo on Kovar pole figures

Determine the texture of the Mo film, should be (110) out-of-plane.
Oops?

Mo (110)



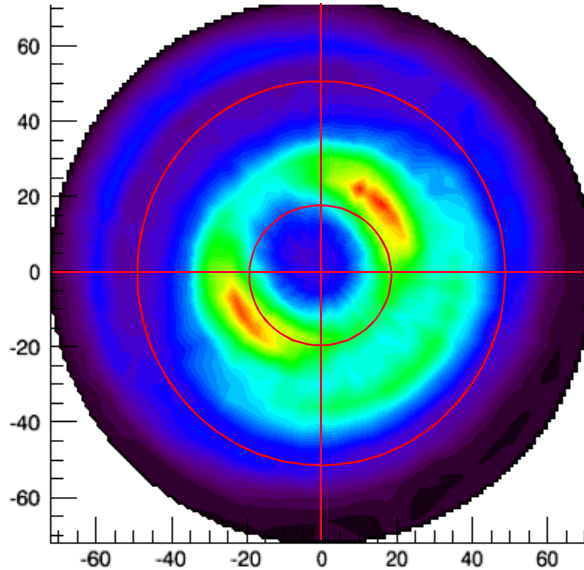
Mo (200)



Sputtered Mo on Kovar pole figures

Intensity at $\chi = 30, 54.7^\circ$ consistent with Mo (110) being out of plane.
Film appears to have some in plane texture.

Mo (211)

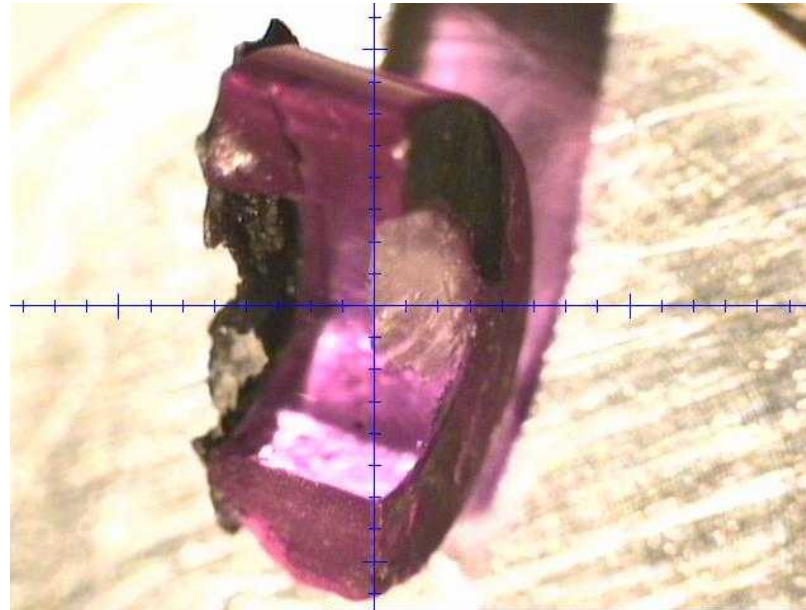


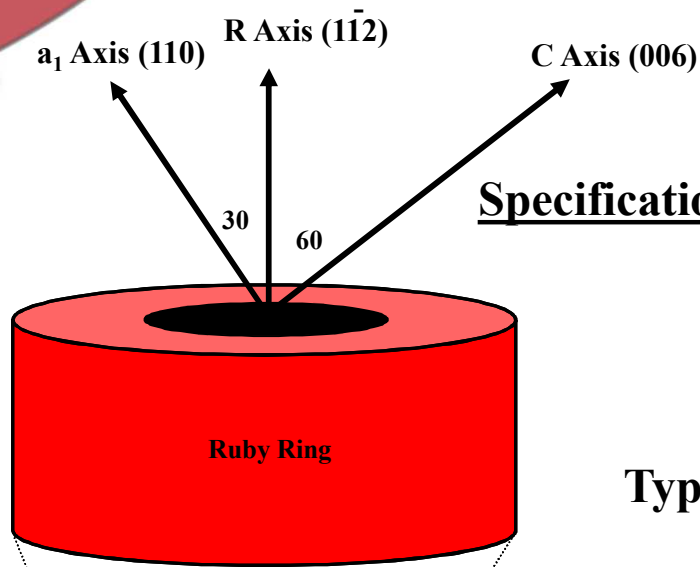
Possible explanations:

- Sample misalignment
- Uneven film thickness
- Sputtering Process

Problem: A synthetic single crystal Ruby ring fractured!

Is there a crystallographic difference between this ring and the users specification that may be a cause of the failure?

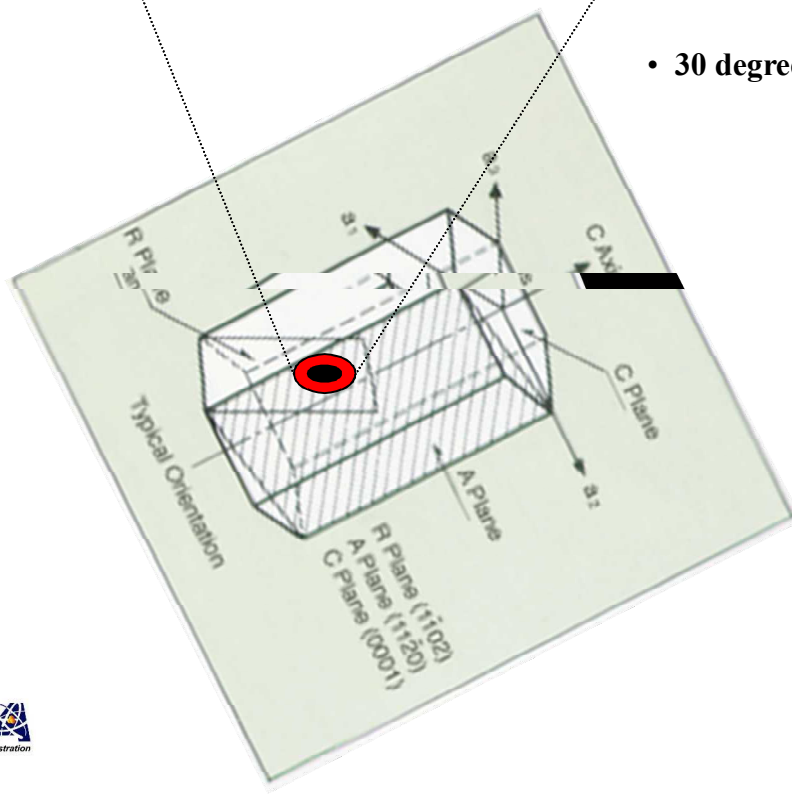




Specification calls for “R Cut” (random orientation)

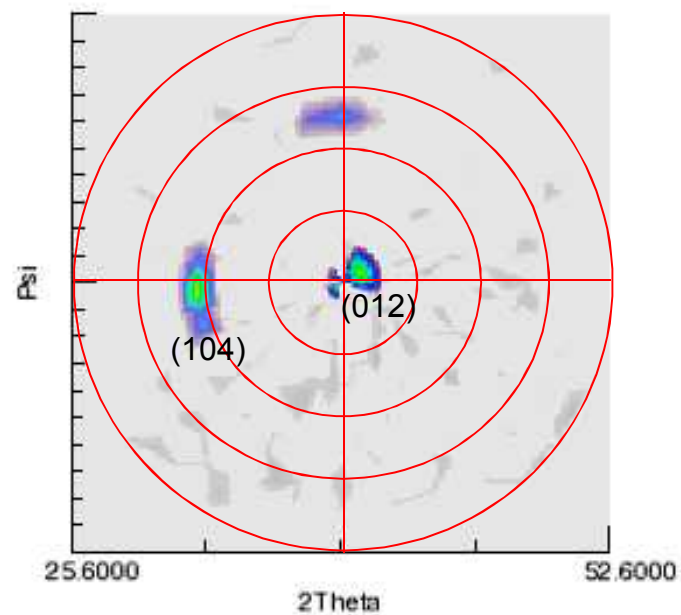
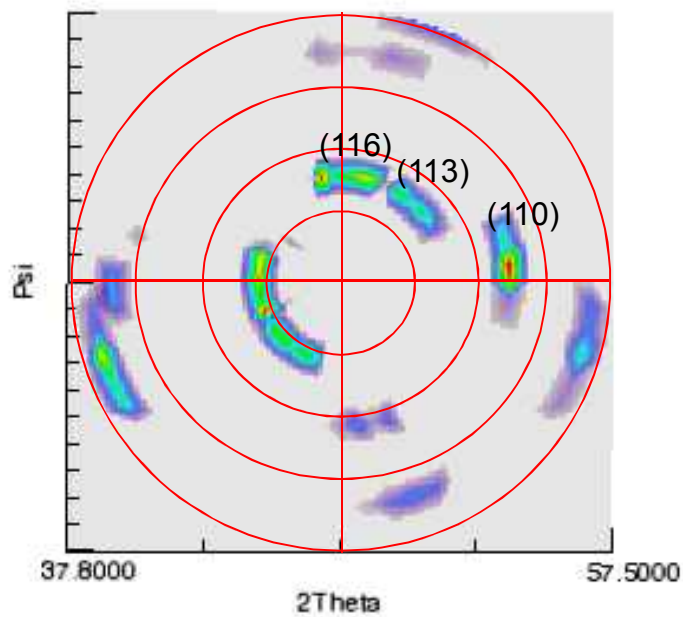
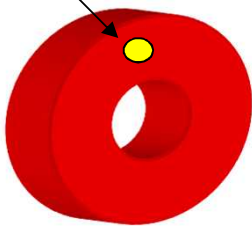
Typical Sapphire (Ruby) Orientations:

- 0 degree to the C-axis
- 90 degree to the C-axis
- 30 degree to the C-axis (known in the industry as “Random Orientation”)



(#5) Lot# 0708001

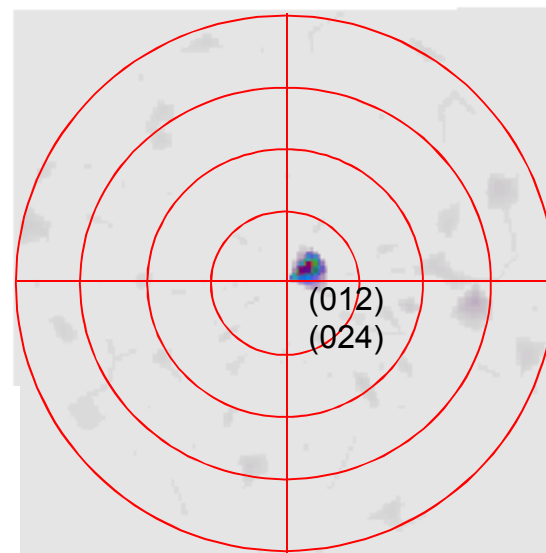
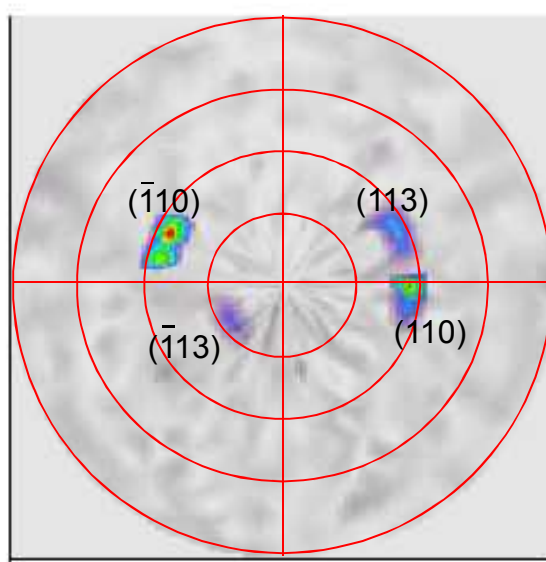
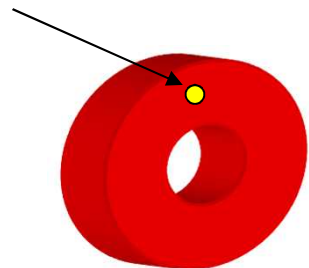
XRD beam location



Looks like a (012) 'R' cut

(#1) Lot 0708001

XRD beam location

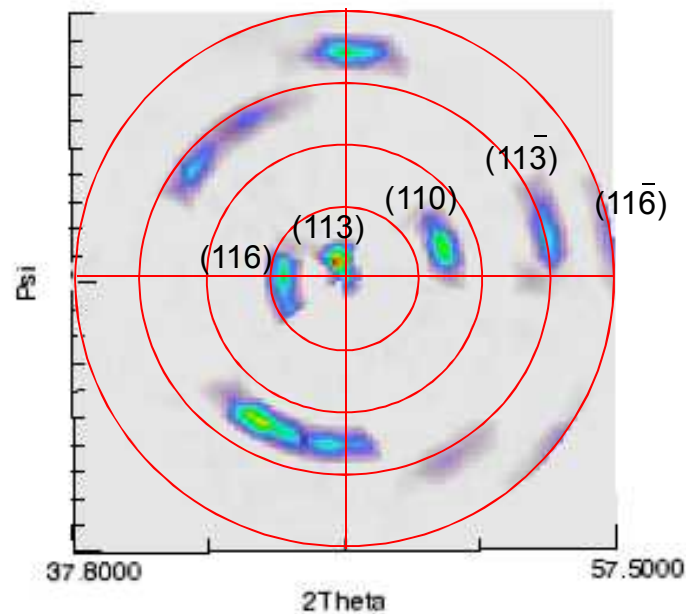
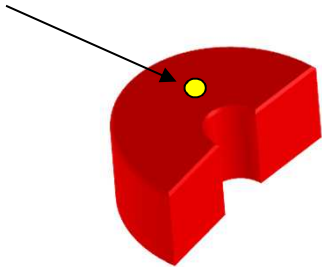


Superimposed (012) and (024)

Looks like a (012) 'R' cut

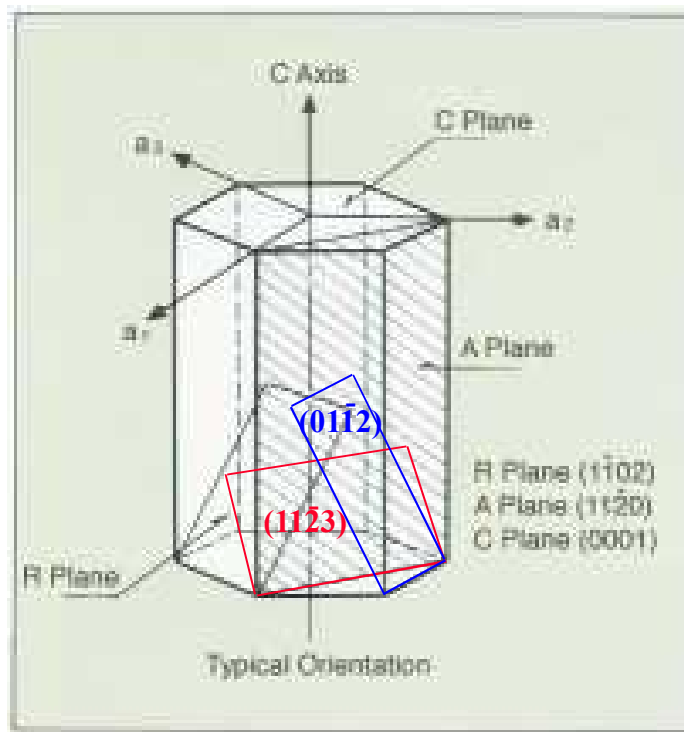
Fractured ruby ring – top surface

XRD beam location



Looks like a (113) cut

What are the differences between these two cuts?



PDF#00-010-0173(RDB): QM=Indexed(I); d=(Unknown); I=Diffractometer

Corundum, syn (Aluminum Oxide)

Al₂O₃ Blue, colorless, yellow purple to violet, green, pink to deep pigeon-blood red

Radiation=CuK α 1 Lambda=1.5405 Filter=Ni

Calibration= 2 θ =25.584-152.454 I/Ic(RIR)=1

Ref:

Natl. Bur. Stand. (U.S.), Circ. 539, v9 p3 (1960)

Rhombohedral - Powder Diffraction, R-3c (167)

Z=6

mp=2049.85°

CELL: 4.758 x 4.758 x 12.991 <90.0 x 90.0 x 120.0> P.S=hR10.00

Density(c)=3.989 Density(m)=4.05 Mwt=101.96

Vol=254.70

F(30)=49.8(0.018,32/0)

Ref:

Typical pole figure projections

- Equal area projection: Surface element sizes remained unchanged.
- Orthogonal projection: Best visualization for area ratios along the line-of-sight.
- Stereographic projection: Direct measurement of spatial angles.

