# X-ray Microdiffraction

## New Technique in Texture and Strain Analysis

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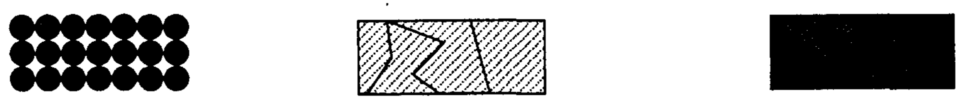
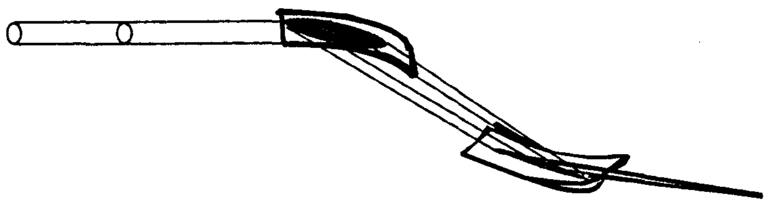
# Mesoscale Materials Science

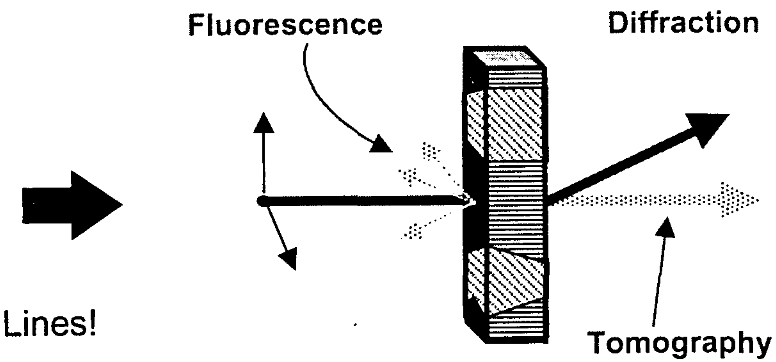
Most "Real" Materials Are Heterogeneous



Microscopic (Atomic Scale) | Mesoscopic | Macroscopic (Continuum)

- Materials Properties Depend Critically On
  - Mesoscopic Structure (grain sizes, texture, impurities ...)
  - Mesoscopic Evolution (grain growth, creep, fracture ...)
  - Typically Tenths-to-Tens of Microns





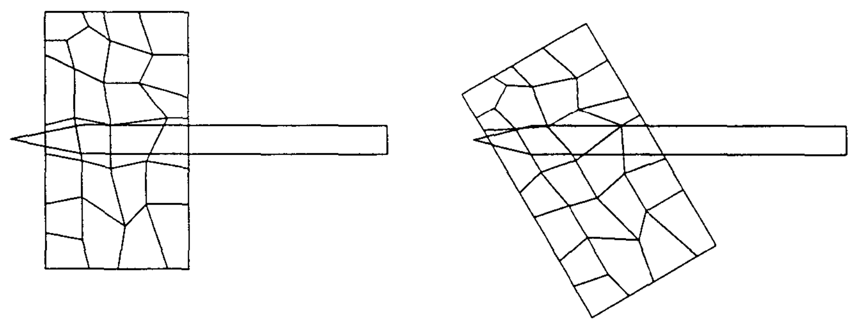

Intense Micron-Size Beams At APS
Power of Millimeter-Size Beams At NSLS Bend Magnet Lines!

# Advantages of X-ray

- Nondestructive: $10^{-4}$ power for same Minimum Detectable Limit (MDL) compared to electron probes
- Minimum sample preparation
- Penetrating probe: Possible 3 dimensional maps
- Orders of magnitude better strain sensitivity

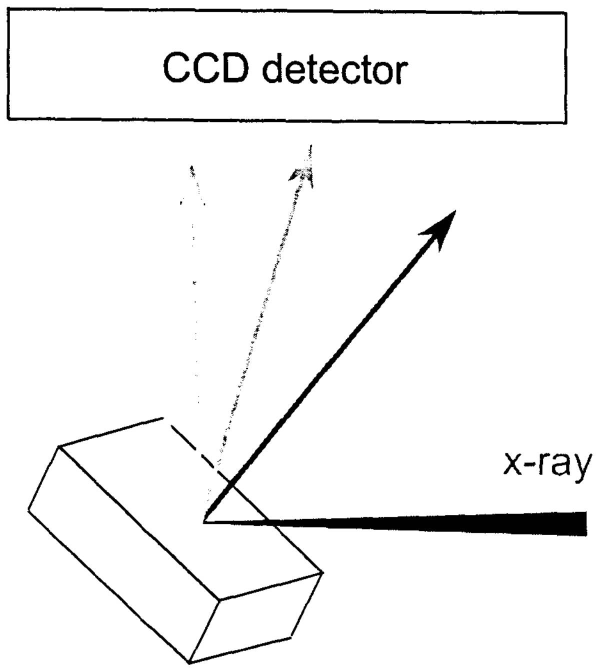
# Problems in Standard Diffraction Technique

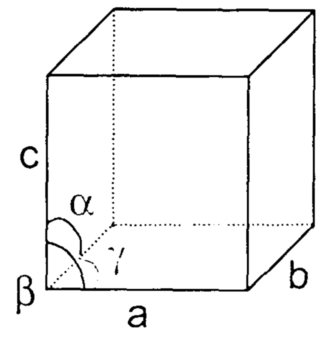
- Rotation changes the illuminated volume due to limited accuracy of diffractometer



- Takes too much time to do 2D or 3D scan
- Solution : Broad Bandpass(white beam) Laue image (with automated indexing)

# Broad Bandpass Diffraction




unit cell

- Broad bandpass (white beam) x-rays excite several reflections at the same time.

⇒ No need to rotate the sample

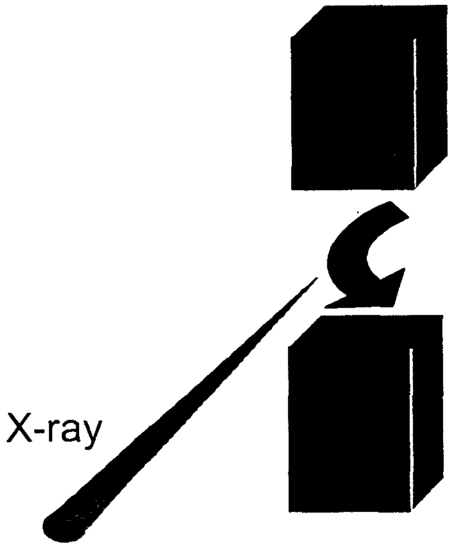
- Angles of 4 reflections and the energy of one reflection yield the unit cell.

# Key Elements in X-ray Microprobe Setup

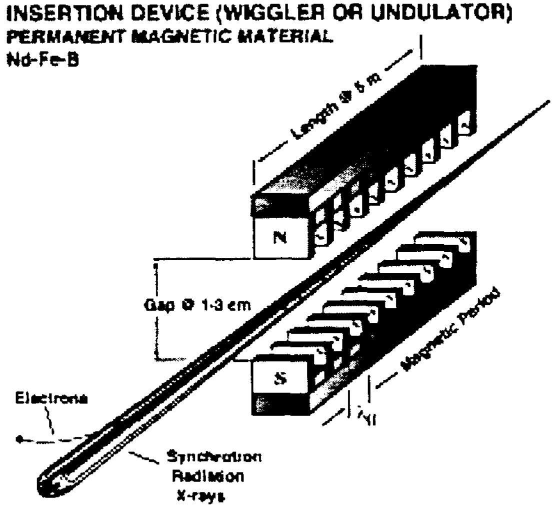
- X-ray source : 3rd generation synchrotron radiation sources with high brilliance, small source size ⇒ Pohang Light Source(PLS), APS, ALS, ESRF, Spring-8
- X-ray optics : Elliptic K-B mirror pair ⇒ Sub-micron focused beam (white and monochromatic)
- Analysis software : Automated indexing, calibration, peak finding, strain calculation, etc.
- Monochromator ⇒ White beam and monochromatic beam along the same axis

⇒ *Quantitative map of microstrain, phase, and texture*

# Synchrotron Radiation

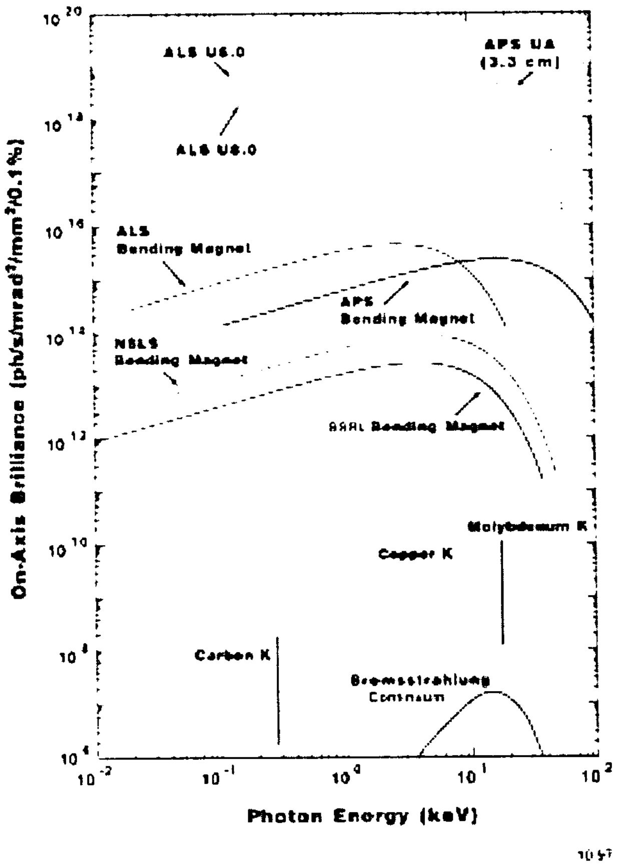


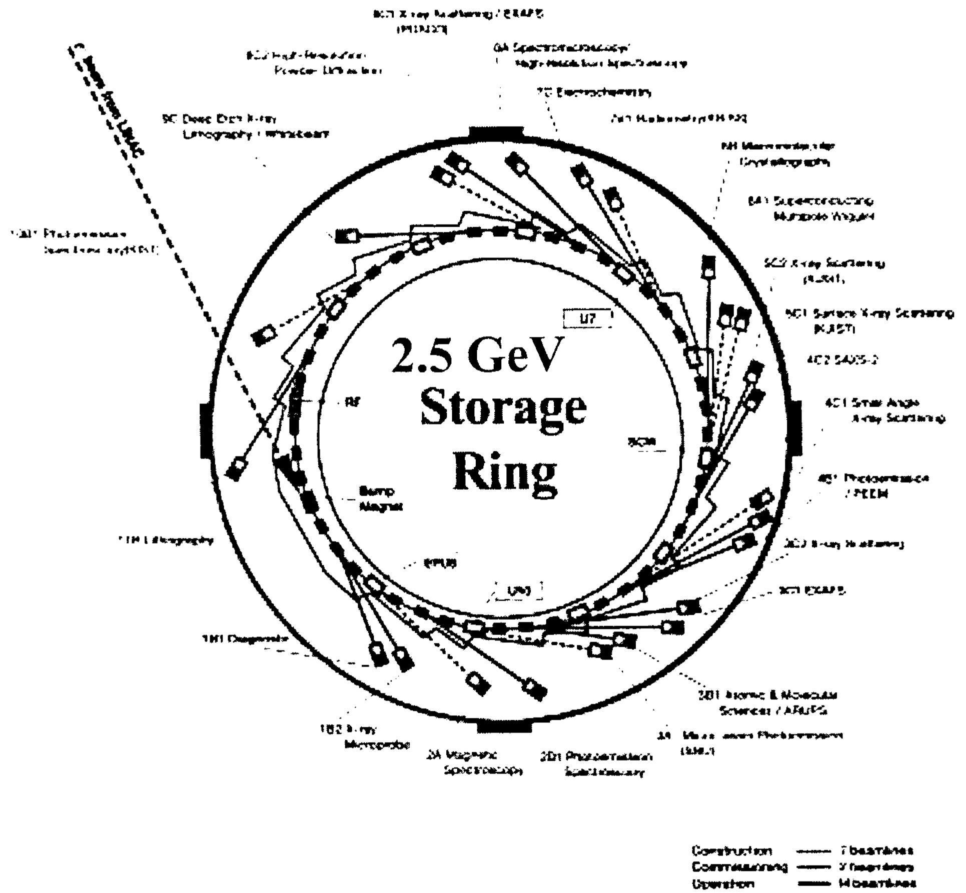

Bending magnet




Undulator

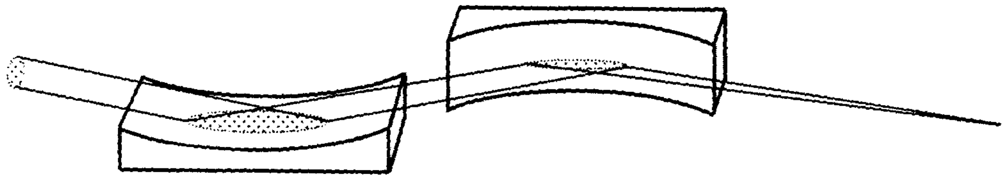
# Pohang Light Source




PLS: similar to ALS beamlines with more higher energy components

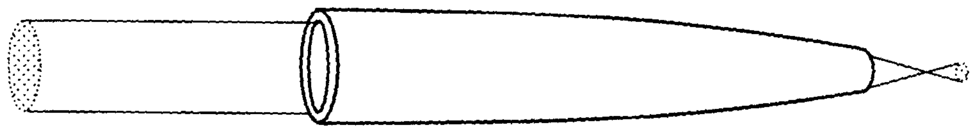
# X-ray Focusing Optics



## Kirkpatrick-Baez mirrors

Polychromatic

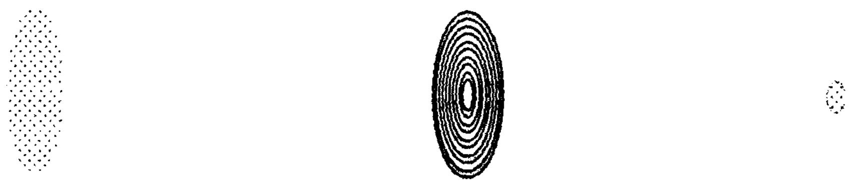
Hard to make and align



## Glass capillary

Polychromatic

Large divergence, small working distance

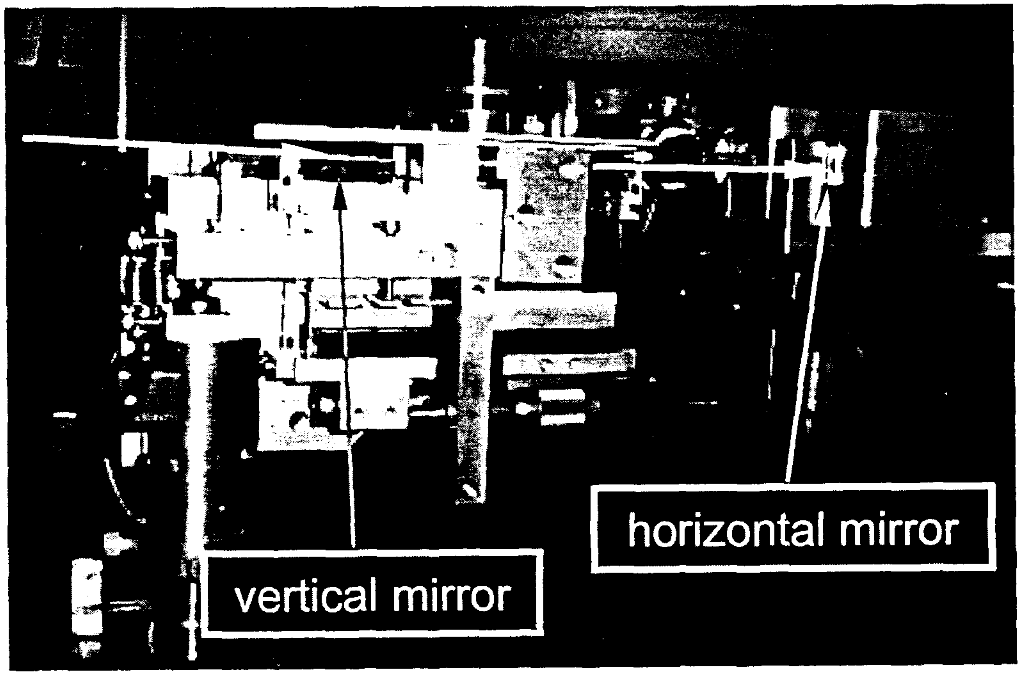


## Fresnel zone plate

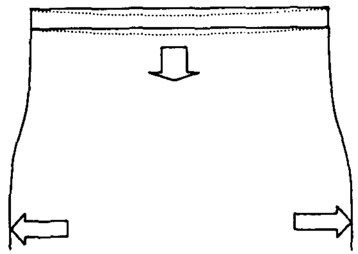
Easy to align

Monochromatic, low efficiency

# Kirkpatrick-Baez Mirrors



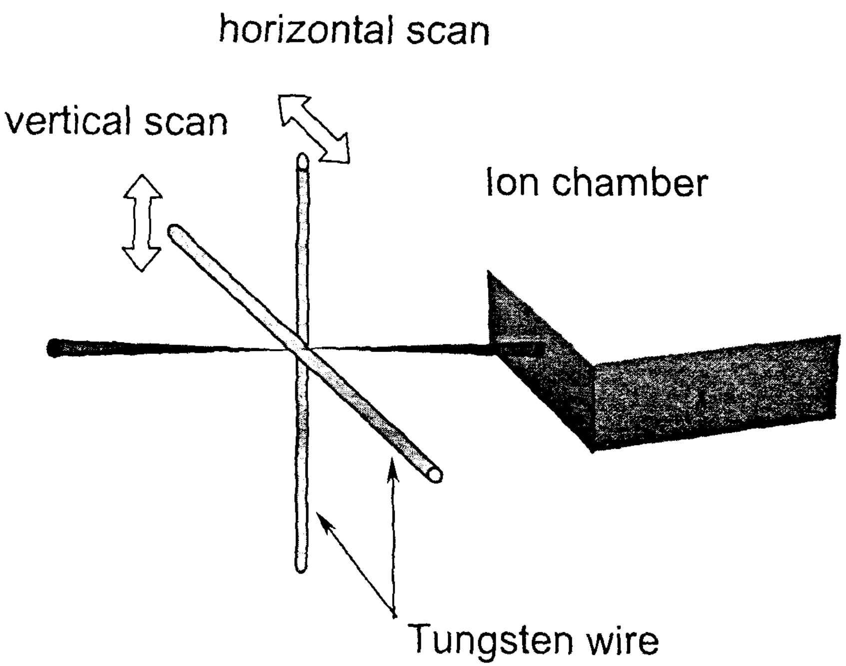

Elliptically figured mirror with benders

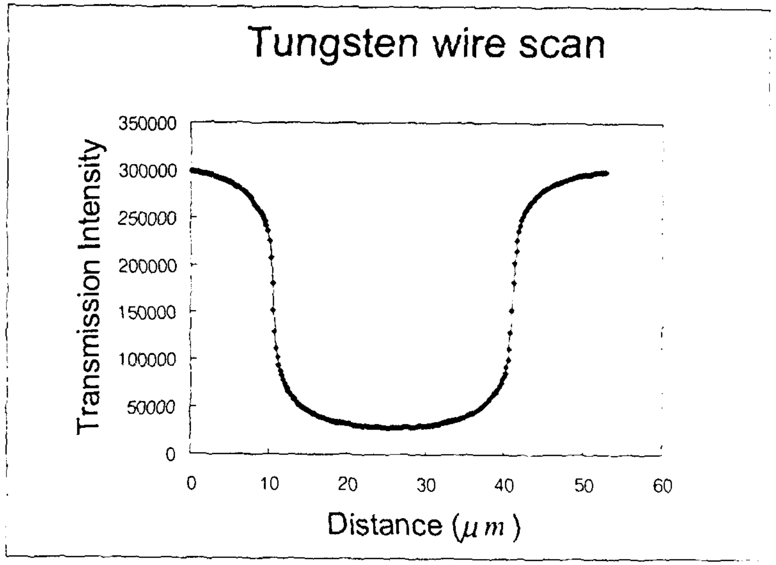


Focused beam size at PLS 1B2: $2 \times 0.7\mu m^2$

# Measurement of the Beam Size

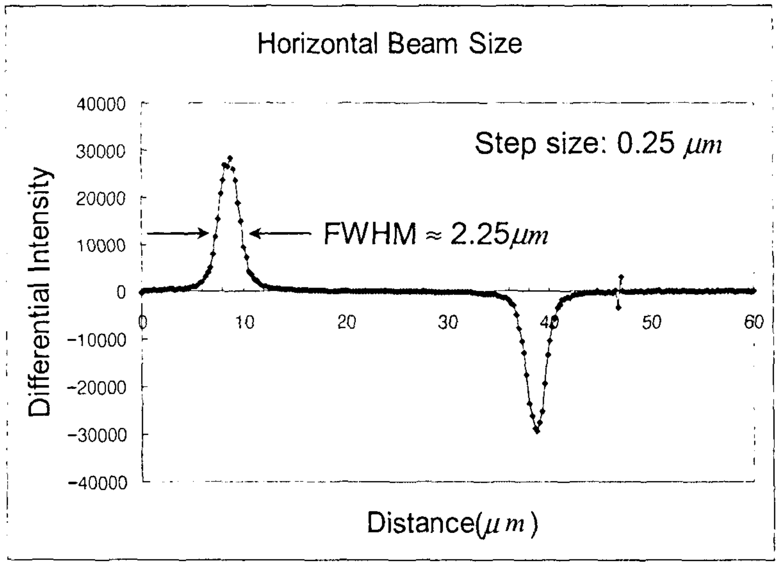
Scan 40 $\mu m$ thick tungsten wire

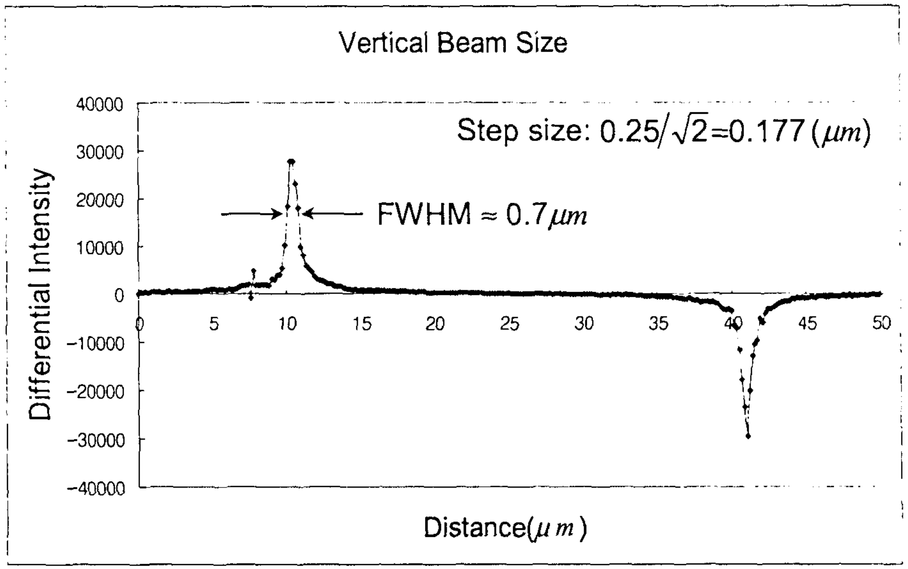


# Measurement of the Beam Size

## Tungsten-wire scan profiles (Transmission Intensities)



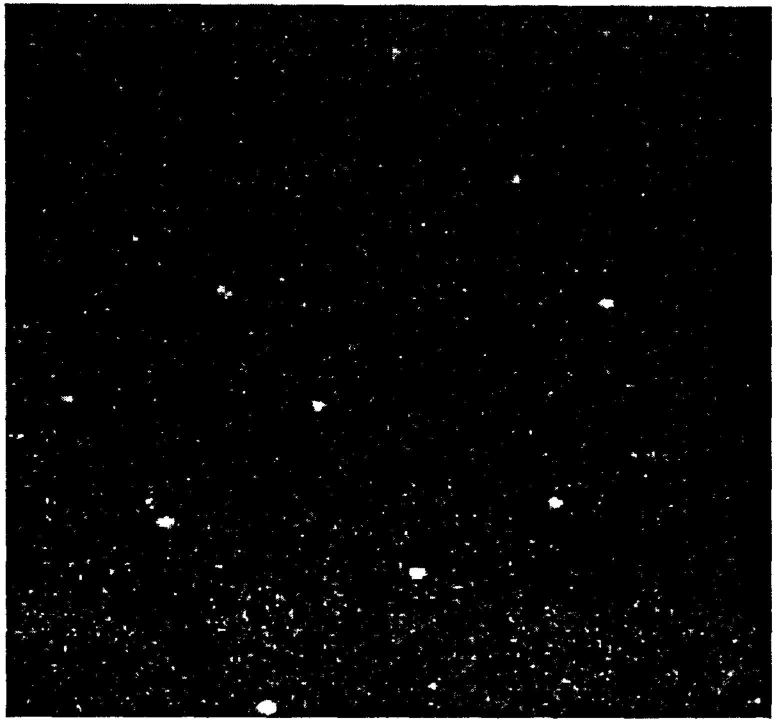




Nominal Beam Size : $2.25 \times 0.7\ \mu m^2$

# Automated Indexing of Multigrain Crystals

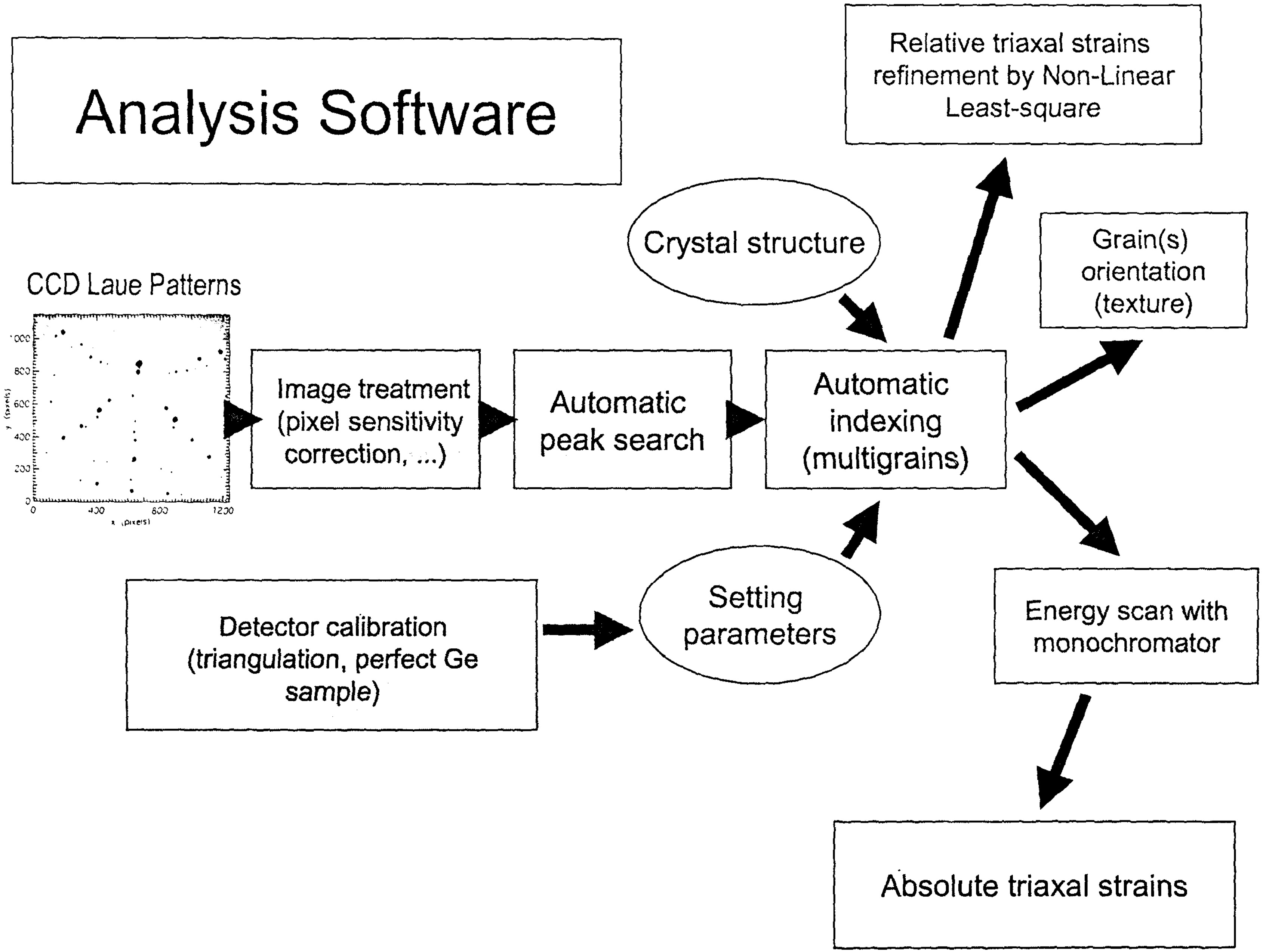
- For multigrain crystals, automated indexing is essential to find indices.
- Our algorithm finds indices of several grains

Yellow and red dots represent Laue spots coming from two different grains



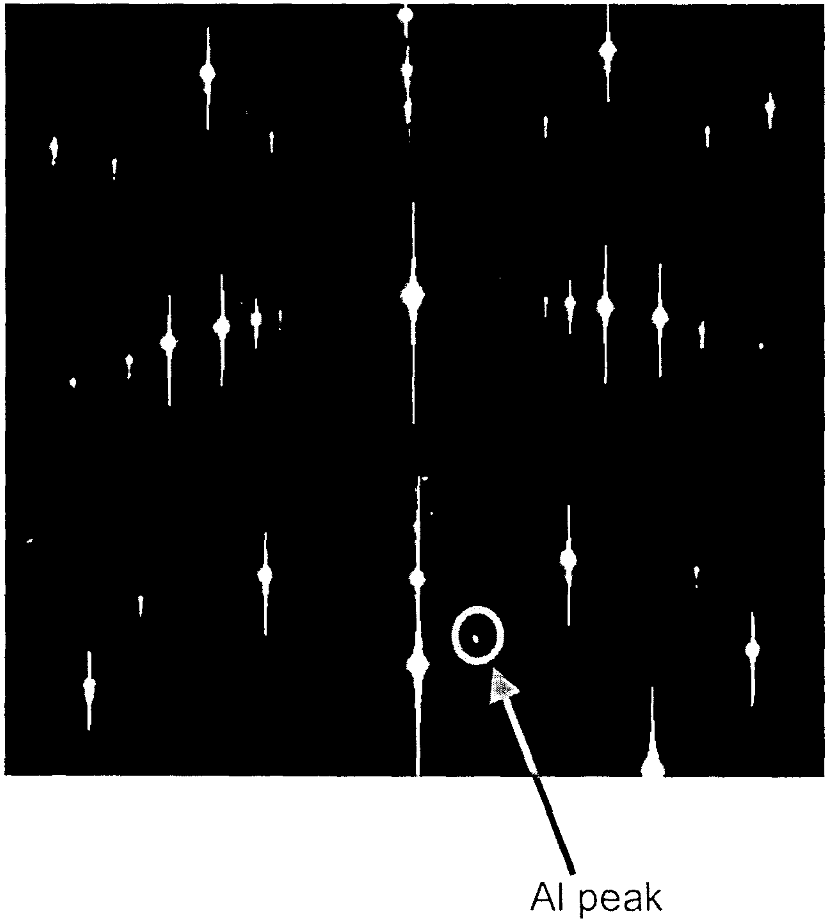
Laue image from two grains of Si

# Analysis Software

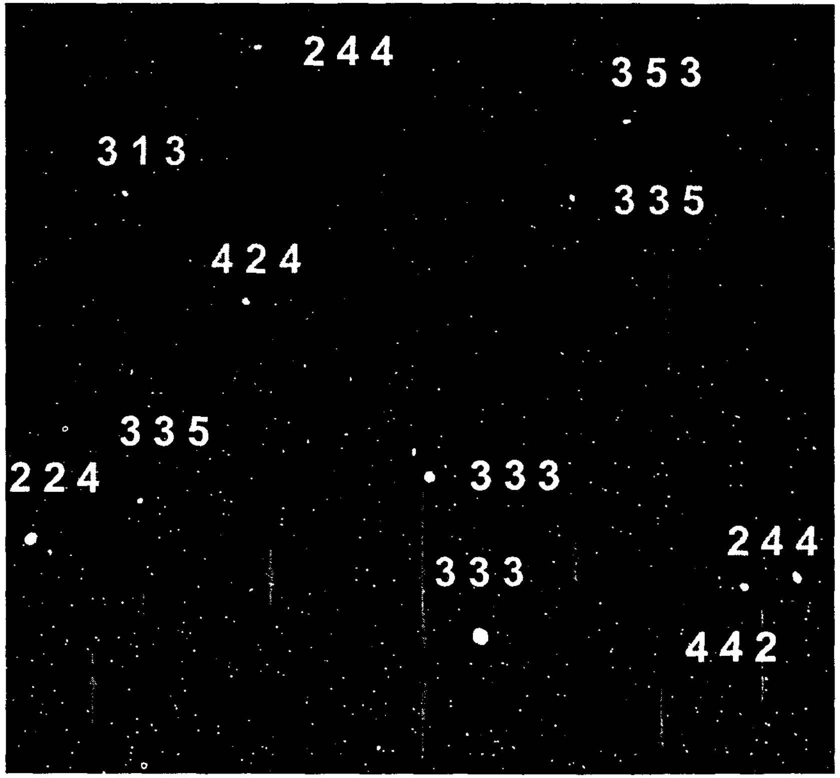


# Laue Image Treatment and Indexing

White beam CCD Raw image

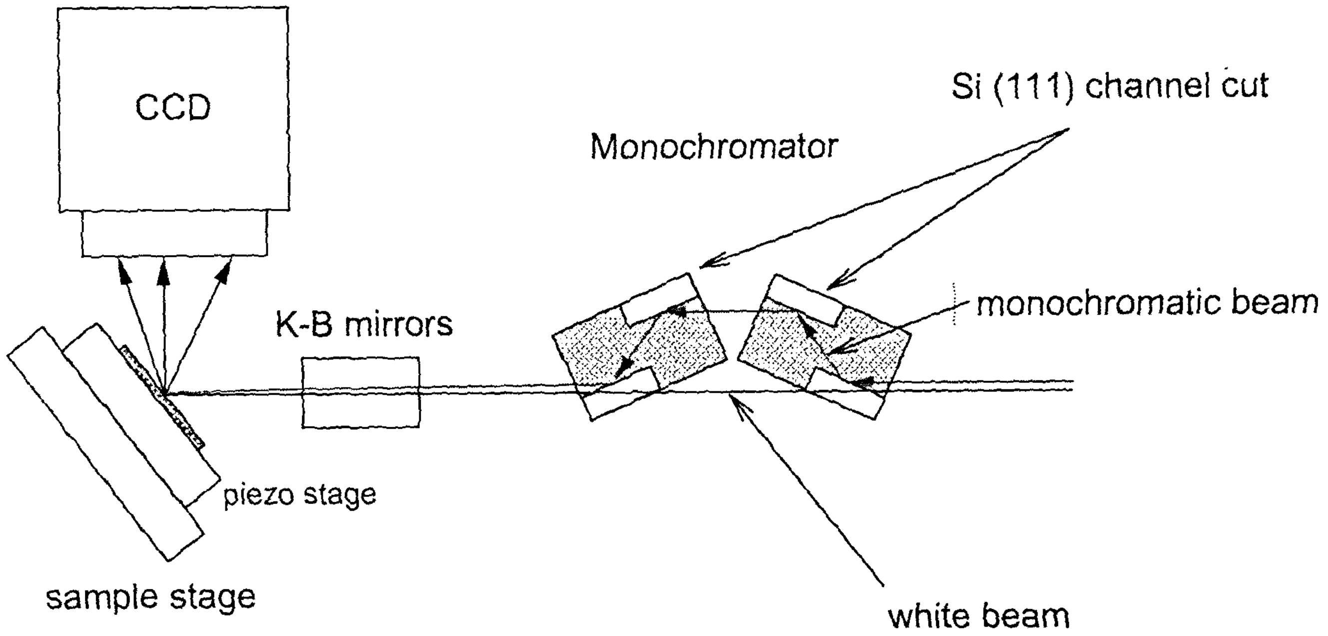



Indexed pattern

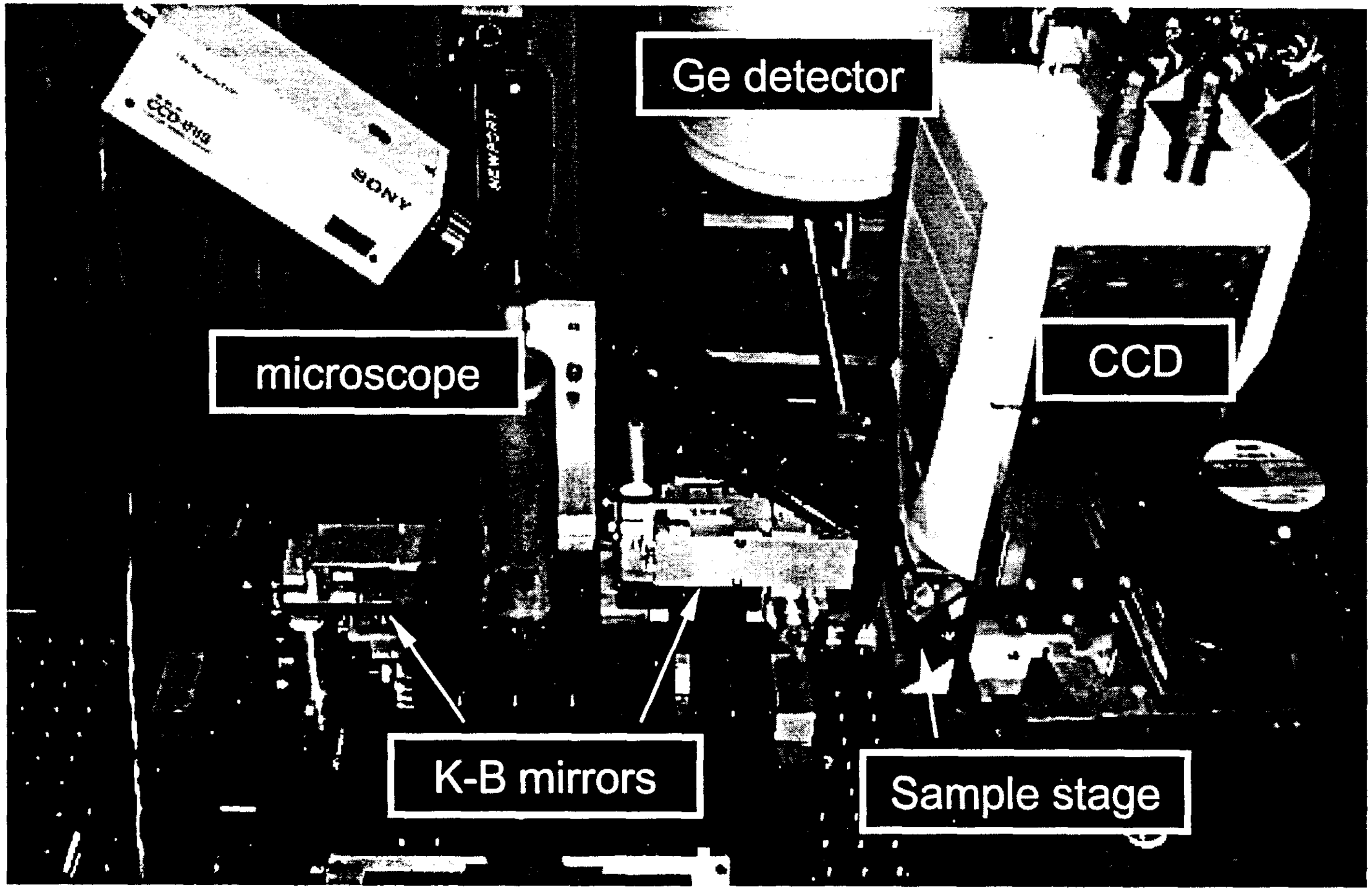



Red and green indices indicate two seperate grains

# PLS 1B2 Microprobe Setup



# X-ray microprobe setup at PLS

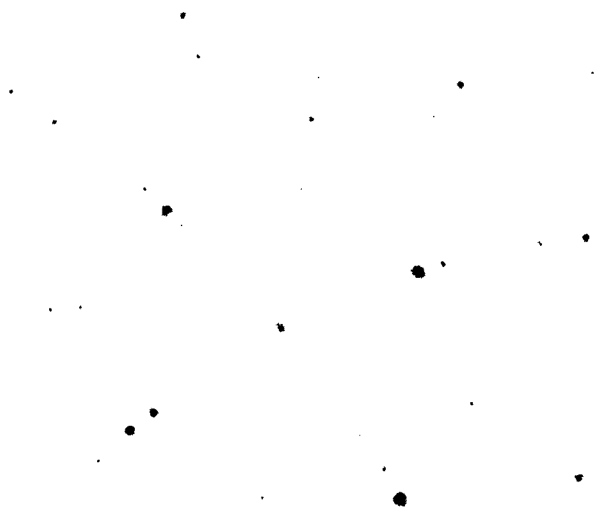


# Comparison of PLS and APS

- Laue image of Ge (111) crystal
  - Numbers of reflections indicates usefulness as a light source for microdiffraction

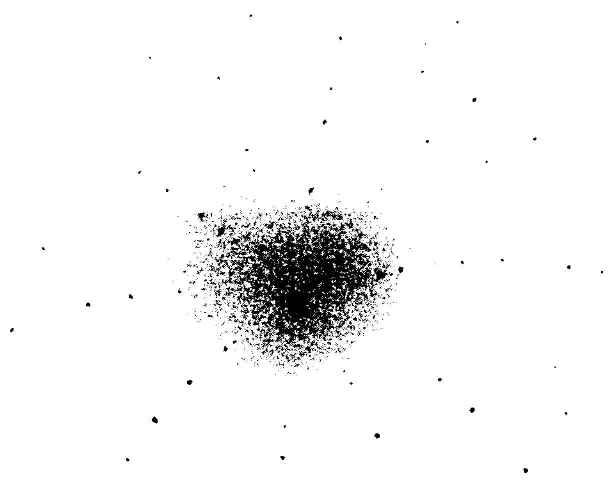
APS: CCD with 1:1 fiber optics

~ 50 peaks ( $E_{max}$ ~ 24 keV)

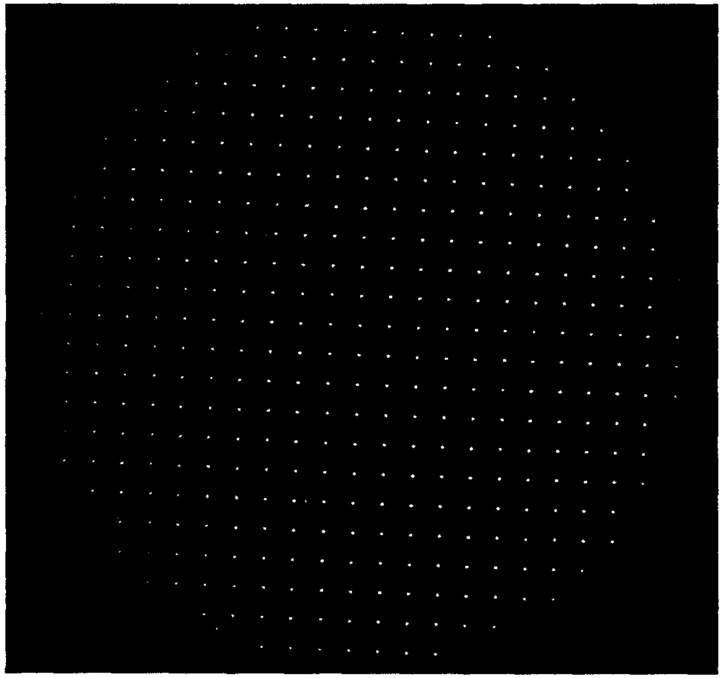


PLS: CCD with 5:1 fiber optics

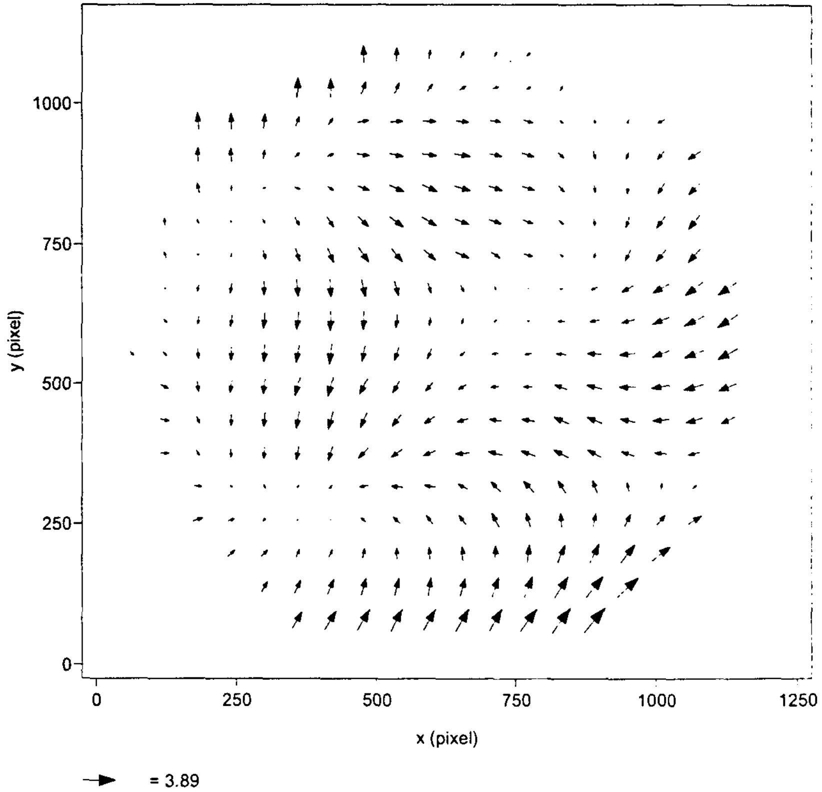
~ 50 peaks ( $E_{max}$ ~ 16 keV)



# Distortion map of a CCD with 5:1 tapered glass fiber optics

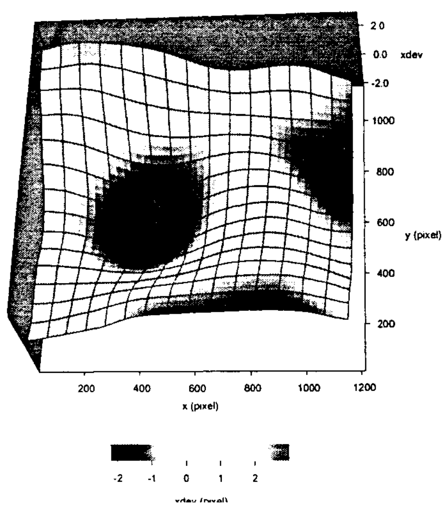


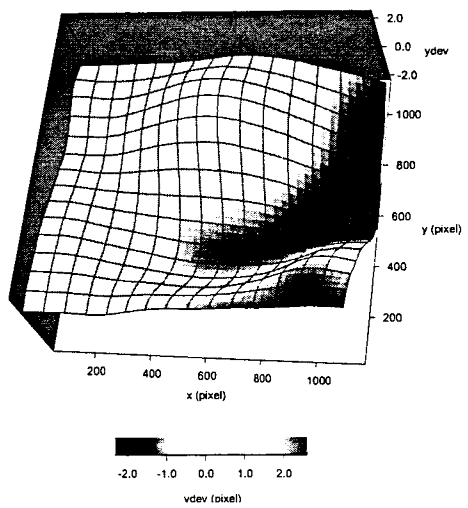
Picture of square grids



# Distortion Correction

- Method:
  1. From the transmission picture of a plate with holes in square grids, find the deviations in x and y coordinates.
  2. For all of the measured peak positions, find the deviations in x and y direction by interpolating the result from 1.
  3. Calculate the correct peak positions.




- Calculated strain in a Ge crystal:
  - Before the correction:

deviatoric strain in crystal reference frame, a b c (*10^3)

| | | |
|---|---|---|
| -1.56972 | -0.23949 | -0.43044 |
| -0.23949 | 1.28806 | 0.64864 |
| -0.43044 | 0.64864 | 0.28200 |

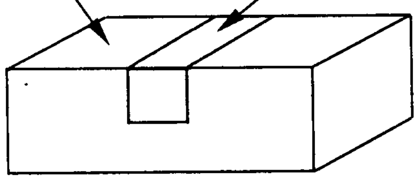
  - After the correction:

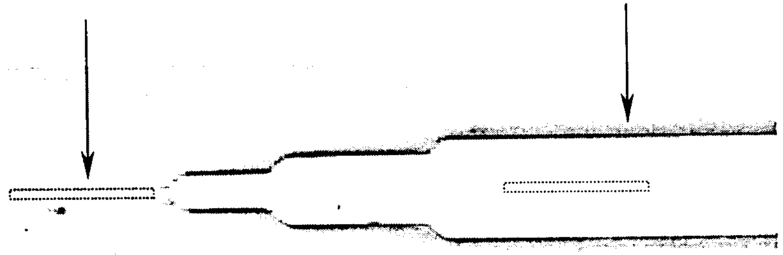
deviatoric strain in crystal reference frame, a b c (*10^3)

| | | |
|---|---|---|
| 0.06132 | -0.01108 | -0.00347 |
| -0.01108 | -0.07170 | 0.00649 |
| -0.00347 | 0.00649 | 0.01038 |

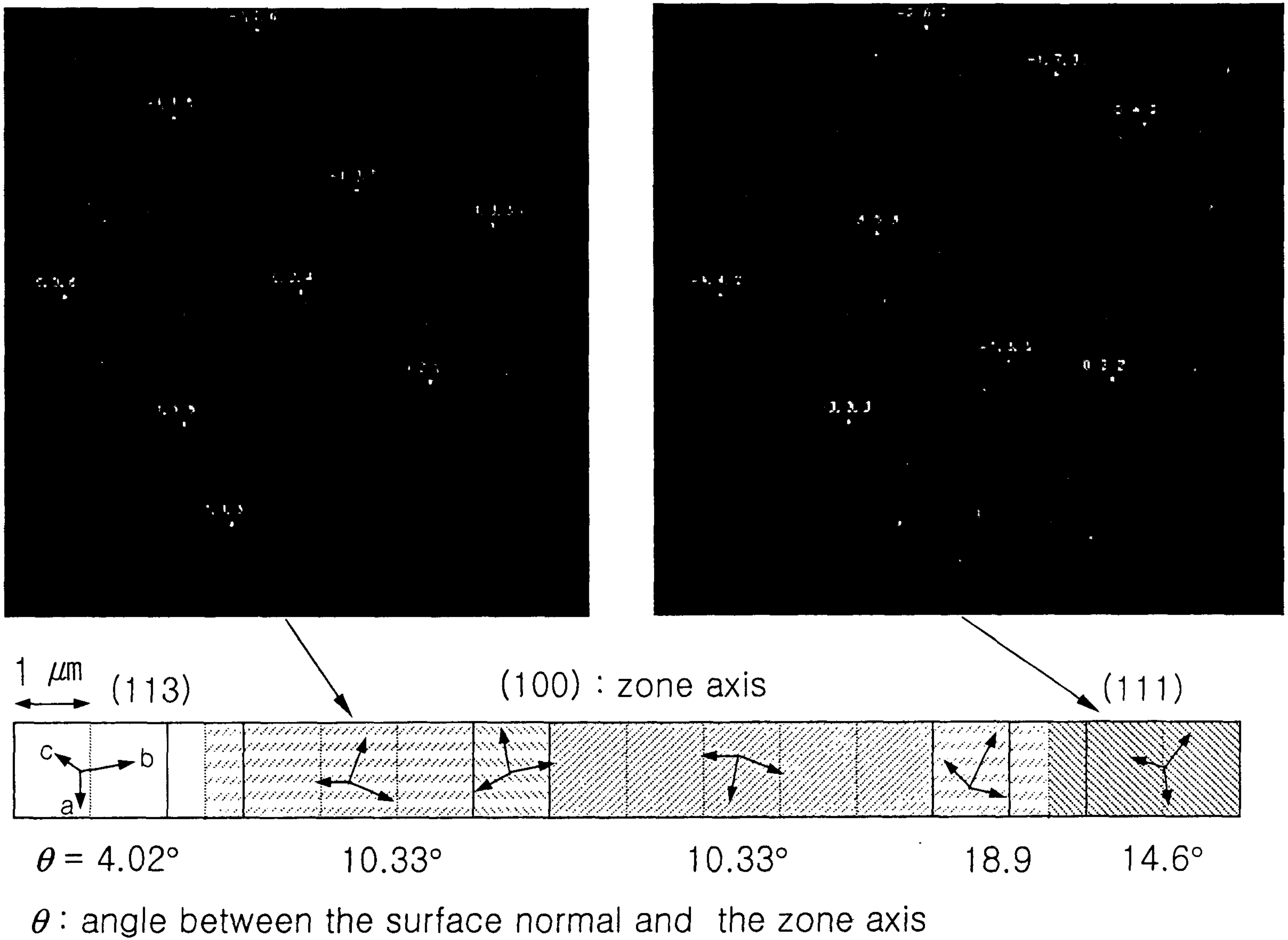
# Cu wires on VLSI chip

Damascene-fabricated Cu lines



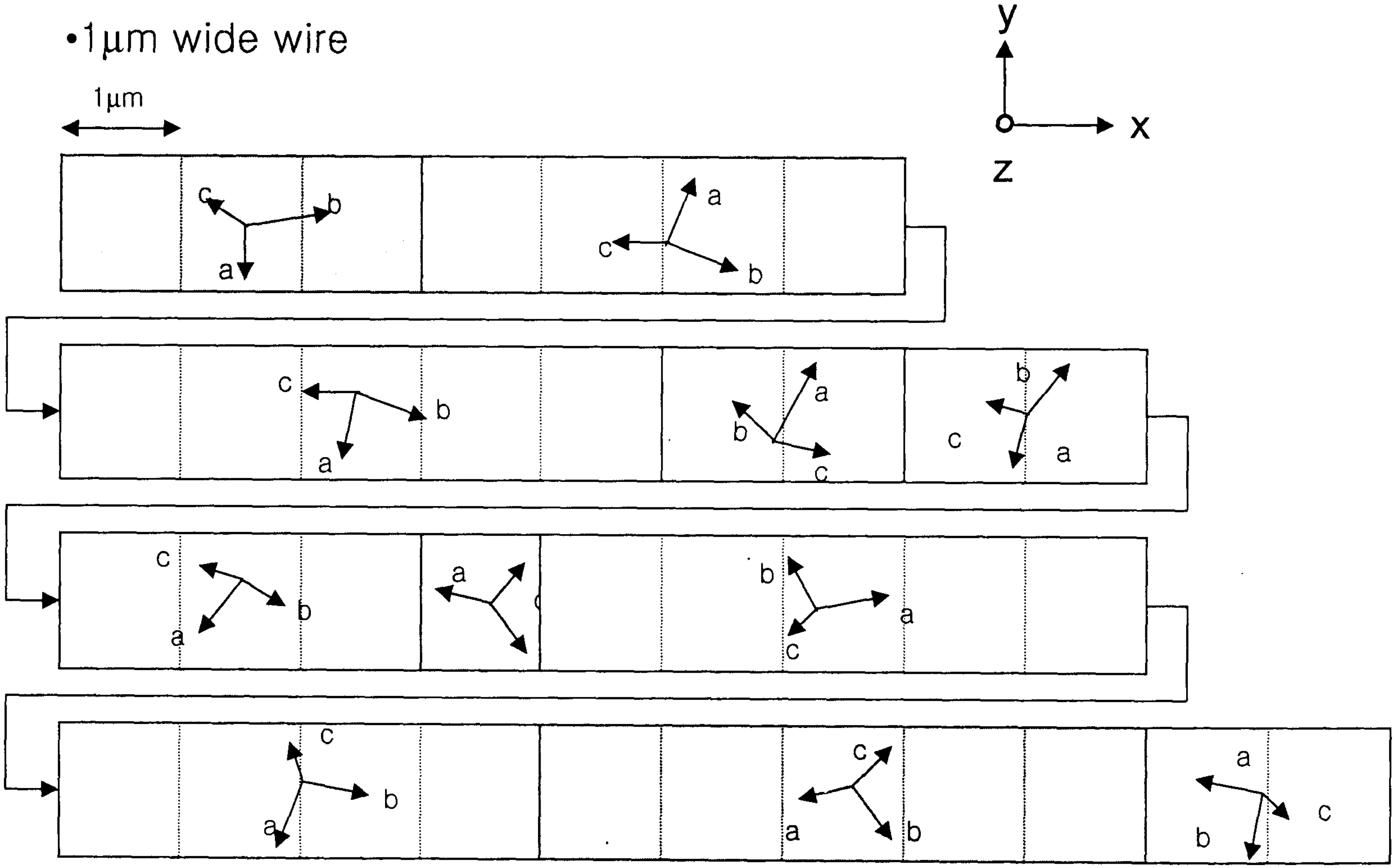



# 1μm wide Cu line의 Texture

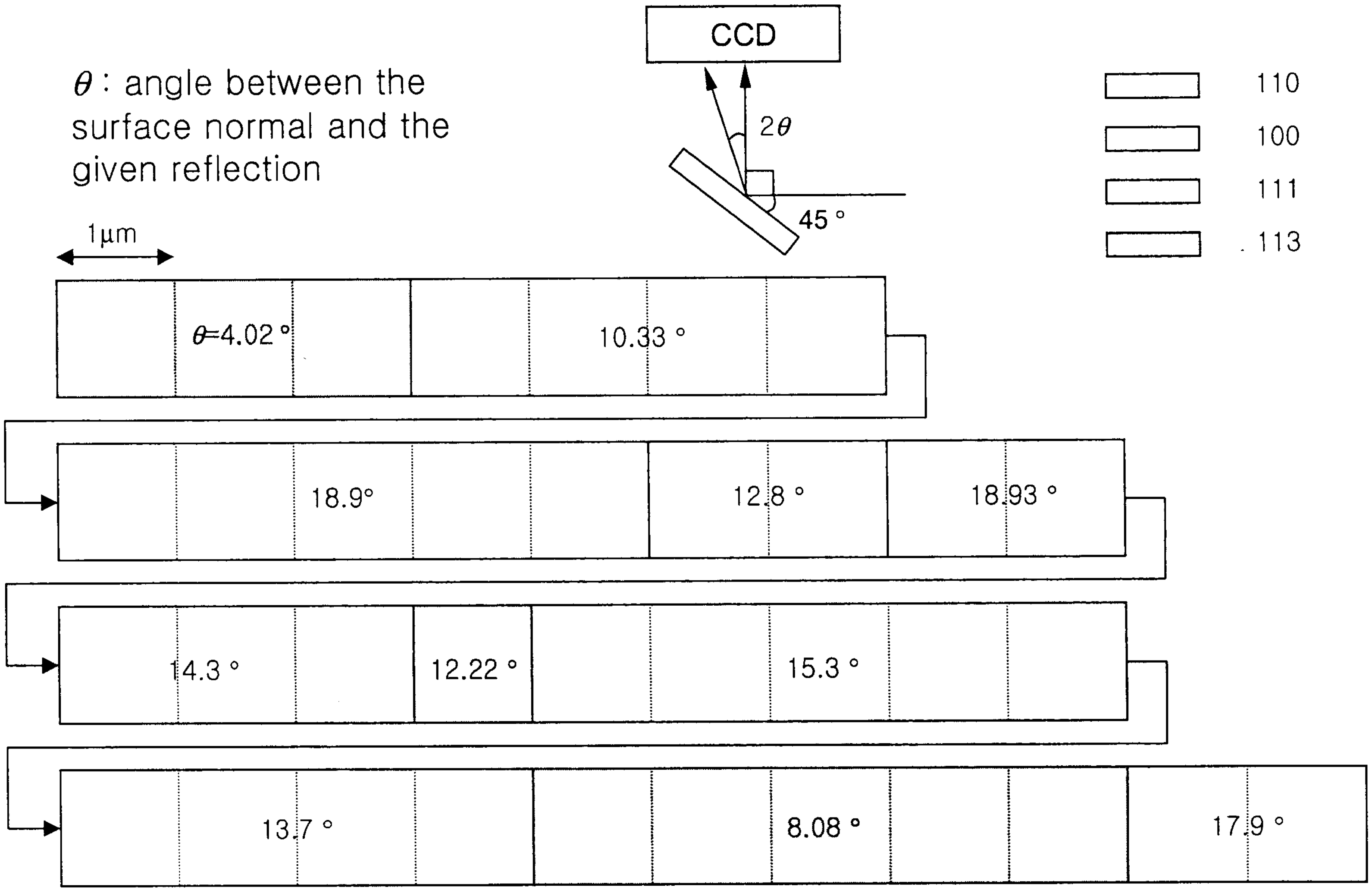


# 1μm Cu wire의 orientation

•1μm wide wire

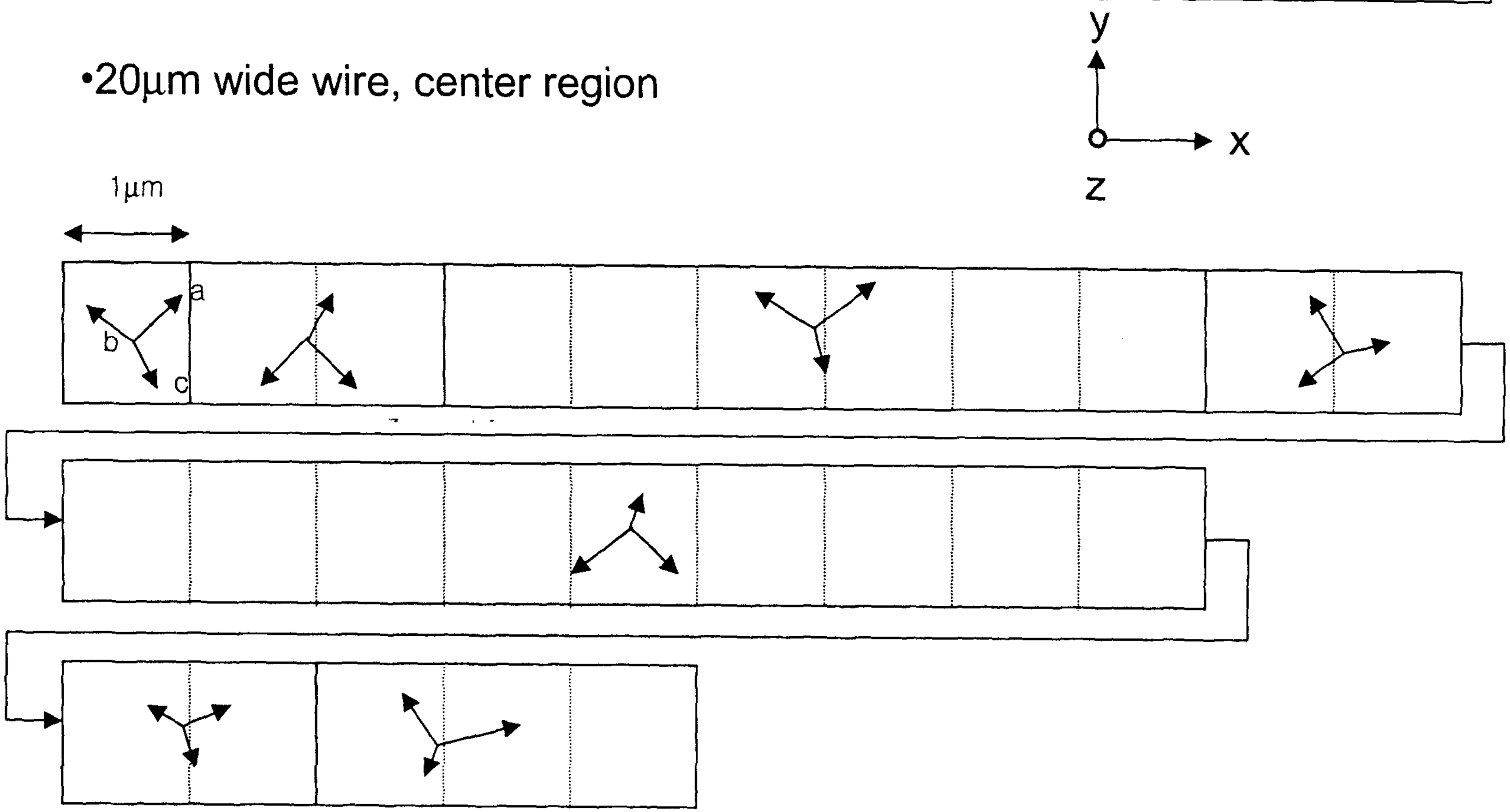


# 1μm Cu wire의 texture



# 20μm Cu wire의 orientation

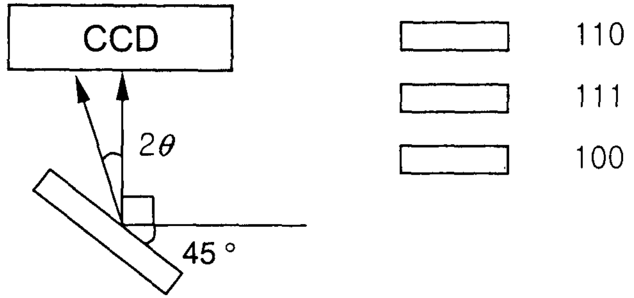
•20μm wide wire, center region

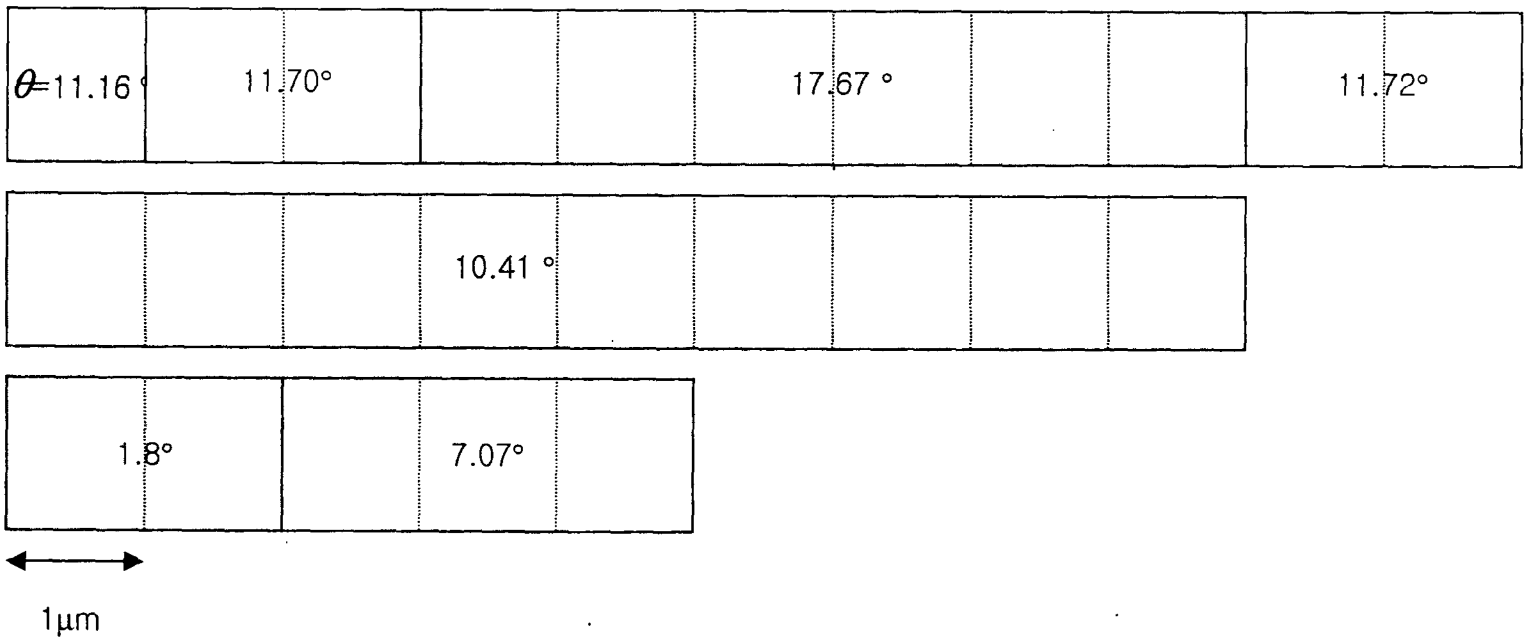


# 20μm Cu wire의 texture

• 20μm wide wire, center region

θ: angle between the surface normal and the given reflection

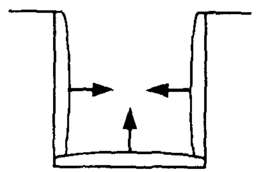


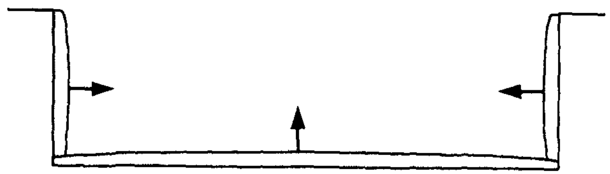
# Grain growth from side walls in the trench structure

Mixture of textures

(111) texture is dominant



1μm wire



20μm wire

The (111) fiber texture growth from side walls results in random textures from the sample surface.

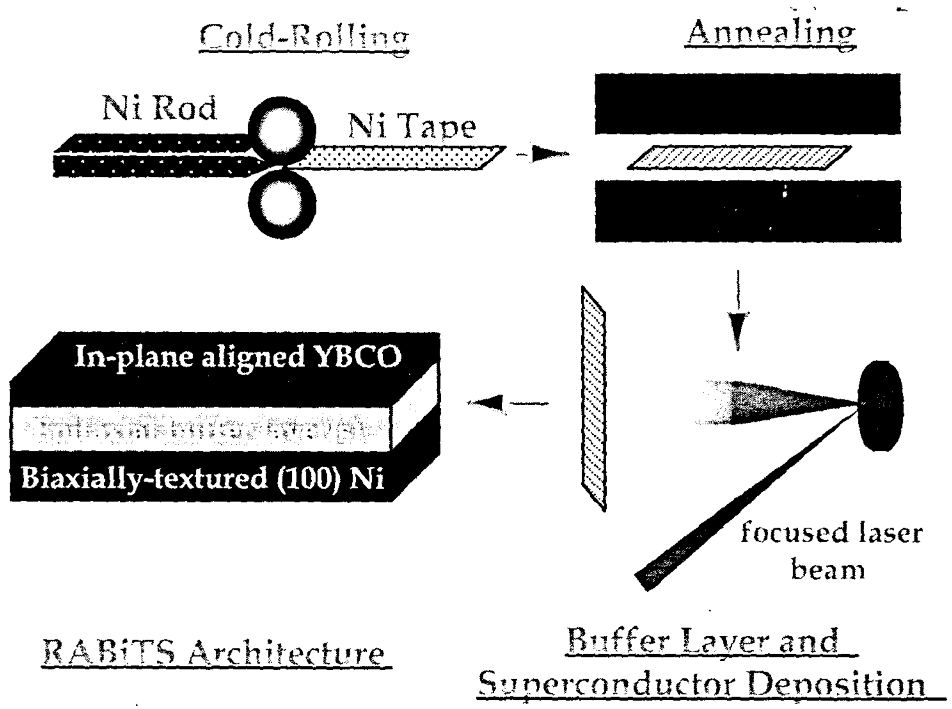
# Rolling-Assisted Biaxially-Textured Substrate (RABiTS) Approach to HTS Conductors

–Grow epitaxial $YBa_2Cu_3O_7$ films rolled-textured metal tapes using pulsed-laser deposition

»biaxially-textured metal substrates by rolling and recrystallization

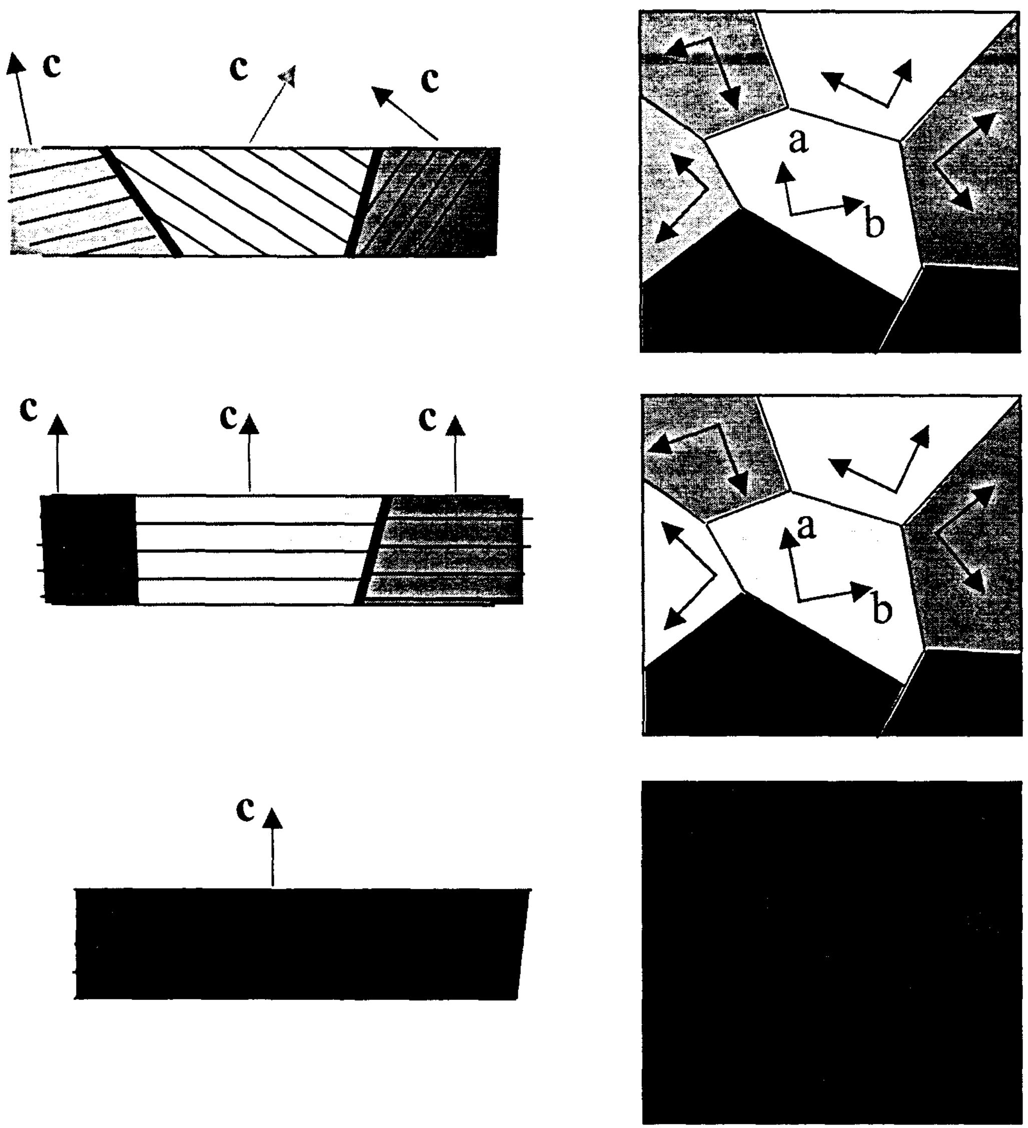
»epitaxial oxide buffer layers on metals

RABiTS™ Process for Superconducting Tapes



# Why is Epitaxial Alignment of YBCO Films Important?

Side View | Top View | Critical Current Density H=0, T=77K

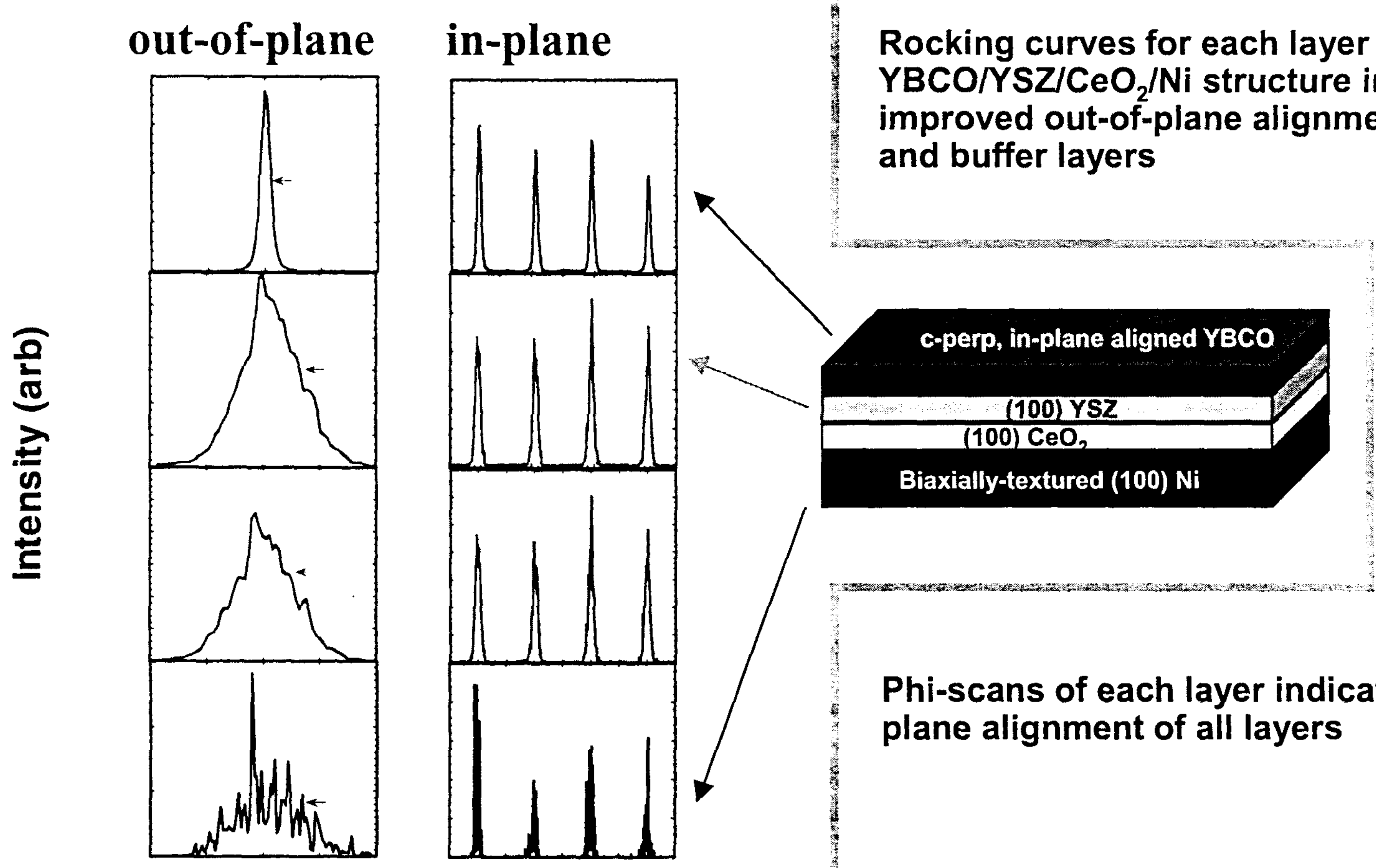



Polycrystalline
$J_c \sim 10^3$ amps/cm$^2$

Textured c⊥
$J_c \sim 10^4$ amps/cm$^2$

3-D Epitaxial c ⊥
$J_c \geq 10^6$ amps/cm$^2$

# In-Plane Alignment of YBCO and Oxide Buffer Layers

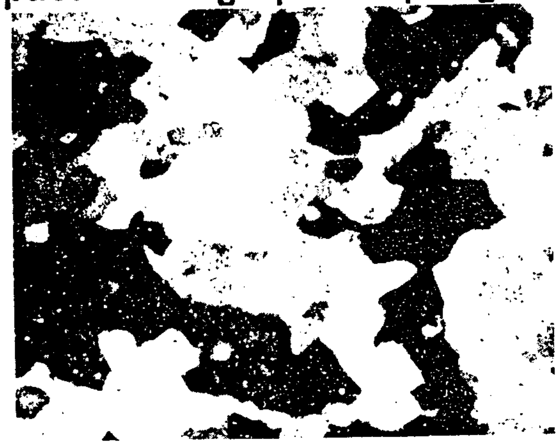


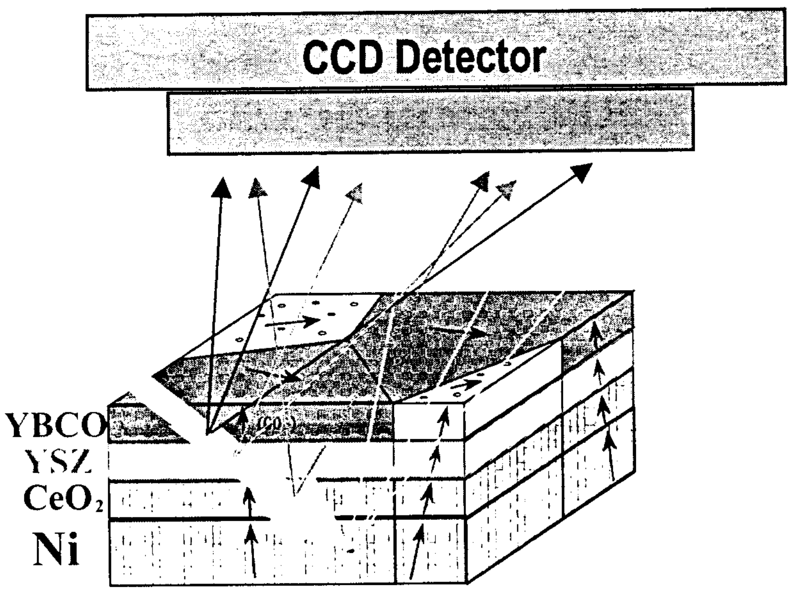

Rocking curves for each layer in the YBCO/YSZ/$CeO_2$/Ni structure indicates improved out-of-plane alignment of the YBCO and buffer layers

Phi-scans of each layer indicate in-plane alignment of all layers

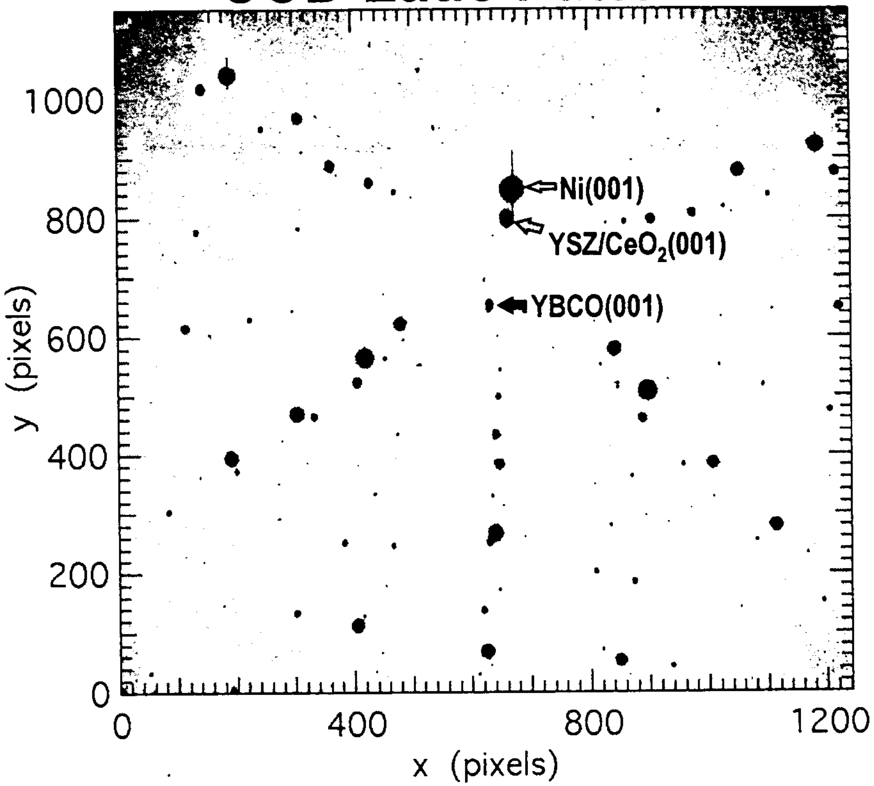
# X-Ray Microprobe Characterization of Individual Grains in RABiTS

Optical Photograph ~50 μm grains



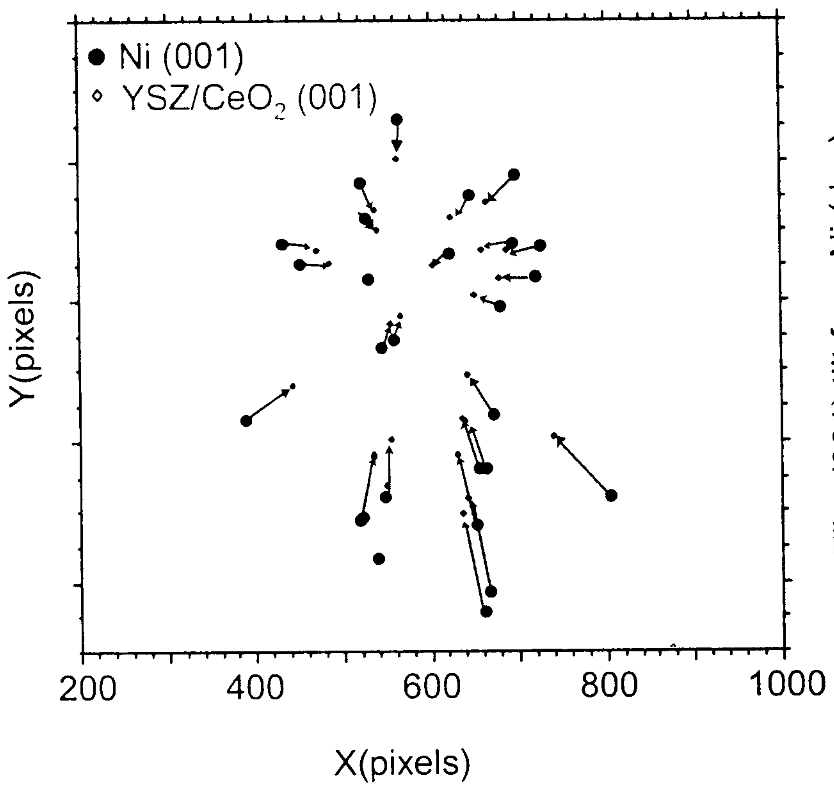



## CCD Laue Patterns

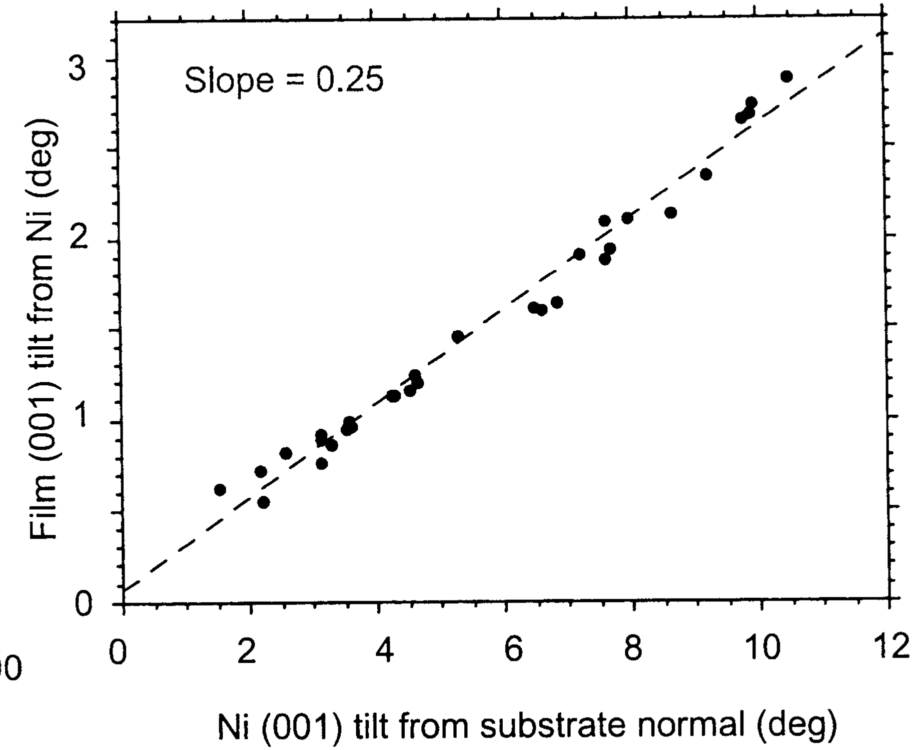


# Epitaxial Tilt of Buffer Layers for Individual Grains in YSZ/CeO2/ Rolled Ni

## Location of (001) poles on CCD

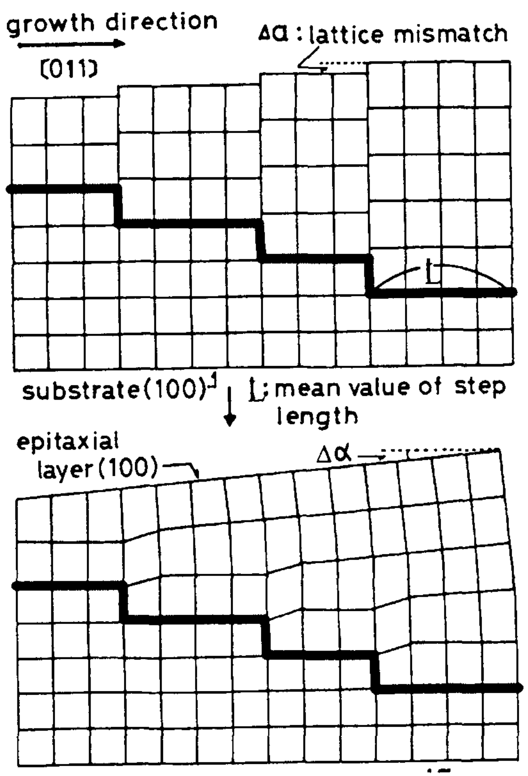



## Film tilt vs. Ni (001) misorientation



# Theoretical Models for Tilt of Epitaxial Films

- **Driving forces:**
  - Elastic strain due to lattice mismatch
  - Interfacial misfit dislocations
  - Surface energy of film
- **Mechanisms**
  - Elastic deformation at ledges (Nagai, 1974)
  - Out-of plane Burger's vector component for dislocations
  - Coherent tilt (Asymmetric Tilt Boundary)



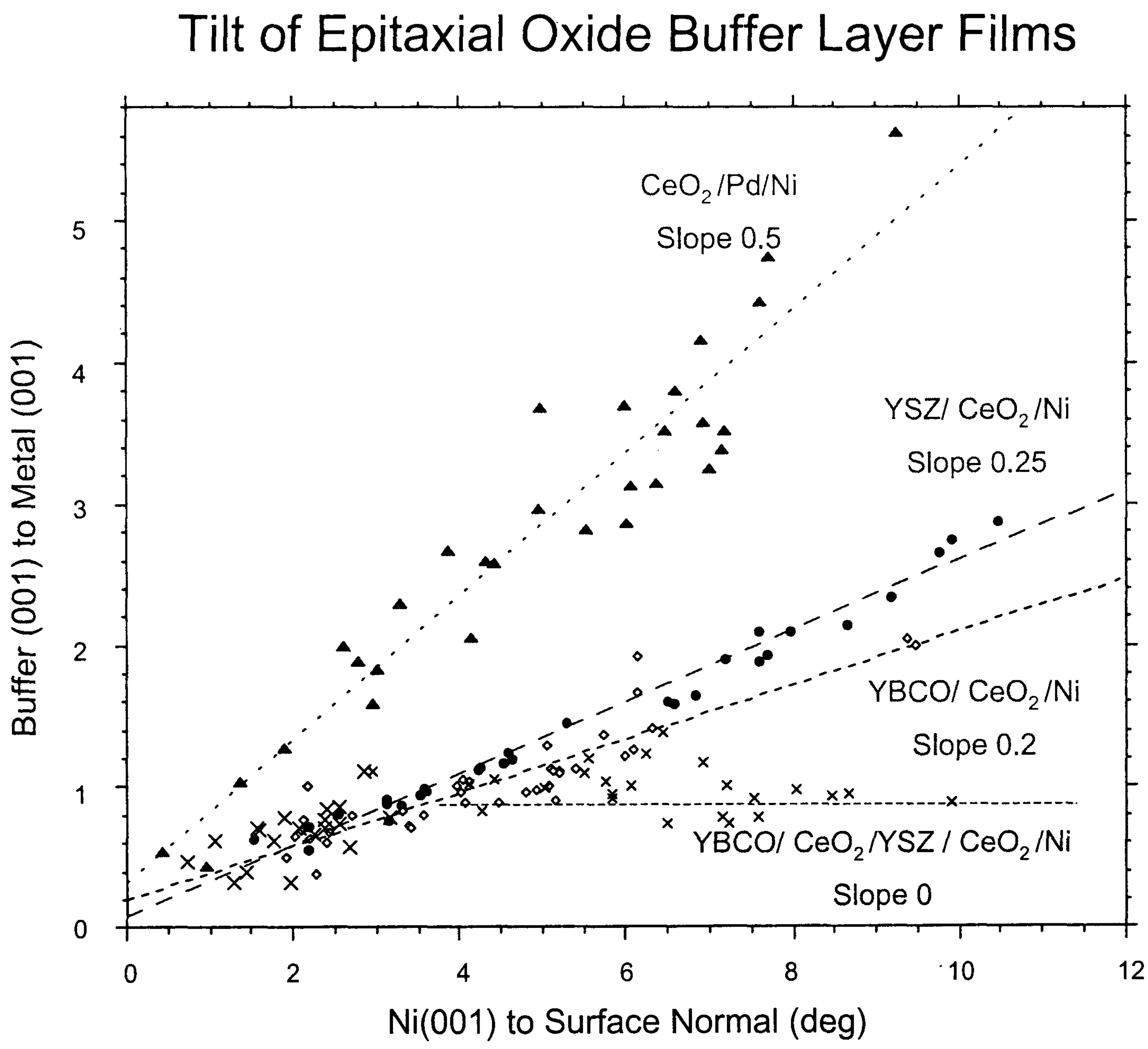

Substrate miscut $\alpha = \tan^{-1}(a_{sub}/L)$

Step height difference $\Delta a = a_{film} - a_{sub}$

Film tilt $\Delta\alpha = \tan^{-1}(\Delta a/L) = \tan^{-1}(\Delta a \tan\alpha / a_{sub})$

Tilt towards surface normal for $a_{film} < a_{sub}$
Tilt depends ~linearly on miscut
Slope $\Delta\alpha/\alpha$ depends on step height difference

$a_{film}$ $CeO_2$ (2.70Å); $a_{sub}$ Ni(3.52Å) ⇒Slope=0.23



Tilt of Epitaxial Oxide Buffer Layer Films
CeO2 /Pd/Ni
Slope 0.5
YSZ/ CeO2 /Ni
Slope 0.25
YBCO/ CeO2 /Ni
Slope 0.2
YBCO/ CeO2 /YSZ / CeO2 /Ni
Slope 0
Buffer (001) to Metal (001)
Ni(001) to Surface Normal (deg)
0
1
2
3
4
5
0
2
4
6
8
10
12

# Future of X-ray Microdiffraction

- One of the most useful tools in local texture, strain analysis : electromigration, grain growth, fracture, deformation mechanism
  - Sub-micron spatial resolution
  - $10^{-5}$ resolution in strain

$\Rightarrow$ 2D or 3D distribution map of orientation, strain

- Analysis tool for small scale complex structures : Devices on VLSI chips, polycrystalline thin films, nano-particles