3-D X-ray Structural Microscopy

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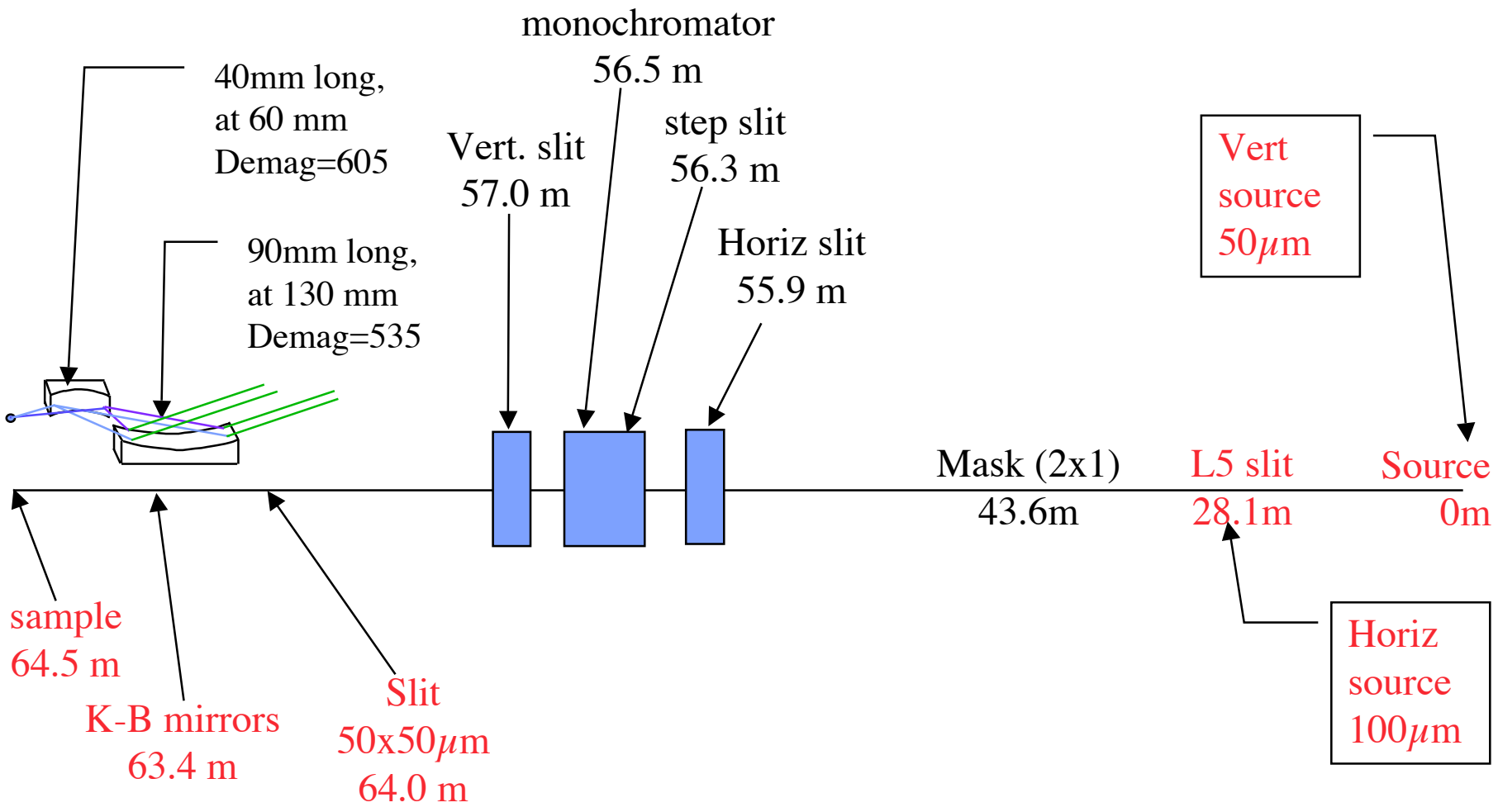
Capabilities

- Measure spatially resolved crystallographic information
- Orientation and strain
- White or monochromatic x-rays
- Fluorescence scanning

Beamline Layout



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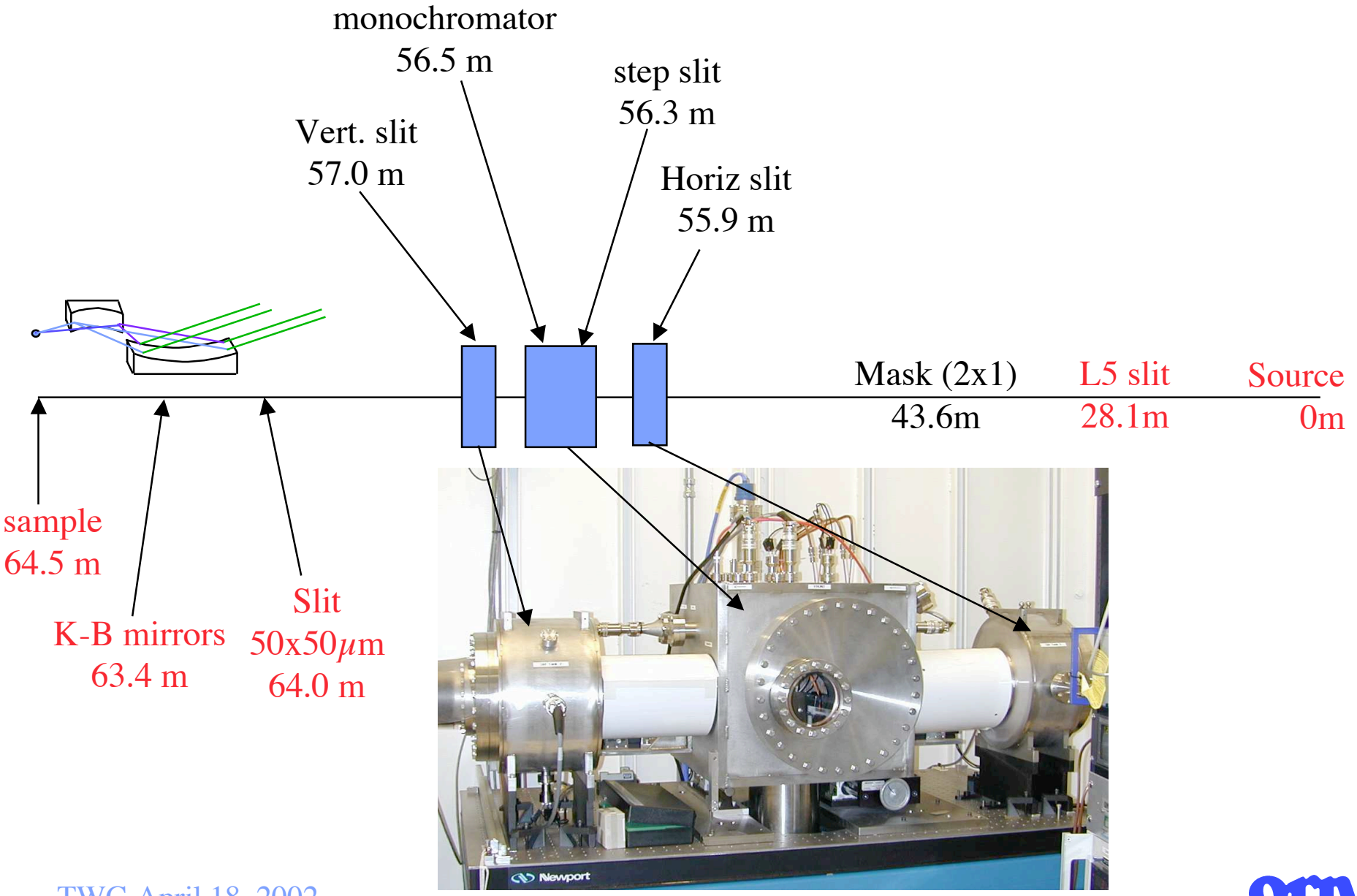
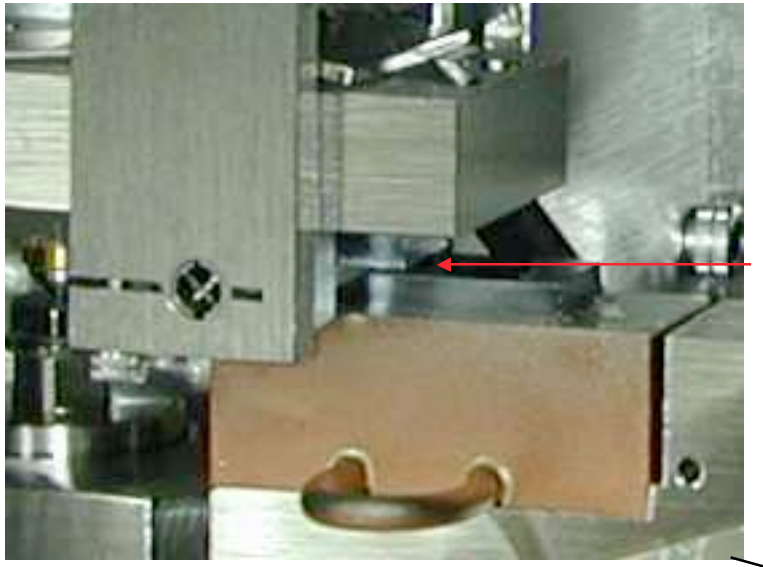
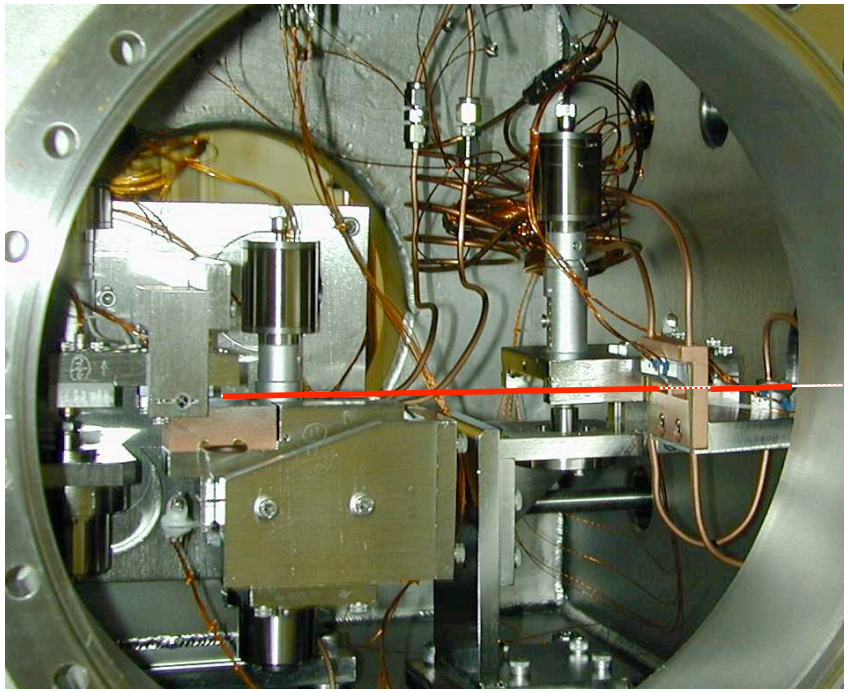
Beamline Layout



Monochromator & Step slit





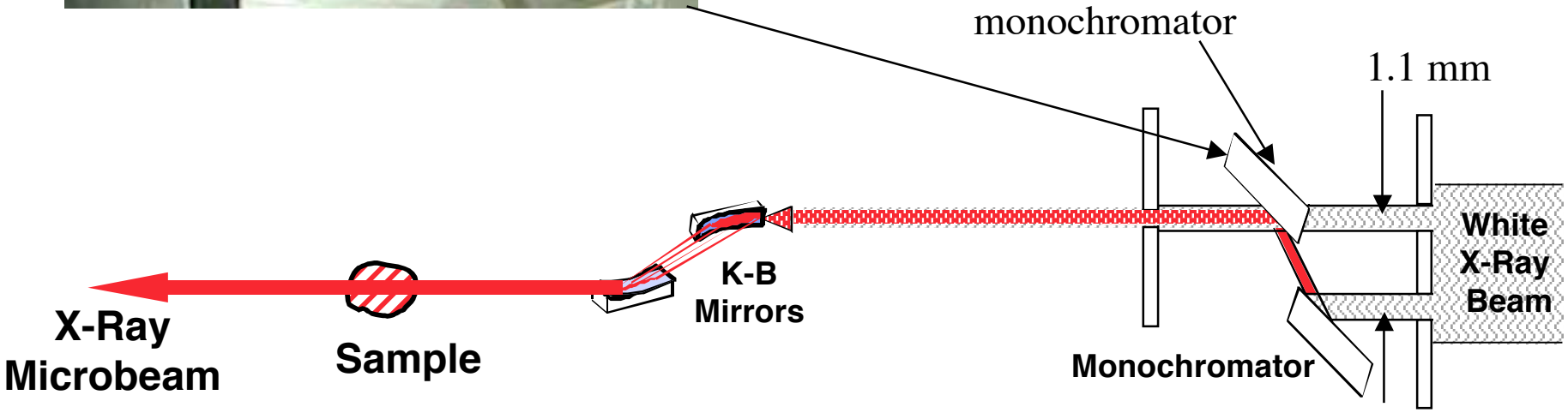


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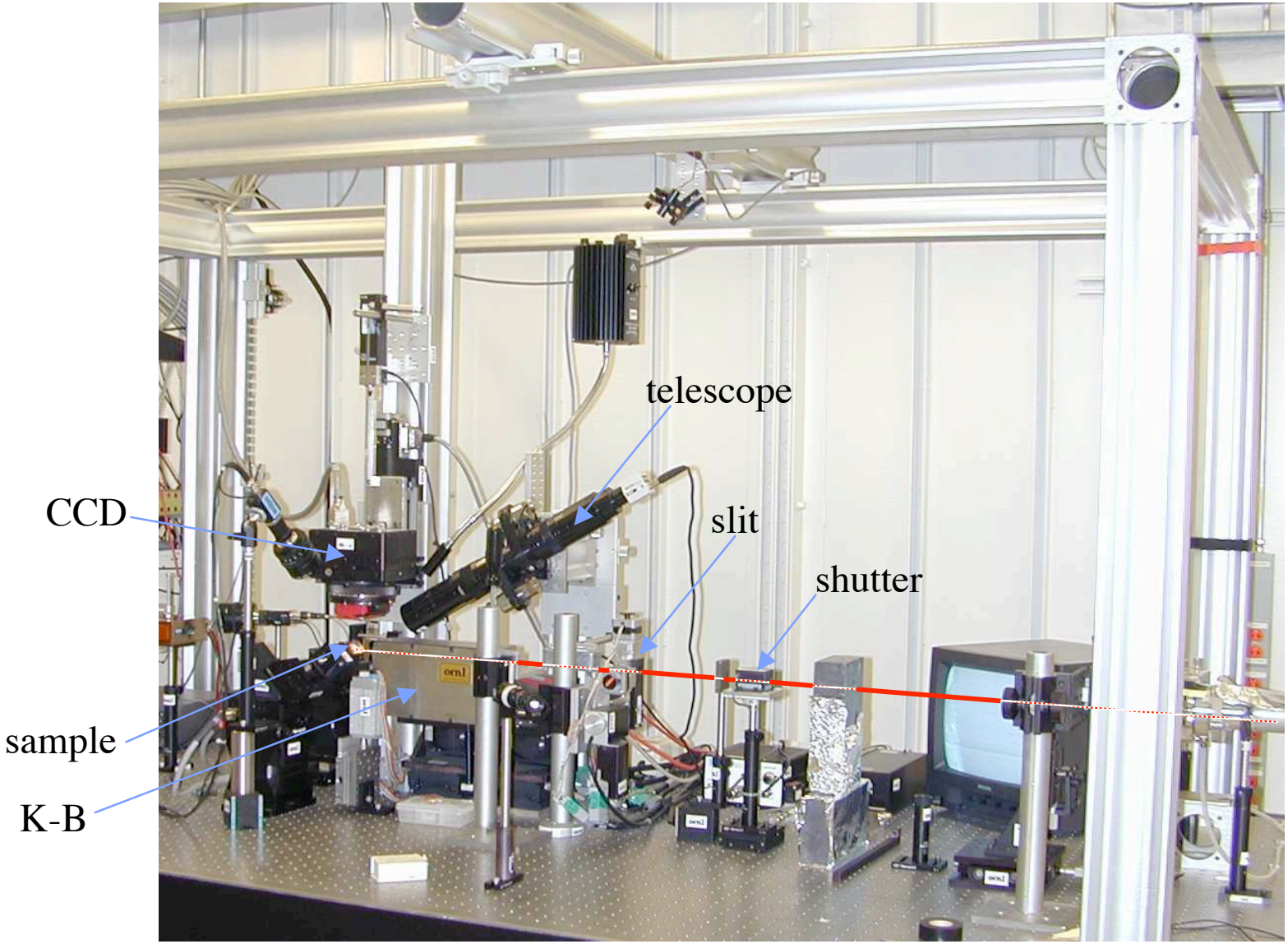
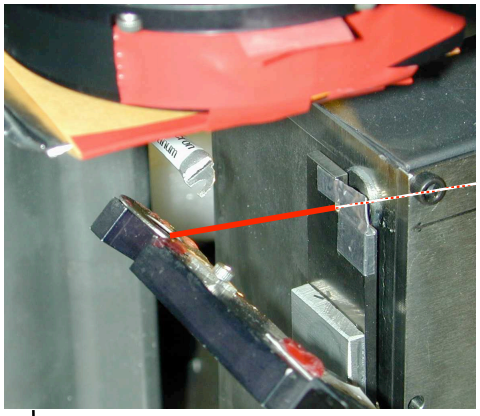
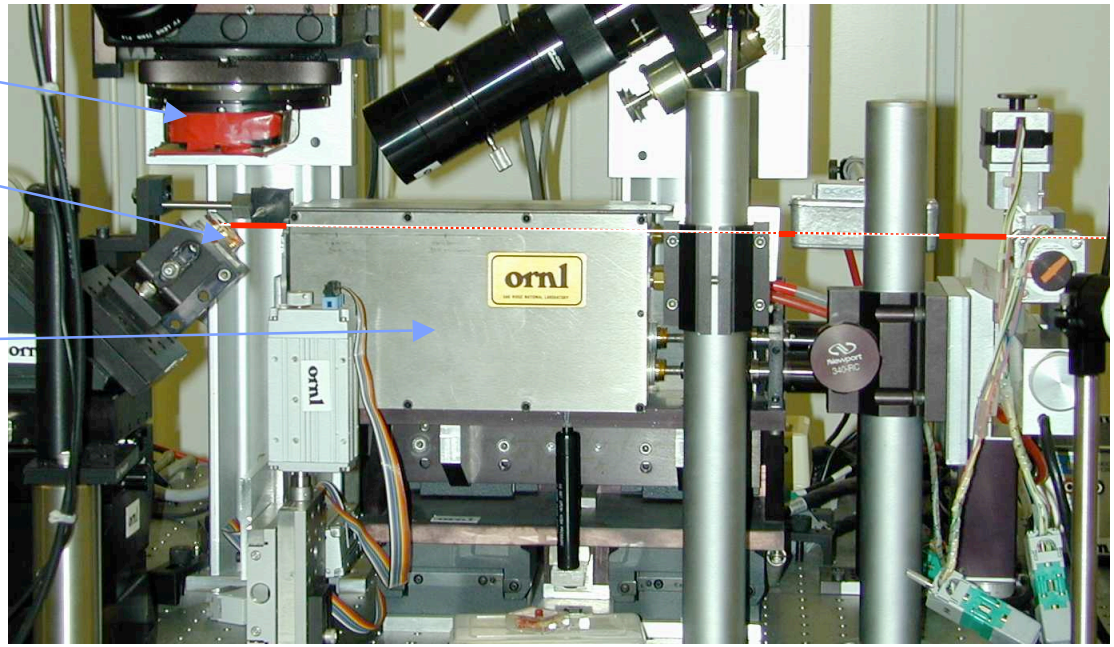
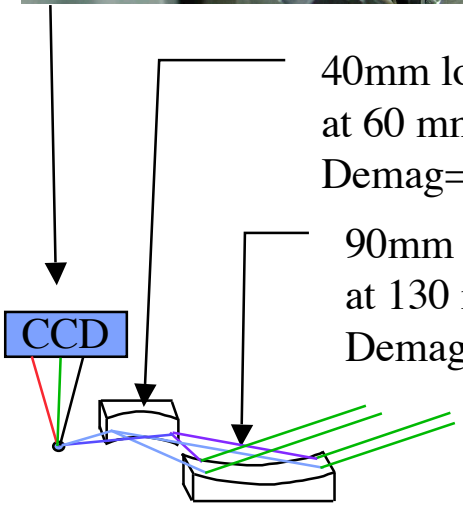
Table in end station, 34ID-E

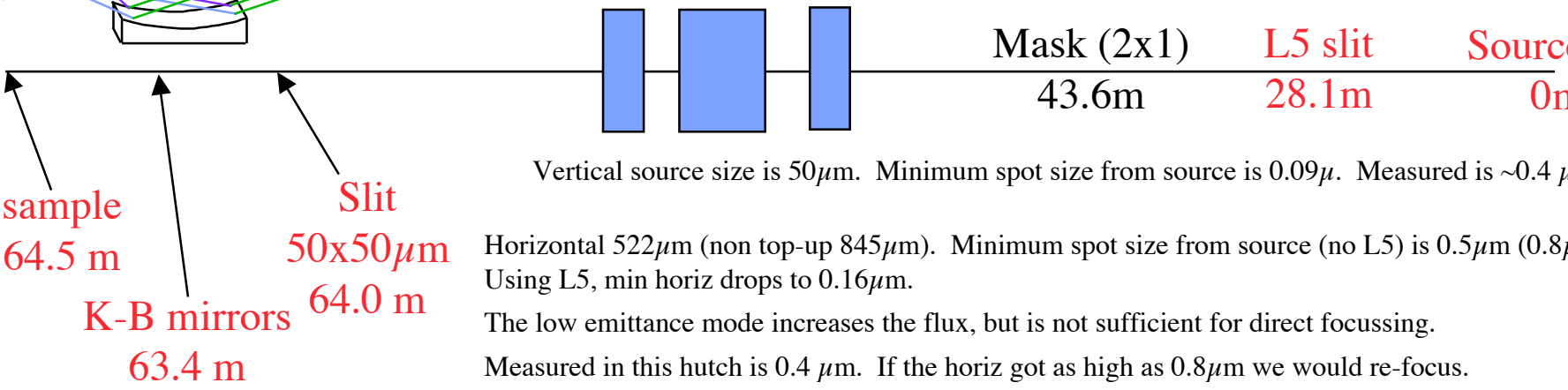


End near focus









Vertical source size is 50μm. Minimum spot size from source is 0.09μ. Measured is ~0.4 μm

Horizontal 522μm (non top-up 845μm). Minimum spot size from source (no L5) is 0.5μm (0.8μm). Using L5, min horiz drops to 0.16μm.

The low emittance mode increases the flux, but is not sufficient for direct focussing.

Measured in this hutch is 0.4 μm. If the horiz got as high as 0.8μm we would re-focus.

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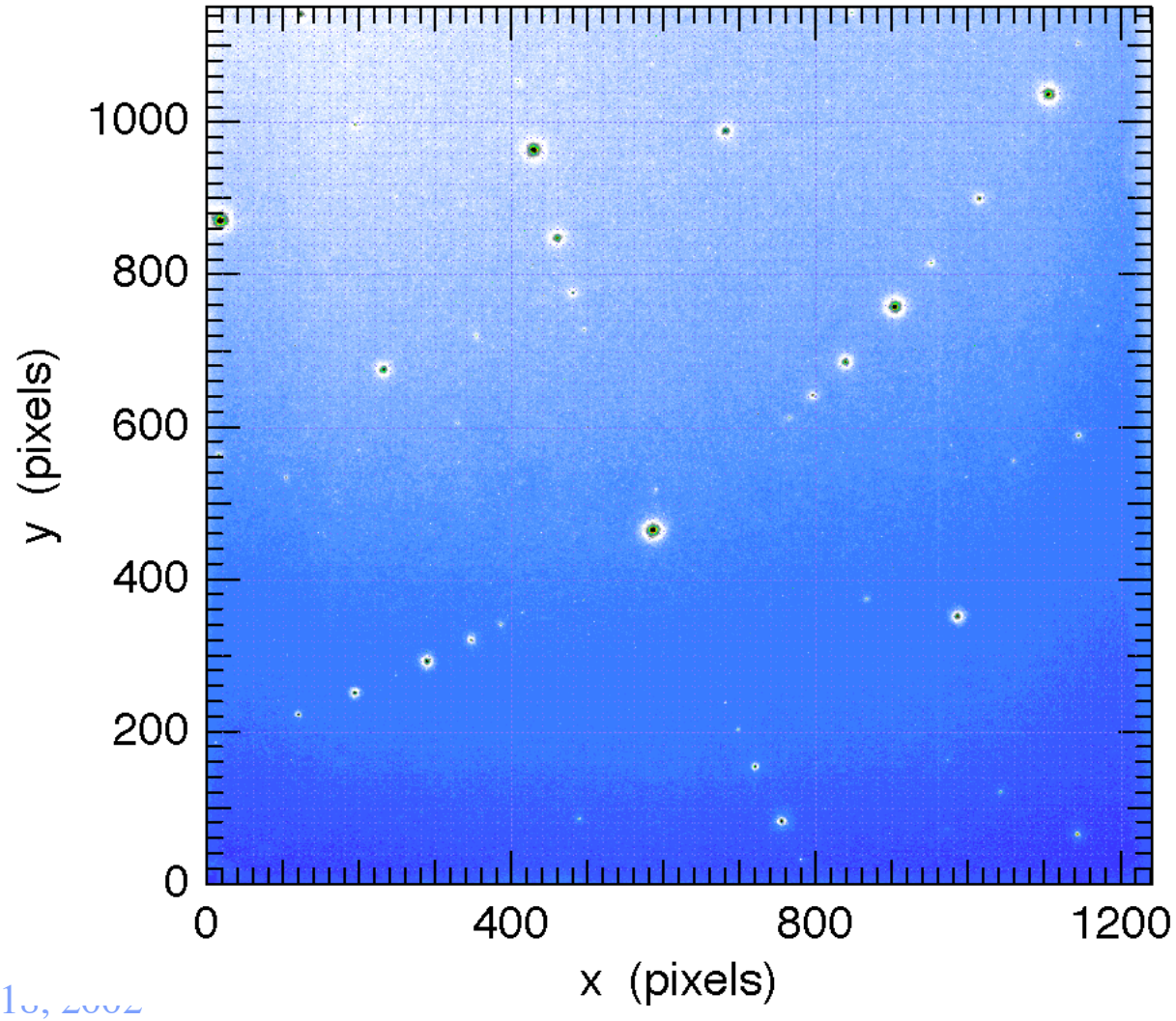
Simple Laue Pattern
Si (001) at 30 mm used for calibration



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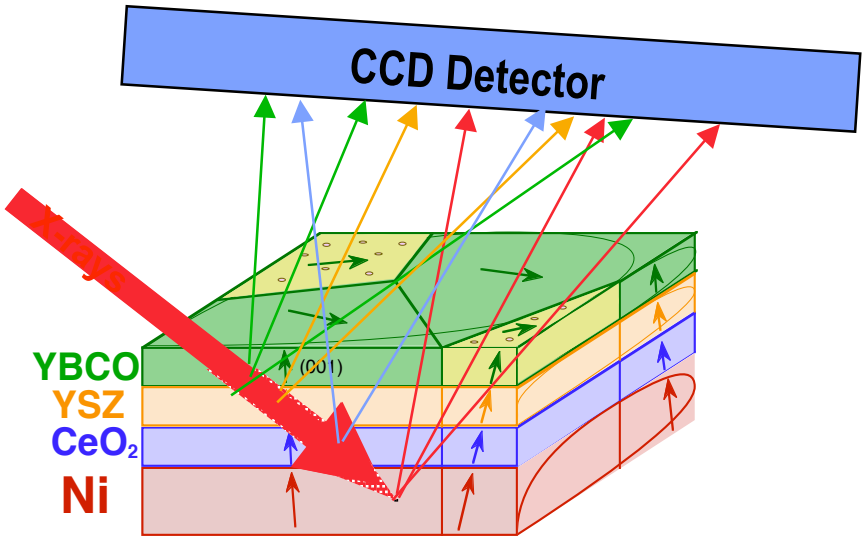
X-Ray Microprobe Study of Local Structure in RABiTS

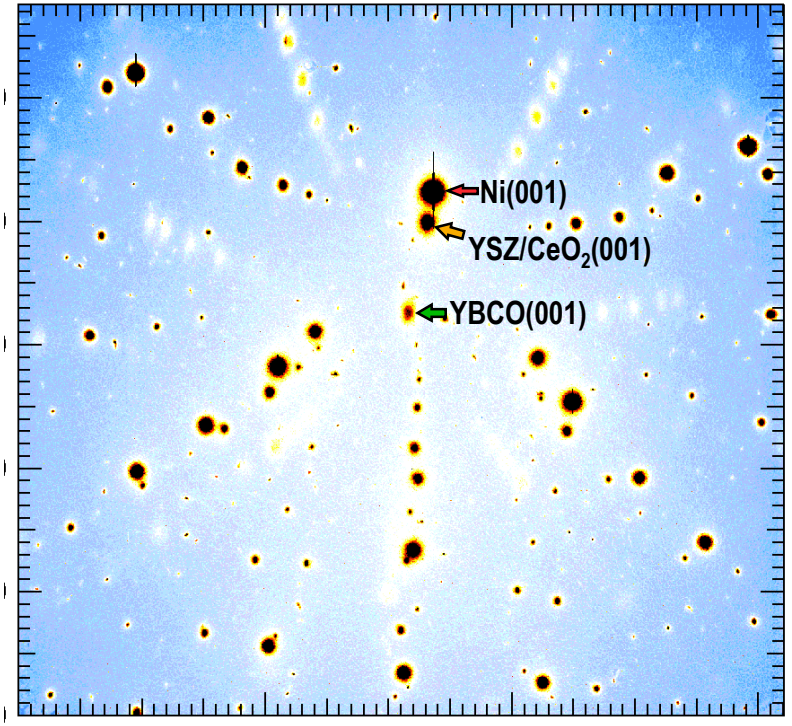
(Rolling Assisted Biaxially-Textured Substrates for High Current Superconductors)

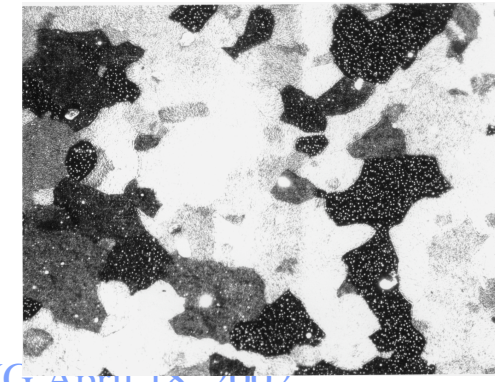


Cube-textured Ni by rolling & annealing
{001} <100>, ~7° Mosaic

Buffer oxides & YBCO deposited epitaxially

CCD Laue Pattern





Optical Photograph
~50 μm grains

Obtain crystallographic orientation and deviatoric strain of each layer with ~1μm spatial resolution.

Observe: Tilts of epitaxial layers.

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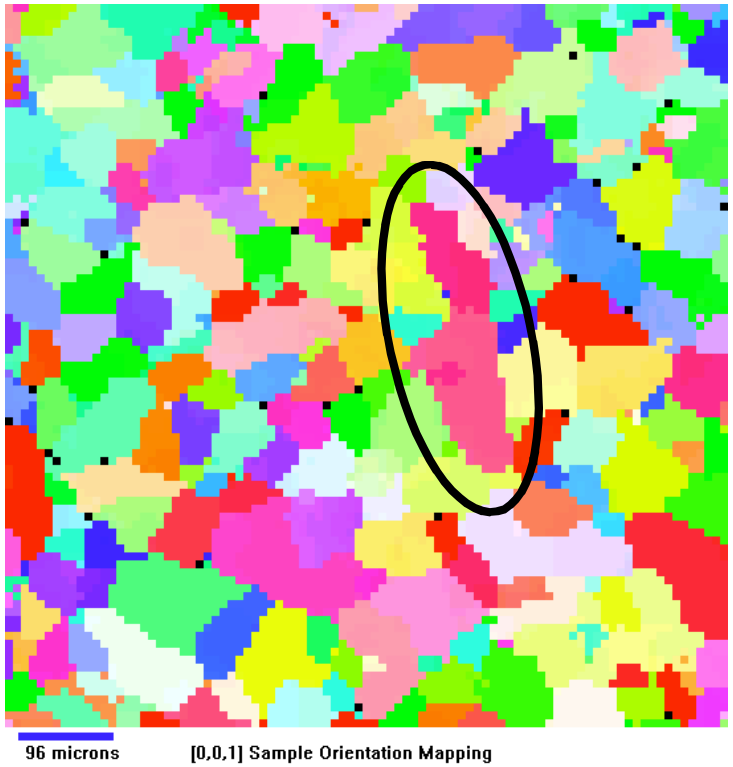
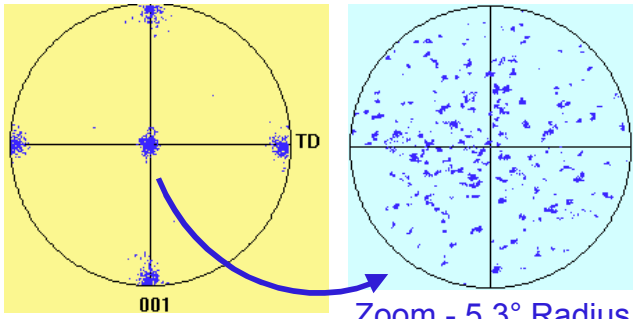
(001) Orientation Maps & Discrete Pole Figures of Multilayer Sample

Area = 0.72 x 0.72 mm; 8 □m steps; Raw images = 13.5 GB in 15 hrs.; 3.5 min for Ni + 110 min for film + 13 hours readout

Ni Substrate Orientation



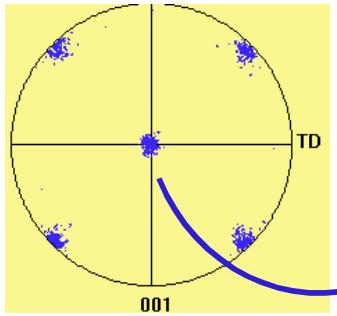
CeO_2 / YSZ / CeO_2 Film



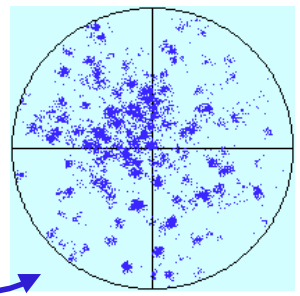
(001) Pole Figure
90° Radius

Zoom - 5.3° Radius

5.3°



(001) Pole Figure
90° Radius



Zoom - 5.3° Radius

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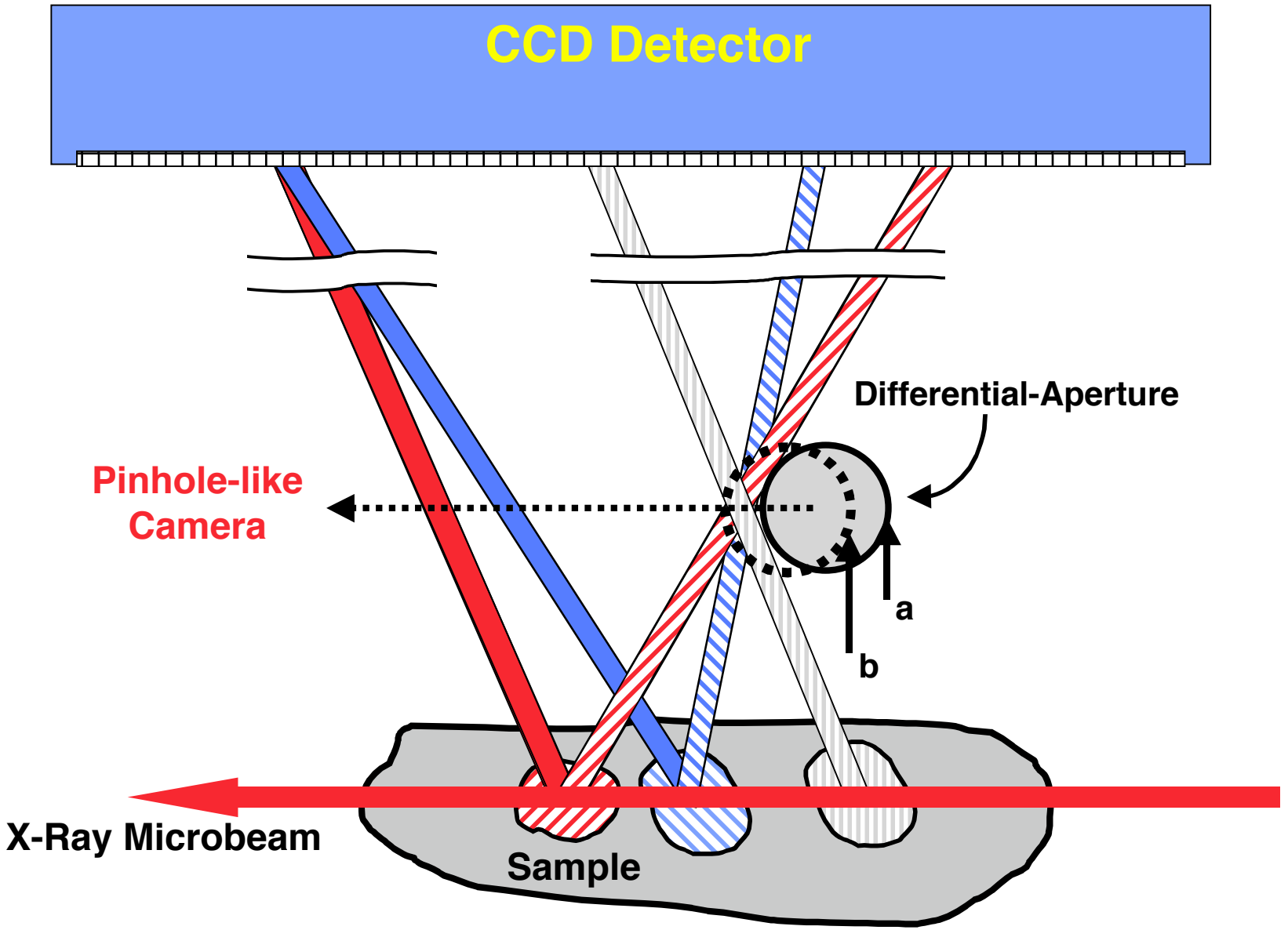
Multiple Grains, Overlapping Peaks → Full Inter- and Intra-Granular Diffraction Information with Micron 3D Resolution



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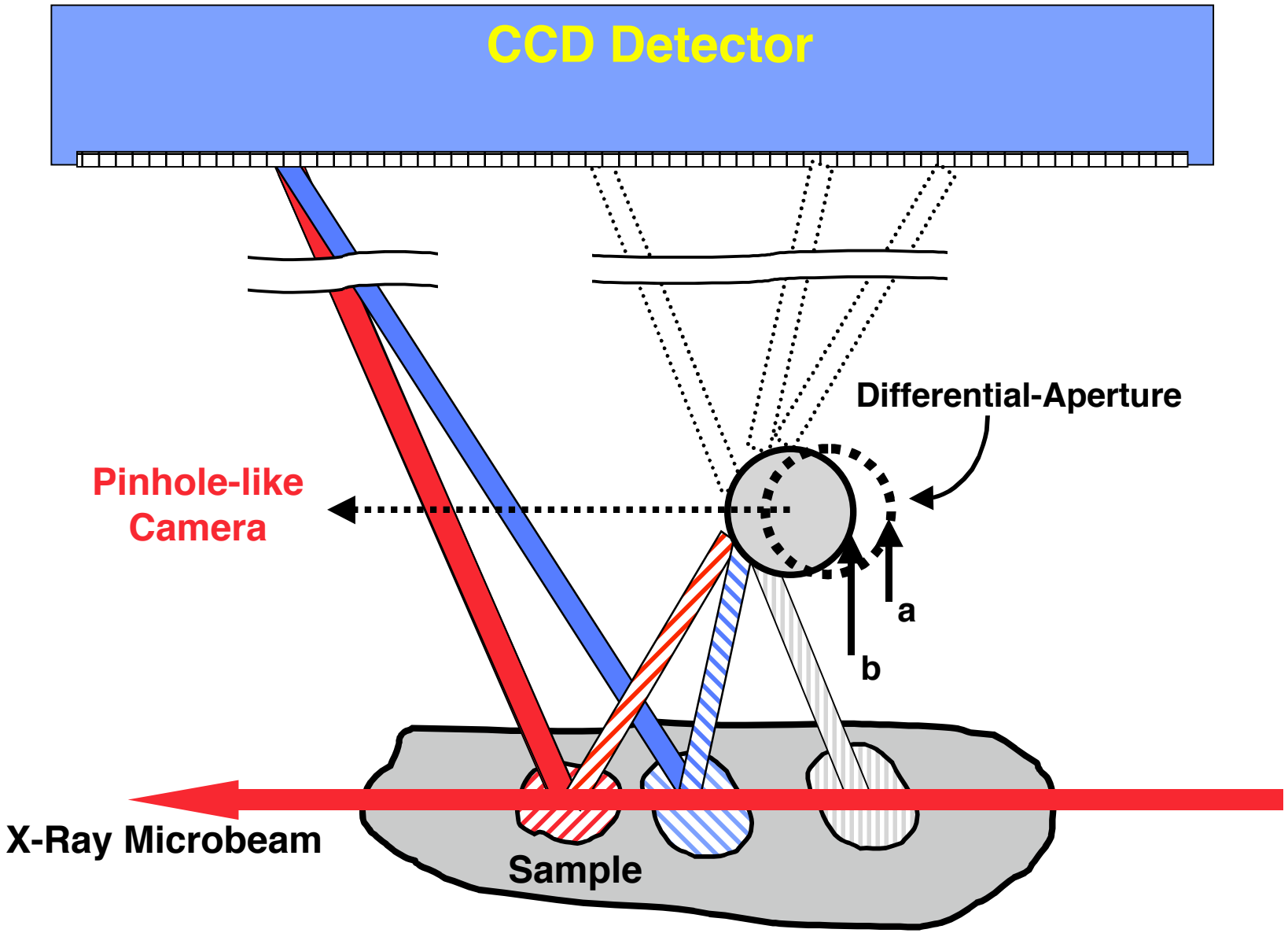
Multiple Grains, Overlapping Peaks → Full Inter- and Intra-Granular Diffraction Information with Micron 3D Resolution



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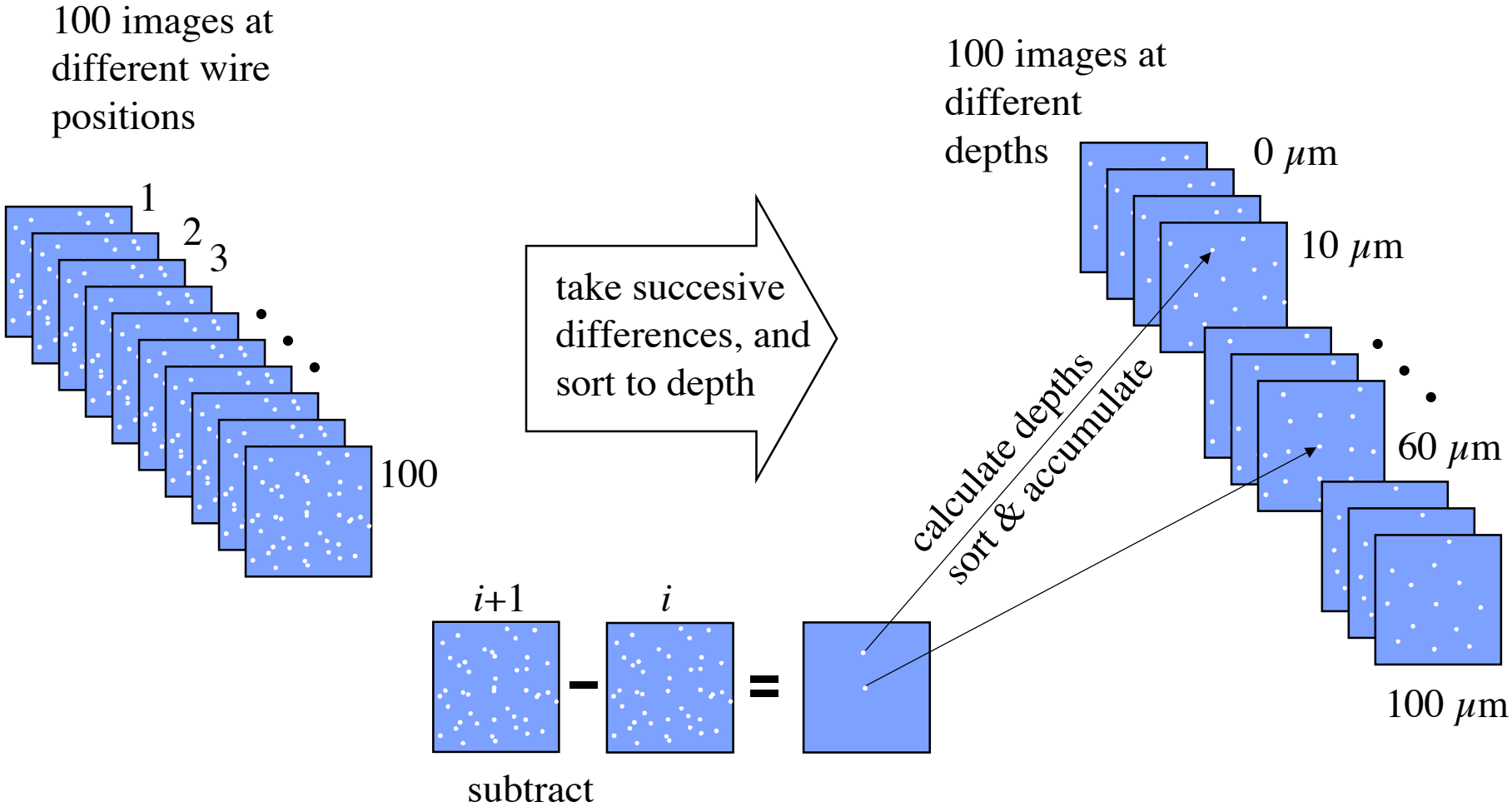
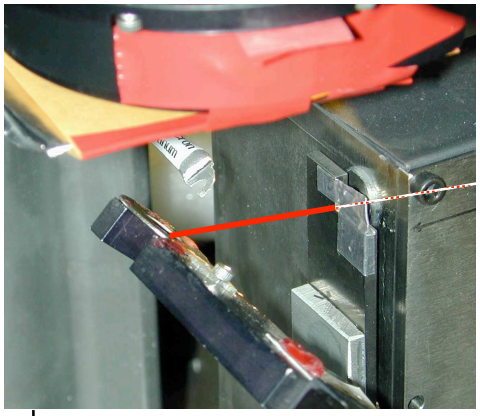
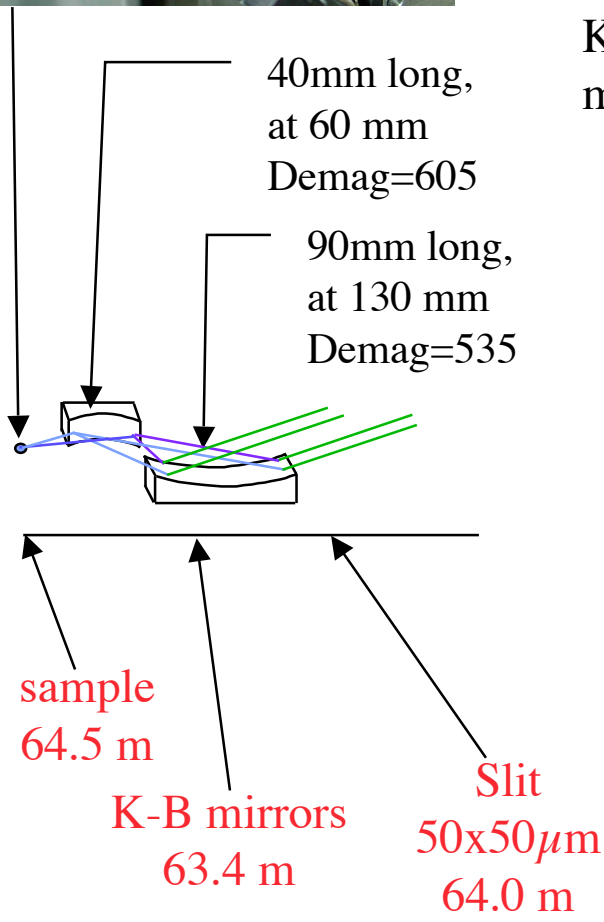
Sorting from wire -> depth



End near focus with wire

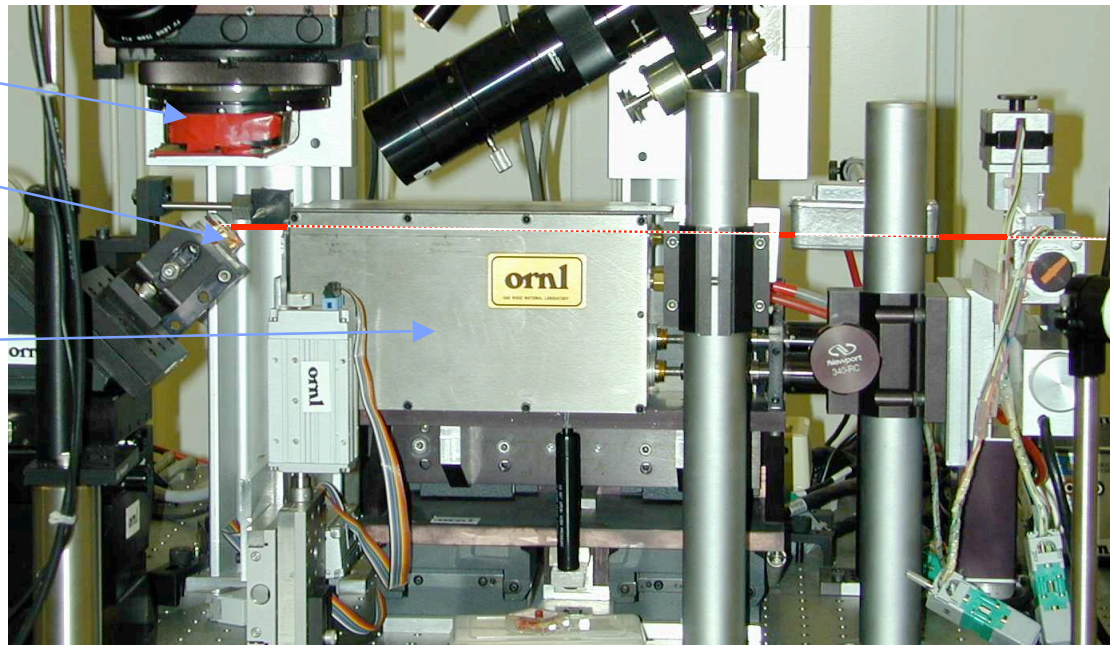




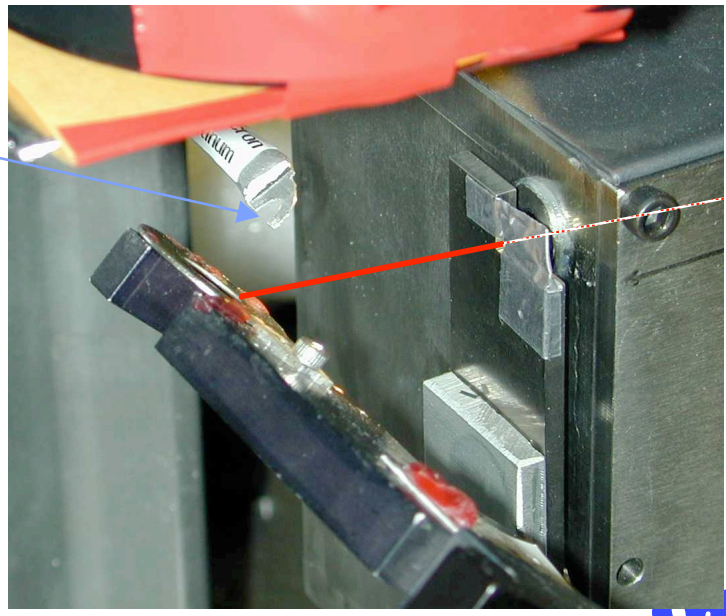
CCD

sample

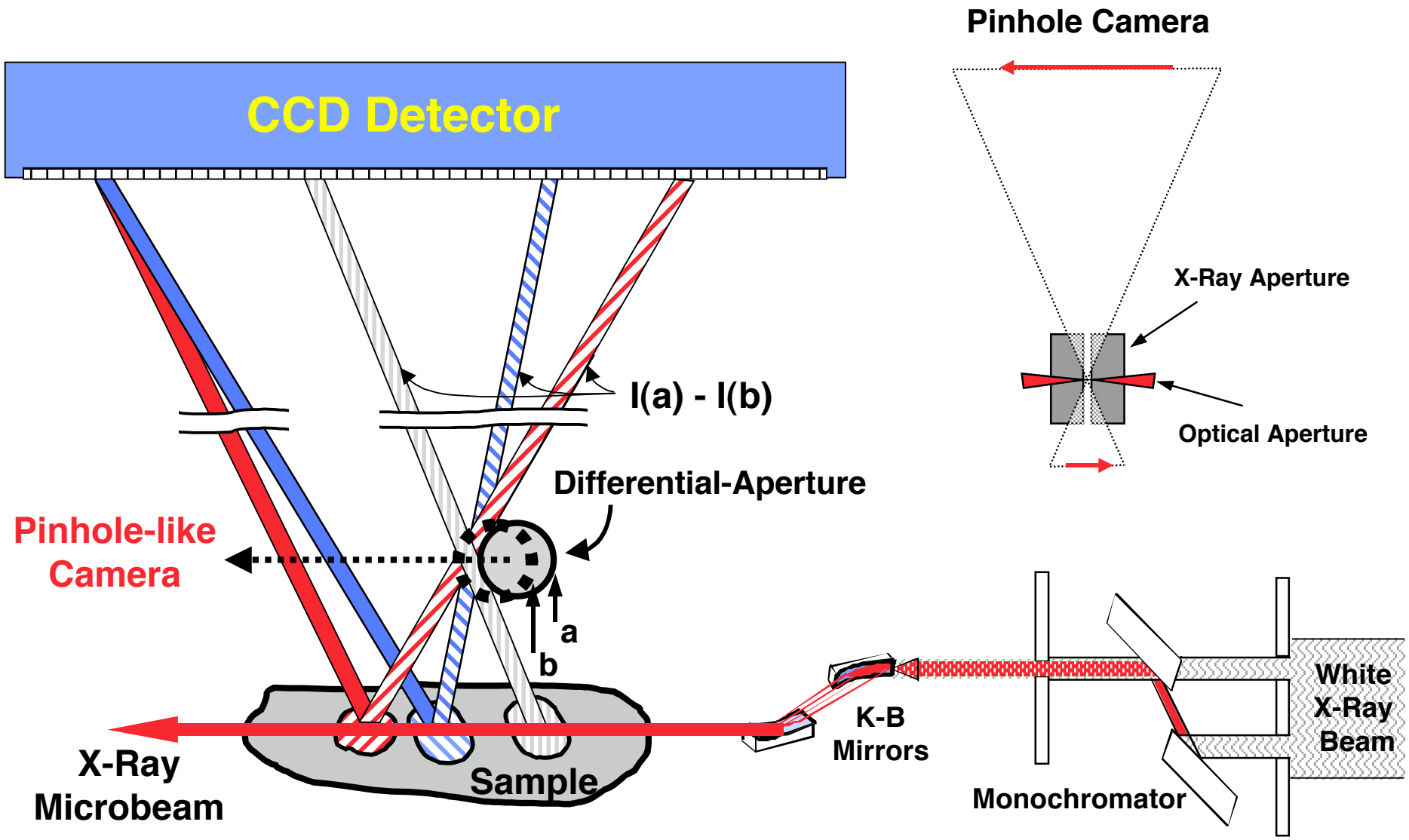
K-B mirrors



25 μm
Pt wire



3-D X-Ray Structural Microscopy Using Microbeams



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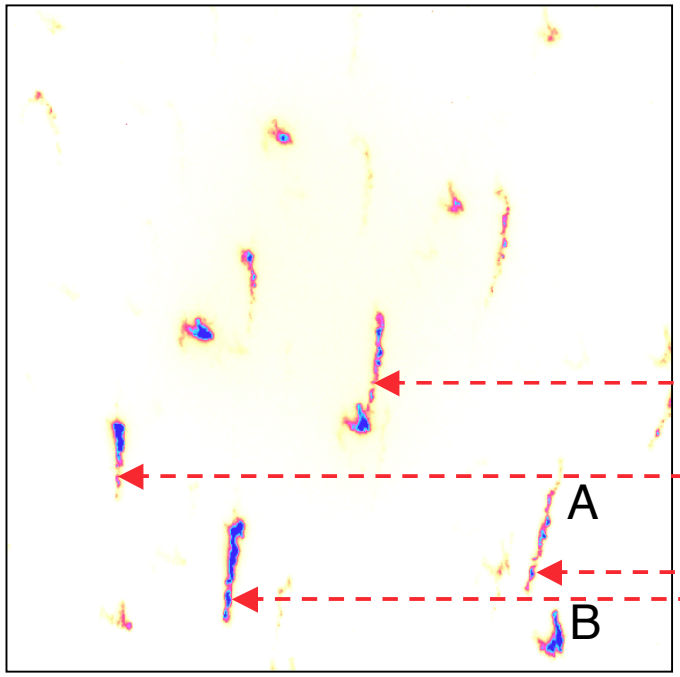
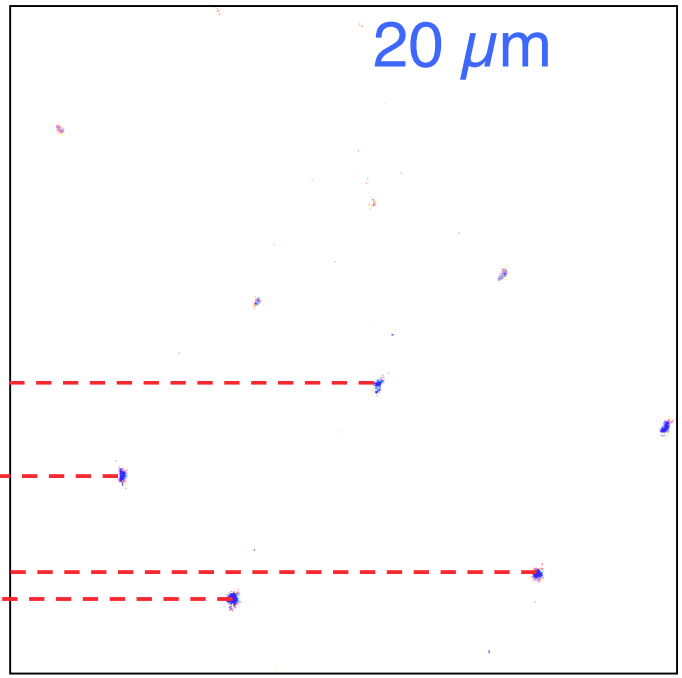
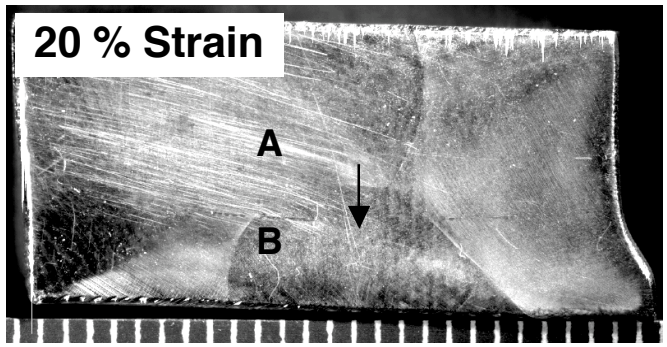
Depth Resolved Laue Pattern at G-B in Plastically Deformed Al

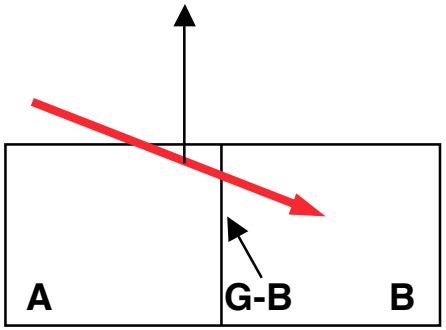
Grain-Boundary Laue Pattern

Depth Resolved Laue Pattern









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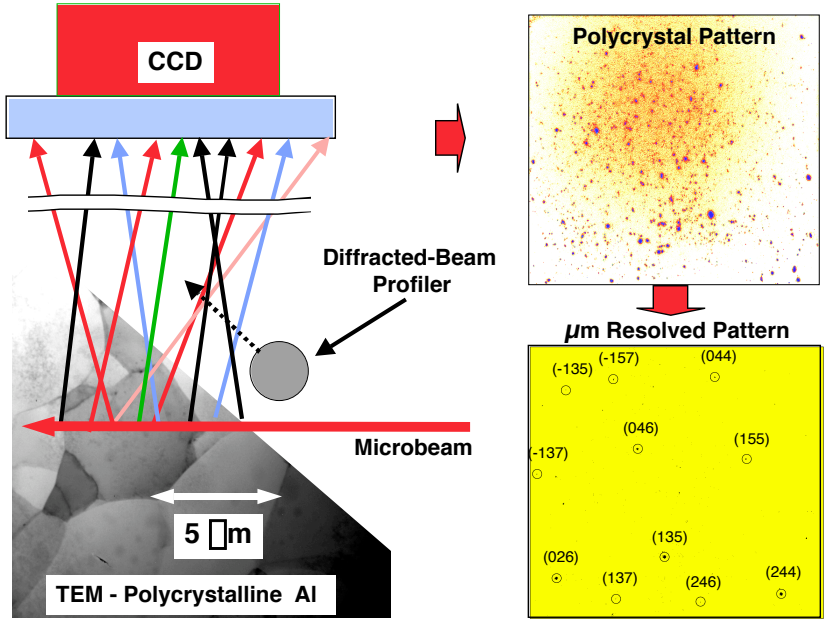
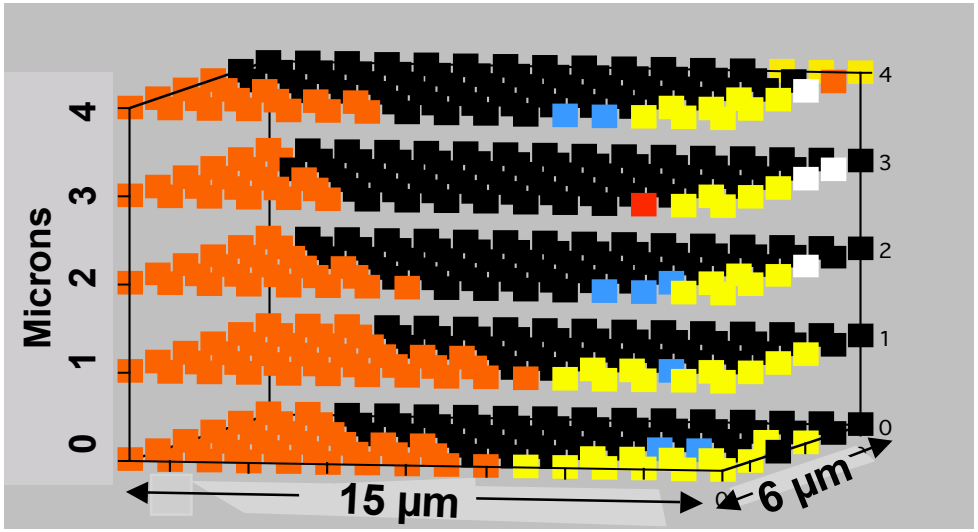
DAXM Measurement of Grain-Orientation and Size



Micron Resolution, Intra- and Inter-Granular Size and Orientation Mapping

3-D Grain Size and Orientation Mapping in Al



Colors Indicate Local Crystallographic Orientation with Micron Resolution

Differential-Aperture Depth Profiling

Depth Resolved Laue Diffraction from One-Micron Depth Interval

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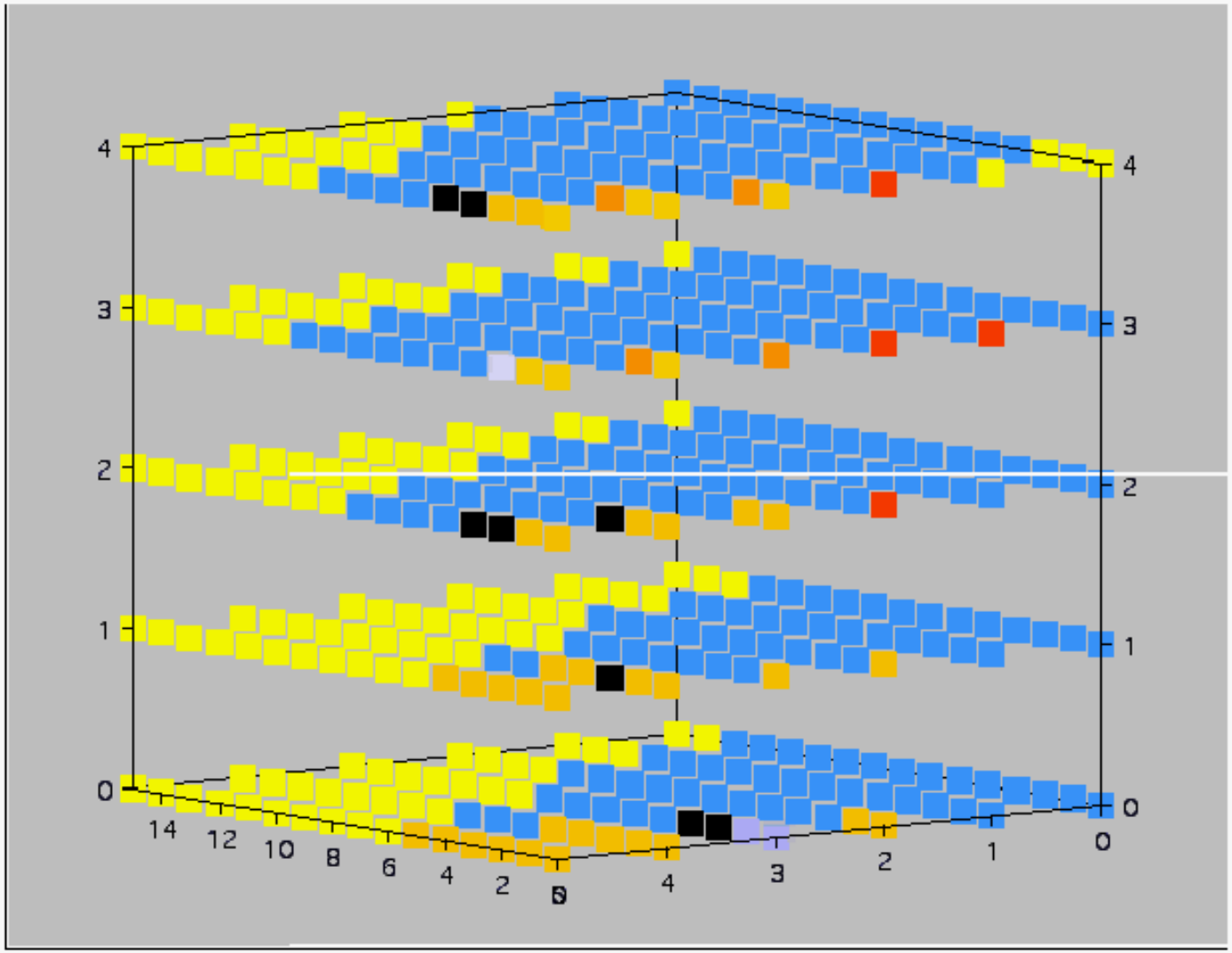
3-D grain of Aluminum



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Conclusions

3-D X-ray structural Microscopy

- **3-D structural microscopy works**
  - provides orientation, and strain, and structure too.
  - is NOT dependent upon perfect crystals, works for continuously strained material
- **2-D measurements provide clear answers on imperfect materials**
  - thin films, multi-layers with resolution better than 0.5 μm.
- **Resolution in depth is also sub-micron.**
  - makes identifying Laue pattern simpler.
- **Main limitation is quality of optics, 0.1μm white beams within reach.**

In General

- **New classes of submicron resolution x-ray studies now possible**
  - **Role of local stress, grain growth, combinatorial studies, segregation, fracture, dynamics, etc. …**
- **Improved optics, instrumentation and analysis techniques under development. Intrinsic limitations have not been approached.**

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