Development of Submicron Resolution 2D and 3D X-Ray Structural Microscopy

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Overview:

- 2D and 3D studies of materials on mesoscopic length-scales of tenths-to-hundreds of microns requires a penetrating structural probe with submicron point-to-point spatial resolution.
- Diffracted beam profiling of white beam Laue patterns from x-ray microbeams provides the basis of point-to-point 3D x-ray structural microscopy with submicron spatial resolution and high angular resolution [Larson, Yang, Ice, Budai, and Tischler *Nature* 415, 887 (2002)].
- The differential-aperture x-ray microscopy (DAXM) method is general and applicable to single crystal, polycrystalline, composite, deformed, and functionally-graded materials, etc.
- Non-destructive, micron-resolution, 2D and 3D measurement capabilities for crystal structure, orientation, strain and grain-size have been demonstrated using multilayered structures and polycrystalline aluminum.
- Monochromatic DAXM techniques have been developed as well that provide absolute lattice parameter measurements with 3D submicron spatial resolution [Yang, Larson, Ice, Tischler, Budai, Chung, Lowe, *Appl. Phys. Lett.* 82, 3856 (2003)].
- This capability provides a direct – and previously missing – link between *actual materials microstructure and evolution*, and increasingly powerful numerical simulations and multi-scale modeling of the structure and dynamics of materials on mesoscopic length scales.

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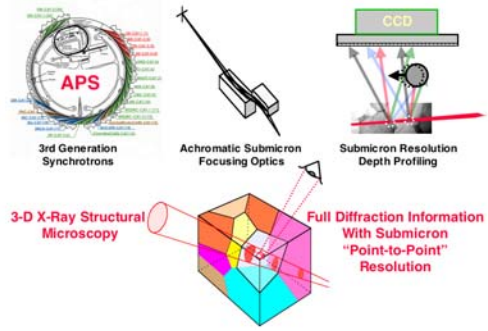
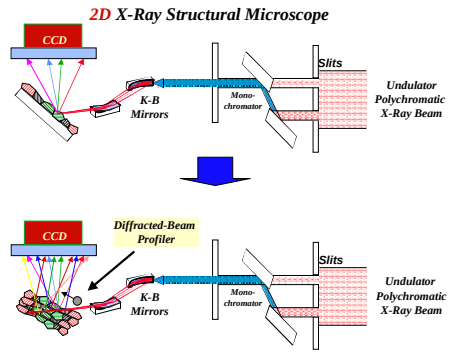
New Oportunities in Mesoscale Materials Physics Using High-Brilliance Sources, White Beams, Achromatic Optics, and Depth Profiling

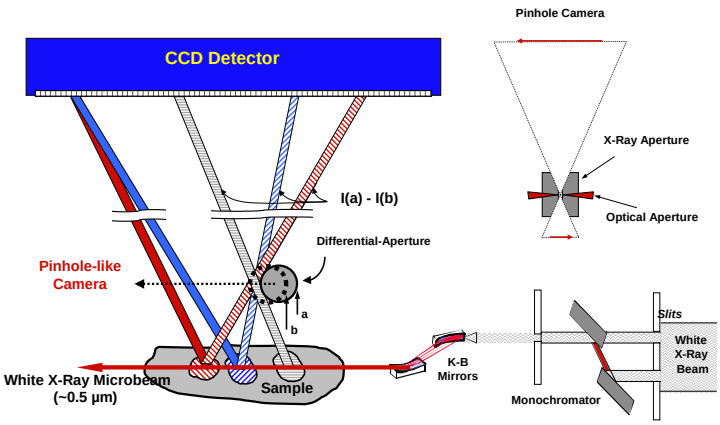


X-Ray Structural Microscopy Using Microbeams



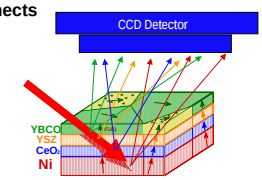
Multiple Grains, Overlapping Peaks ➡ Full Inter- and Intra-Granular Diffraction Information with Micron 3D Resolution

Submicron Resolution Differential-Aperture Depth Profiling



Examples of 2D Mesoscale Science Studies:

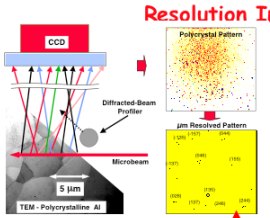
- Electromigration and Deformation in IC Interconnects
- Al_2O_3 Thermal Barrier Coatings
- Epitaxial growth of oxide films



X-Rays Compared with EBSD:

- Buried layers as well as surface
- Higher angular resolution (~0.01°)

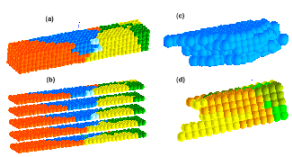
Differential-Aperture X-Ray Microscopy (DAXM) Measurement of Local Crystal Structure With Submicron Resolution In Three-Dimensions



Differential-Aperture Depth Profiling

Depth Resolved Laue Diffraction Pattern from One-Micron Depth Interval

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



Colors Indicate Local Crystallographic Orientation with Micron Resolution

Grain-Orientation and Morphology in Polycrystalline Al

Combinatorial Study of Epitaxial Growth and Crystal Tilts in Oxide Films on Textured Metals

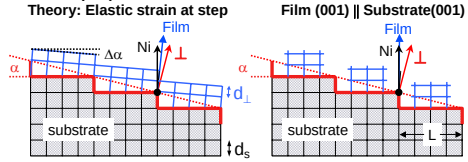
[Budai, Yang, Tamura, Chung, Tischler, Larson, Ice, Park and Norton, *Nature Materials* **2**, 487 (2003)]

PLD growth of CeO_2/Ni buffer layers (RABiTS - superconducting uses).

Growth Modes Change with SubstrateTemperature

High Temperature (> 600°C)
Ledge Growth
Film (001) tilts toward ⊥
Theory: Elastic strain at step

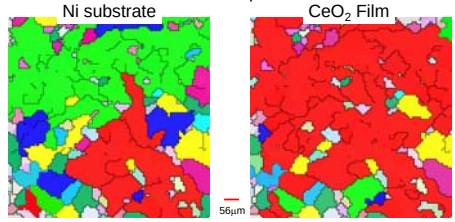
Low Temperature (< 500°C)
Island Growth
Suppressed Kinetics
Film (001) || Substrate(001)



Crystallographic Tilts of Film Leads to Enhanced Texture &Percolation

Percolation Orientation Maps: Boundaries > 5°

Ni substrate | CeO_2 Film



Percolation threshold = 5.2° Percolation threshold = 4.4°

Tilts ⇒ Improved Texture ⇒ Enhanced J_c