X-Ray Diffraction (XRD) and Electron Backscatter Diffraction (EBSD) Combinatorial Measurements

Mark D. Vaudin

NIST Ceramics Division

NCMC-5

Monday, April 26th, 4:20 p.m.

Combinatorial XRD

- **X-Ray Diffraction**
- Gives detailed, semi-quantitative, phase information (and crystal texture)
- Modern instruments have motorized stages, small intense x-ray beams, high speed area detectors
- Clear potential for Combi approach

Combinatorial EBSD

- **<u>E</u>lectron <u>B</u>ack <u>S</u>catter <u>D</u>iffraction**
- Gives phase information, crystal orientation
- Technique performed in Scanning Electron Microscope at fast scan rates
- Clear potential for Combi approach

Availability at NIST

- XRD: currently purchasing Bruker D8 with area detector, X,Y,Z stage, focussing optics (10 / 2004)
- EBSD: 2 HKL systems at NIST in MSEL (now)
  - JEOL 6400: LaB_6 source
    - medium EBSD resolution ≈ 500 nm
  - Hitachi 4700: cold field emission source
    - high EBSD resolution ≈ 20 nm
- EBSD: 1 Noran PhaseID system in CSTL (now)
  - Hitachi 4500: cold field emission source (John Small)
    - high EBSD resolution ≈ 50 nm

Specimen Requirements

- XRD
  - minimal surface prep required
  - up to 75 mm wafer easily accommodated on vacuum chuck

- EBSD
  - flat diffracting surface
  - colloidal silica often gives good diffraction
  - up to 75 mm wafer easily accommodated

Combi data from XRD

- Phase identification
  - identifies unknown phases
  - can obtain data from predetermined positions on specimen
- Crystal orientation and texture
- Resolution: we are hoping for 200 nm
- Note: better resolution require much longer data acquisition times because:
  - 1) more data points collected
  - 2) intensity lower

Rapid analysis of ternary phase diagrams using synchrotron radiation

E.D. Specht[1], A. Rar[1,2], G.M. Pharr[1,2], E.P. George[1,2], P. Zschack[3], H. Hong[3], and J. Ilavsky[4]

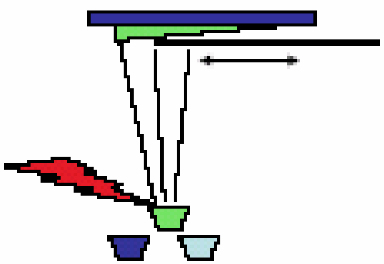
[1] Metals and Ceramics Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee

[2] Dept. of Materials Science and Engineering, University of Tennessee, Knoxville, Tennessee

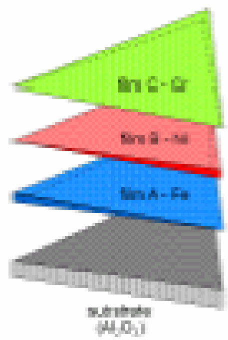
[3] Frederick Seitz Materials Research Laboratory, University of Illinois at Urbana-Champaign

[4] University of Maryland, College Park, Maryland

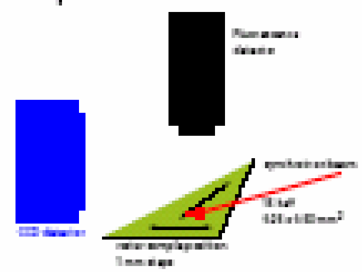
Sliding shutters are used to deposit by e-beam three layers with graded thickness



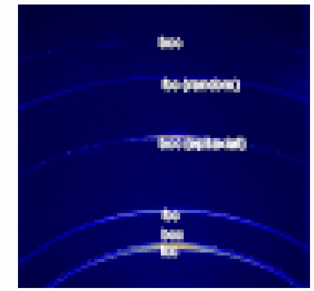
Sample is heated to interdiffuse layers



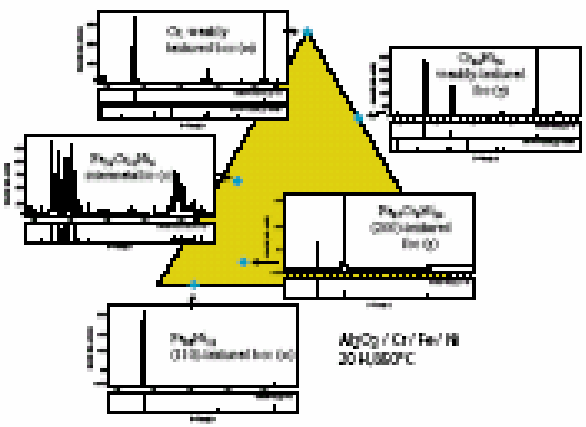
X-ray diffraction and fluorescence are measured from 2600 positions in 4 hours



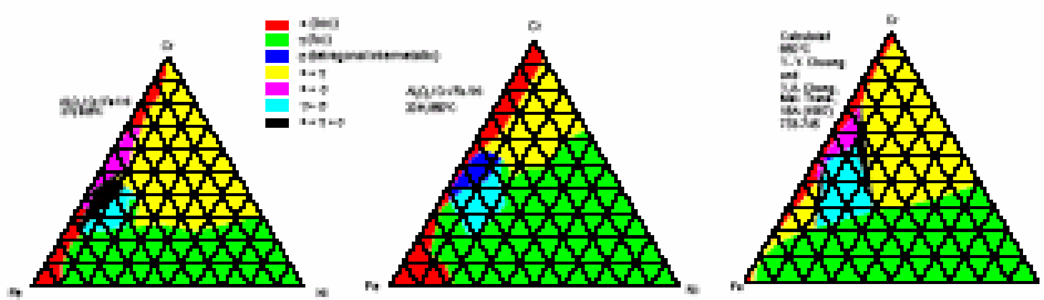
CCD collects diffraction from epitaxial and randomly oriented phases



Phases are identified from integrated CCD images



Composition from fluorescence and phase from diffraction gives ternary diagrams



under-annealed: single-phase σ has not developed

partially oxidized: formation of Cr_2O_3 is shifting phase boundaries toward Cr

Calculated diagram

Conclusions

Synchrotron radiation provides rapid characterization of ternary phase diagrams grown by vapor deposition.

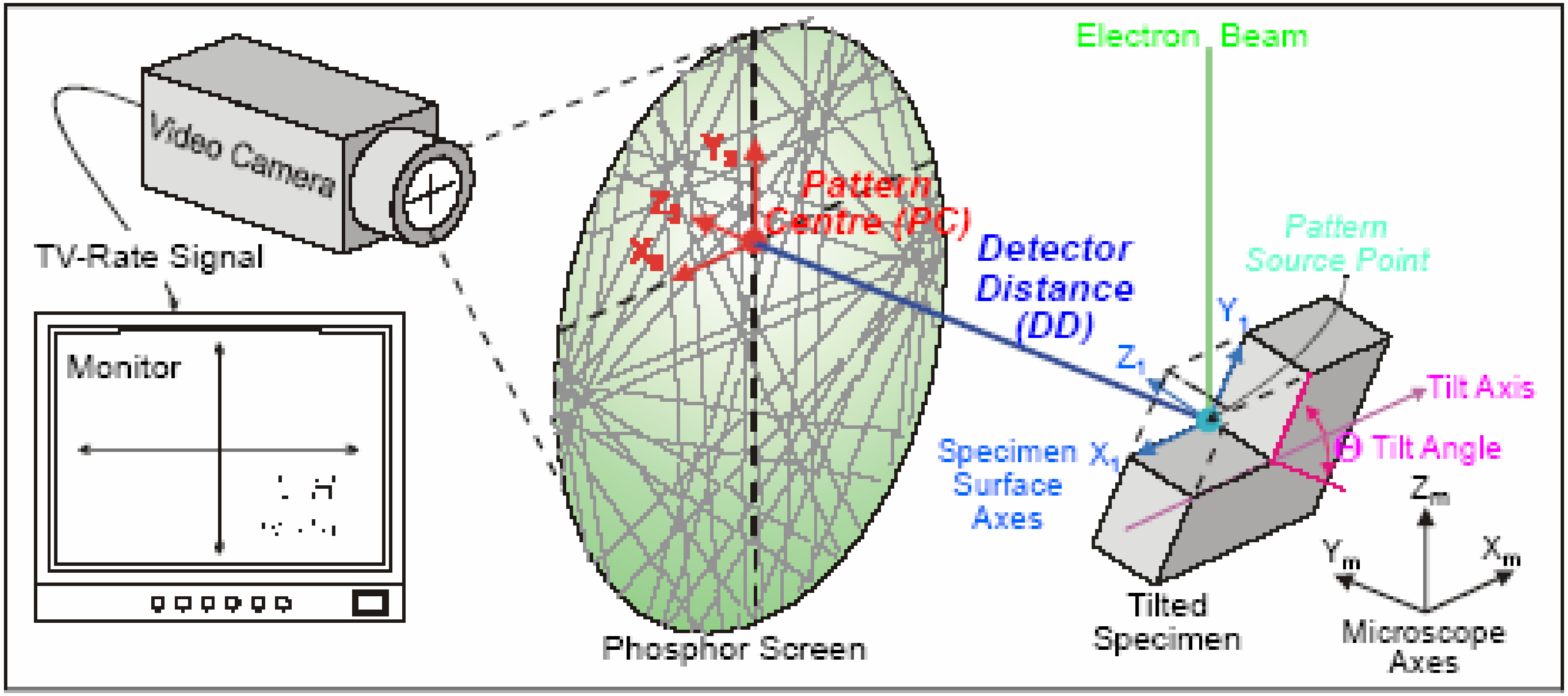
Techniques must be developed for rapid interdiffusion of the deposited layers without oxidation or reaction with the substrate.

Possibilities include melting in patterned substrates and localized heating by lasers, IR, or e-beams.

- Essentially identical experiments can be conducted on in-house XRD equipment
  - Phase diagrams
  - Crystal structure/texture vs composition
- In-house experiment will be slower and have larger step-size
- However, more in-house beam time is available !
- Uni of MD is successfully using a similar tool
- Talk: “Combinatorial exploration of functional materials using thin-film techniques”, Ichiro Takeuchi
- Ongoing collaborations

EBSD Data Collection Method

HKL Technology EBSD detector installed in SEM with a field emission gun.



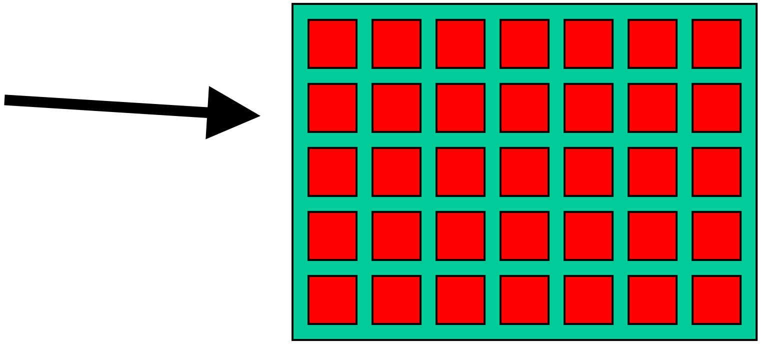
Each point interrogated produces a unique Kikuchi diffraction pattern.

Data available from EBSD

- Phase identification
  - distinguishes between known phases
  - (with EDS, can identify unknowns)
- Can determine phase volume fraction in scanned area
- Also, orientation and texture
  - orientation maps
  - most common use to date

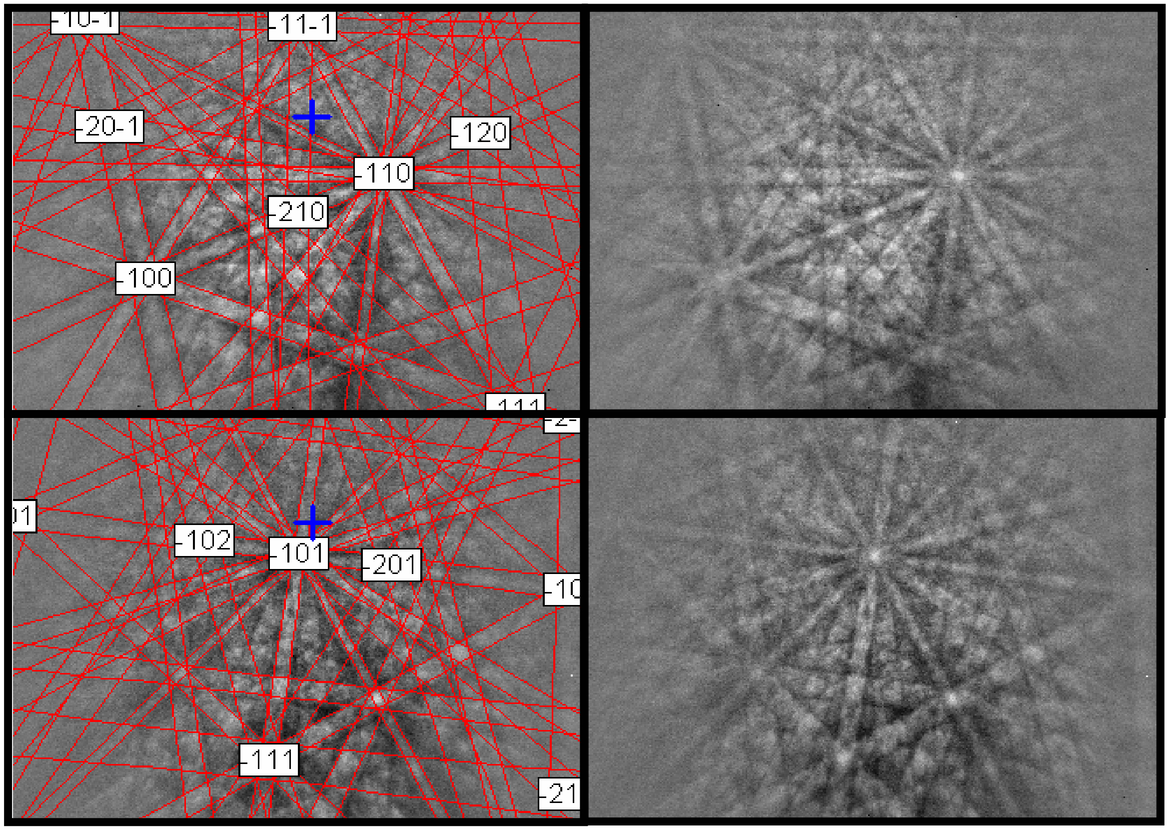
Combi data from EBSD

- Do scans at a grid of positions on specimen



- Scan speeds of 20 patterns per second are becoming routine (record is > 90 sec^{-1})
- Scan resolution: down to 20 nm on highest resolution machine, 200 nm on LaB_6 machine
- Scan grid completely flexible

Indexing EBSD Patterns

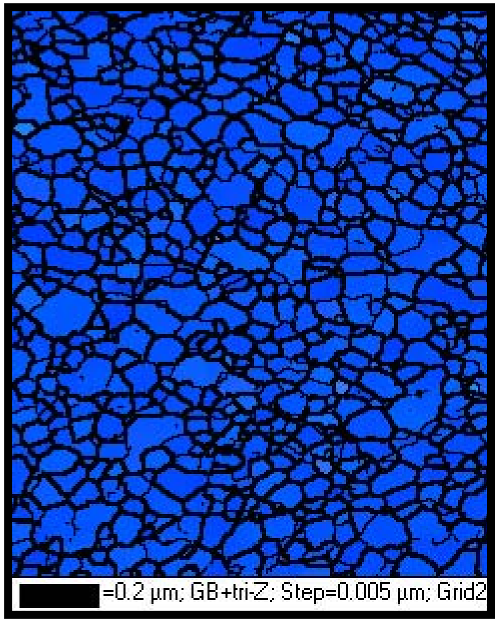


- Kikuchi diffraction patterns collected from individual points.
- Incident electron beam is step through a matrix along the sample surface.
- Each Kikuchi diffraction pattern was indexed according to the FCC structure.
- Euler angles determined for each point.

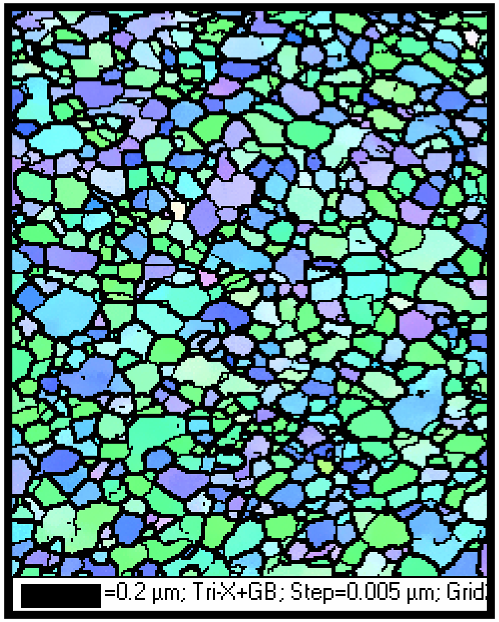
Example EBSD patterns taken from the individual grains of a Pt thin film.

Pt Texture Mapping

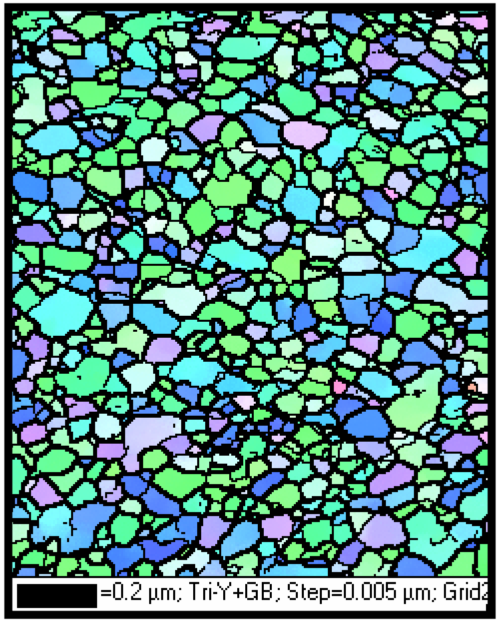
Z- Map



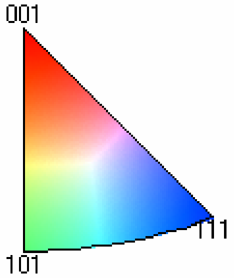
X- Map



Y- Map



Zero solution reduction: 1 point nearest neighbor fill.



Pt exhibits a <111> fiber texture normal to the plane of the film and in plane orientations distributed through 360°.

Conclusions

- XRD and EBSD can both be used in scanning mode = combinatorial potential
- Techniques provide phase and crystallographic information
- XRD has proven track record both at synchrotrons and on conventional machines
- EBSD has been used less but has great potential