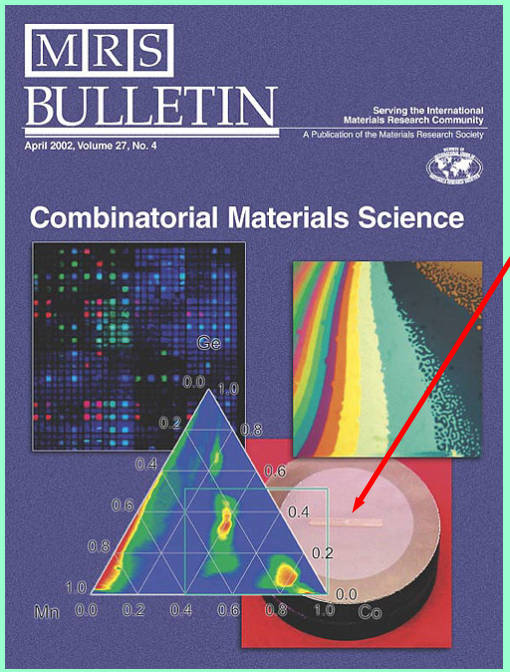
Development of Combinatorial Screening Tools for Inorganic Materials

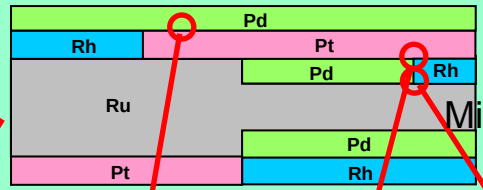
J.-C. Zhao
GE Global Research

Outline

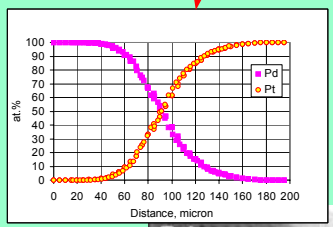
- The diffusion-multiple approach
- Screening tools for various properties
- Summary

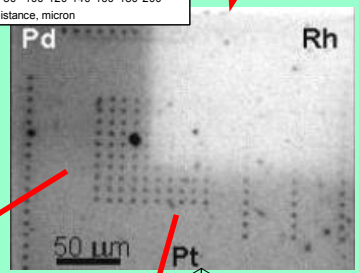
Diffusion-Multiple Approach: Introduction

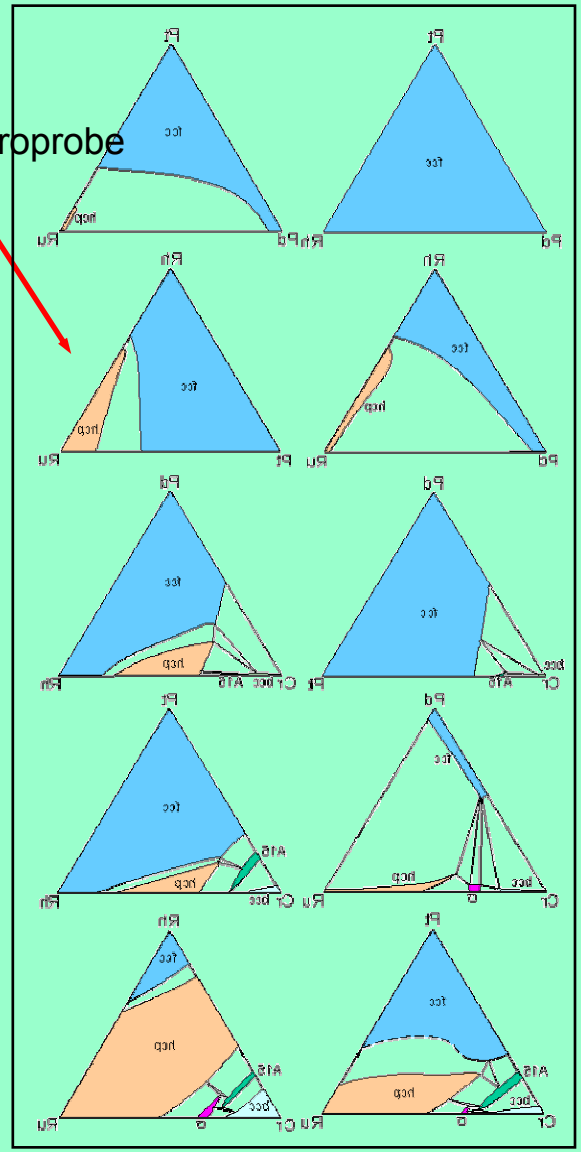




Microprobe

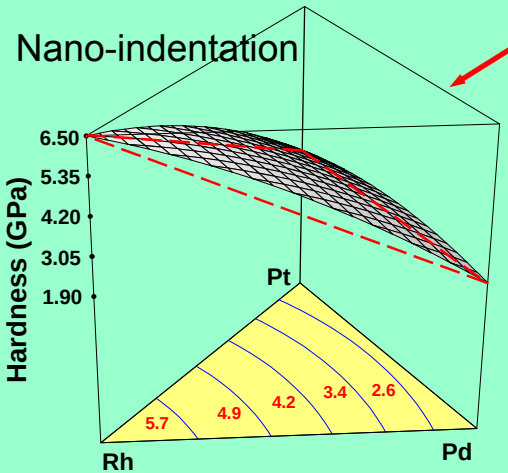


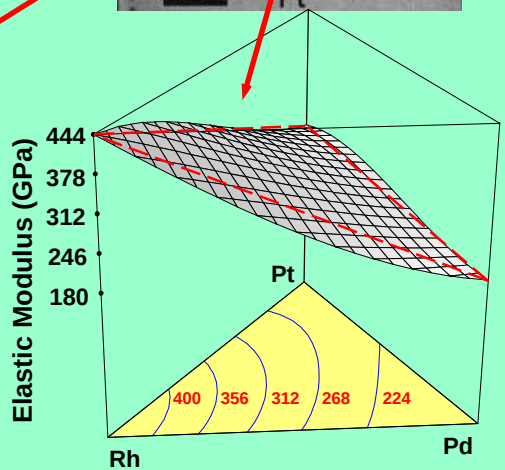




10 Phase diagrams from a single sample

Nano-indentation



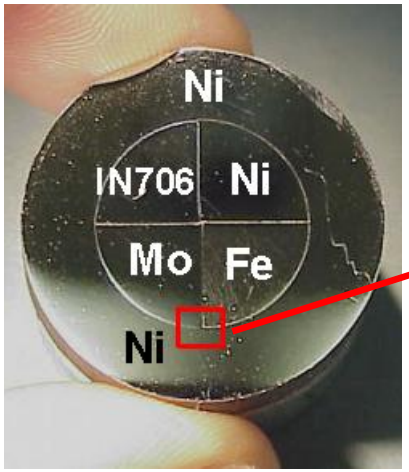


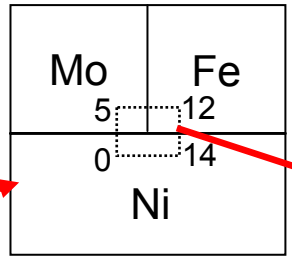
Various Properties to be Screened:

1. thermodynamic properties (ΔH, etc.)
2. thermal expansion coefficient
3. thermal conductivity
4. ductility
5. compressive yield strength
6. magnetic properties
7. melting point
8. lattice parameter variation with composition
9. optical properties
10. electrical conductivity
11. phase diagrams
12. crystal structure
13. elastic modulus
14. hardness
15. diffusion coefficient
16. Band gap
17. Thermoelectric properties, etc.

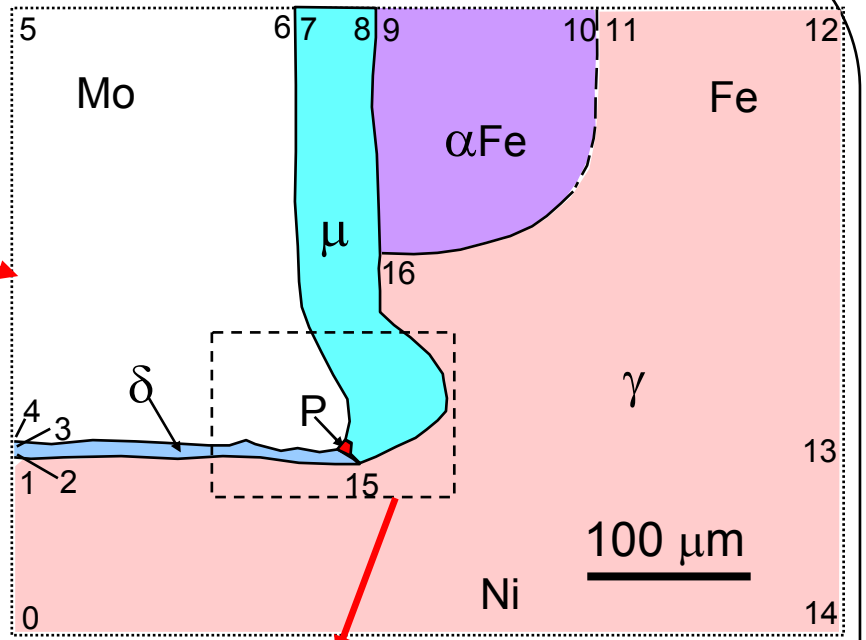
Green = fully developed
Blue = partially developed
Red = not developed

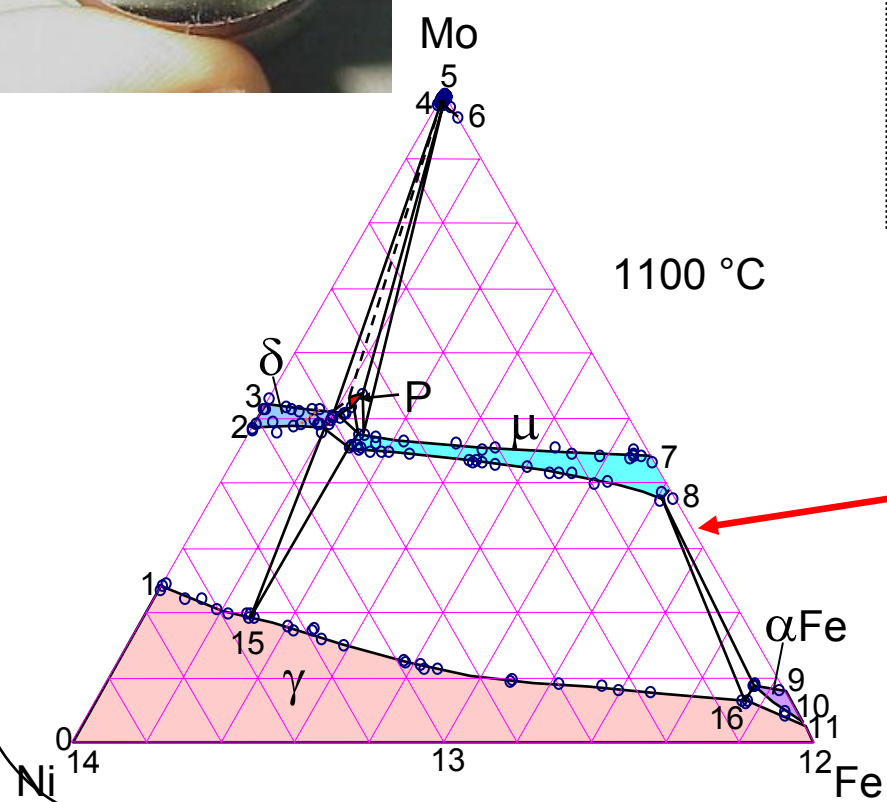
Phase Diagram Mapping: Example from a diffusion multiple

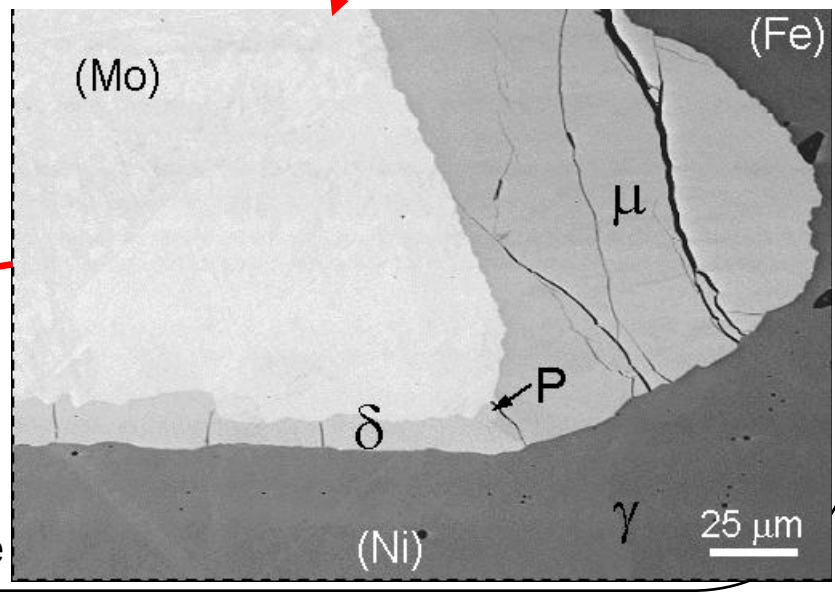




1100°C 1500 hrs

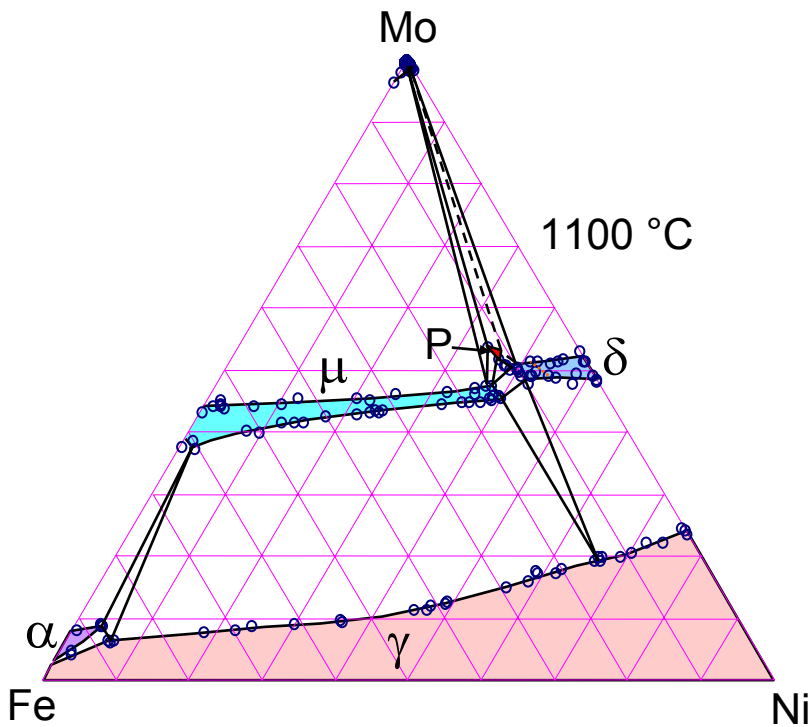




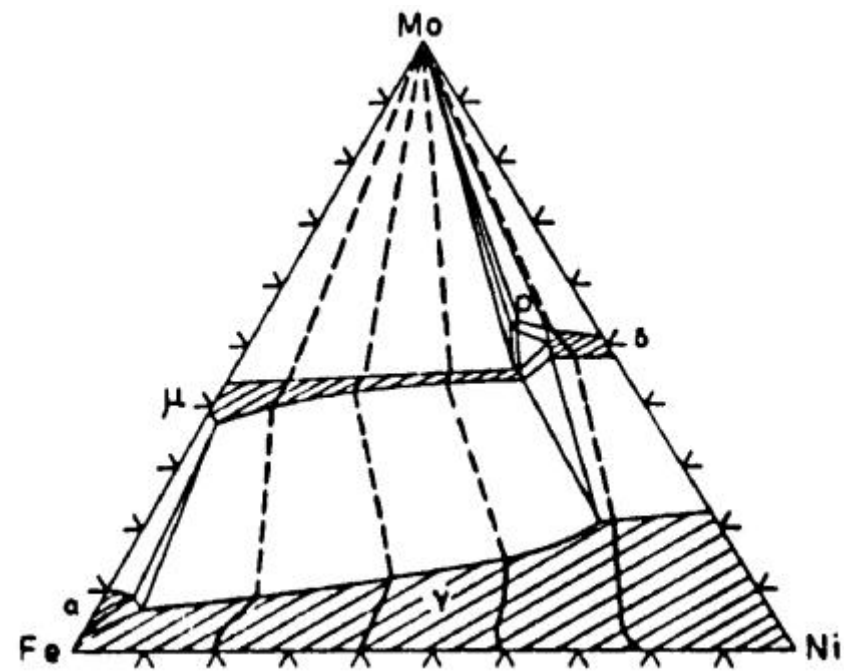


Phase Diagram Mapping: Ni-Fe-Mo system

One diffusion multiple



23 alloys + 7 diffusion couples (van Loo *et al.*)



Diffusion Multiples:

- Much higher efficiency for determining of phase diagrams
- An efficient screening tool for new intermetallic compounds

Phase Diagram Mapping: Phase Equilibria of Superalloys

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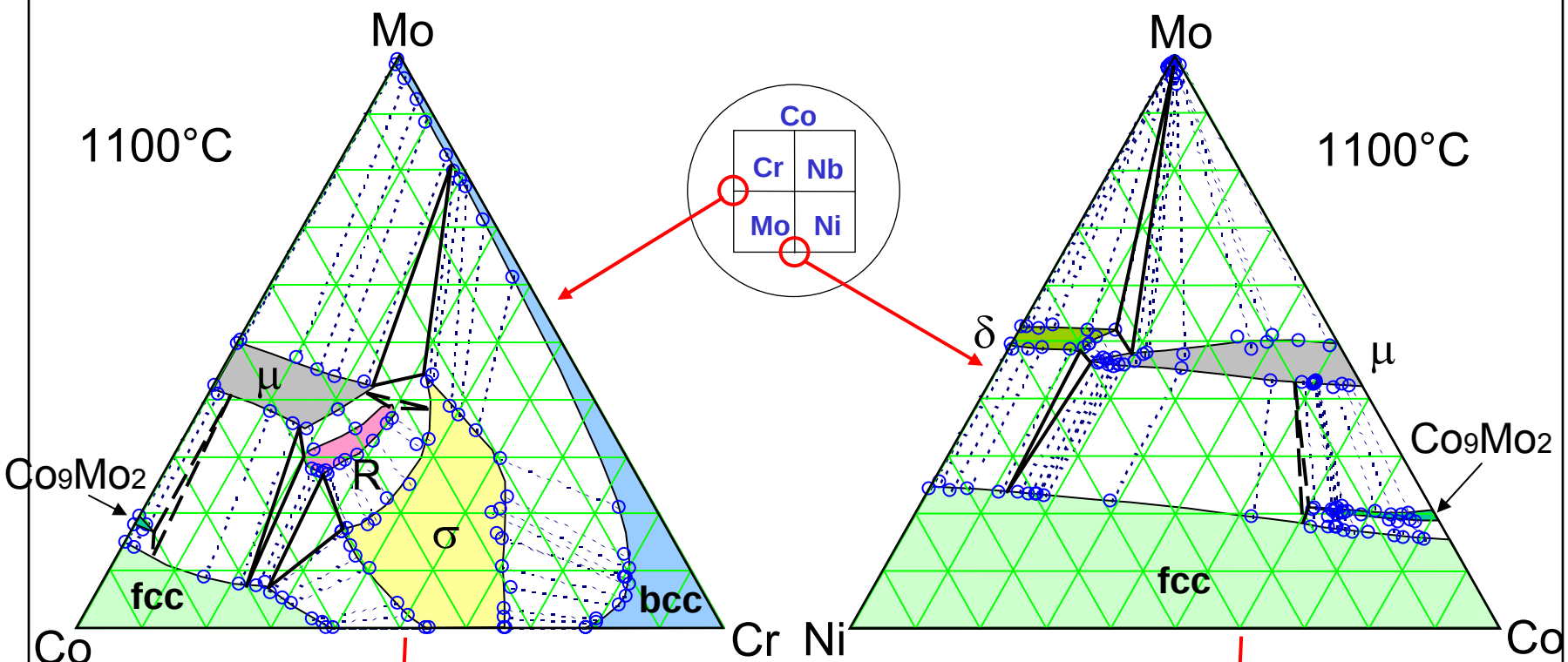
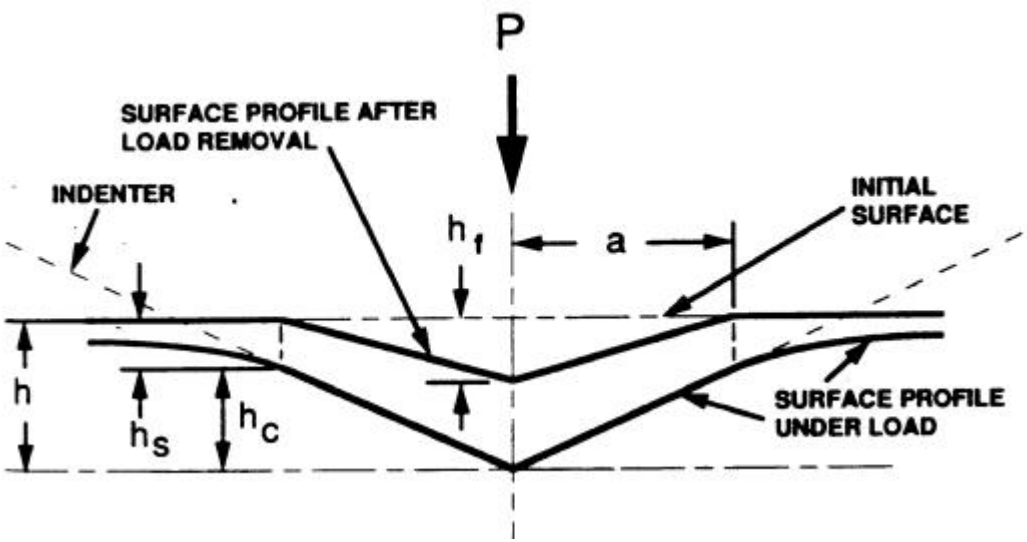
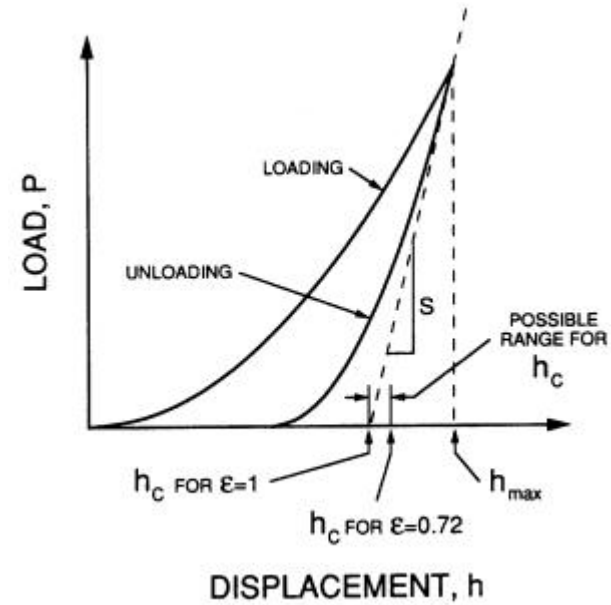


University of Wisconsin - Madison

High-fidelity thermodynamic parameters of TCP phases

Hardness and Elastic Modulus: Nanoindentation



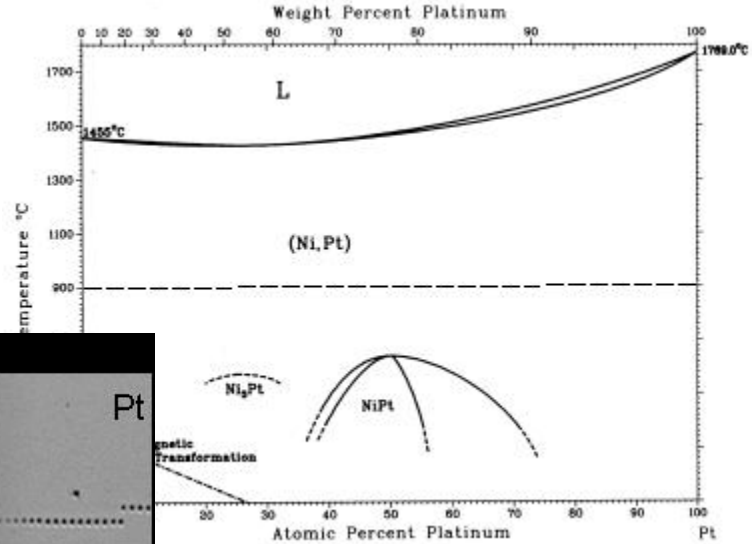


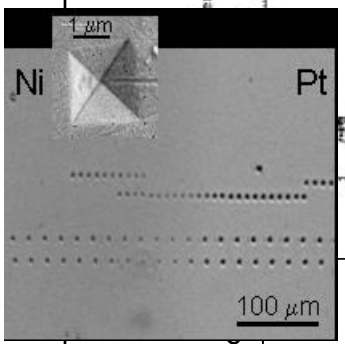
Load and displacement sensing nano-indentation

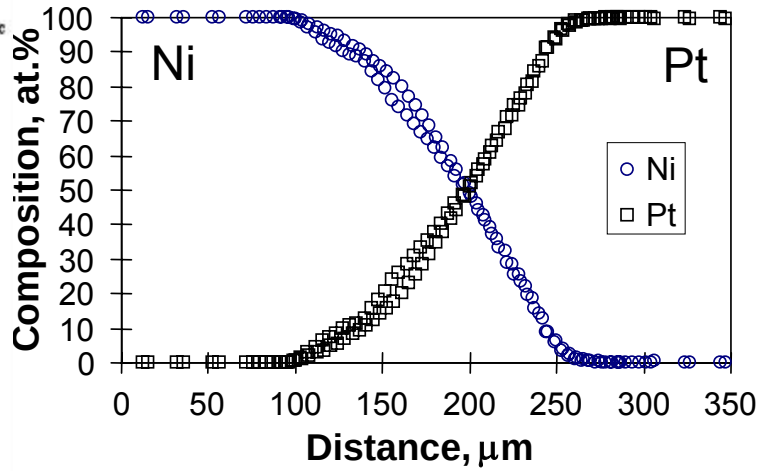
Doerner & Nix, 1986
Oliver & Pharr, 1992

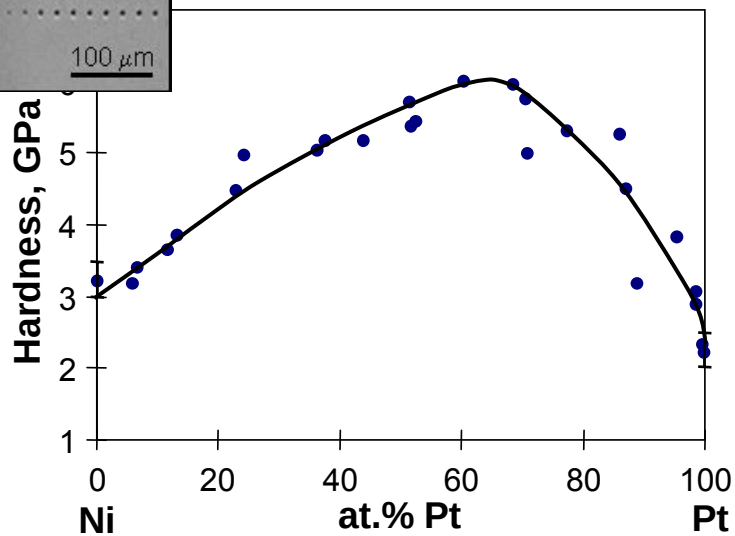
Nano-indentation allows simultaneous evaluation of hardness and elastic modulus

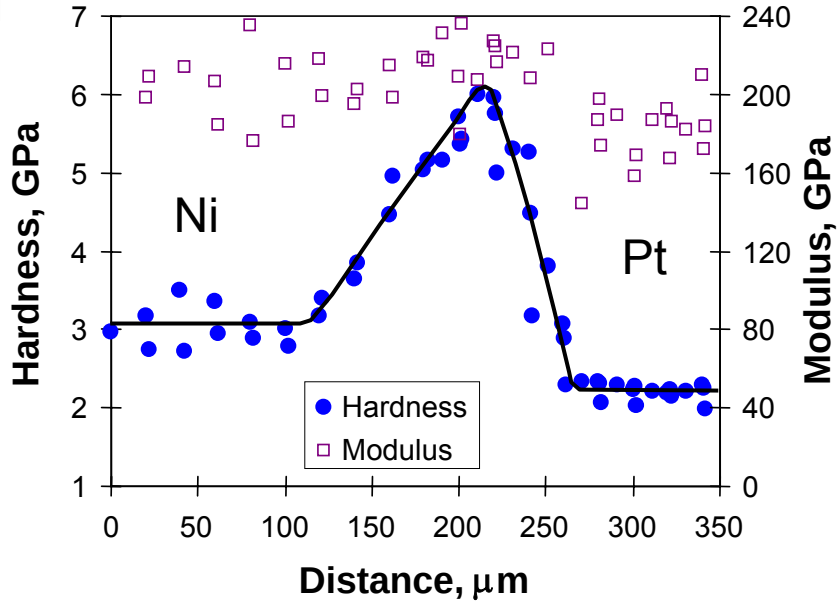
Hardness & Elastic Modulus: Ni-Pt diffusion couple





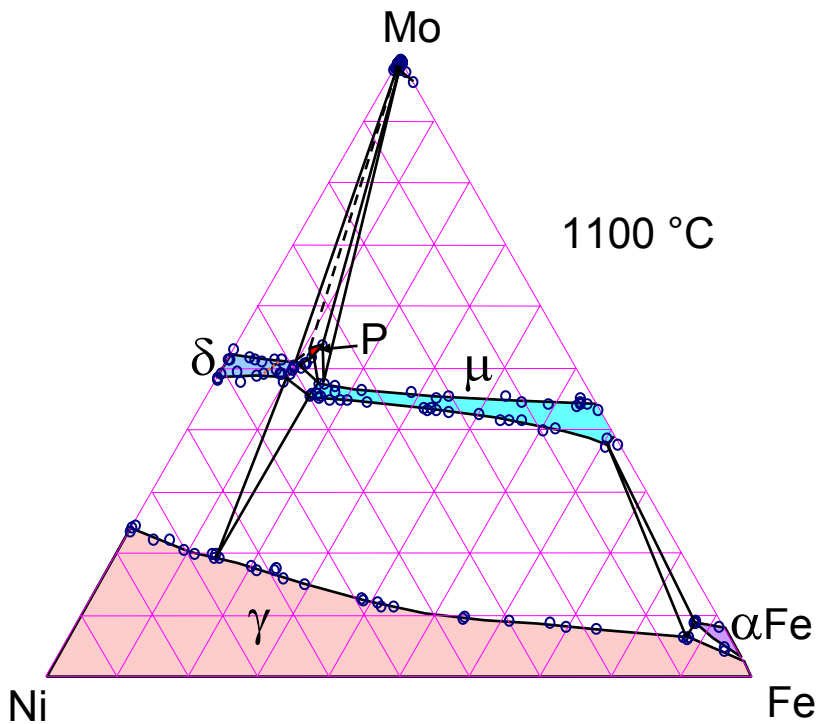


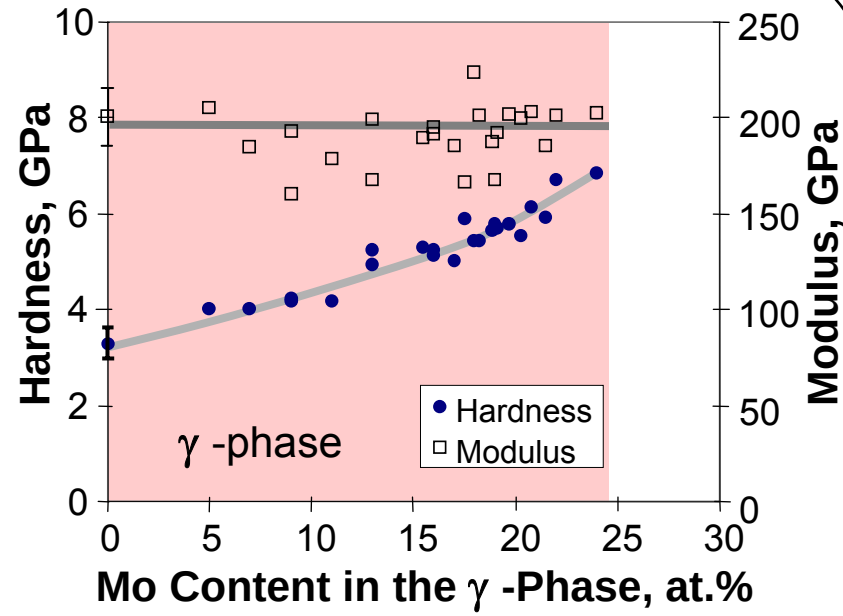


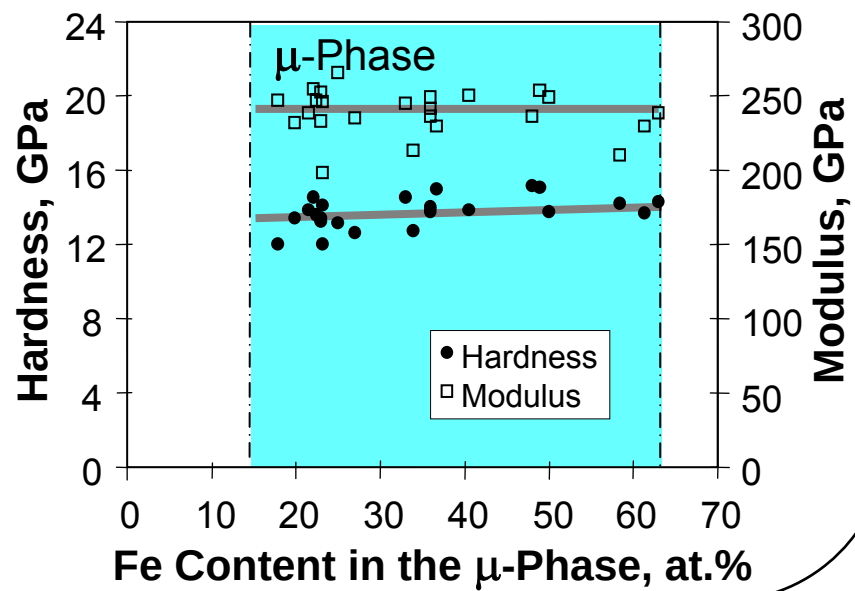


Efficient survey of the solution-hardening effect

Hardness and Modulus: Ni-Fe-Mo

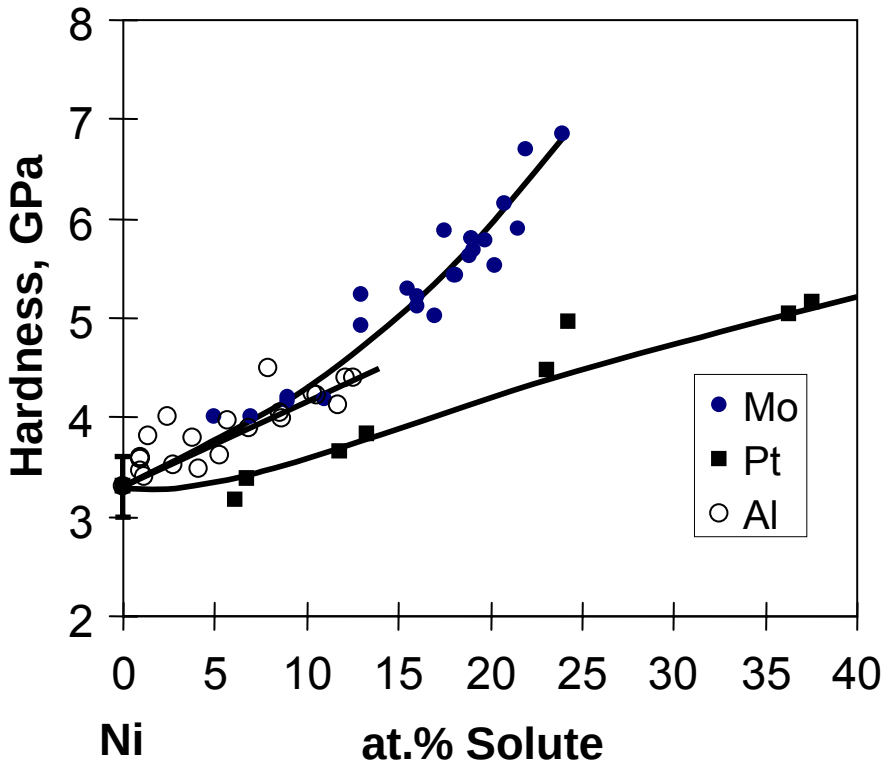


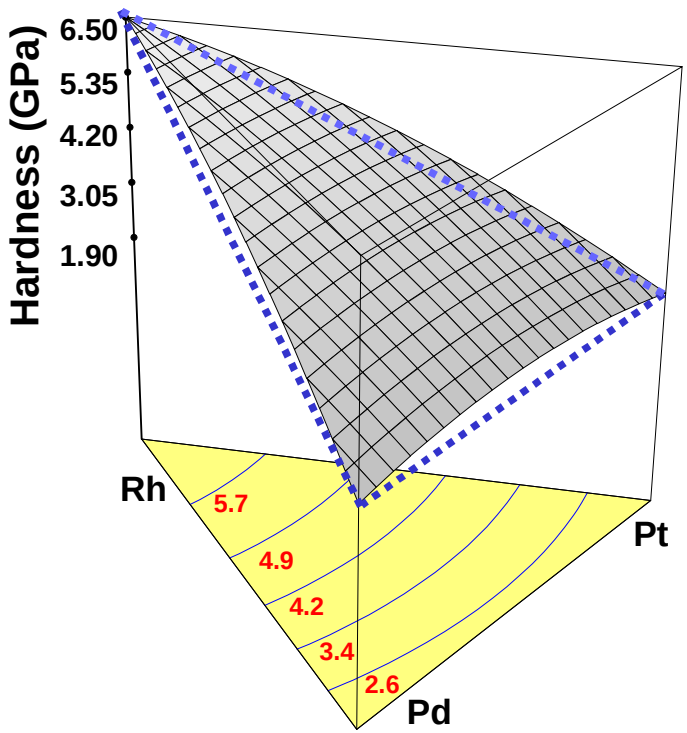




Efficient survey of composition - phase-property relations such as solution hardening effect

Hardness: databases on the solution-hardening effect

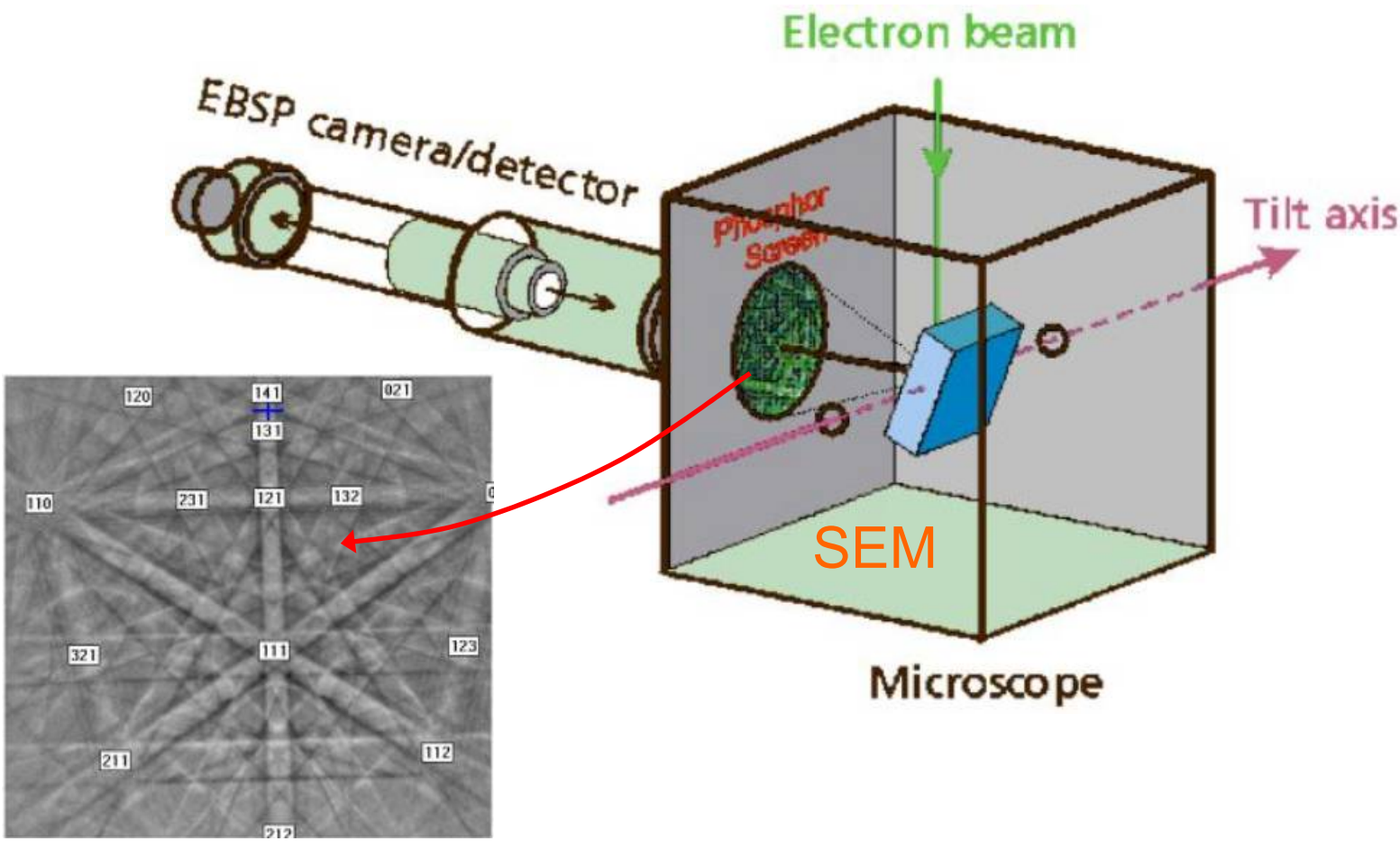




Hardness vs yield (flow) strength
Potency in solution-strengthening

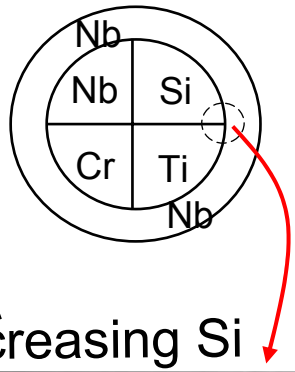
Databases will allow prediction of alloying element effects in current & new alloy systems

Crystal Structure: EBSD

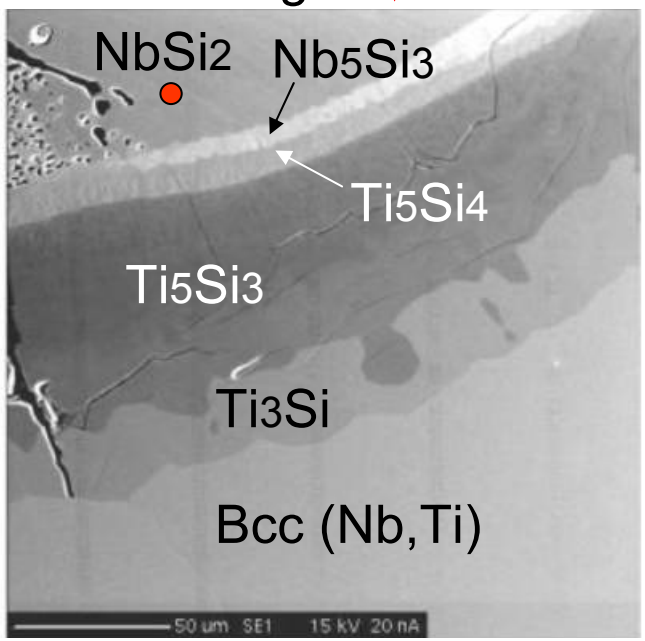


EBSD: A powerful technique for crystal structure identification in scanning electron microscope (SEM)

Crystal Structure: EBSD



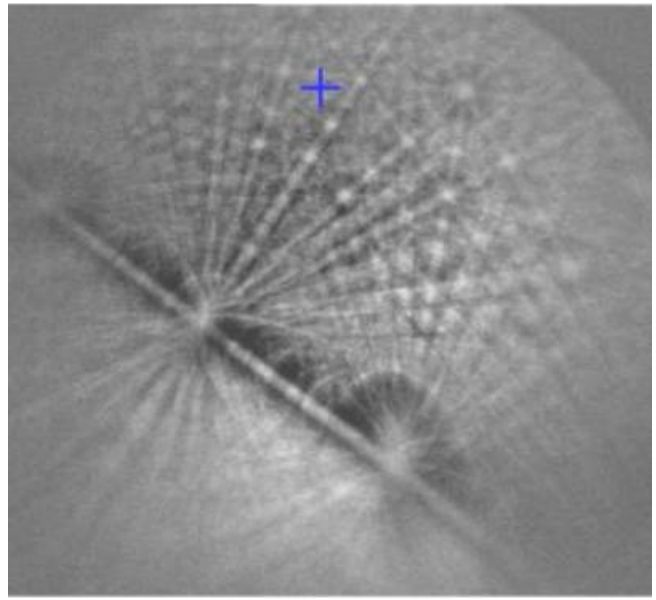
Increasing Si

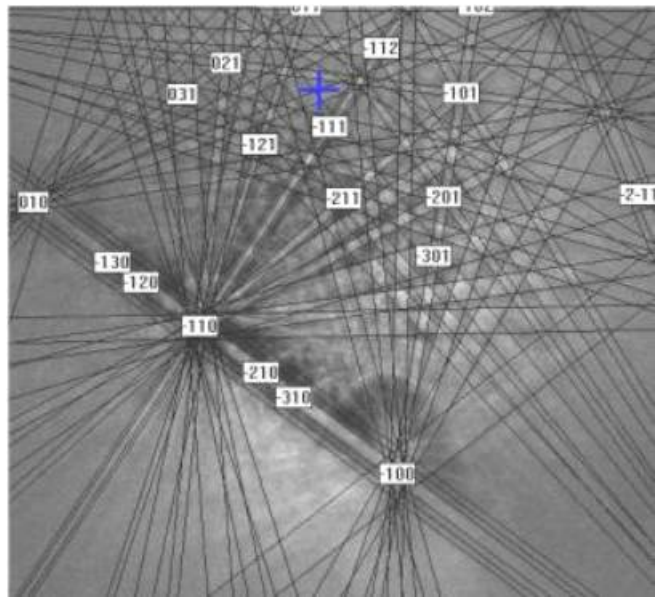


Increasing Nb

Increasing Ti

$NbSi_2$





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Crystal Structure: EBSD

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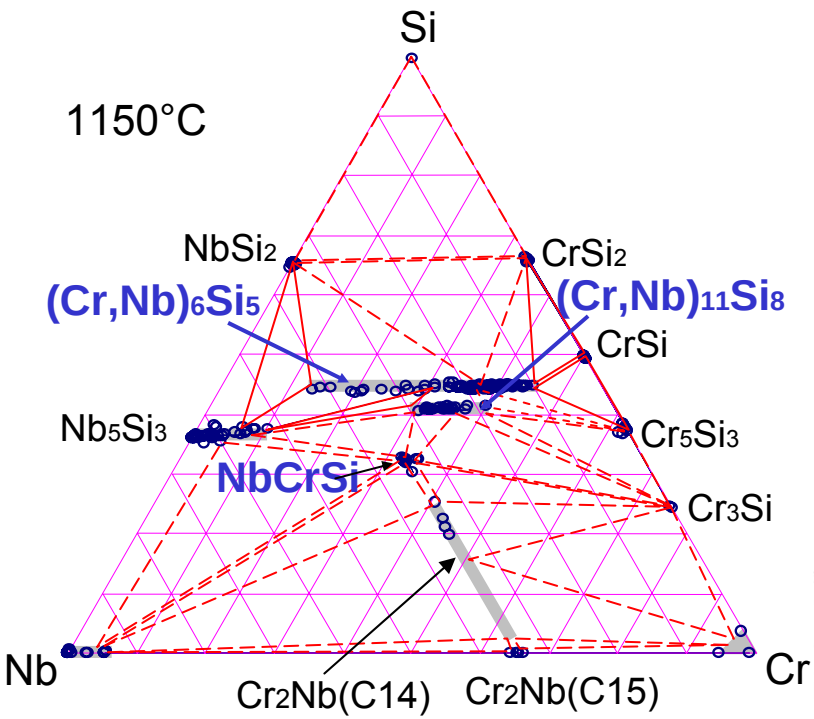
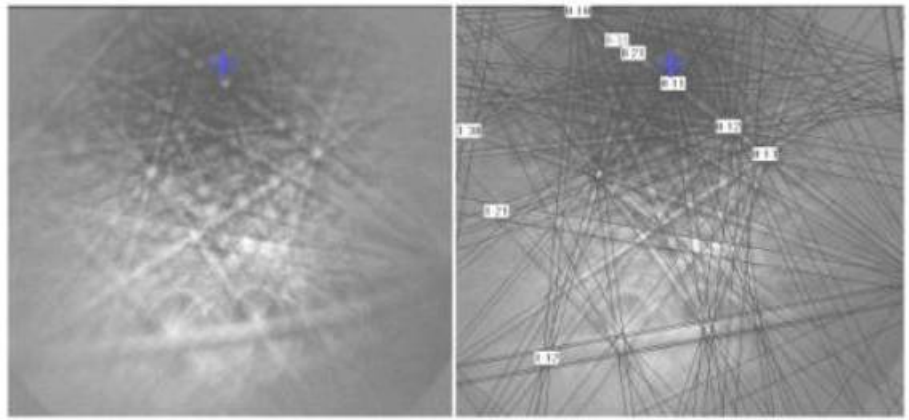
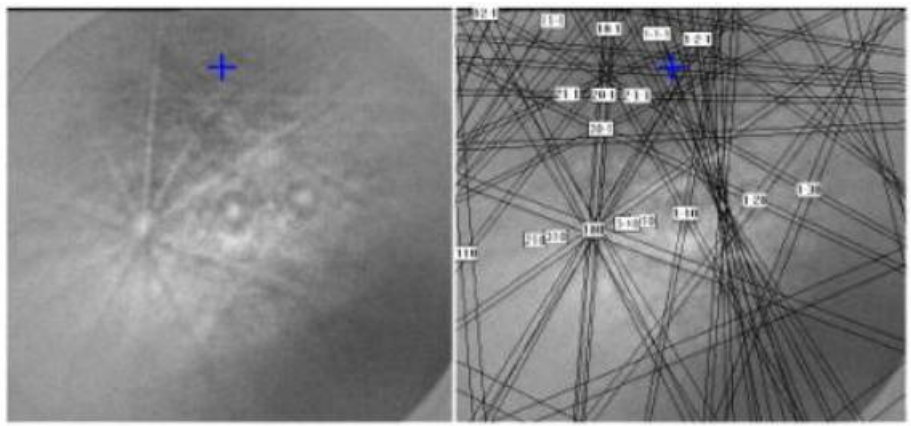


- Phase diagram determined from one diffusion multiple
- The same phase diagram in the literature was determined using **220** alloys

$(Cr,Nb)_6Si_5$: oI44, Ibam, s.g.# 72



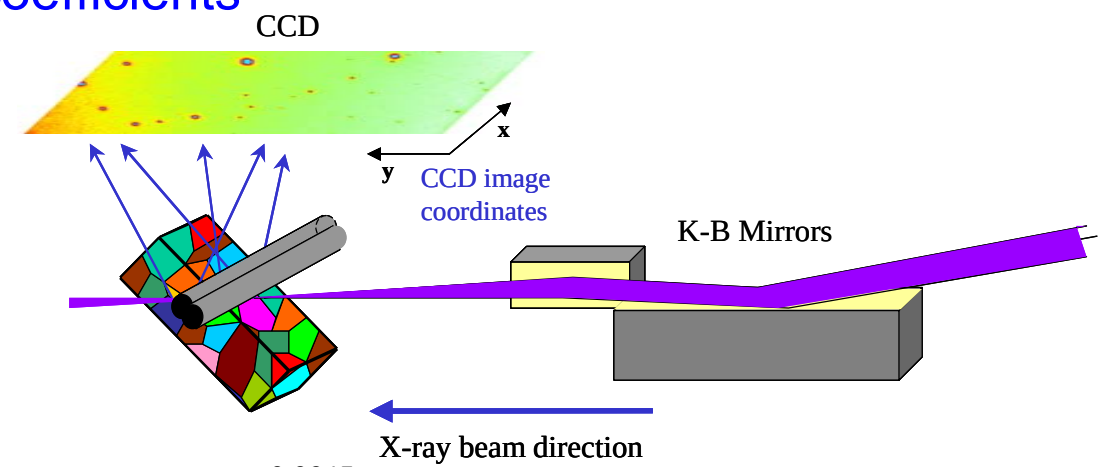
$(Cr,Nb)_{11}Si_8$: oP76, Pnma, s.g. # 62

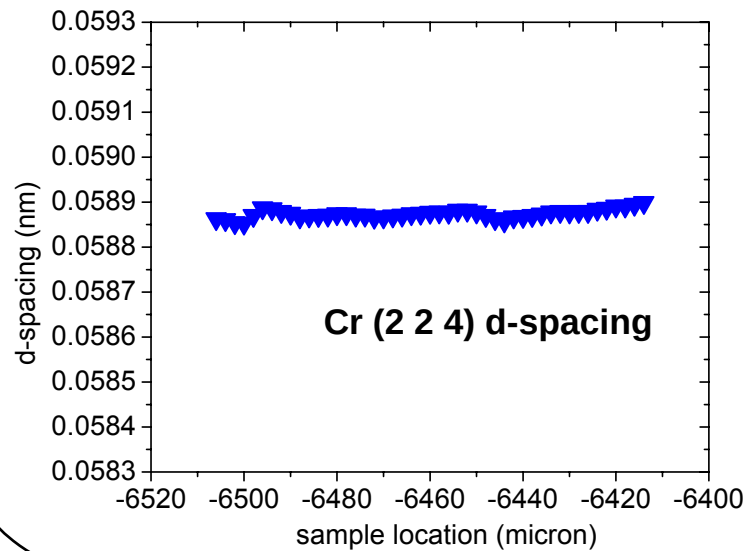


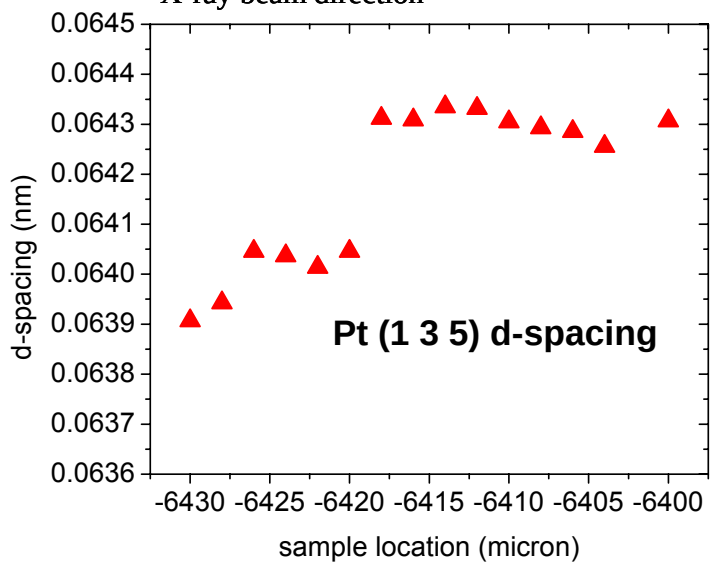
More complex structure ID may need micro-XRD or TEM + FIB

Crystal Structure: Micro-XRD

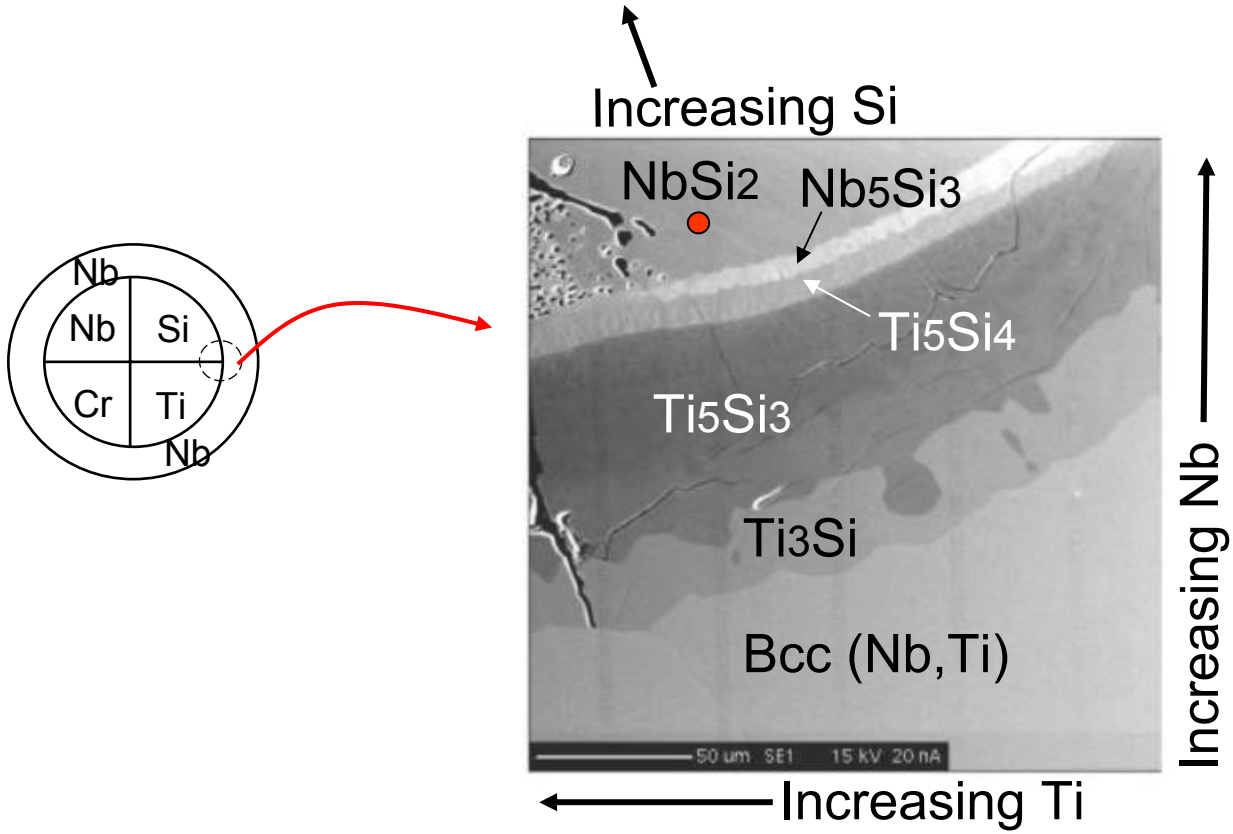
- X-ray micro-diffraction at micron resolution
- Measurement of lattice parameter as a function of composition
- Thermal expansion coefficients
- Density calculations





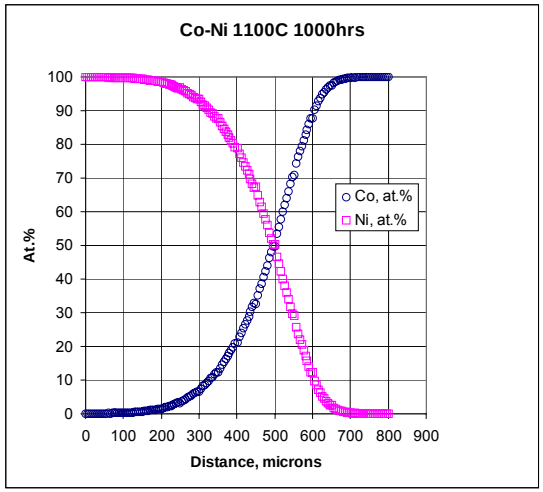


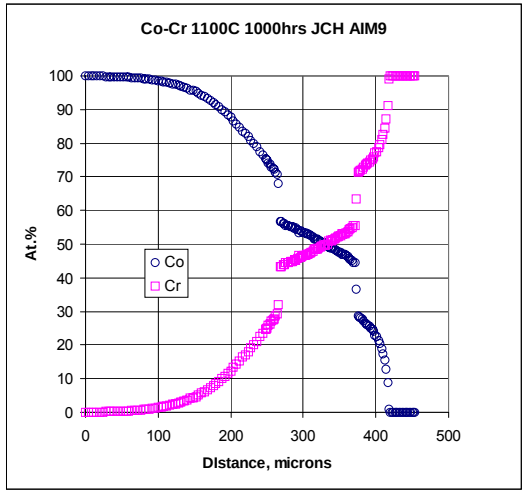
Crystal Structure: FIB + TEM

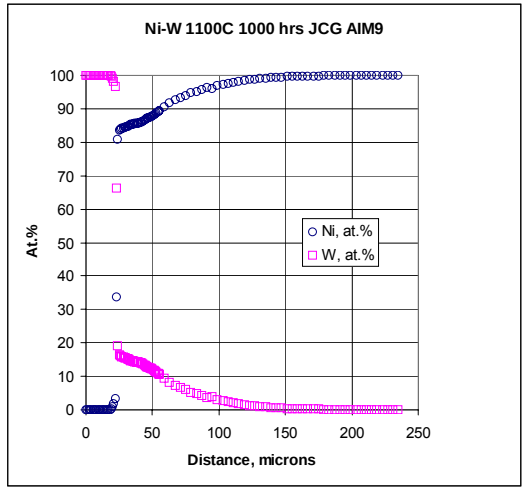


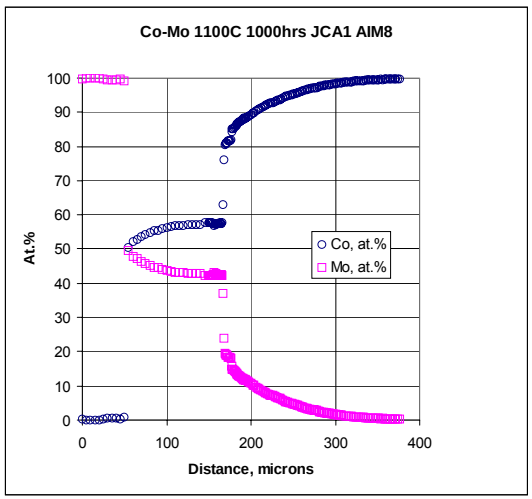
FIB cutting of small phases
TEM phase ID by electron diffraction

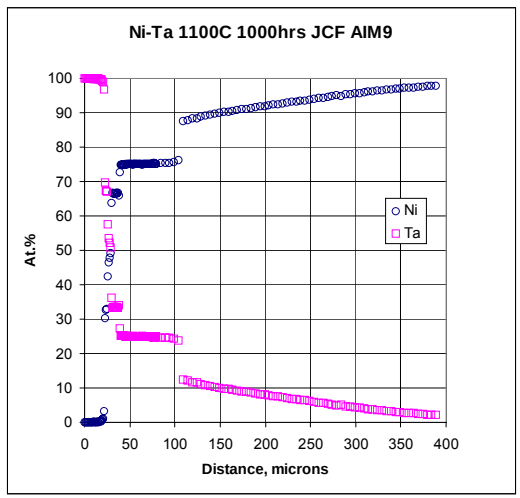
Diffusion Coefficients from Diffusion Profiles











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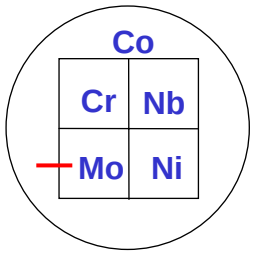
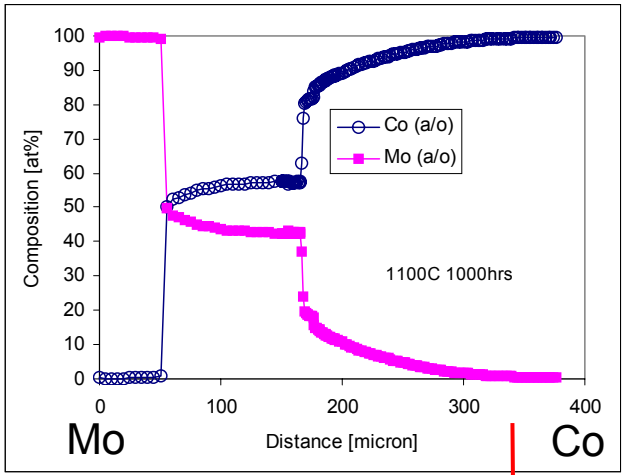
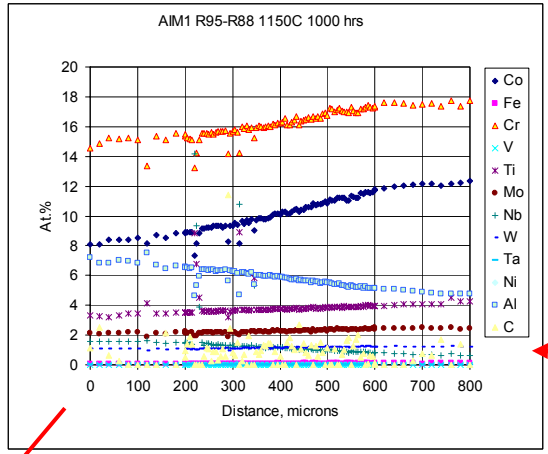
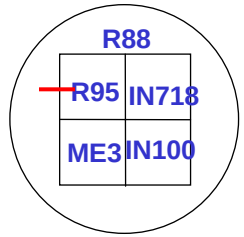
Diffusion Coefficients from Diffusion Profiles





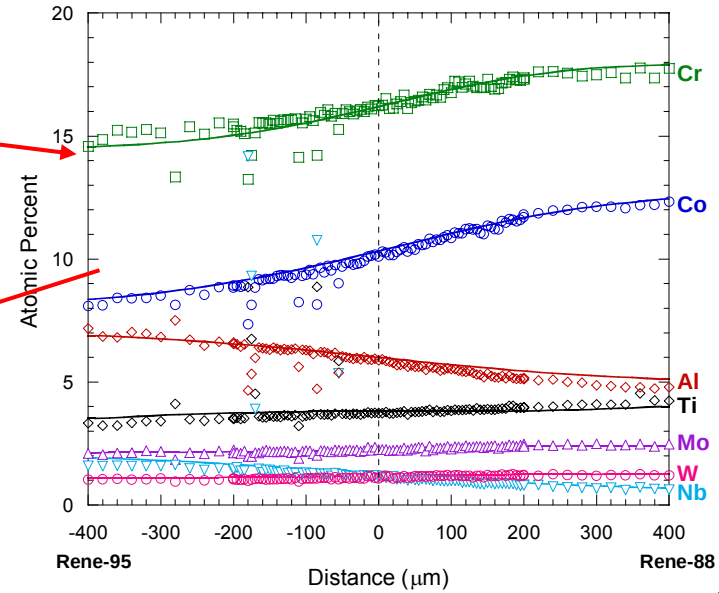




NIST

High-fidelity diffusivity database

γ', H/T, grain size modeling



Campbell, Boettinger, Zhao, Henry

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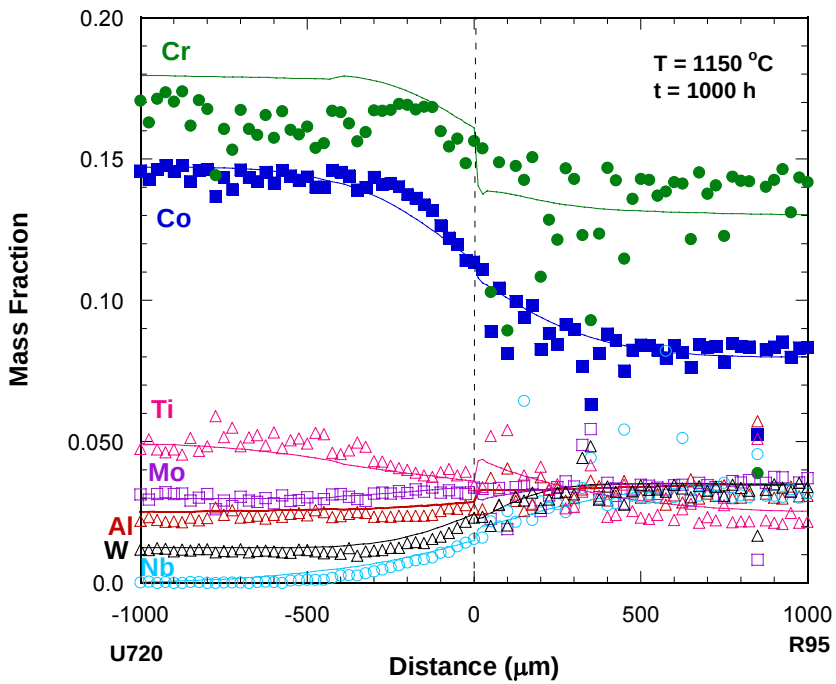
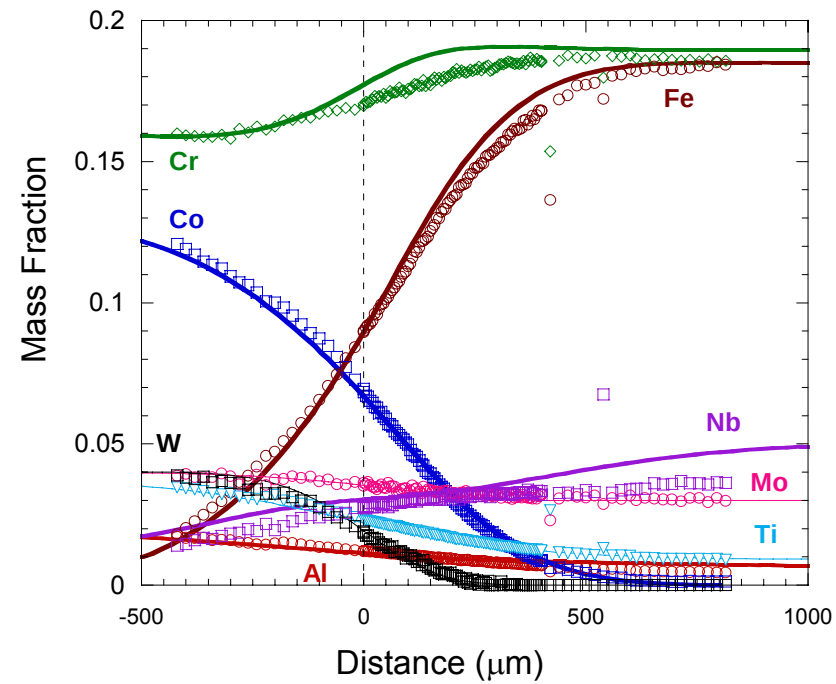
Diffusion Coefficients from Diffusion Profiles

U720/R95; 1000 h at 1150 °C



René-88/IN-718; 1000 h at 1150 °C



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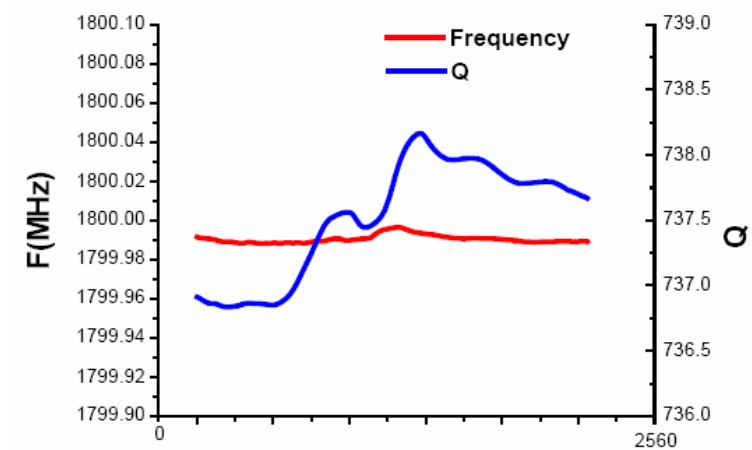
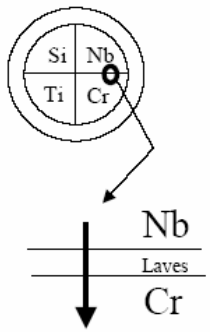
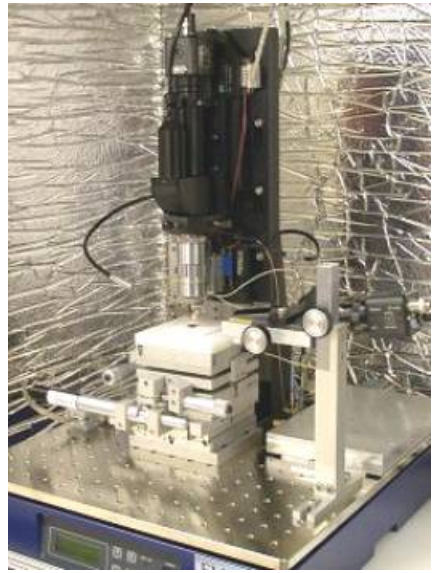
Electrical Conductivity:

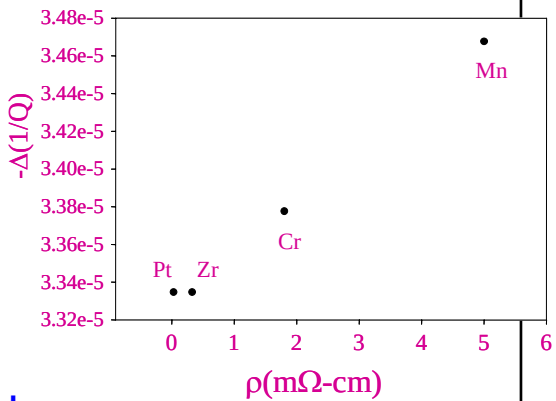






Evanescent Microwave Probe Microscope (EMP):

- Can map electrical conductivity at very high resolution
- Require very flat & porosity-free sample

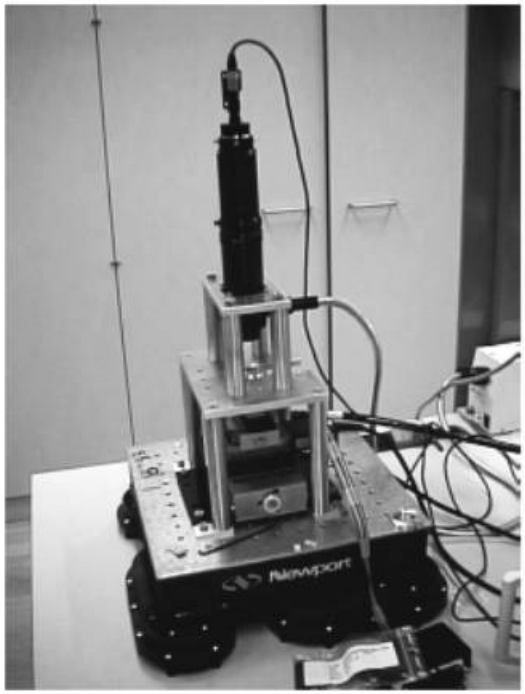


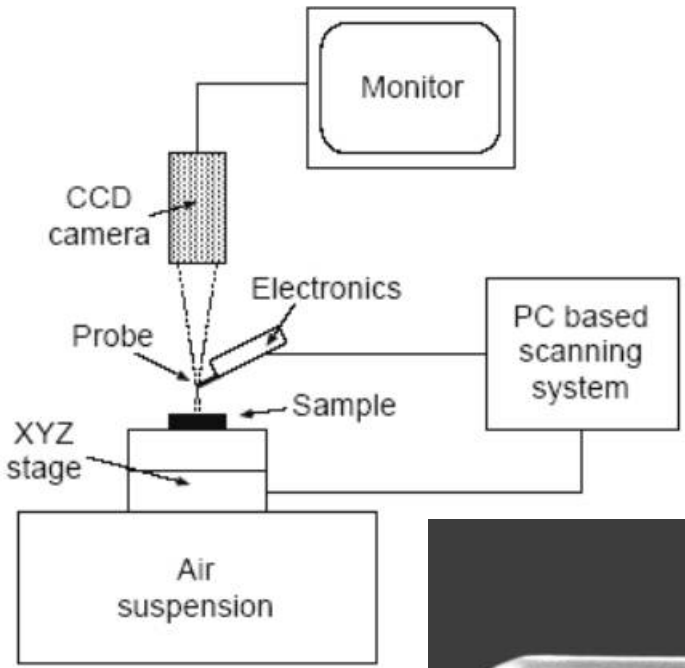
Oak Ridge National Lab:

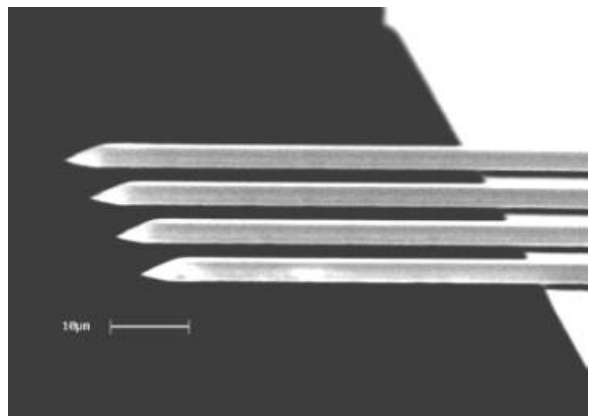
- Current pulse and decay curves
- Mapping electrical conductivity of phases simultaneously

Electrical conductivity is critical for many materials:
superconductors, semiconductors, thermoelectric materials, etc.

Electrical Conductivity: Micro four-point probes





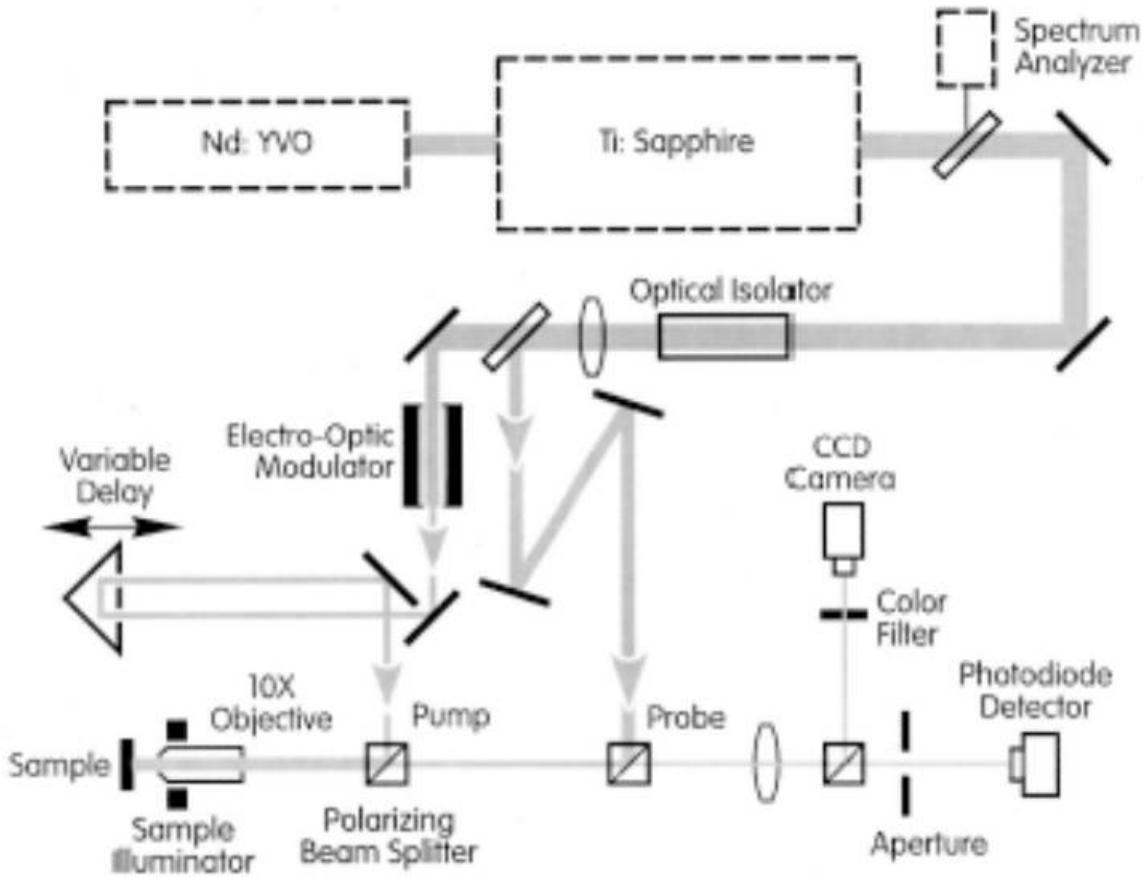


C.L. Peterson

Need to develop a more robust screening tools for electrical conductivity

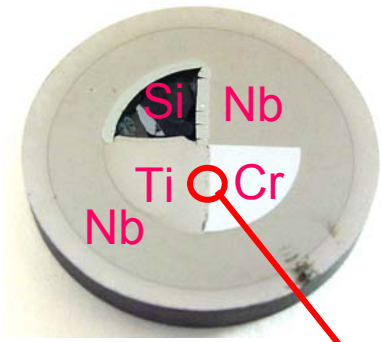
Thermal Conductivity:

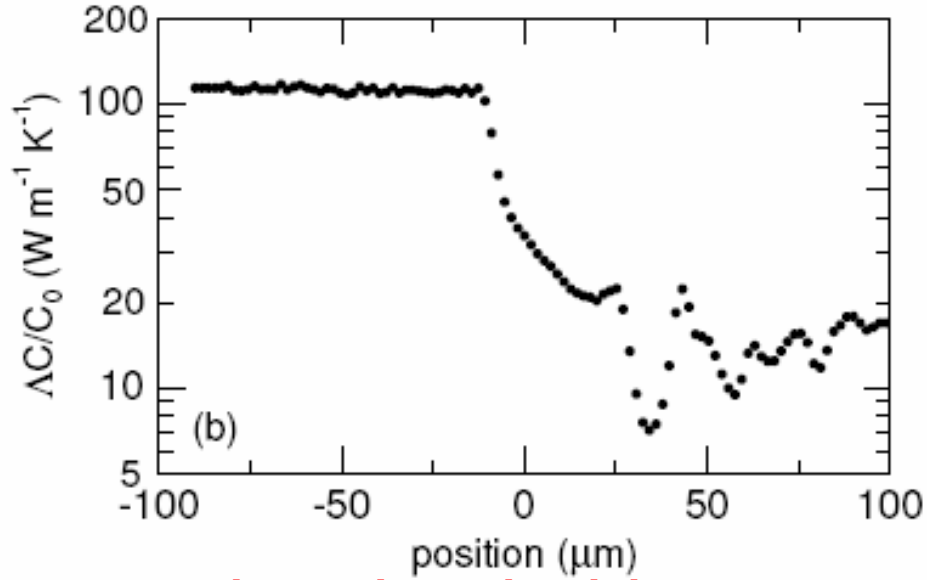
Time-domain thermoreflectance imaging using femtosecond pulsed laser

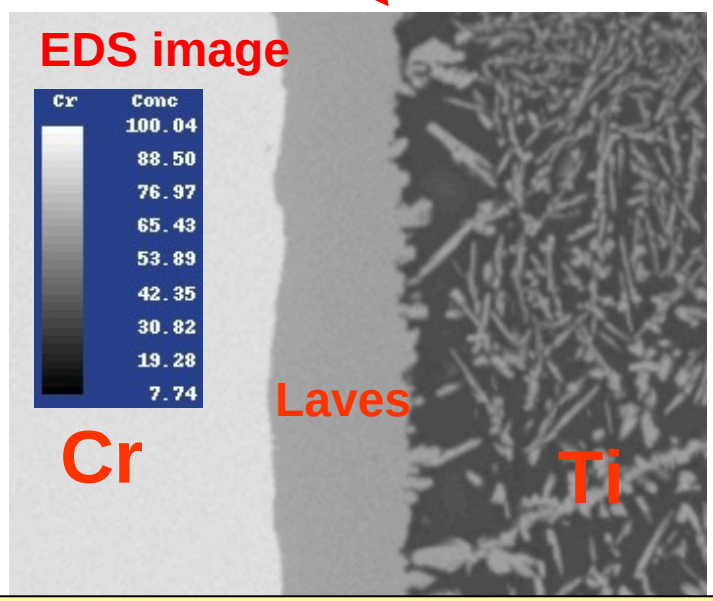


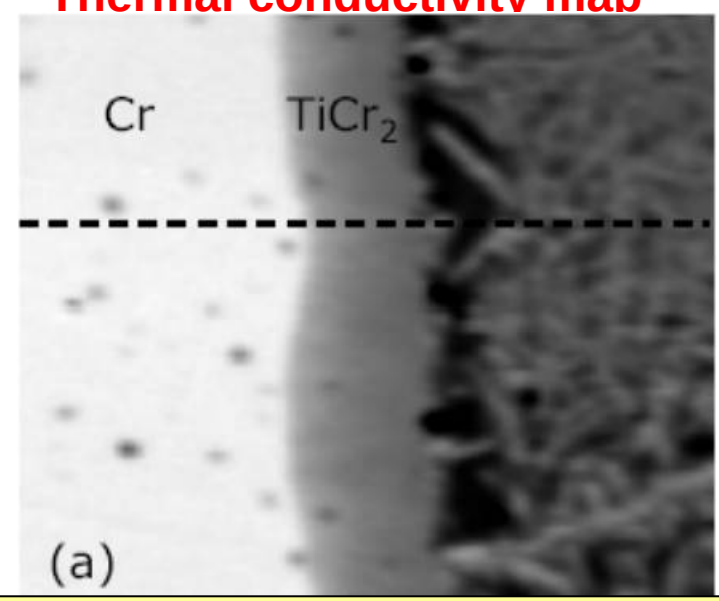
Huxtable, Cahill, Fauconnier, White, Zhao

Thermal Conductivity:



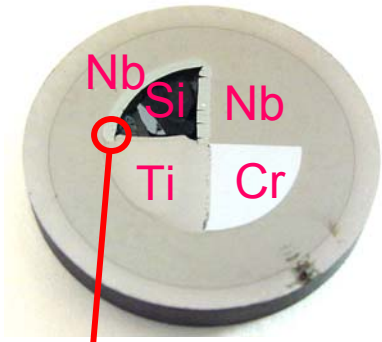




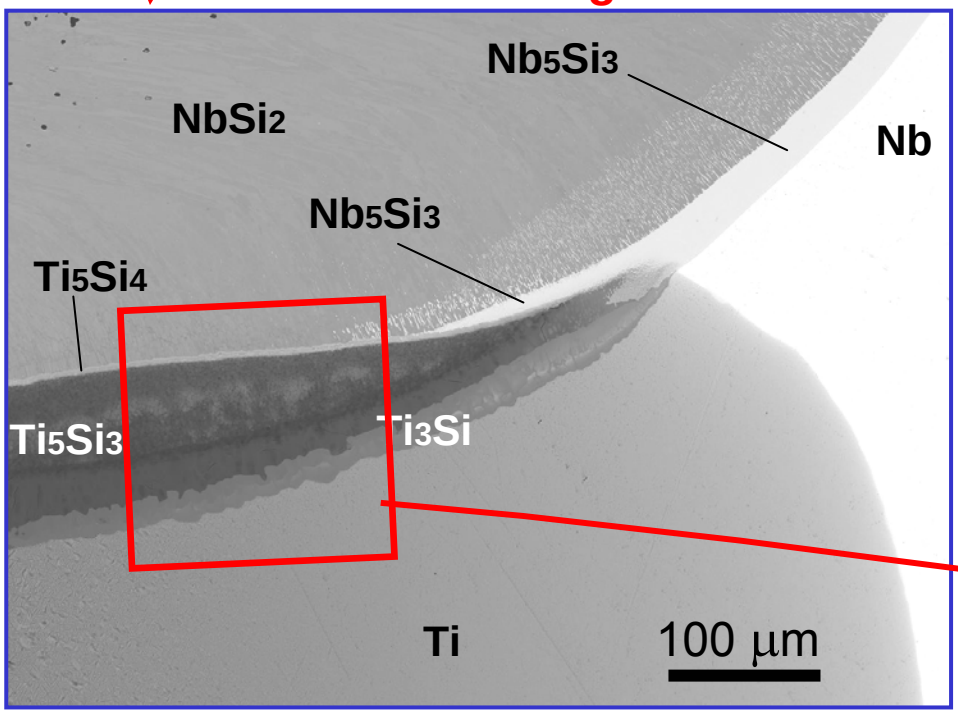


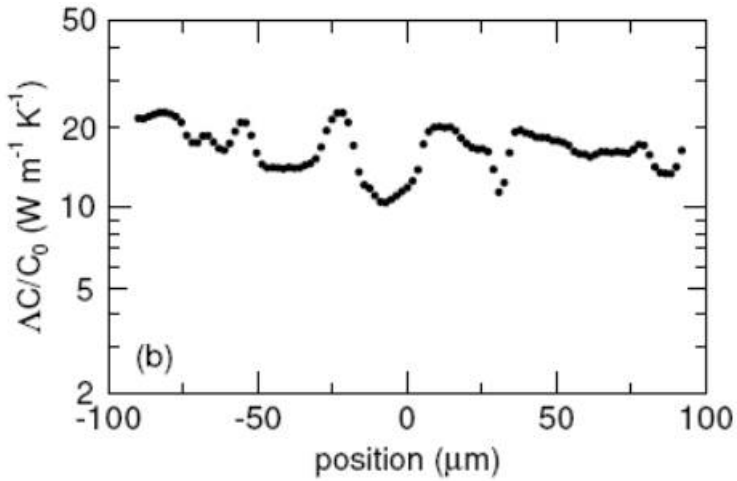
3.5 μm resolution, ±10% accuracy, 10,000 points/hour

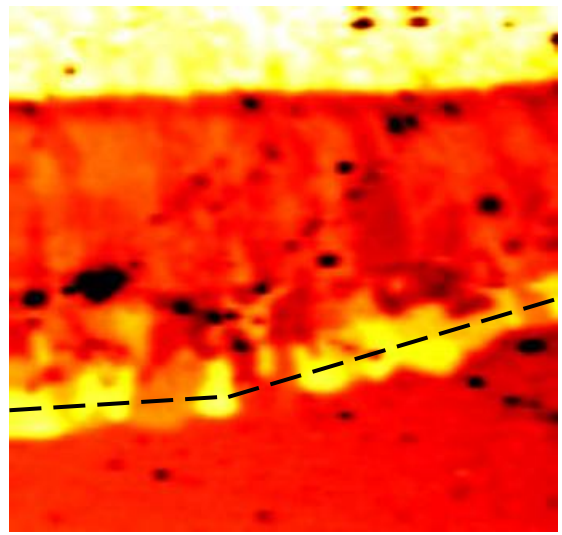
Thermal Conductivity:



SEM Image







Thermal conductivity map

Huxtable, Cahill, Fauconnier, White, Zhao

Magnetic Properties:

SCANNING SQUID MICROSCOPY

John R. Kirtley
IBM T. J. Watson Research Center, Yorktown Heights, New York 10598;
e-mail: kirtley@watson.ibm.com

John P. Wikswo, Jr.
Department of Physics and Astronomy, Vanderbilt University, Nashville,
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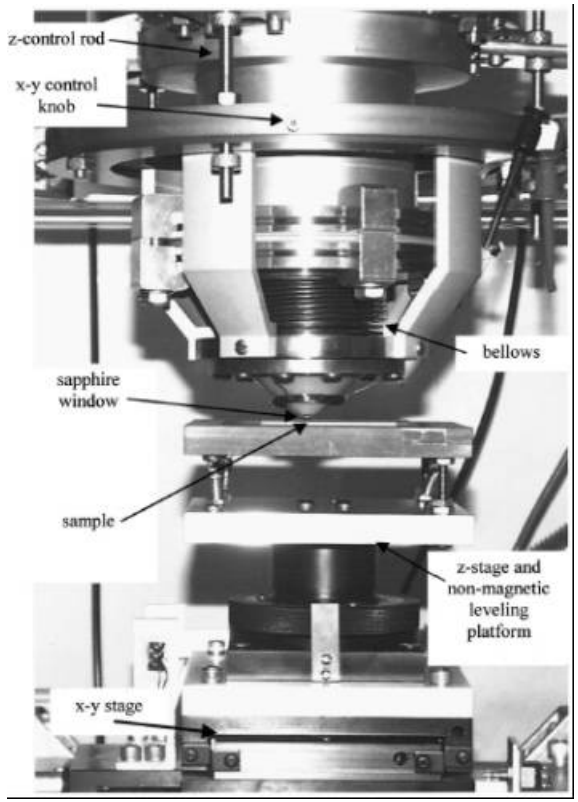
MAGNETIC FORCE MICROSCOPY

U. Hartmann
Institute of Experimental Physics, University of Saarbrücken, P.O. Box 151150,
D-66041 Saarbrücken, Germany; e-mail: u.hartmann@rz.uni-sb.de



Closed-cycle refrigerator-cooled scanning SQUID microscope for room-temperature samples

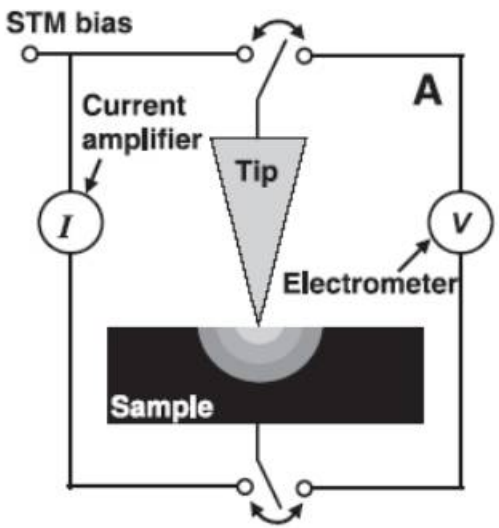
E. F. Fleet,[a] S. Chatraphorn, and F. C. Wellstood
Center for Superconductivity Research, University of Maryland at College Park, College Park, Maryland 20742

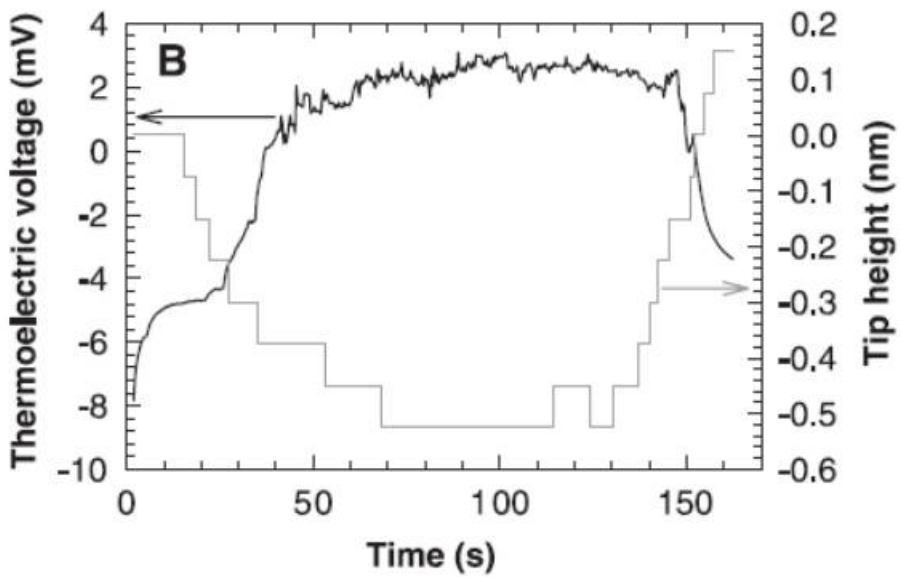
L. A. Knauss and S. M. Green[b]
Neocera, Incorporated, Beltsville, Maryland 20705-4215

Thermoelectric properties:

Profiling the Thermoelectric Power of Semiconductor Junctions with Nanometer Resolution

Ho-Ki Lyeo,[1] A. A. Khajetoorians,[1] Li Shi,[2,3]* Kevin P. Pipe,[4] Rajeev J. Ram,[4] Ali Shakouri,[5] C. K. Shih[1,3]*

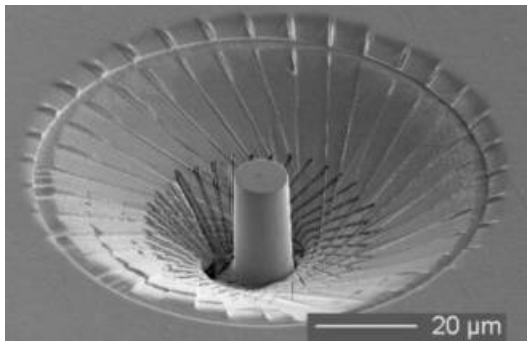


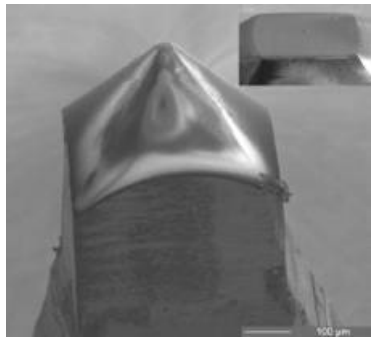


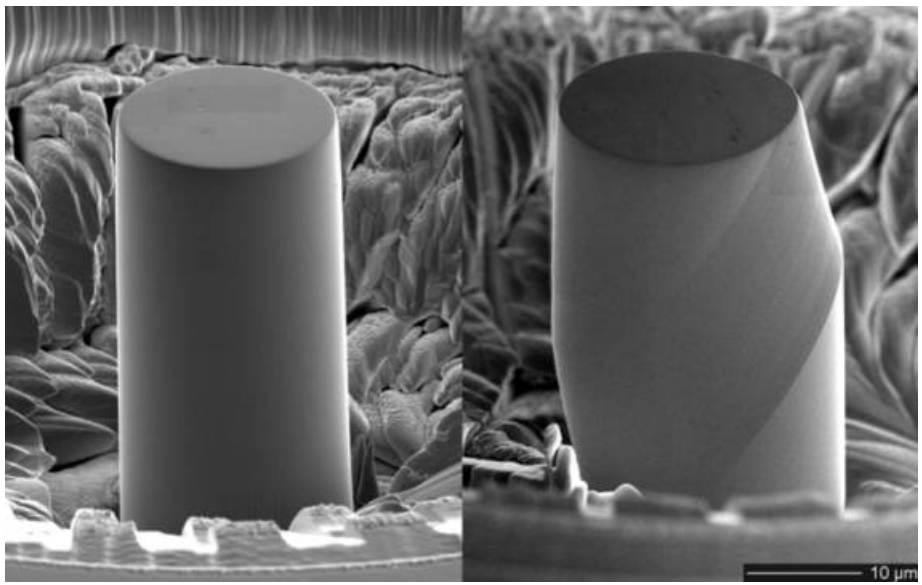
Science, Feb. 6, 2004

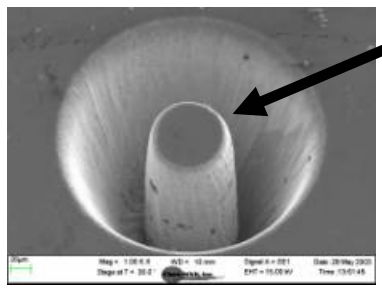
Compressive Yield Strength:

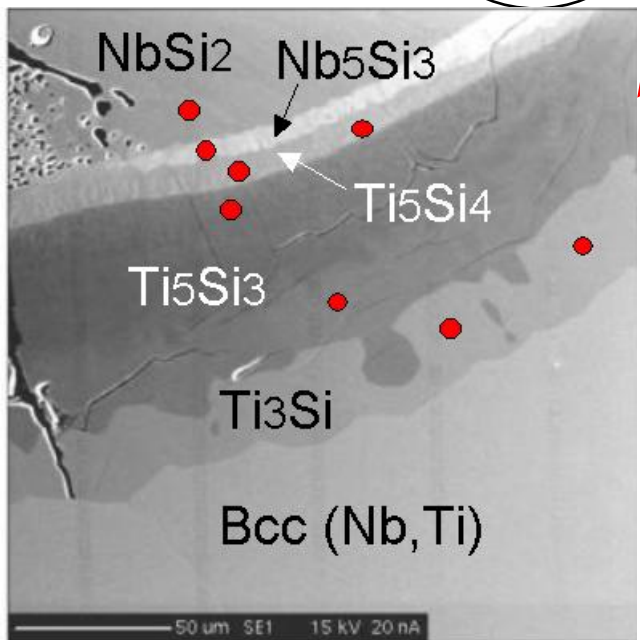
FIB machining of micro samples for micro-mechanical testing – M. Uchic & D.M. Dimiduk, AFRL

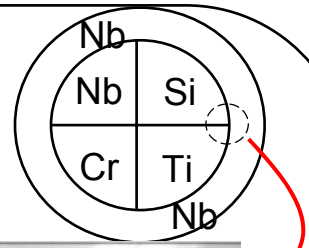












Machining time:
Laser ≈ 30s
FIB > 50h
T. Pollock
(U. Mich.)

Rapid screening of structural intermetallics & superalloys

Other Potential Screening Tools

- Scanning Kelvin Probe - Band gap
- Scanning Tunneling Potentiometry – electrical ?
- Electromotive Force (emf) Measurements – ΔH ?
- ….

Summary

1. thermodynamic properties (ΔH, etc.)
2. thermal expansion coefficient
3. thermal conductivity
4. ductility
5. yield strength
6. magnetic properties
7. melting point
8. lattice parameter variation with composition
9. optical properties
10. electrical conductivity
11. phase diagrams
12. crystal structure
13. elastic modulus
14. hardness
15. diffusion coefficient
16. Band gap
17. Thermoelectric properties, etc.

Green = fully developed
Blue = partially developed
Red = not developed