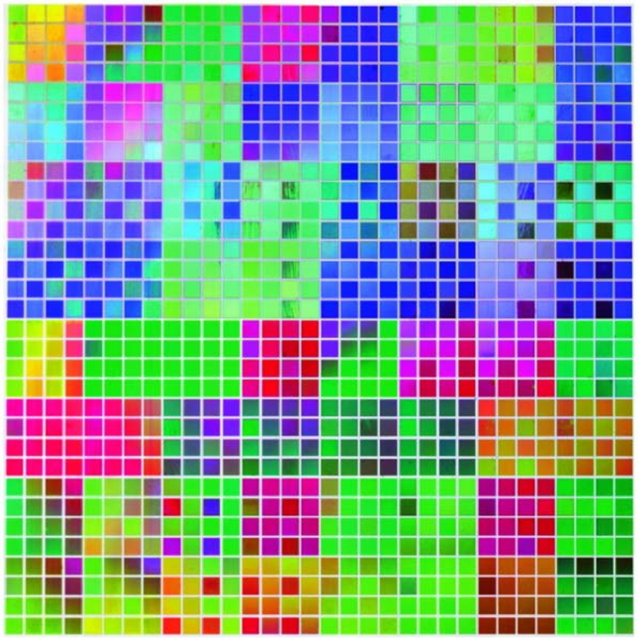
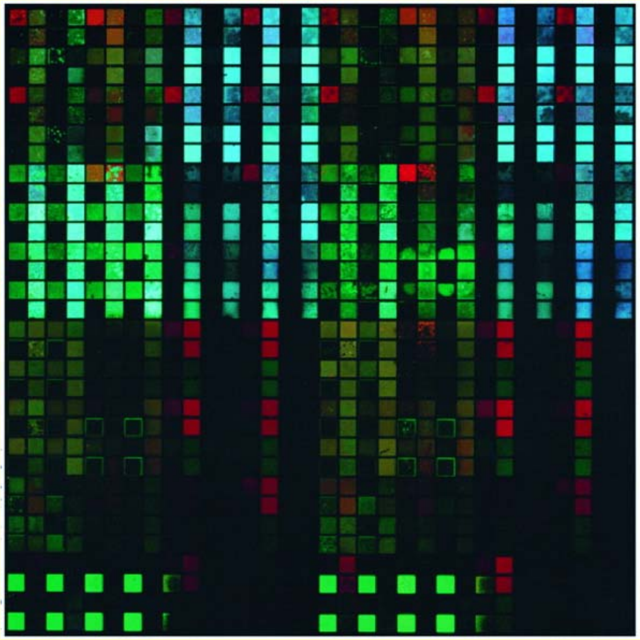
COMBINATORIAL MATERIALS DISCOVERY

"Combinatorial Libraries"

Blue Photoluminescent Phosphors - $Gd_20_3/Ga_2O_3/SiO_2$



Normal light



UV light

- Wang et al., *Science* **275**, 1712 (1998)

PROGRAM OBJECTIVES

- *devise simple methods to produce combinatorial "alloy libraries"*
- *devise screening tools to rapidly assess alloy library properties and identify optimum alloy compositions*
- *compare alloy library specimens to conventionally processed alloys*

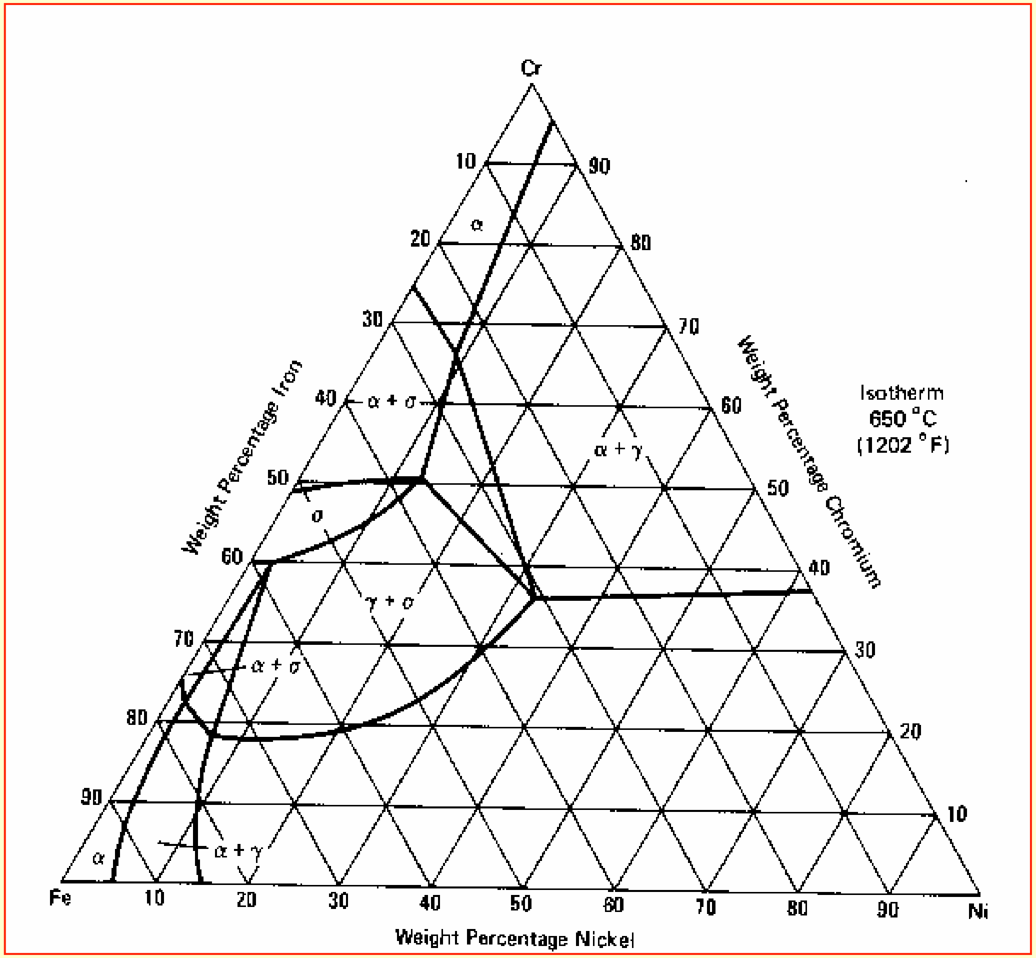
SIGNIFICANCE

- *expedite alloy design process*
- *reduce alloy design costs*
- *explore larger number of alloy compositions*
- *identify and optimize new alloys with improved properties for energy savings*
- *industries affected: aluminum, chemicals, forest products, glass, metal casting, petroleum, steel, forging, heat treating, welding, etc.*

MODEL ALLOY SYSTEM: Fe-Ni-Cr Ternary

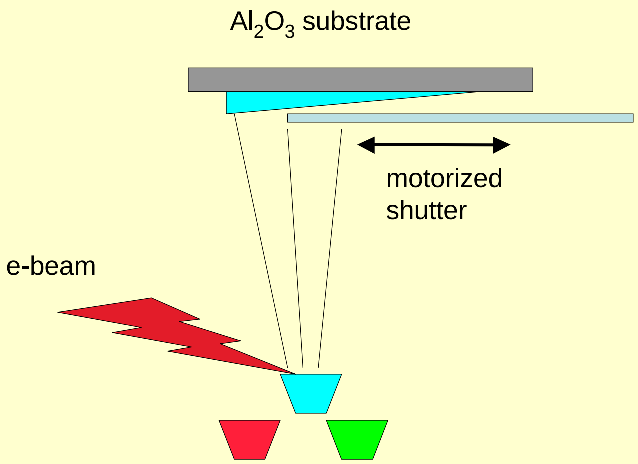
- *Foundation for austenitic and ferritic stainless steels*
- *Well studied and characterized*

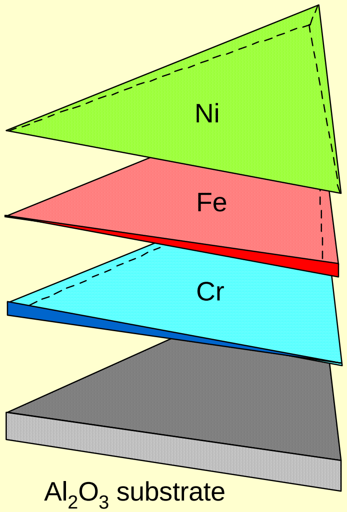
Program emphasis: *development of combinatorial methodologies*

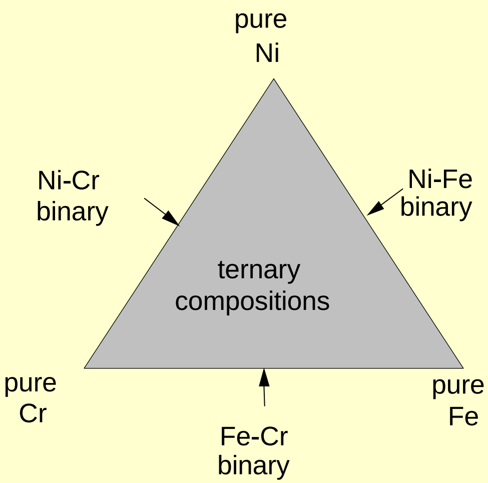


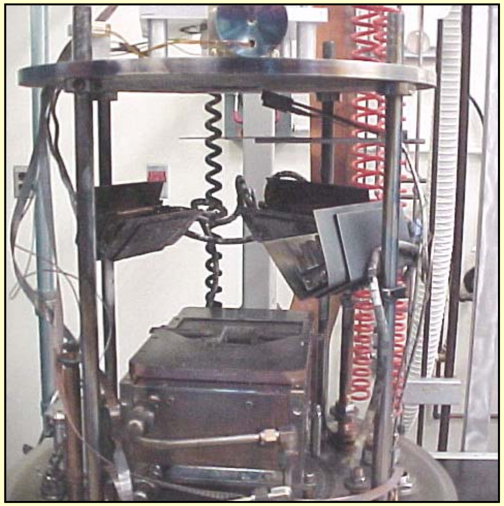
“ALLOY LIBRARY” SPECIMEN PREPARATION

Thin film deposition by shuttered e-beam evaporation









Alloying Techniques:

- *solid state diffusion by annealing*
- *localized heating and melting:*
  - *- focused e-beam welder*
  - *- laser (Nd:YAG)*
  - *- IR heating*

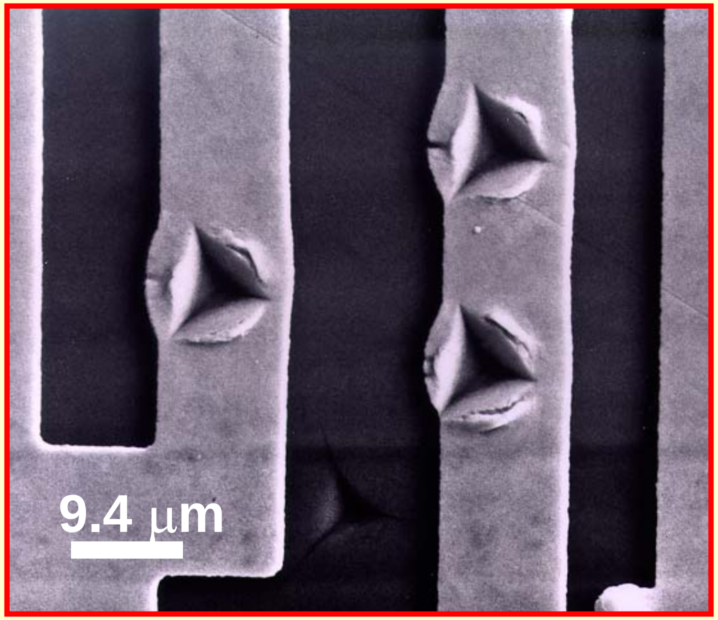
STRUCTURAL CHARACTERIZATION

- electron microprobe analysis: *chemical composition*
- x-ray diffraction: *crystal structure and phase identification (automated systems)*
- synchrotron-based microfocus x-ray analysis: *rapid analysis of chemical composition, crystal structures, and constituent phases*

PROPERTY CHARACTERIZATION

initial emphasis on properties of interest to industrial partners (H- and C-series stainless steel casting alloys)

- basic mechanical properties: *measurement of hardness and elastic modulus by nanoindentation*
- aqueous corrosion: *environmental exposure followed by visual examination and profilometry*
- carburization resistance *carburization followed by electron microprobe analysis*



OTHER POSSIBLE PROPERTY MEASUREMENTS

- weldability - *laser and e-beam induced hot cracking*
- magnetic properties - *magnetic force microscopy*
- wear and abrasion resistance - *wear testing and profilometry*
- etc

ENERGY, ECONOMIC, & ENVIRONMENTAL BENEFIT ANALYSIS

~ Impact by the year 2020 ~

Energy Savings						Environmental Savings	
	Electricity	Gas	Oil	Total	Total	CO_2	NO_x
Vision Industry	billion kWh	billion kWh	million barrels	trillion BTU's	million $/year	thousand tons/year	
Chemical	0.56	10.4	1.6	26	131	438	3.9
Heat treating	0.15	4.7	-	6	34	93	0.8
Steel	-	5.8	-	6	20	76	0.6
Total	0.71	20.9	1.6	38	185	607	5.3

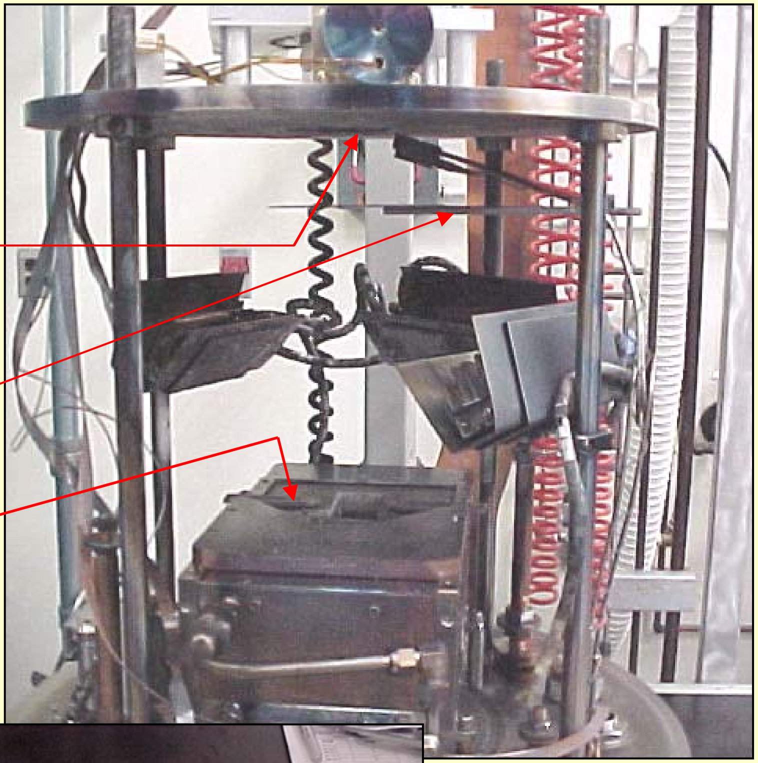
- *calculation assumes 50°C higher use temperature for H-series alloys*
- *impact analysis based on US DOE Office of Industrial Technologies' Energy Savings Worksheet spreadsheet model*

Progress:Year 1
(April 1, 2002 - March 31, 2003)

- *Designed and constructed controllable shutter system for the electron beam vapor deposition system*
- *Developed procedures for depositing wedge profiled films of iron, chromium, and nickel on sapphire substrates*
- *Determined optimum annealing conditions for alloying by solid-state diffusion*
- *Examined quality of the first ternary alloy library by synchrotron x-ray diffraction and fluorescence (APS at ANL)*
- *Began to explore alternative methods for alloying by electron beam, laser, & IR melting*

Graduate student making thin films in e-beam deposition system

ELECTRON BEAM DEPOSITION SYSTEM

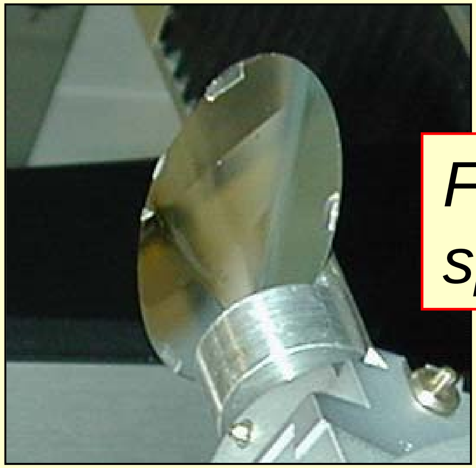


sample

main shutter

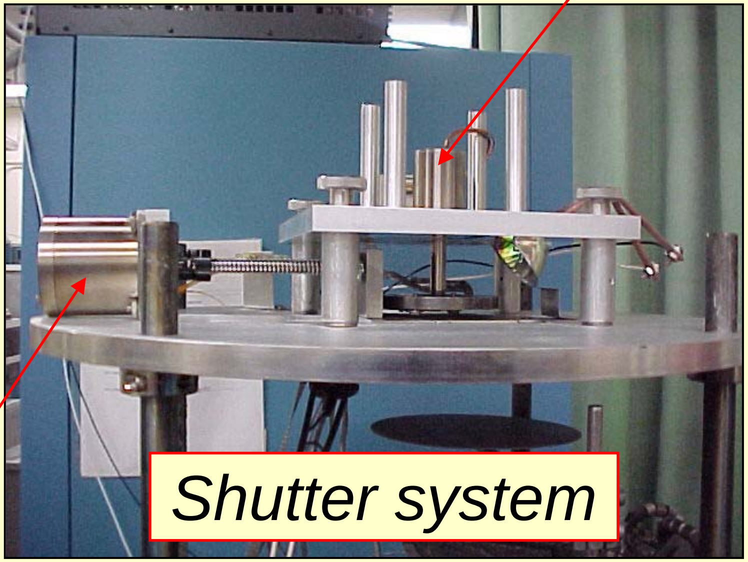
crucibles for source metal

Sample holder



Finished specimen

rotation motor

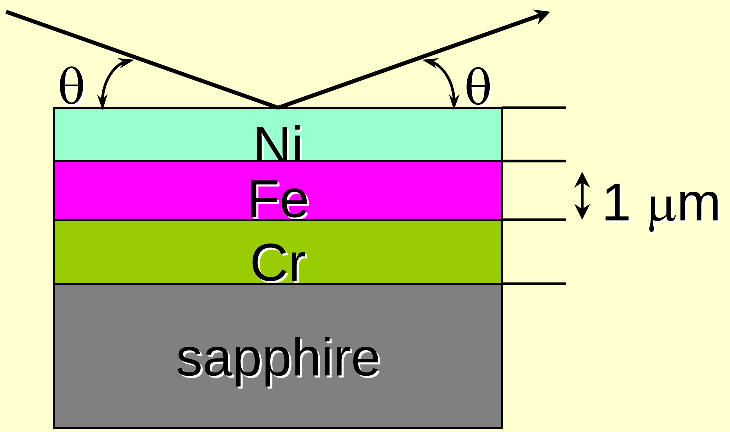


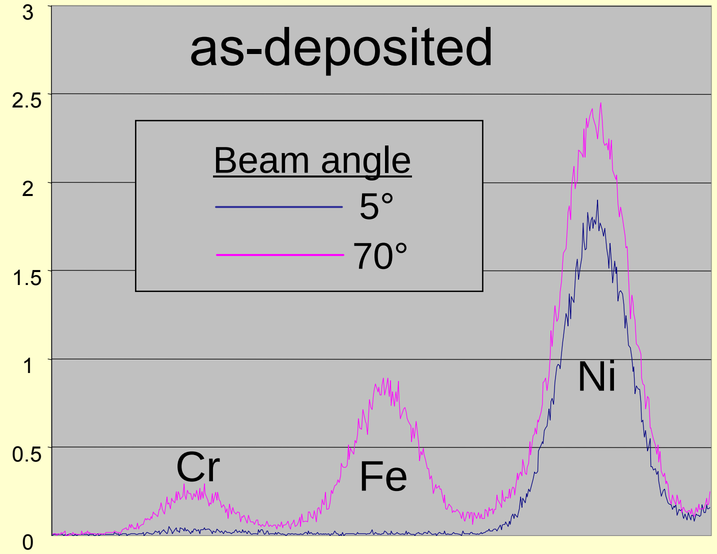
translation motor

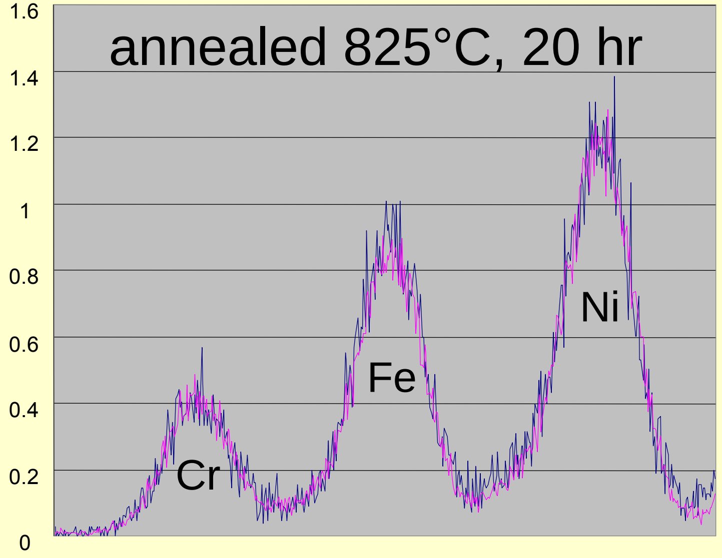
Shutter system

ANNEALING STUDIES

Angular resolved x-ray fluorescence

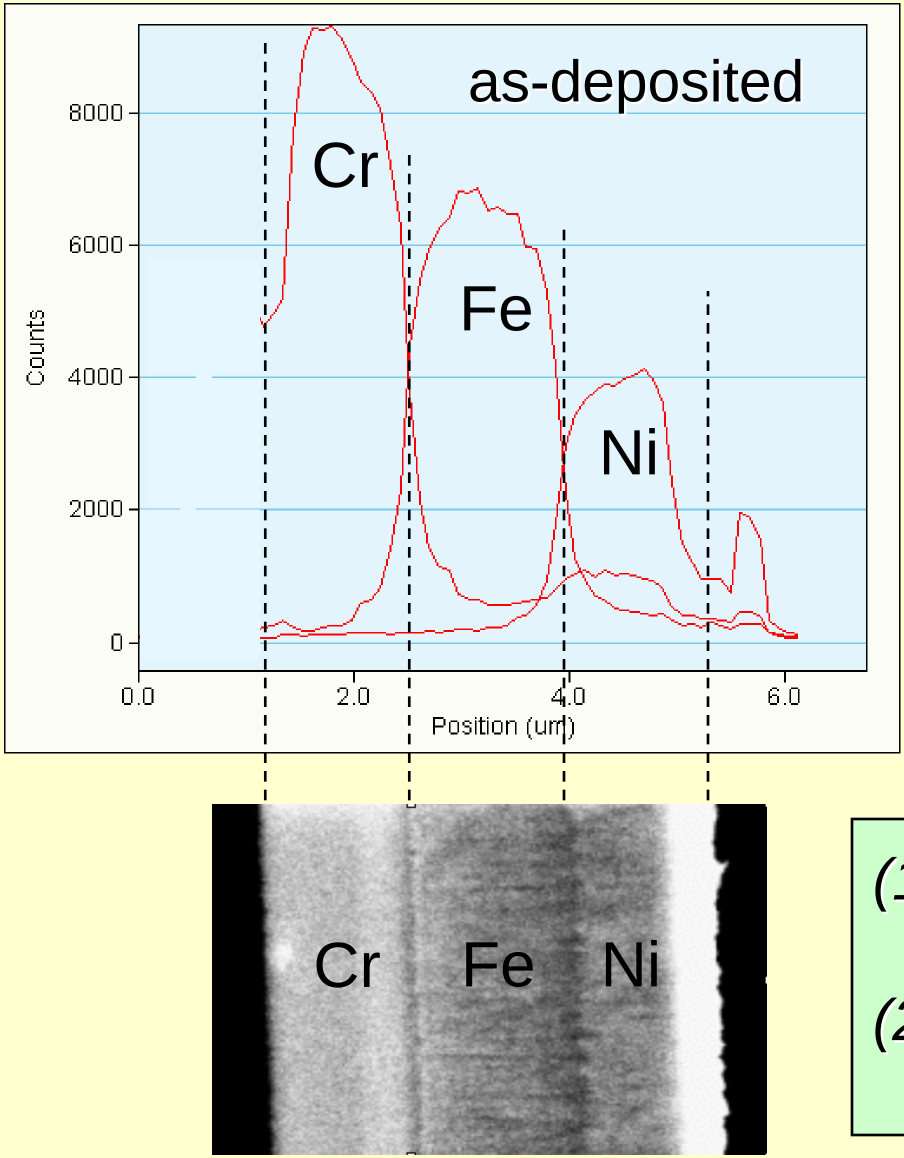


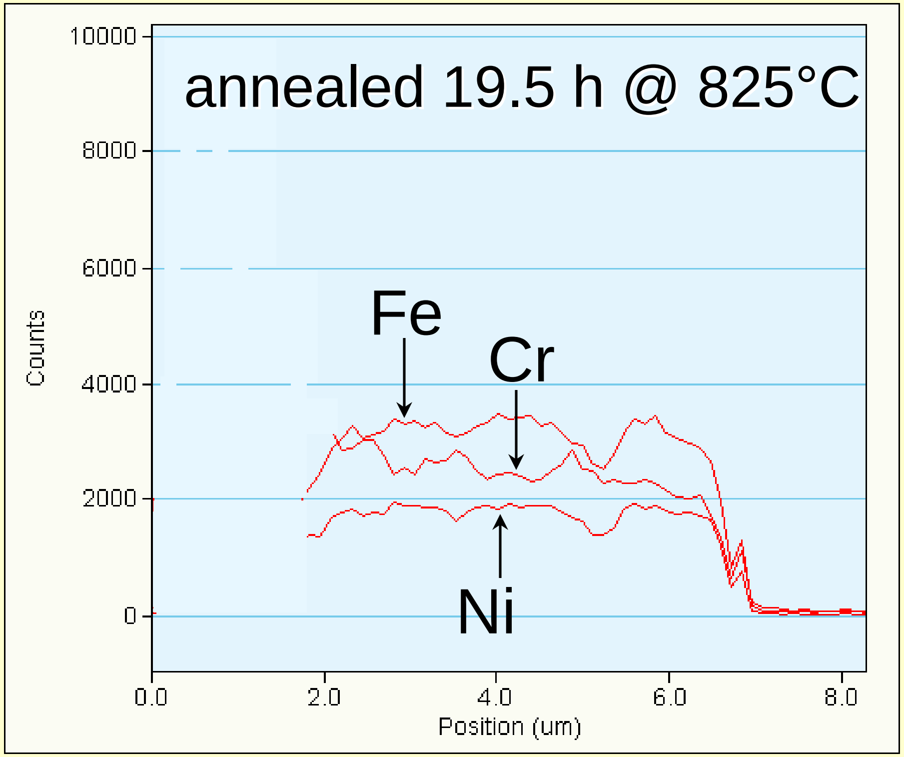




insensitivity of 825°C results to x-ray beam angle shows that alloying is complete after 20 hrs at 825°C

CROSS-SECTIONAL EDS



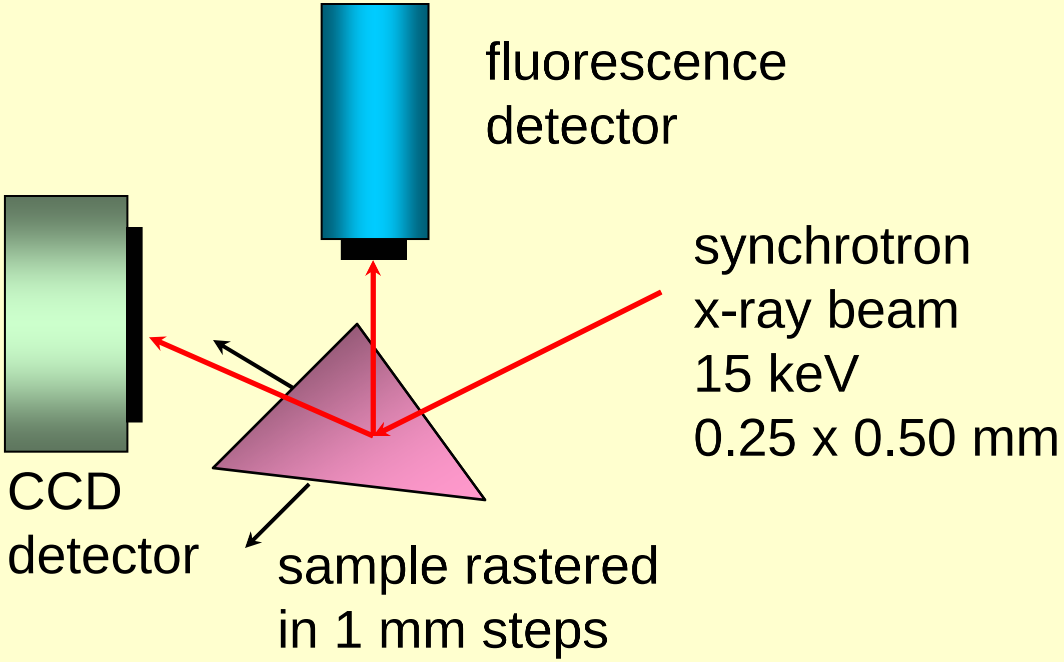


(1) good intermixing after 20-24 hr anneal at 825-850 °C

(2) for T>900°C, Cr evaporation causes difficulties

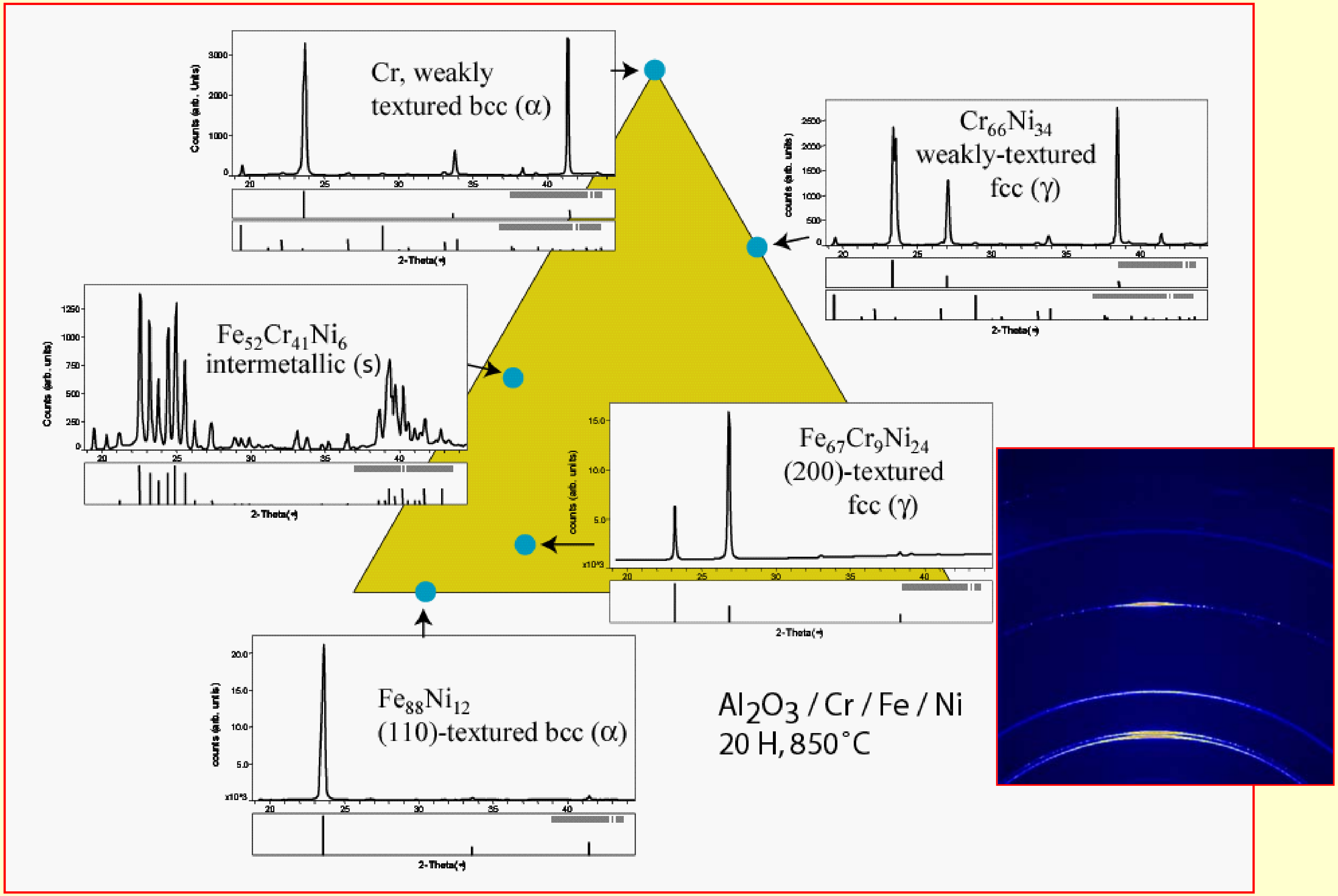
RAPID STRUCTURAL ASSESSMENT BY SYNCHROTRON TECHNIQUES

Advanced Photon Source at Argonne National Lab

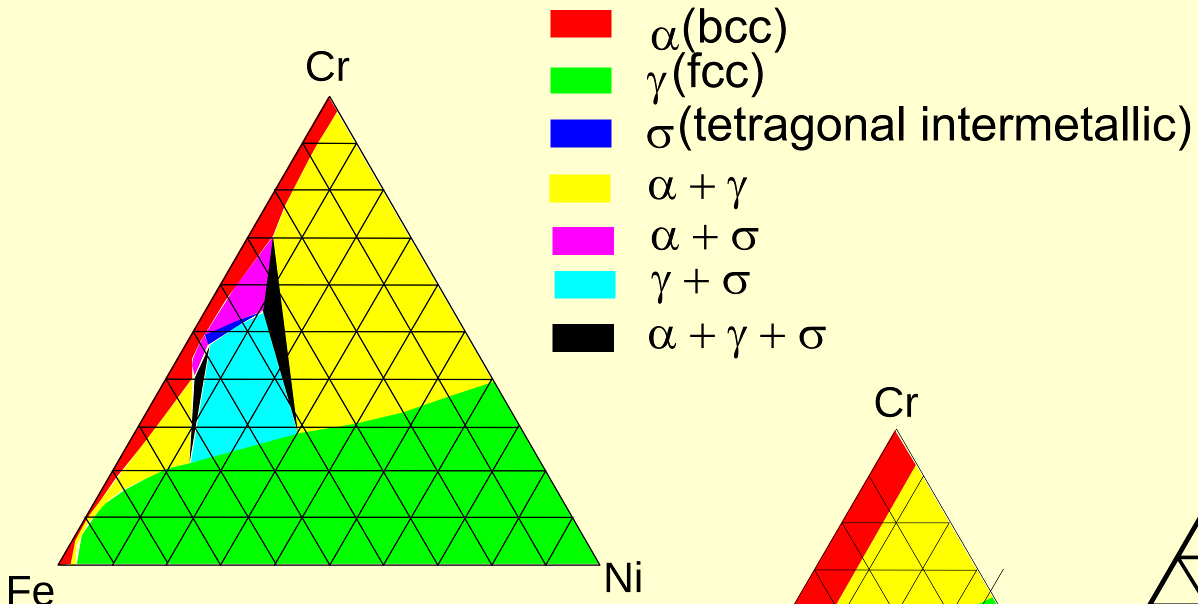


- *x-ray diffraction and fluorescence measured from 2600 positions in 4 hours*

PHASE IDENTIFICATION FROM DIFFRACTION

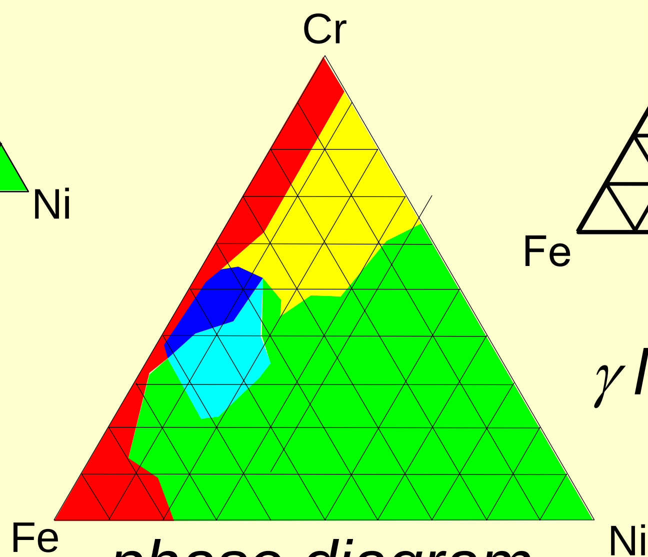


PHASES & LATTICE PARAMETERS

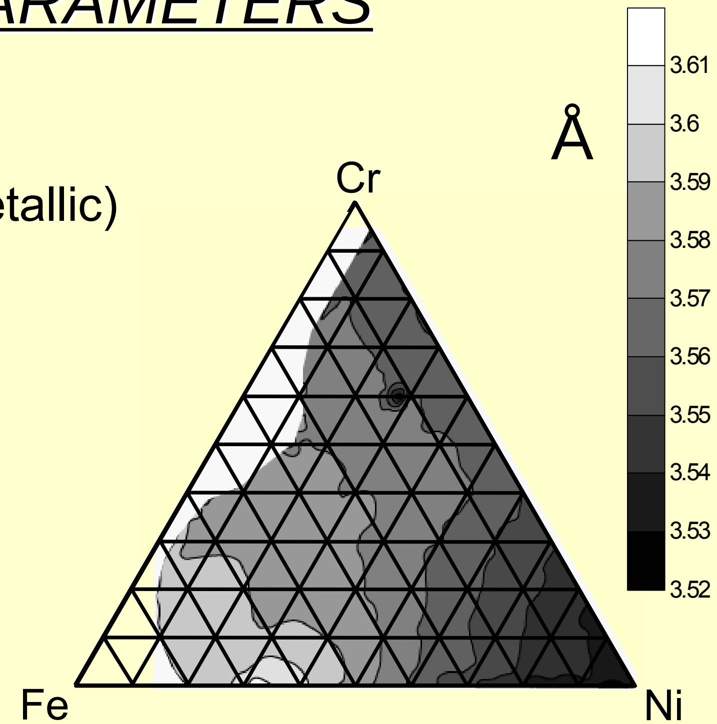


phase diagram from handbook

T = 850°C



phase diagram as measured



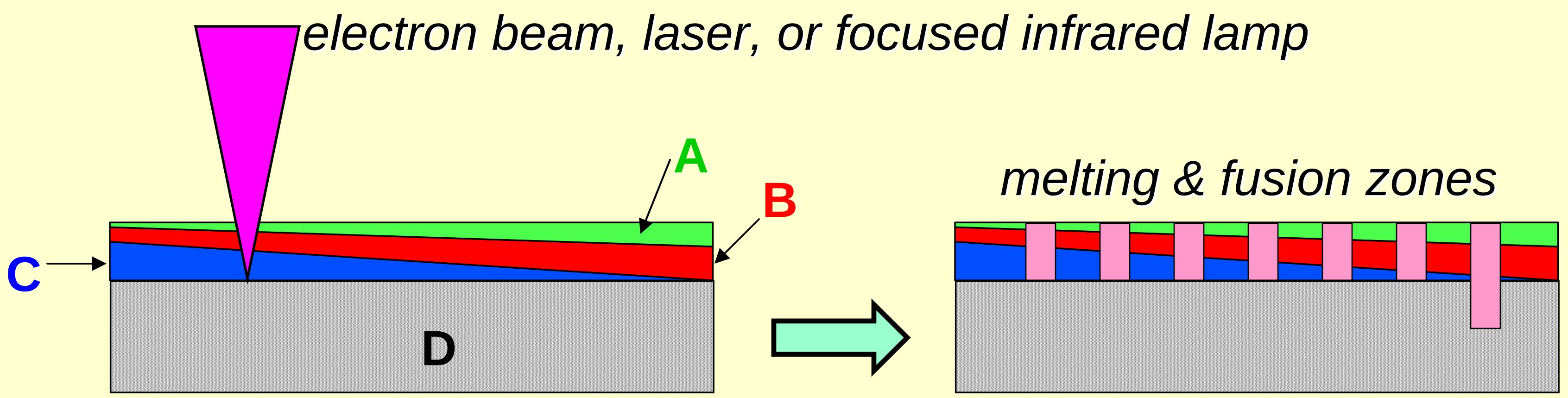
γ lattice parameters

- paper submitted to *Journal of Materials Research*

OTHER ALLOY LIBRARY PREPARATION TECHNIQUES

- *Electron beam melting with a focused electron-beam welding machine*
- *Laser melting with pulsed Nd:YAG laser*
- *Rapid melting with a focused IR lamp*
- *Co-deposition by simultaneous magnetron sputtering from three separate elemental targets (Fe, Ni, Cr)*
- *Complete melting using patterned substrates*

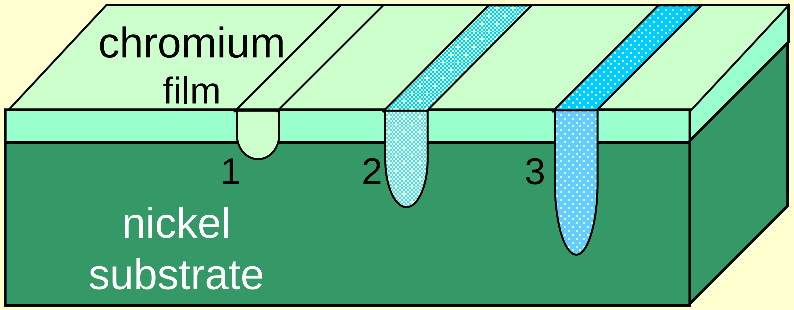
BASIC CONCEPT



Advantages:

- *eliminates guesswork in choosing annealing conditions*
- *not limited by T_m of lowest melting component*
- *high vapor pressure materials not a problem*
- *simulates solidification microstructures*
- *facilitates quarternary alloying*

FOCUSED ELECTRON BEAM MELTING: Cr on Ni



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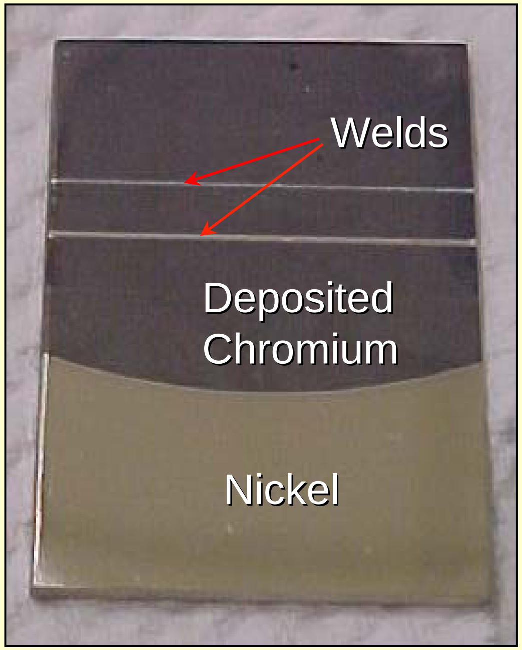
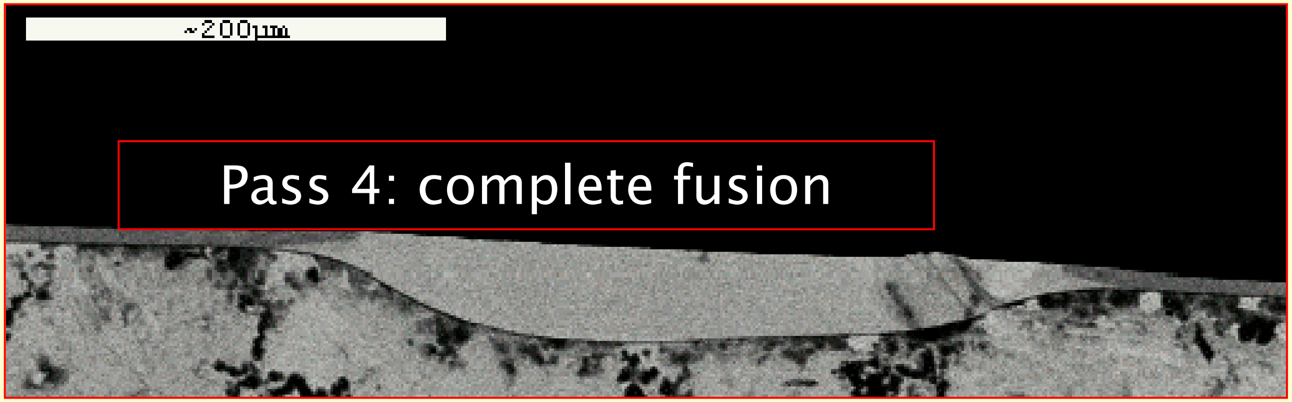
are needed to see this picture.

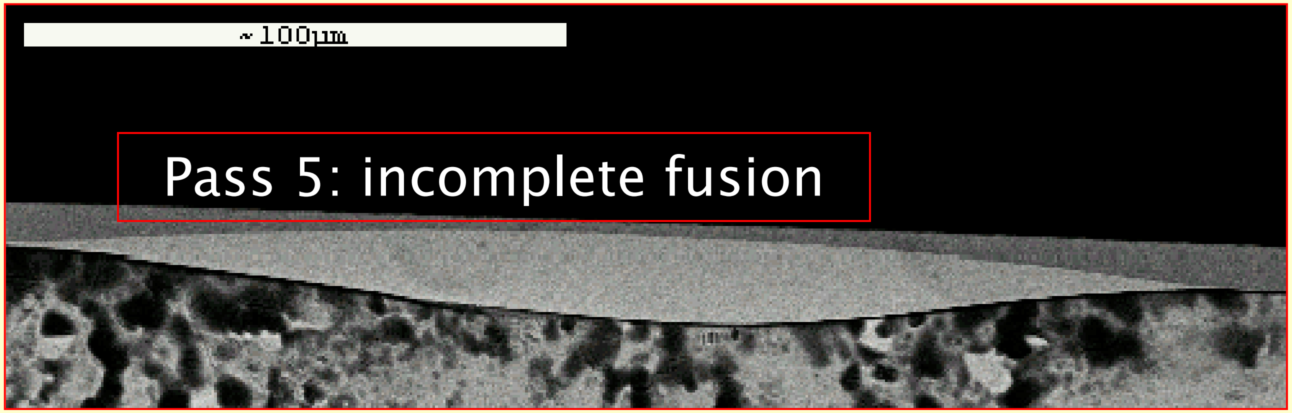


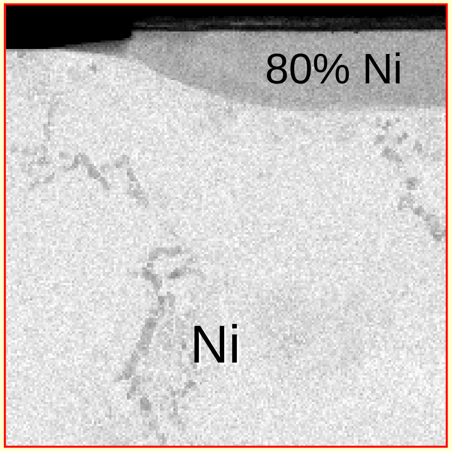
	Voltage (V)	Current (mamps)	Speed (in/min)
Pass 1*	75	0.2	50
Pass 2	75	0.5	50
Pass 3	75	1.0	50
Pass 4	75	1.5	50
Pass 5	150	0.7	50
Pass 6*	150	0.3	20
Pass 7*	150	0.3	5
*no marking visible on surface after weld			

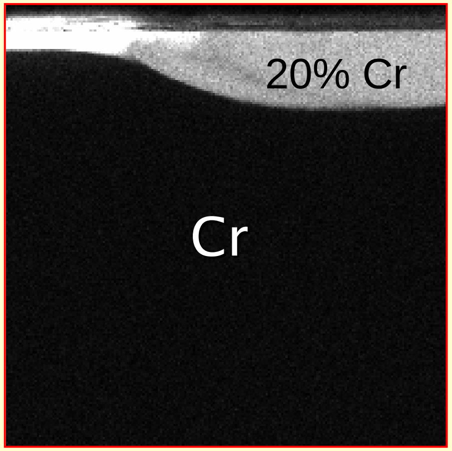
e-BEAM WELD PASSES IN CROSS SECTION

EDS element maps: Pass 4



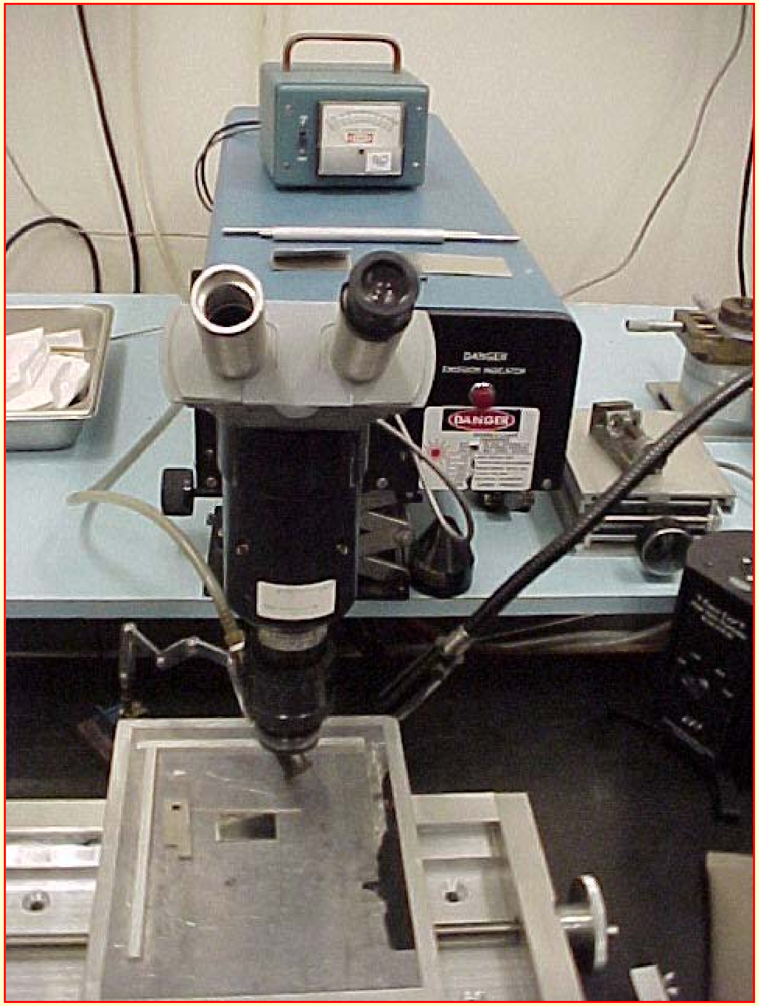






- *composition very uniform in fusion zone*

LASER MELTING



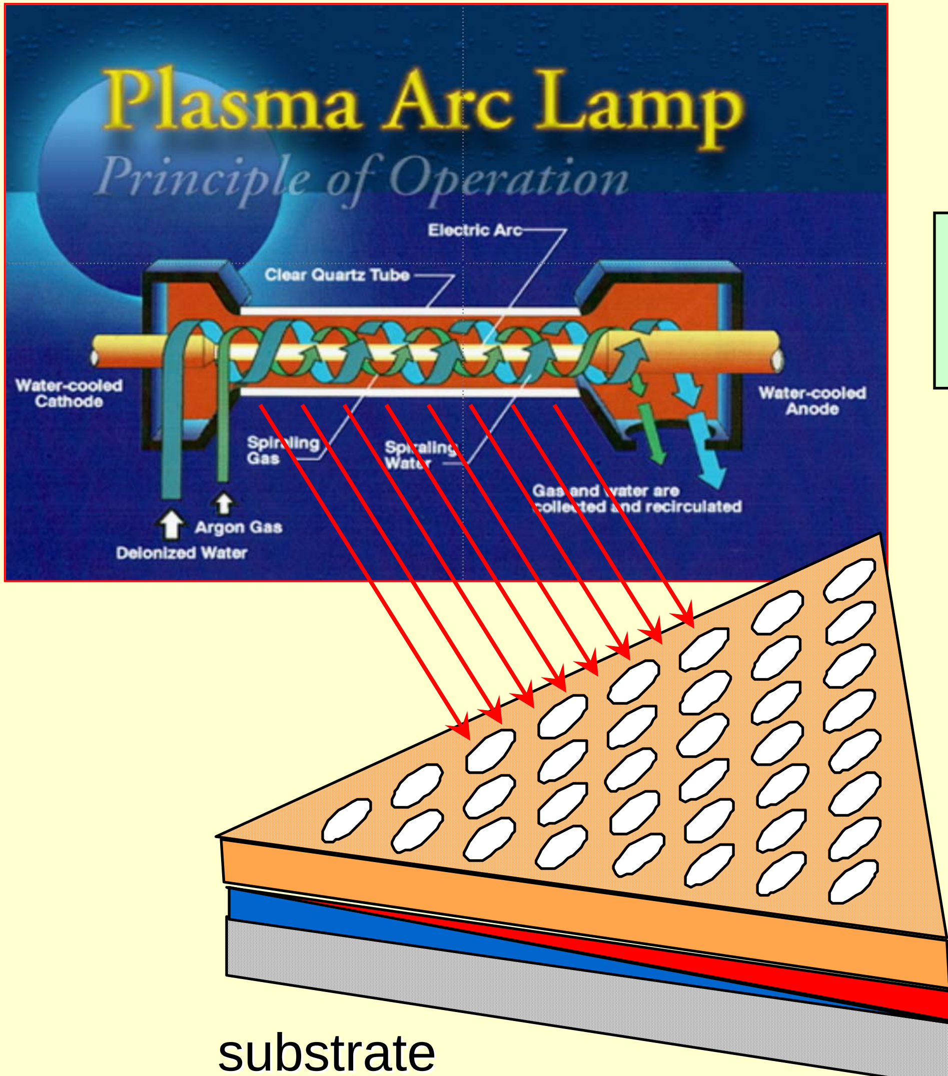
Nd:YAG Focused Pulsed Laser

Control variables:

- *power*
- *pulse rate*
- *pulse length*
- *focus*
- *melt time*

Advantage: works in air or inert gas atmosphere

INFRARED MELTING



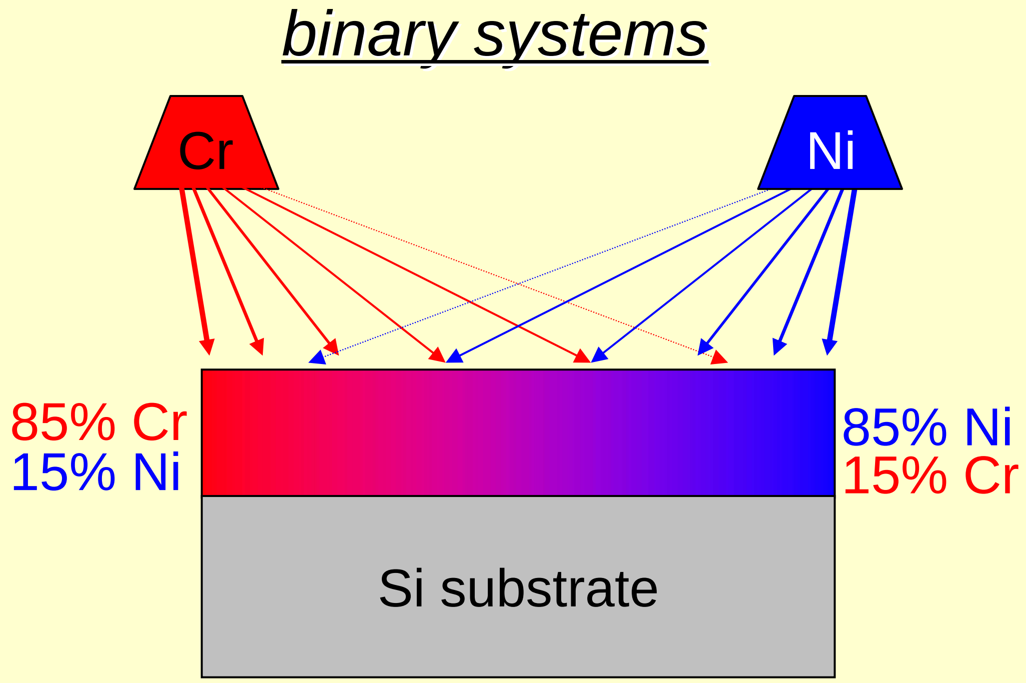
<u>Advantage</u>: rapid alloying of entire library at one time

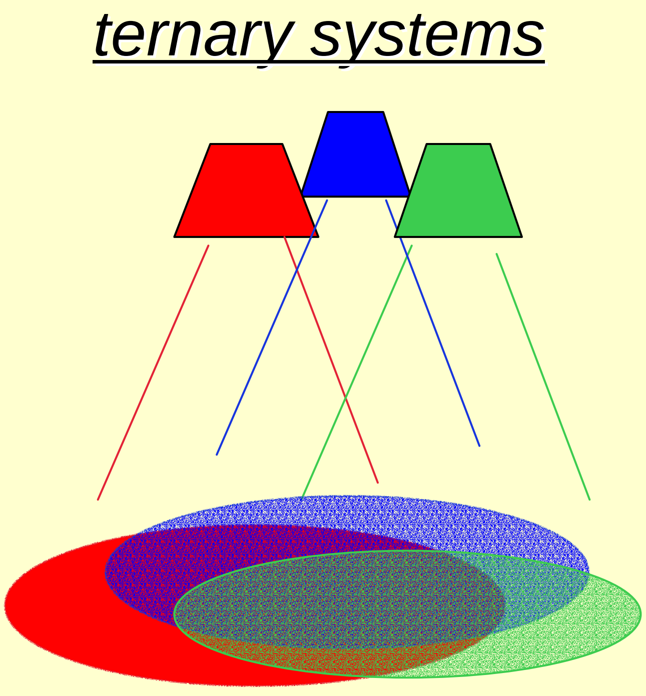
perforated reflective mask

thin films

substrate

CODEPOSITION BY MAGNETRON SPUTTERING



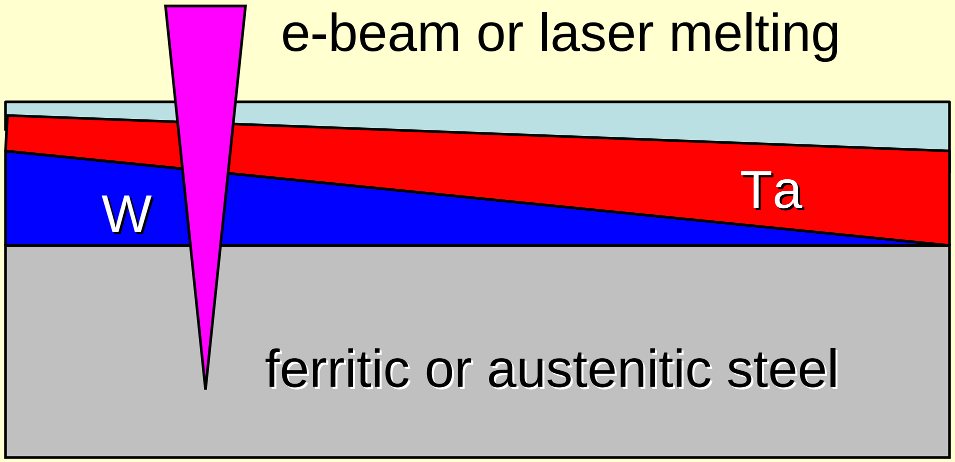


- ***Advantage**:* *no alloying required - mixing occurs during film deposition*
- ***Disadvantage**:* *cannot cover full range of composition*

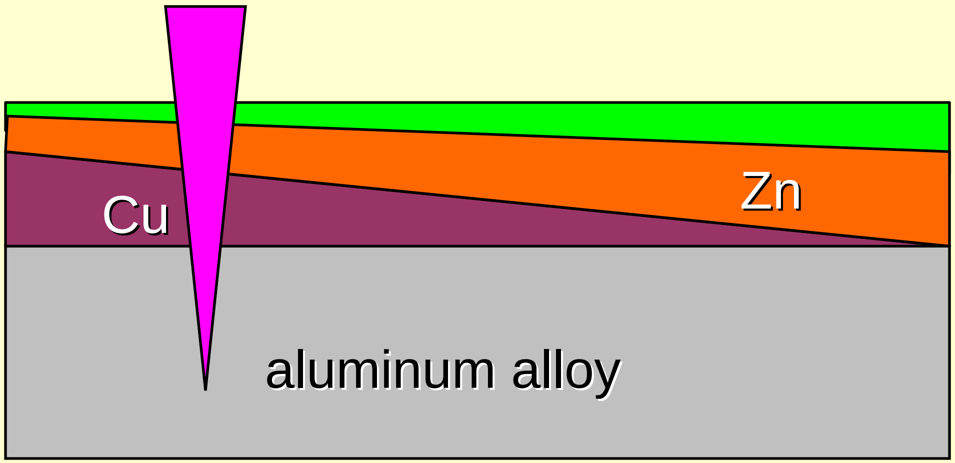
PLANS FOR YEAR 2

- *Prepare high-quality ternary alloy library specimens for chemical and structural characterization and nanoindentation testing.*
- *Further develop techniques for rapid assessment of chemical compositions, phases and structures based on electron microprobe analysis, x-ray diffraction, and synchrotron radiation methods.*
- *Prepare conventional alloys for comparison of properties to alloy libraries.*
- *Develop methods for measuring mechanical properties of alloy libraries by nanoindentation.*
- *Further develop e-beam, laser and IR melting techniques for preparing alloy libraries.*

POSSIBLE APPLICATION TO COMMERCIAL ALLOYS



Ferritic and austenitic steels alloyed with Nb, Ta, W, & V



Commercial aluminum alloyed with Cu, Zn, Mg, Zr, Ti, Sc, etc.