



# High-Throughput Electric-Field-Assisted Sintering and Characterization Techniques for Materials Discovery

March 2023

*Changing the World's Energy Future*

Michael J Moorehead, Arin Seth Preston, Zilong Hua, Jorgen Fredrick Rufner



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**March 2023**

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**<http://www.inl.gov>**

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Advanced Nuclear  
Materials Scientist

# High-Throughput Electric-Field-Assisted Sintering and Characterization Techniques for Materials Discovery

Co-authors: Arin Preston, Zilong Hua, Jorgen Rufner

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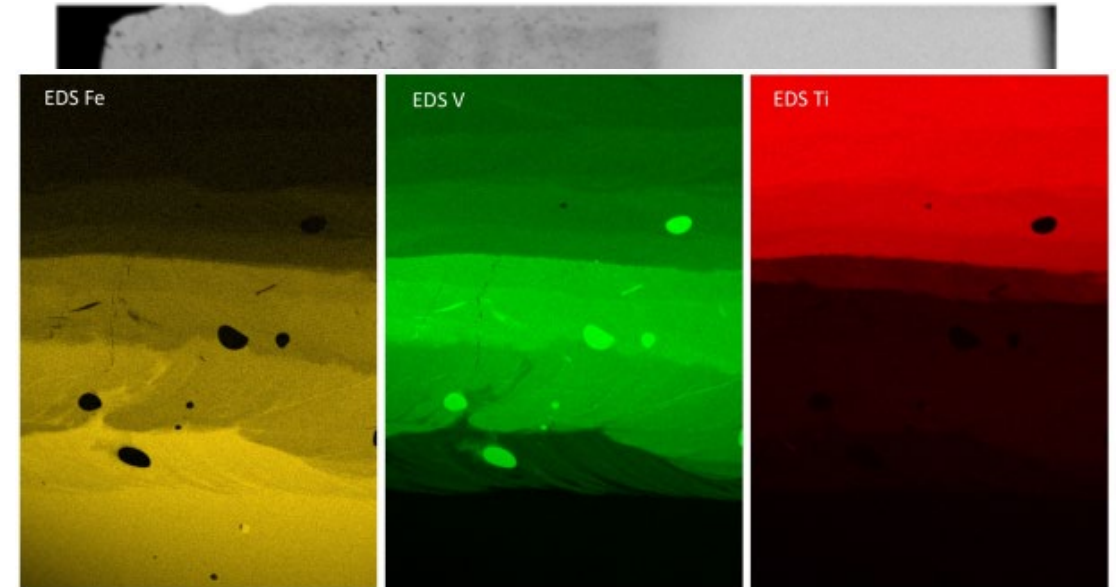
Idaho National Laboratory



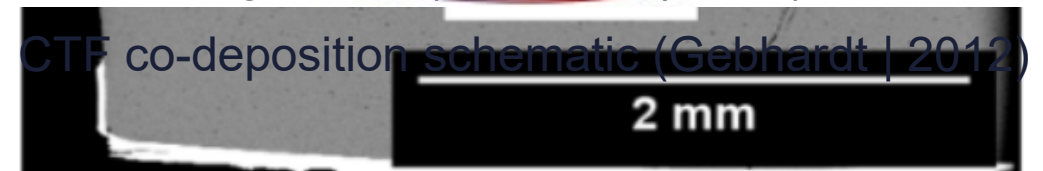
# High-Throughput Materials Synthesis

- Diffusion multiples
  - Small volume, limited composition control
- Combinatorial thin films (CTFs)
  - Limited to ~microns of material
- Additively manufactured gradients
  - Non-uniform, limited composition control

**“Bulk” high-throughput synthesis needed for arbitrary compositions and volumes**



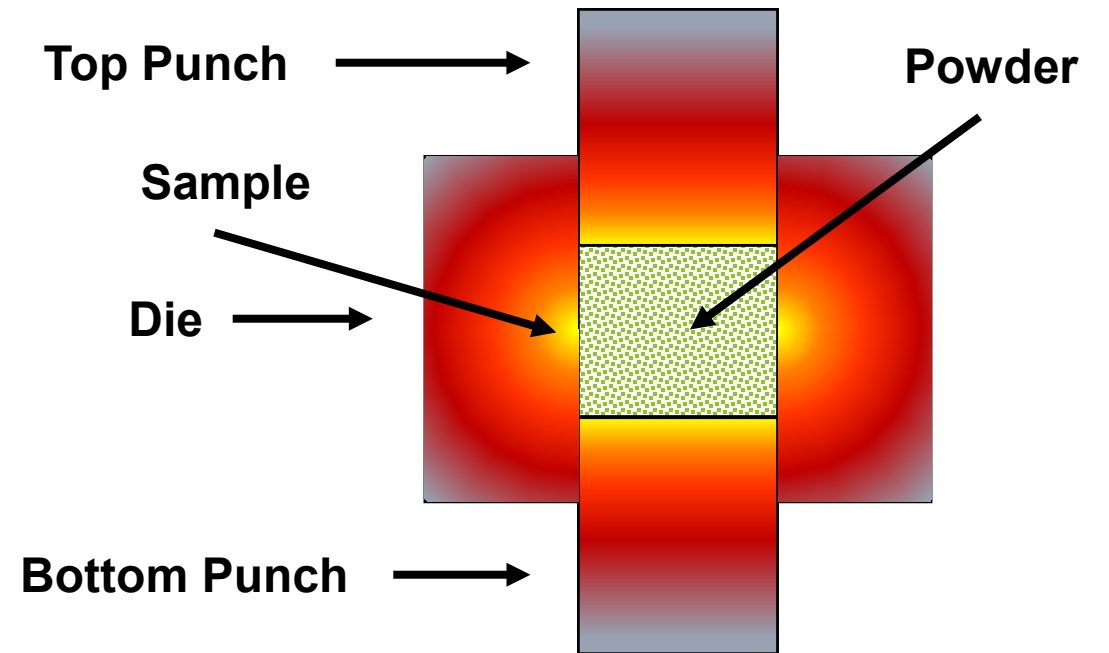
420 martensitic steel to vanadium to Ti-6V-4Al gradient (Reichardt | 2017)



Co-Cr-Fe-Mn-Ni diffusion multiple (Wilson | 2016)

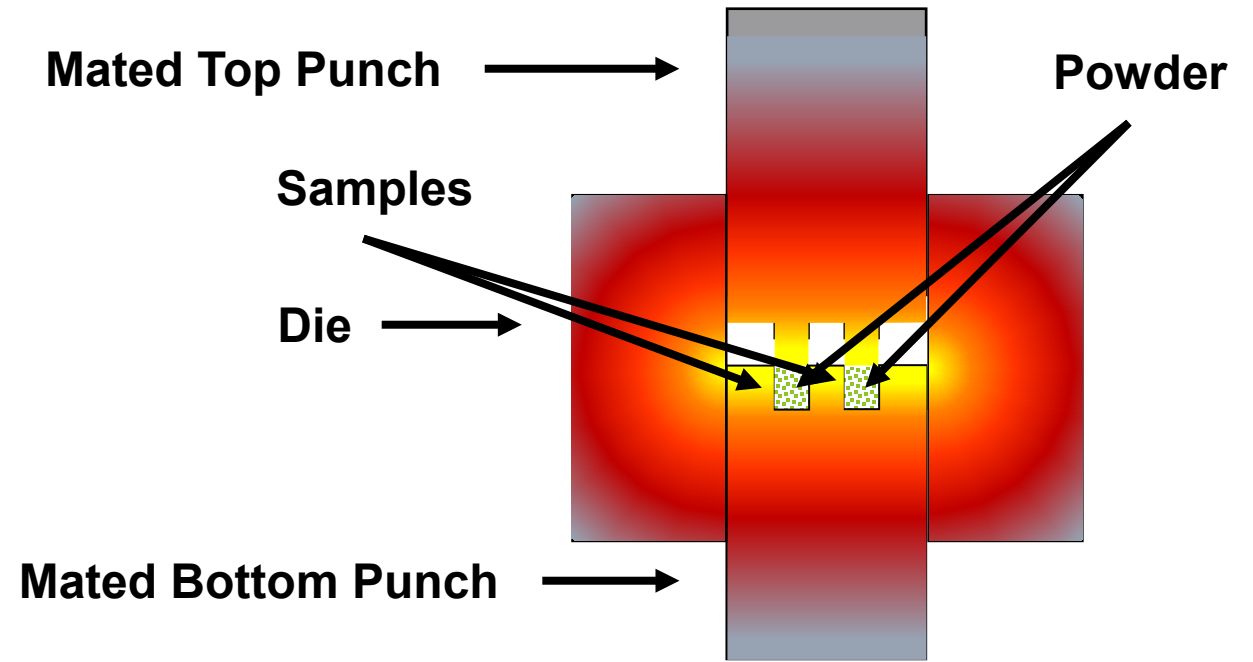
# Current Material Synthesis via EFAS

- Single-Sample Production
  - Two flat punches
  - Single powder (mixture)



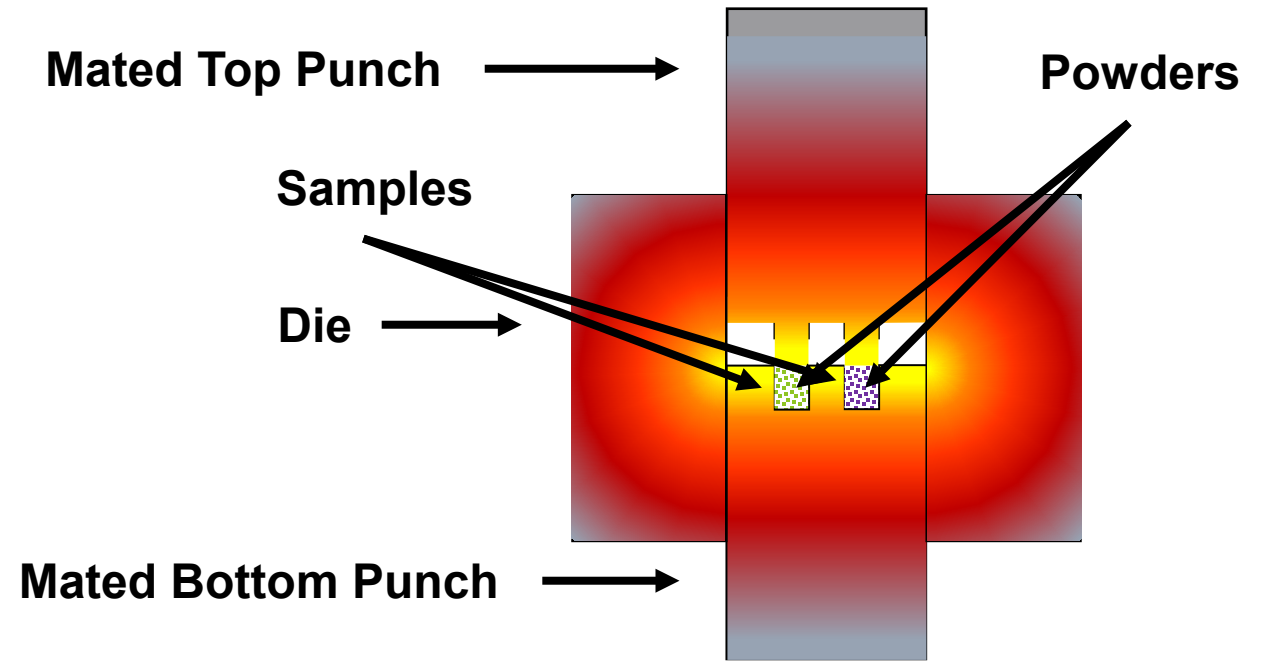
# Current Material Synthesis via EFAS

- Single-Sample Production
  - Two flat punches
  - Single powder (mixture)
- Multi-Sample Production
  - Mated top/bottom punches
  - Single powder (mixture)



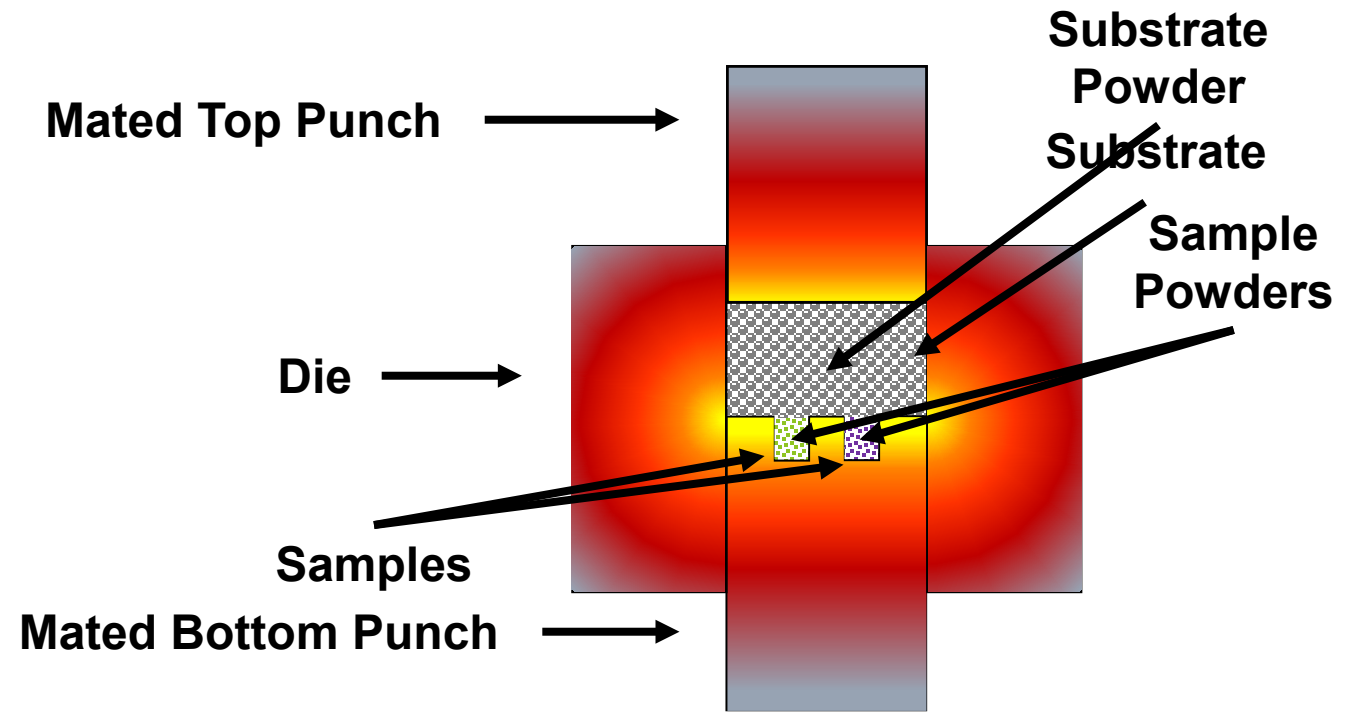
# Current Material Synthesis via EFAS

- Single-Sample Production
  - Two flat punches
  - Single powder (mixture)
- Multi-Sample Production
  - Mated top/bottom punches
  - Single powder (mixture)
- Limitations:
  - Different materials in cavities
  - Different shaped cavities requires machining new pair of punches



# Parallelized EFAS Sample Production

- Multi-Sample Production
  - Single custom punch
  - Varying powder mixtures
  - Varying sample geometries



# Parallelized EFAS Sample Production

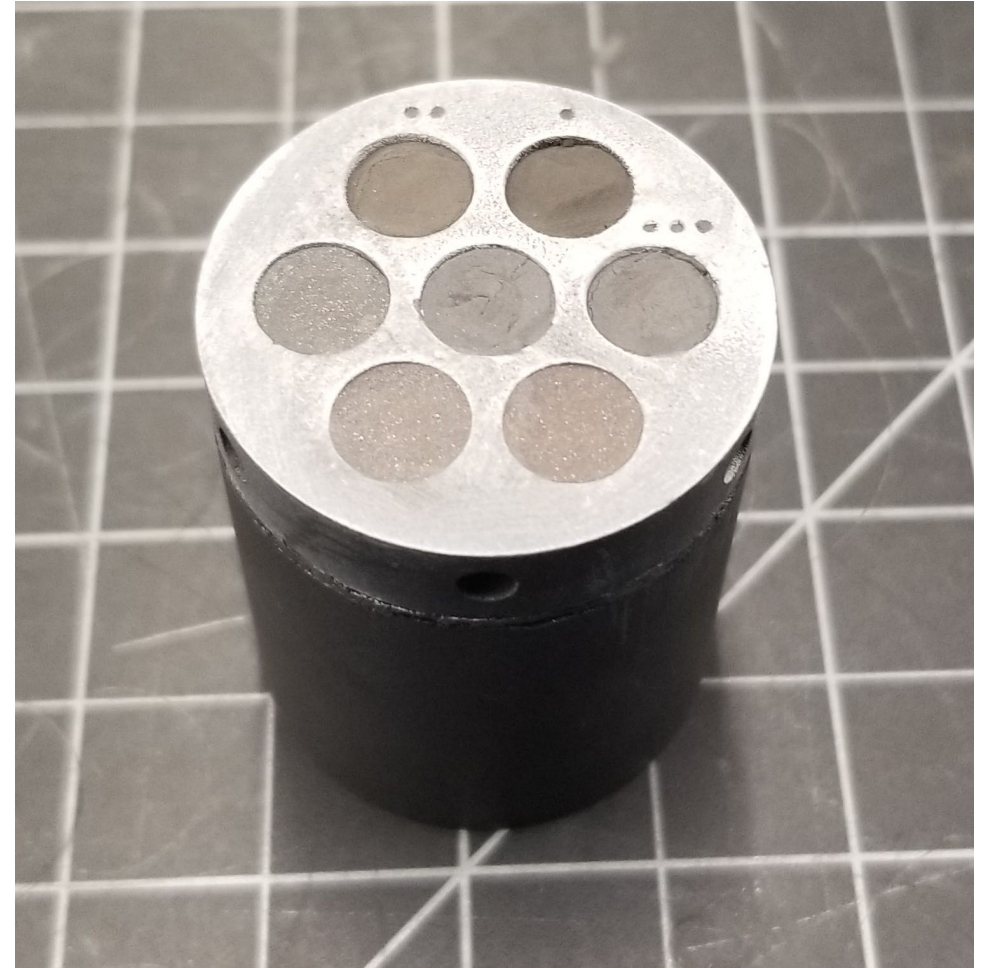


1 <b>H</b> Hydrogen 1.008	Atomic Symbol Name Weight																2 <b>He</b> Helium 4.0026	
3 <b>Li</b> Lithium 6.94	4 <b>Be</b> Beryllium 9.0122																	18 <b>Ar</b> Argon 39.948
11 <b>Na</b> Sodium 22.990	12 <b>Mg</b> Magnesium 24.305																	35 <b>Br</b> Bromine 79.904
19 <b>K</b> Potassium 39.098	20 <b>Ca</b> Calcium 40.078	21 <b>Sc</b> Scandium 44.956	22 <b>Ti</b> Titanium 47.867	23 <b>V</b> Vanadium 50.942	24 <b>Cr</b> Chromium 51.996	25 <b>Mn</b> Manganese 54.938	26 <b>Fe</b> Iron 55.845	27 <b>Co</b> Cobalt 58.933	28 <b>Ni</b> Nickel 58.693	29 <b>Cu</b> Copper 63.546	30 <b>Zn</b> Zinc 65.38	31 <b>Ga</b> Gallium 69.723	32 <b>Ge</b> Germanium 72.630	33 <b>As</b> Arsenic 74.922	34 <b>Se</b> Selenium 78.971	35 <b>Br</b> Bromine 79.904	36 <b>Kr</b> Krypton 83.798	
37 <b>Rb</b> Rubidium 85.468	38 <b>Sr</b> Strontium 87.62	39 <b>Y</b> Yttrium 88.906	40 <b>Zr</b> Zirconium 91.224	41 <b>Nb</b> Niobium 92.906	42 <b>Mo</b> Molybdenum 95.95	43 <b>Tc</b> Technetium (98)	44 <b>Ru</b> Ruthenium 101.07	45 <b>Rh</b> Rhodium 102.91	46 <b>Pd</b> Palladium 106.42	47 <b>Ag</b> Silver 107.87	48 <b>Cd</b> Cadmium 112.41	49 <b>In</b> Indium 114.82	50 <b>Sn</b> Tin 118.71	51 <b>Sb</b> Antimony 121.76	52 <b>Te</b> Tellurium 127.60	53 <b>I</b> Iodine 126.90	54 <b>Xe</b> Xenon 131.29	
55 <b>Cs</b> Caesium 132.91	56 <b>Ba</b> Barium 137.33	57-71	72 <b>Hf</b> Hafnium 178.49	73 <b>Ta</b> Tantalum 180.95	74 <b>W</b> Tungsten 183.84	75 <b>Re</b> Rhenium 186.21	76 <b>Os</b> Osmium 190.23	77 <b>Ir</b> Iridium 192.22	78 <b>Pt</b> Platinum 195.08	79 <b>Au</b> Gold 196.97	80 <b>Hg</b> Mercury 200.59	81 <b>Tl</b> Thallium 204.38	82 <b>Pb</b> Lead 207.2	83 <b>Bi</b> Bismuth 208.98	84 <b>Po</b> Polonium (209)	85 <b>At</b> Astatine (210)	86 <b>Rn</b> Radon (222)	
87 <b>Fr</b> Francium (223)	88 <b>Ra</b> Radium (226)	89-103	104 <b>Rf</b> Rutherfordium (267)	105 <b>Db</b> Dubnium (268)	106 <b>Sg</b> Seaborgium (269)	107 <b>Bh</b> Bohrium (270)	108 <b>Hs</b> Hassium (277)	109 <b>Mt</b> Meitnerium (278)	110 <b>Ds</b> Darmstadtium (281)	111 <b>Rg</b> Roentgenium (282)	112 <b>Cn</b> Copernicium (285)	113 <b>Nh</b> Nihonium (286)	114 <b>Fl</b> Flerovium (289)	115 <b>Mc</b> Moscovium (290)	116 <b>Lv</b> Livermorium (293)	117 <b>Ts</b> Tennessine (294)	118 <b>Og</b> Oganesson (294)	
For elements with no stable isotopes, the mass number of the isotope with the longest half-life is in parentheses.																		
		57 <b>La</b> Lanthanum 138.91	58 <b>Ce</b> Cerium 140.12	59 <b>Pr</b> Praseodymium 140.91	60 <b>Nd</b> Neodymium 144.24	61 <b>Pm</b> Promethium (145)	62 <b>Sm</b> Samarium 150.36	63 <b>Eu</b> Europium 151.96	64 <b>Gd</b> Gadolinium 157.25	65 <b>Tb</b> Terbium 158.93	66 <b>Dy</b> Dysprosium 162.50	67 <b>Ho</b> Holmium 164.93	68 <b>Er</b> Erbium 167.26	69 <b>Tm</b> Thulium 168.93	70 <b>Yb</b> Ytterbium 173.05	71 <b>Lu</b> Lutetium 174.97		
		89 <b>Ac</b> Actinium (227)	90 <b>Th</b> Thorium 232.04	91 <b>Pa</b> Protactinium 231.04	92 <b>U</b> Uranium 238.03	93 <b>Np</b> Neptunium (237)	94 <b>Pu</b> Plutonium (244)	95 <b>Am</b> Americium (243)	96 <b>Cm</b> Curium (247)	97 <b>Bk</b> Berkelium (247)	98 <b>Cf</b> Californium (251)	99 <b>Es</b> Einsteinium (252)	100 <b>Fm</b> Fermium (257)	101 <b>Md</b> Mendelevium (258)	102 <b>No</b> Nobelium (259)	103 <b>Lr</b> Lawrencium (260)		



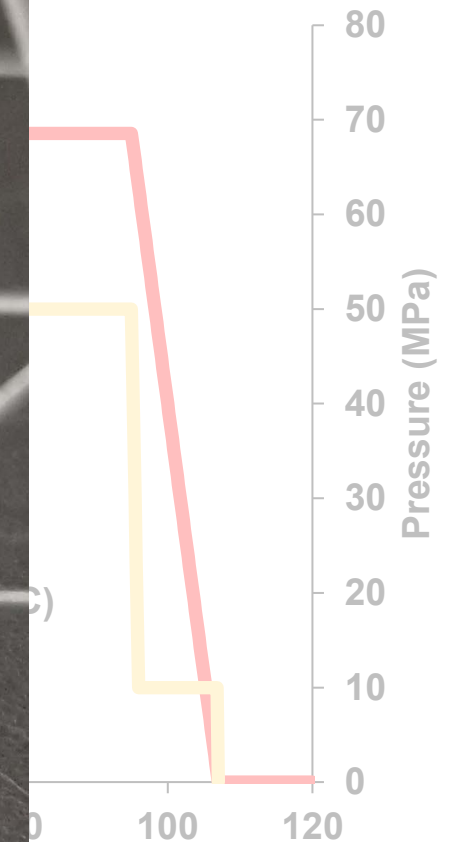
# HEA Case Study

- Material preparation
  - 99.5%+ pure elemental powders, <10-micron powder size
  - 5-g powder blends mixed for 15 minutes via Turbula mixer in parallel
  - <1 g of each alloy mixture added to wells
  - Nickel shim stock placed atop wells
  - 15 g of Ni powder added above shim stock



# HEA Case Study

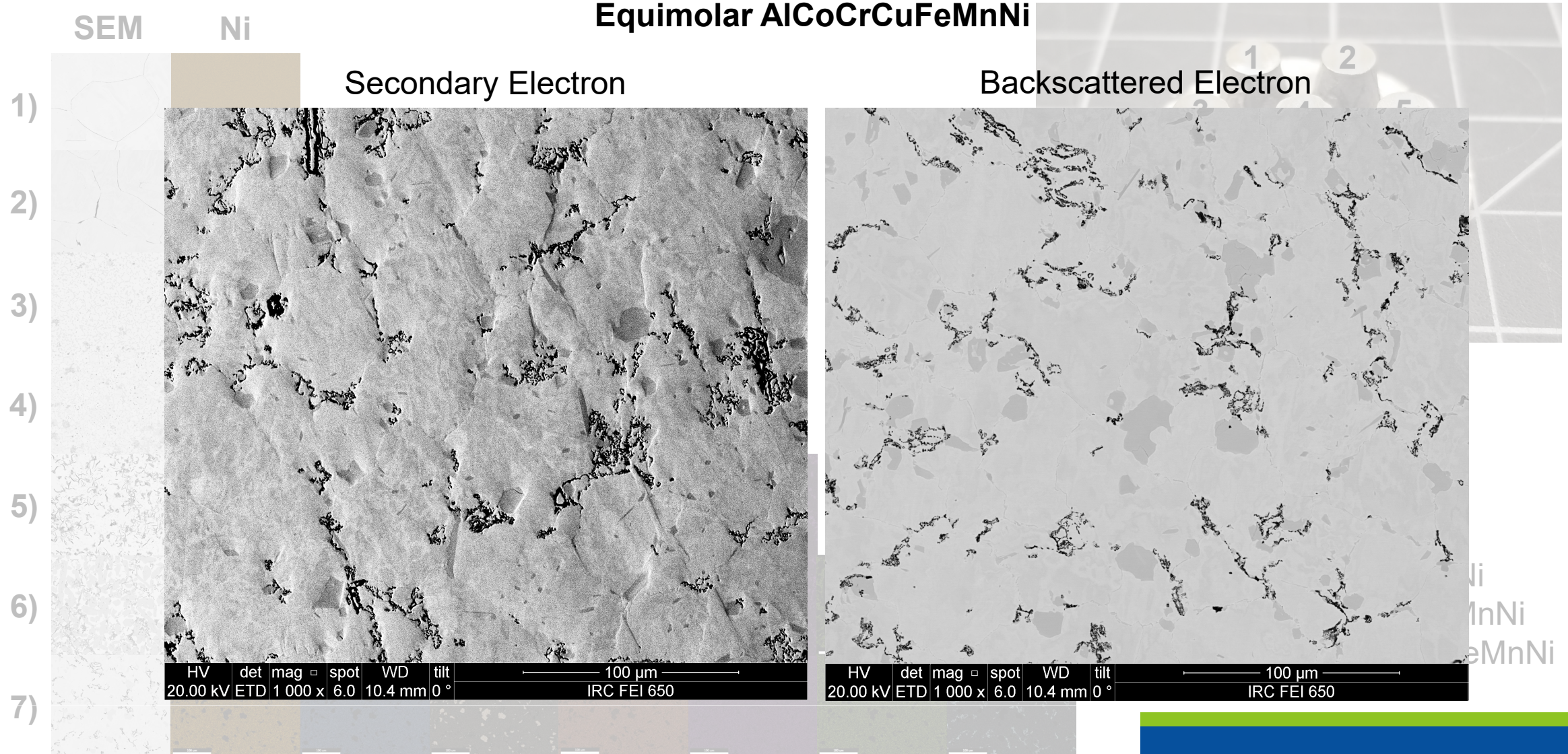
- EFAS pro
- Press  
MPa/r
- Temp
- 90
- 11
- 12
- 1-hou
- Press  
down





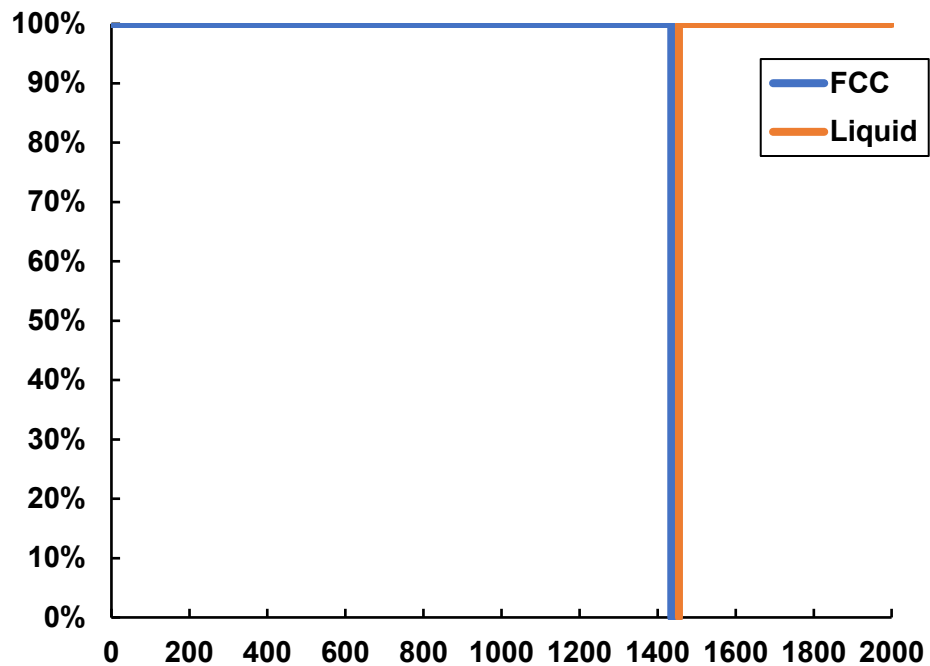
# HEA Case Study: SEM/EDS

Equimolar AlCoCrCuFeMnNi

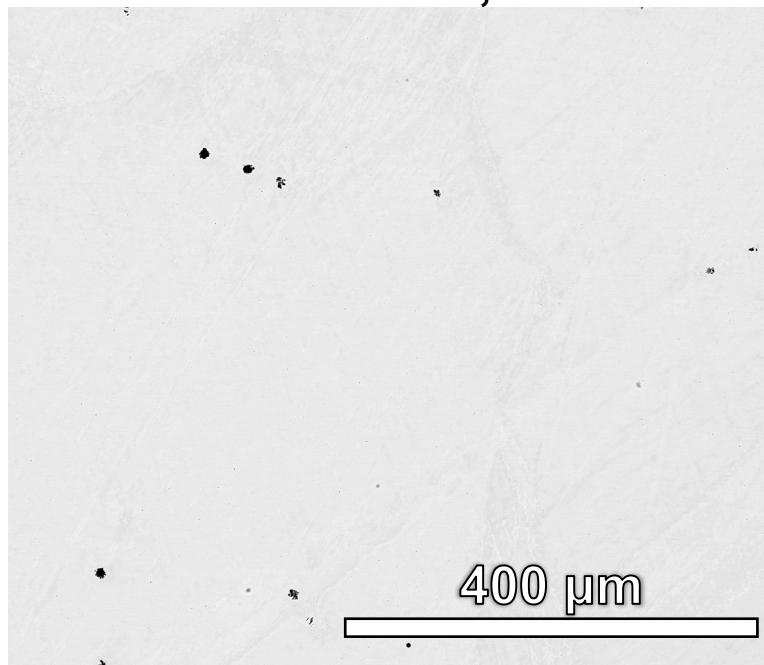


Ni

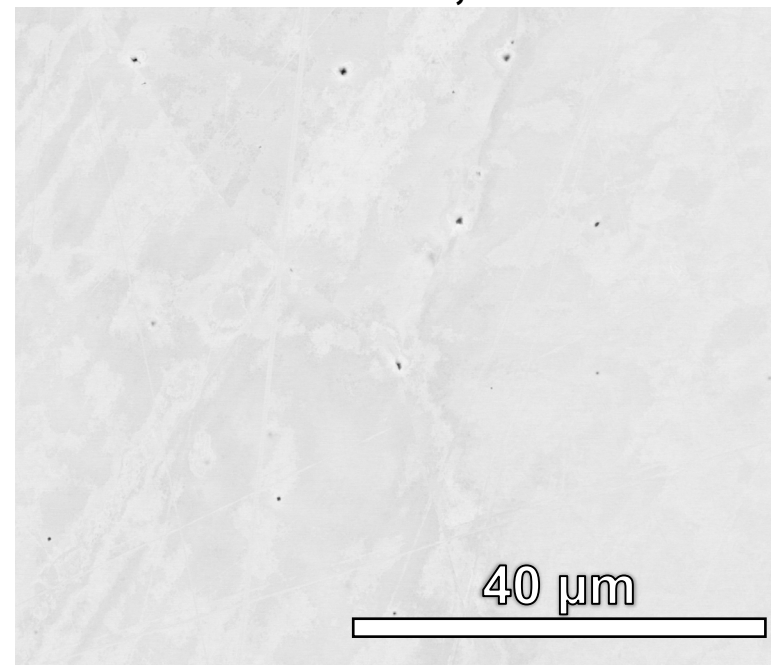
CALPHAD



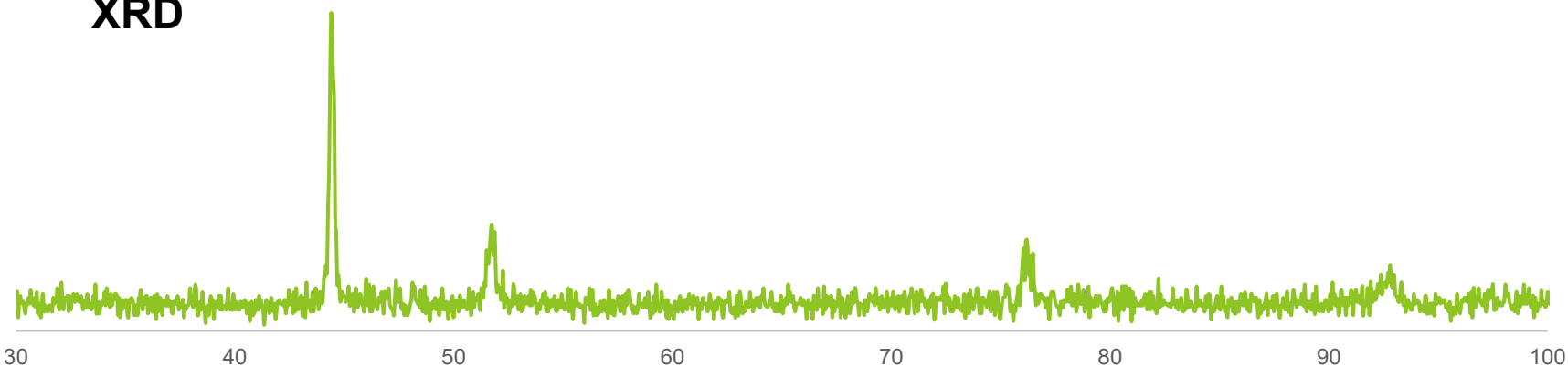
Backscatter, 200x



Backscatter, 2000x



XRD



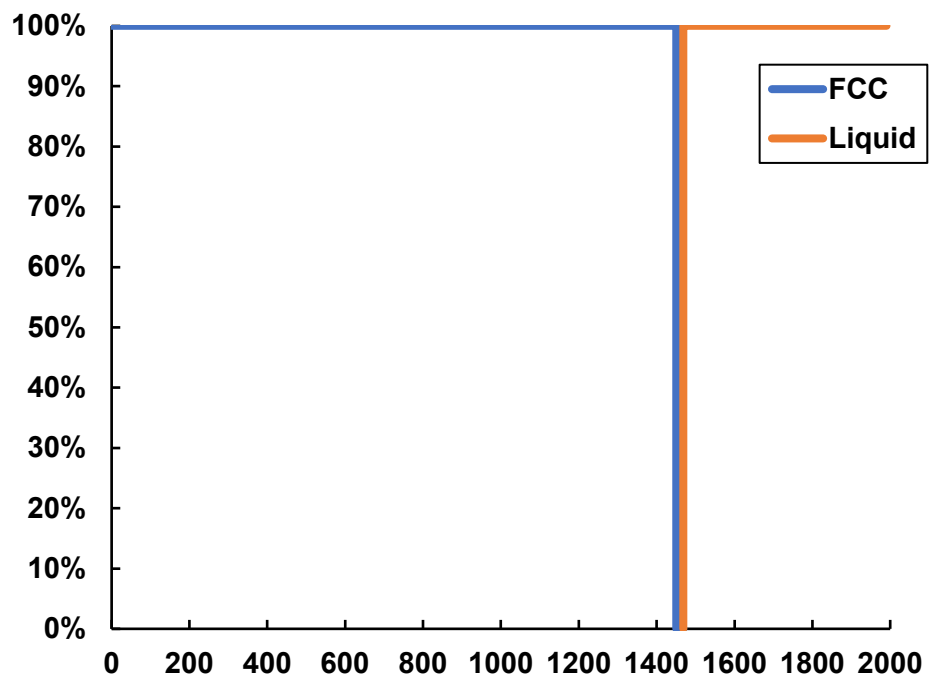
CALPHAD: FCC

XRD: FCC

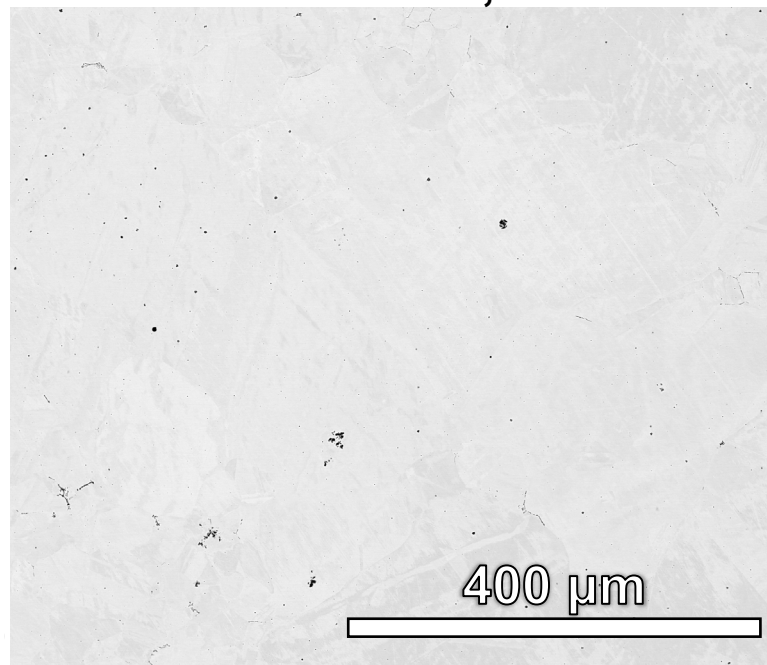


# NiCo

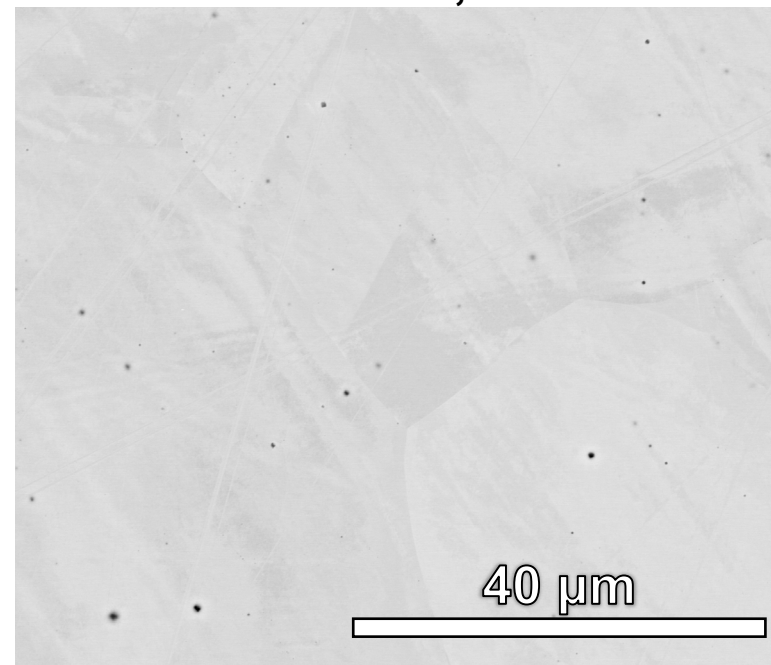
**CALPHAD**



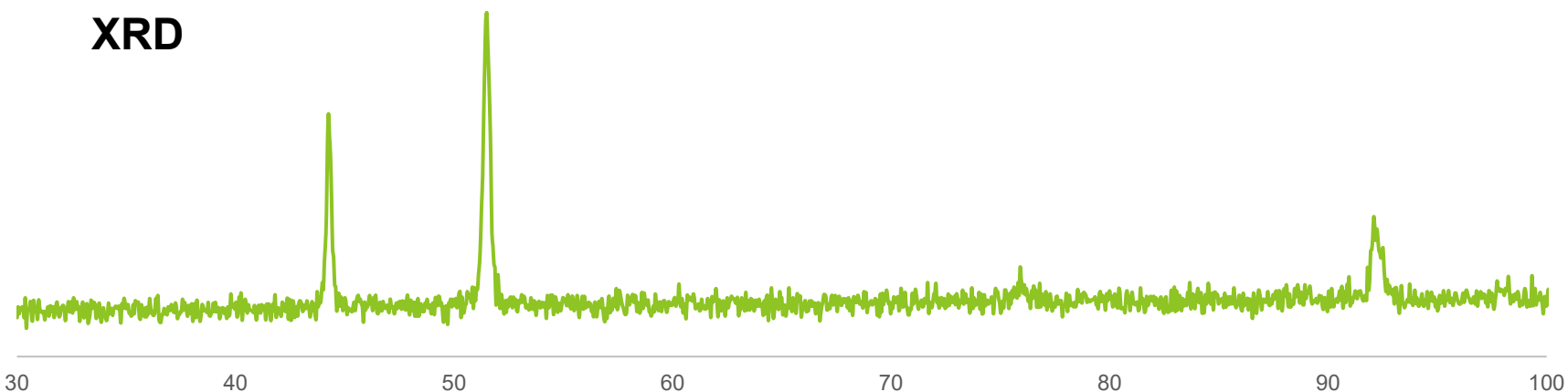
**Backscatter, 200x**



**Backscatter, 2000x**



**XRD**

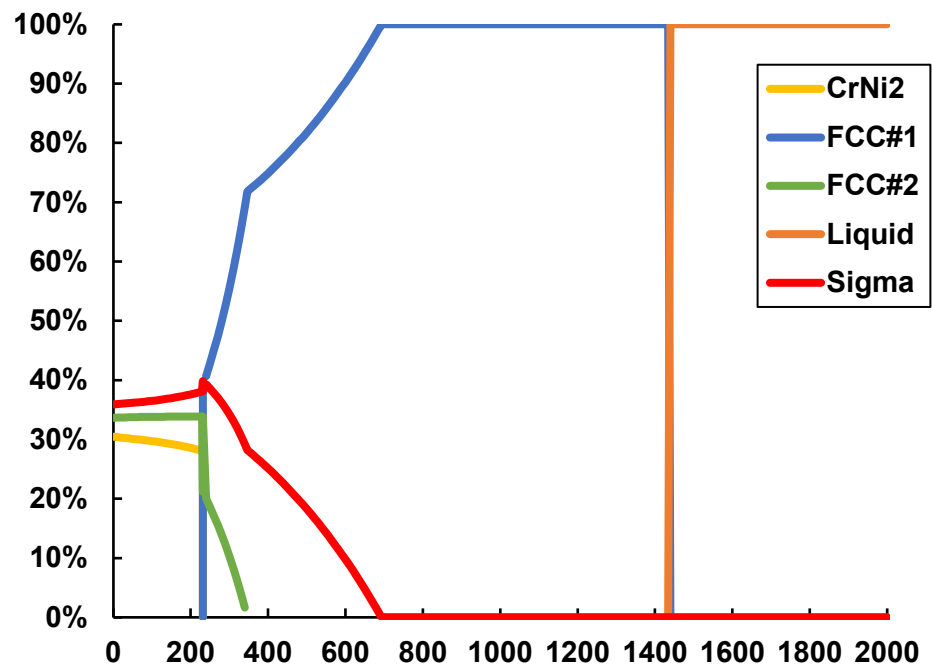


**CALPHAD: FCC**

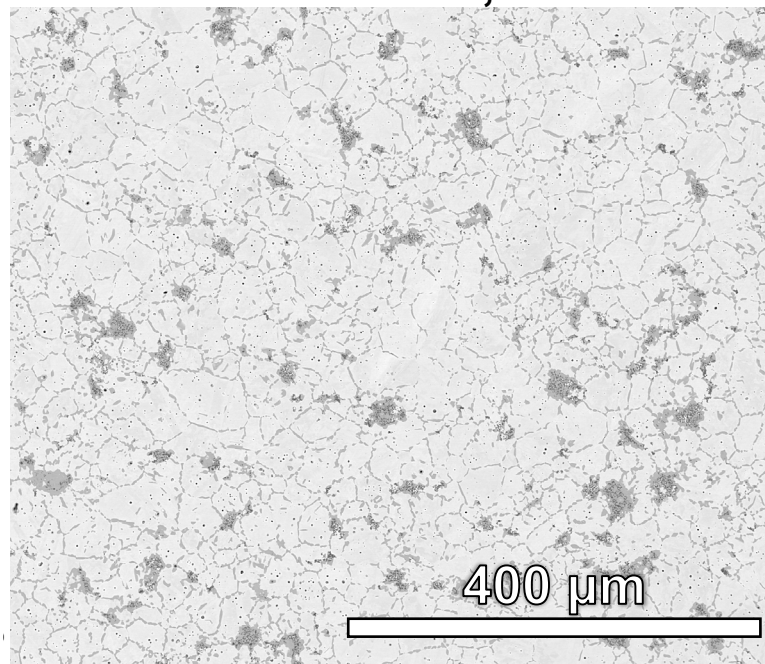
**XRD: FCC**

# NiCoCr

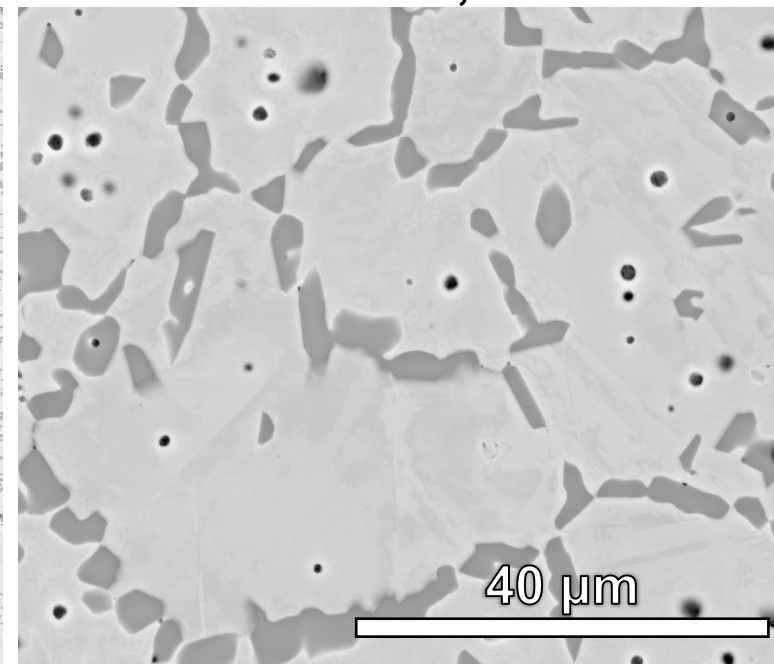
CALPHAD



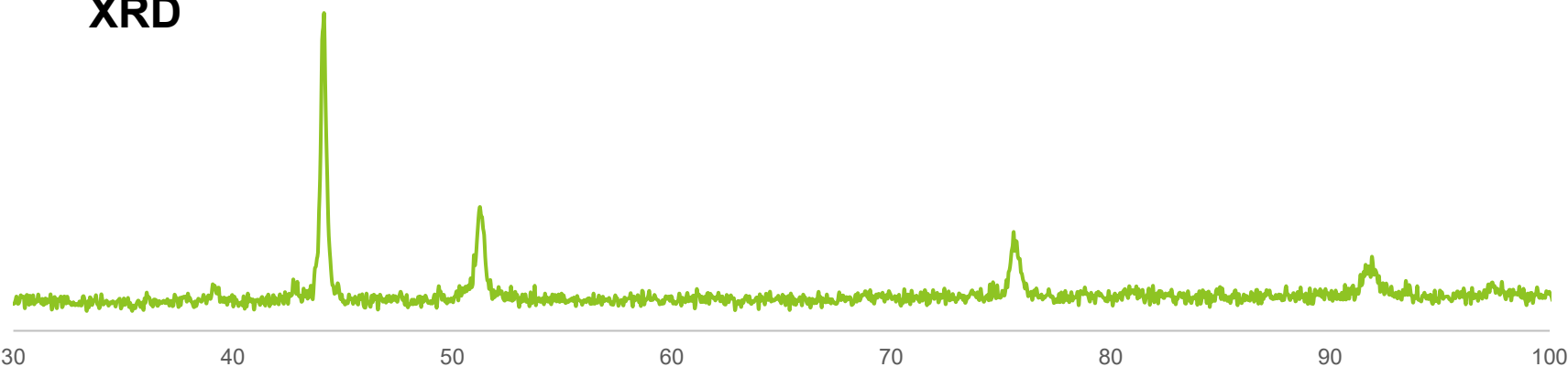
Backscatter, 200x



Backscatter, 2000x



XRD

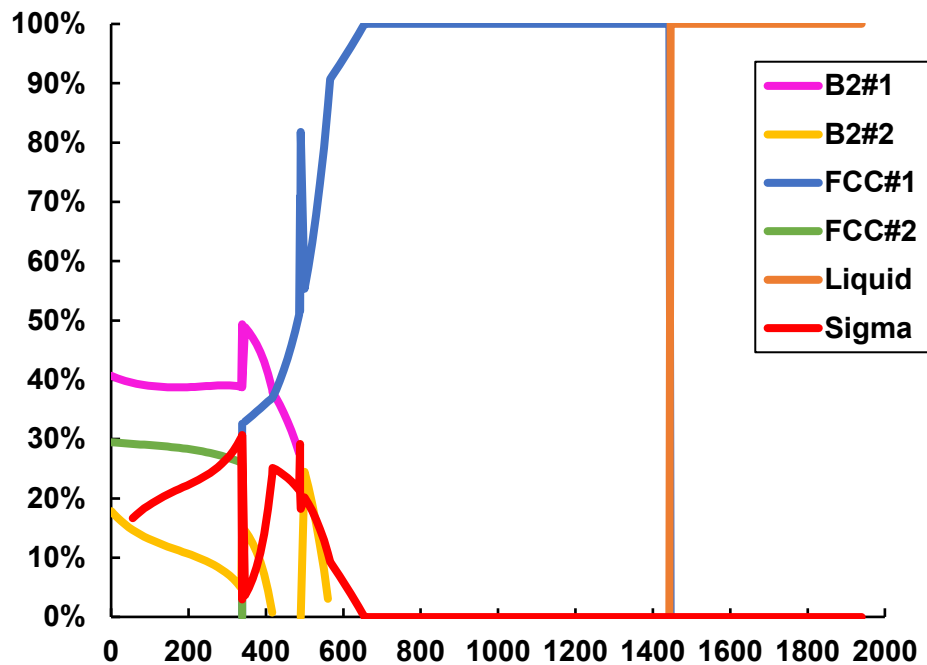


CALPHAD: FCC +  $\sigma$

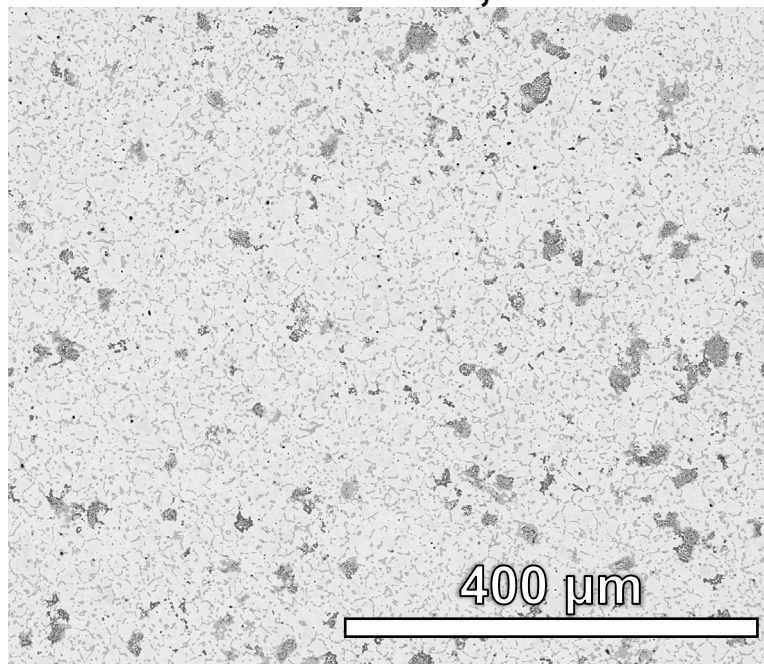
XRD: FCC +  $\sigma$

# NiCoCrFe

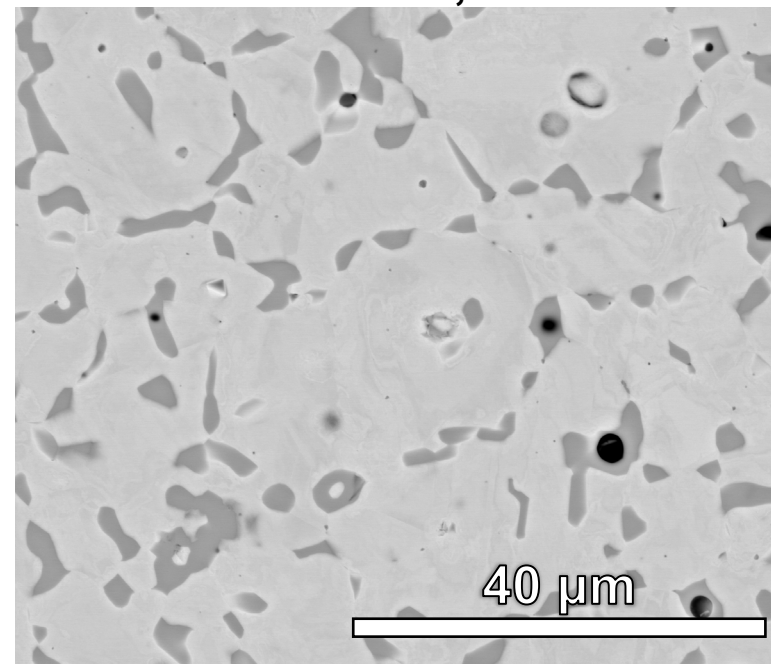
CALPHAD



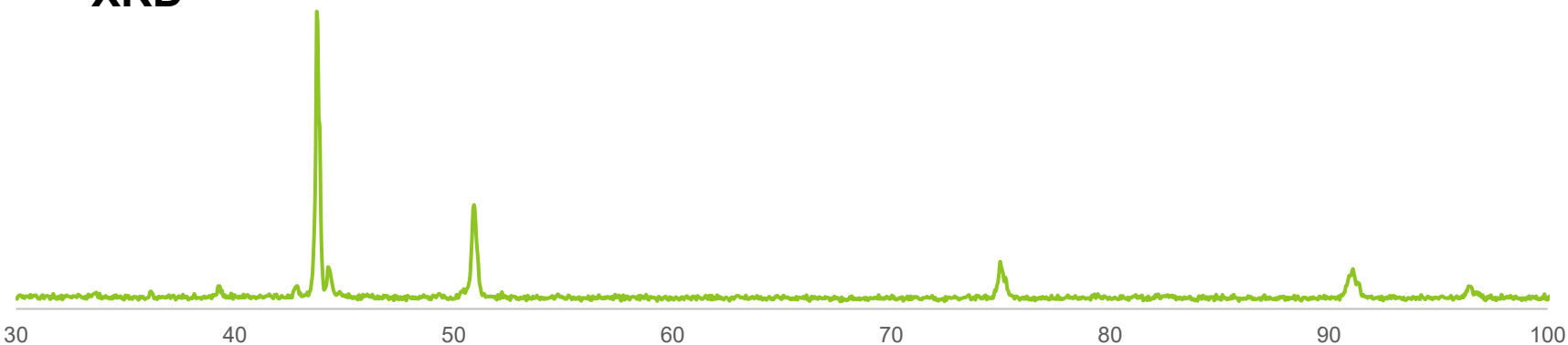
Backscatter, 200x



Backscatter, 2000x



XRD



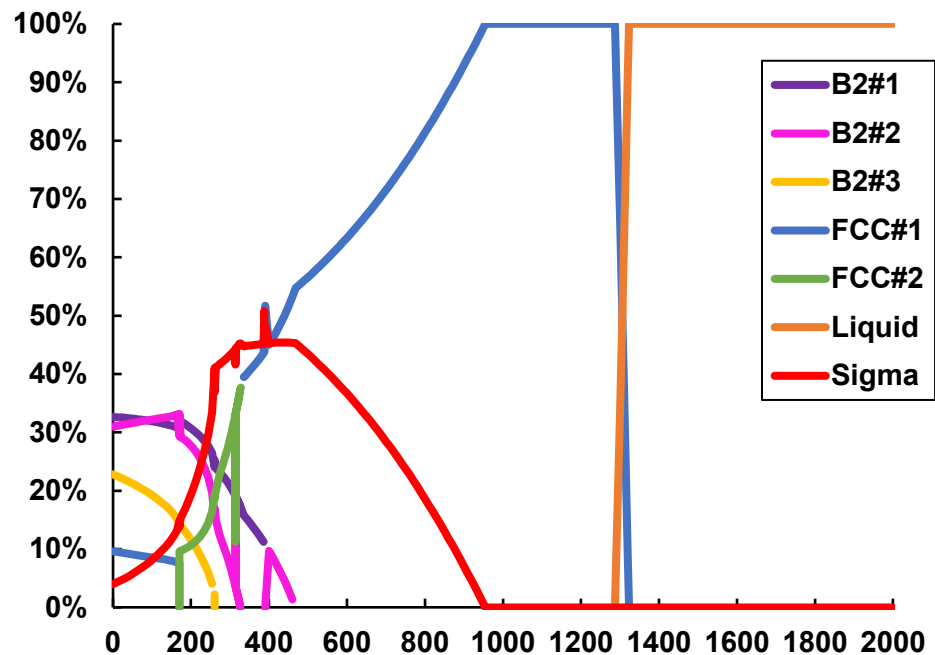
CALPHAD: FCC +  $\sigma$

XRD: FCC +  $\sigma$

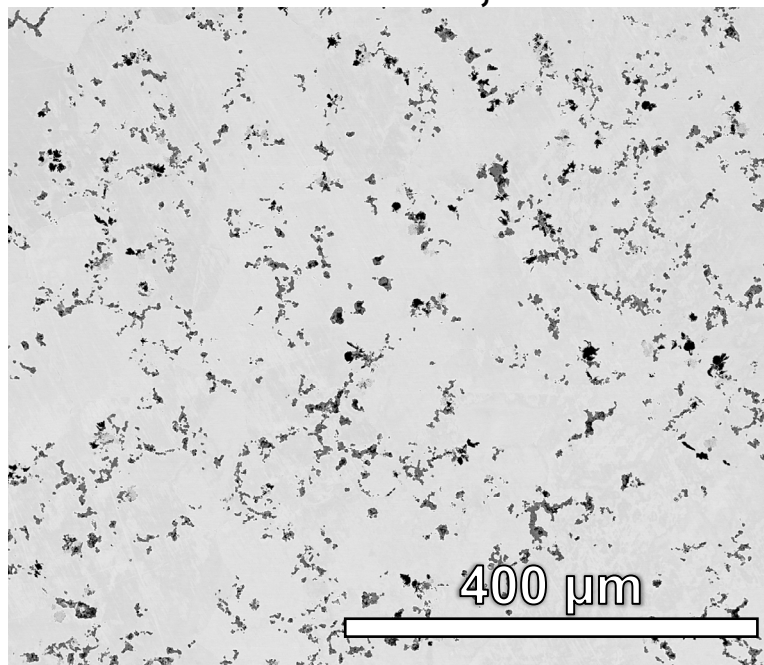


# NiCoCrFeMn

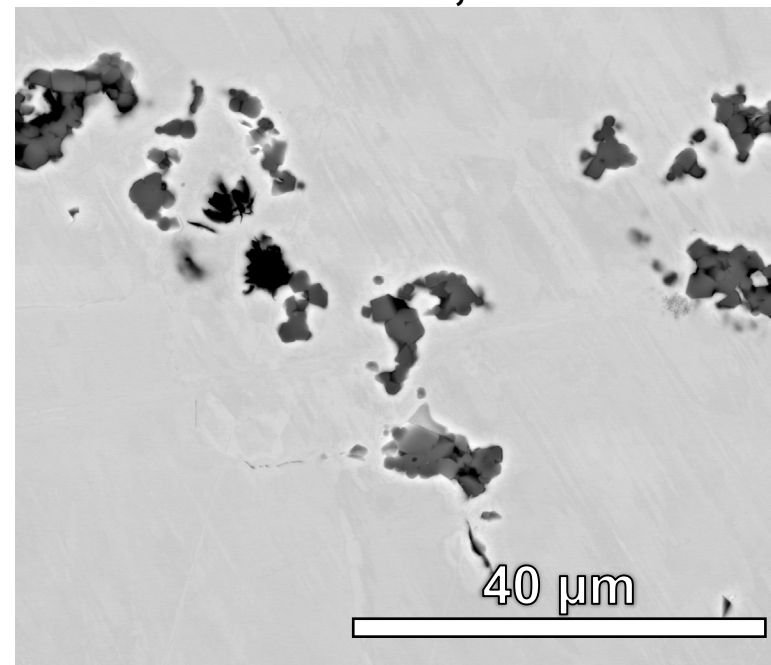
CALPHAD



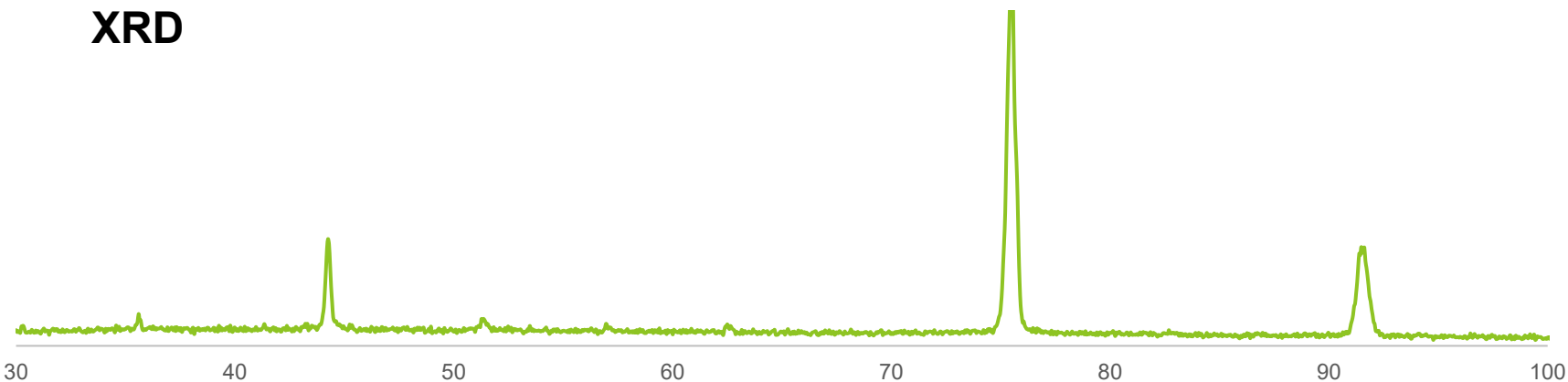
Backscatter, 200x



Backscatter, 2000x



XRD

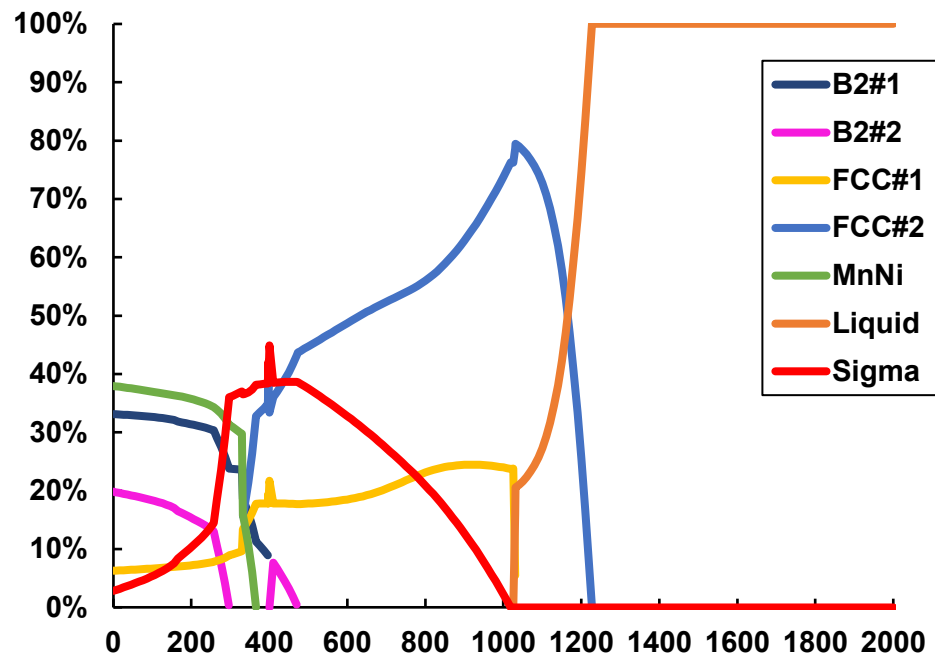


CALPHAD: FCC +  $\sigma$

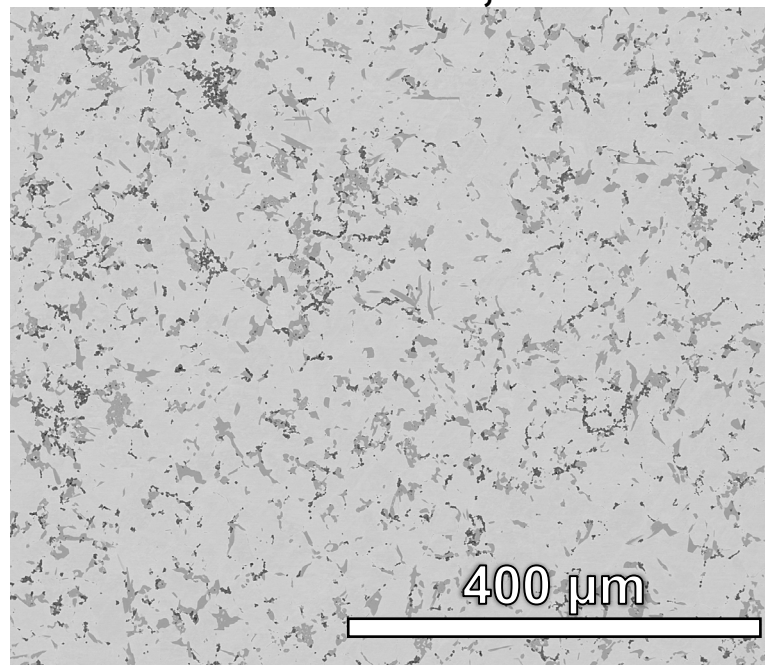
XRD: FCC +  $\sigma$

# NiCoCrFeMnCu

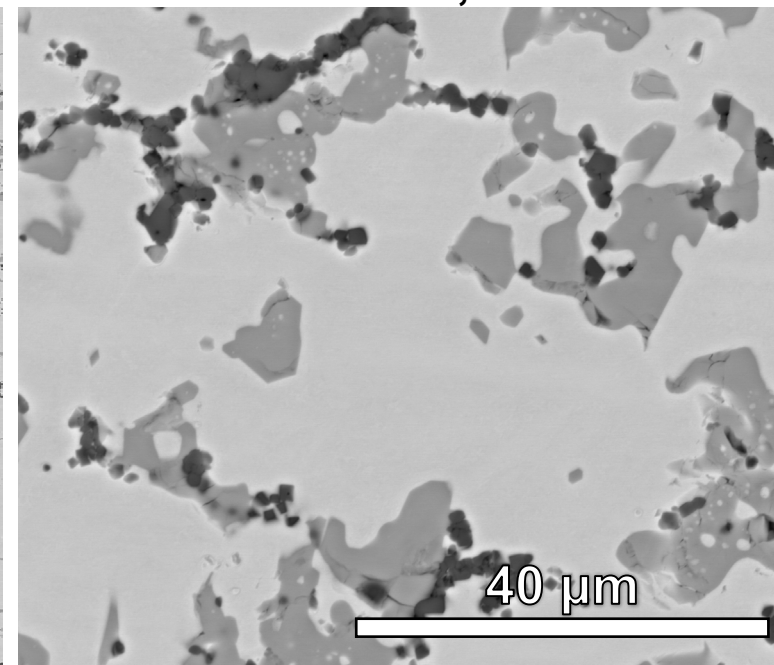
CALPHAD



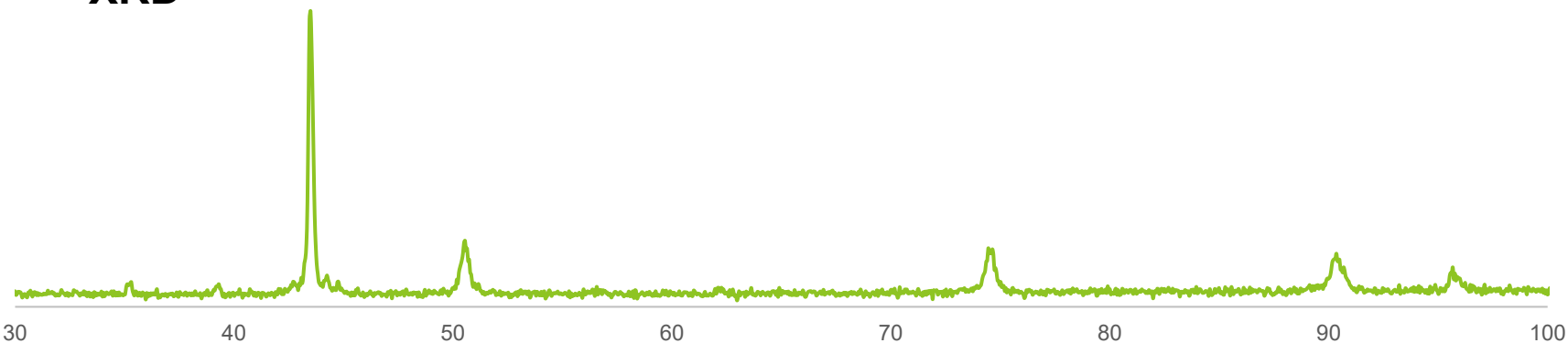
Backscatter, 200x



Backscatter, 2000x



XRD

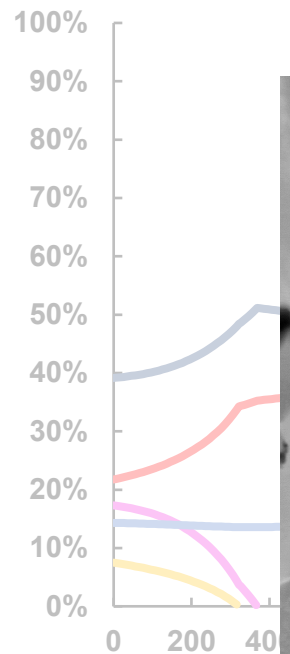


CALPHAD: FCC + FCC +  $\sigma$

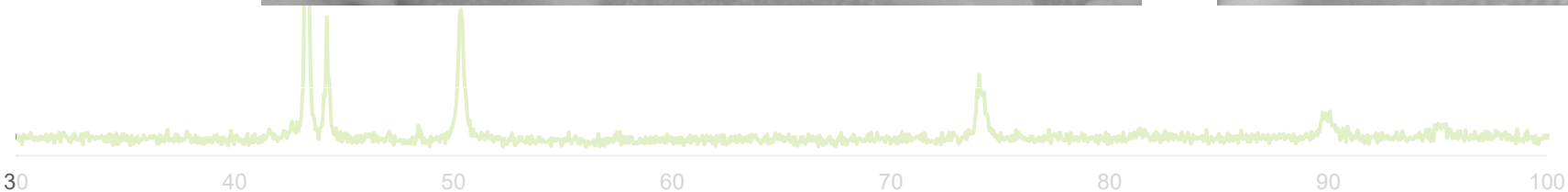
XRD: FCC(x2?) +  $\sigma$

# NiCoCrFeMnCuAl

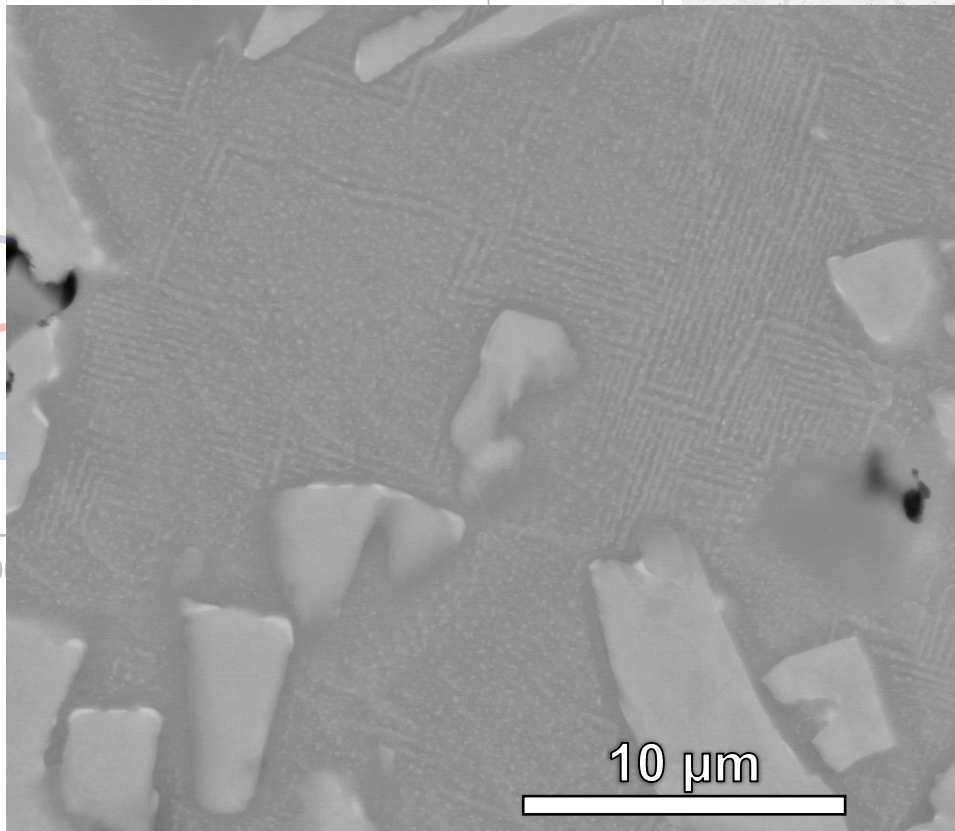
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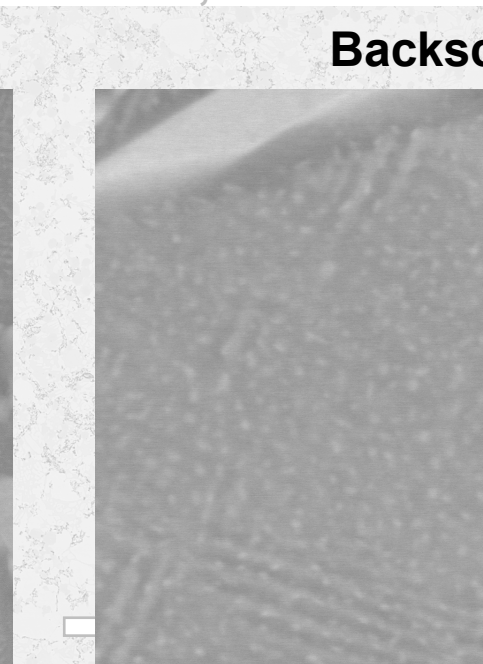
XRD



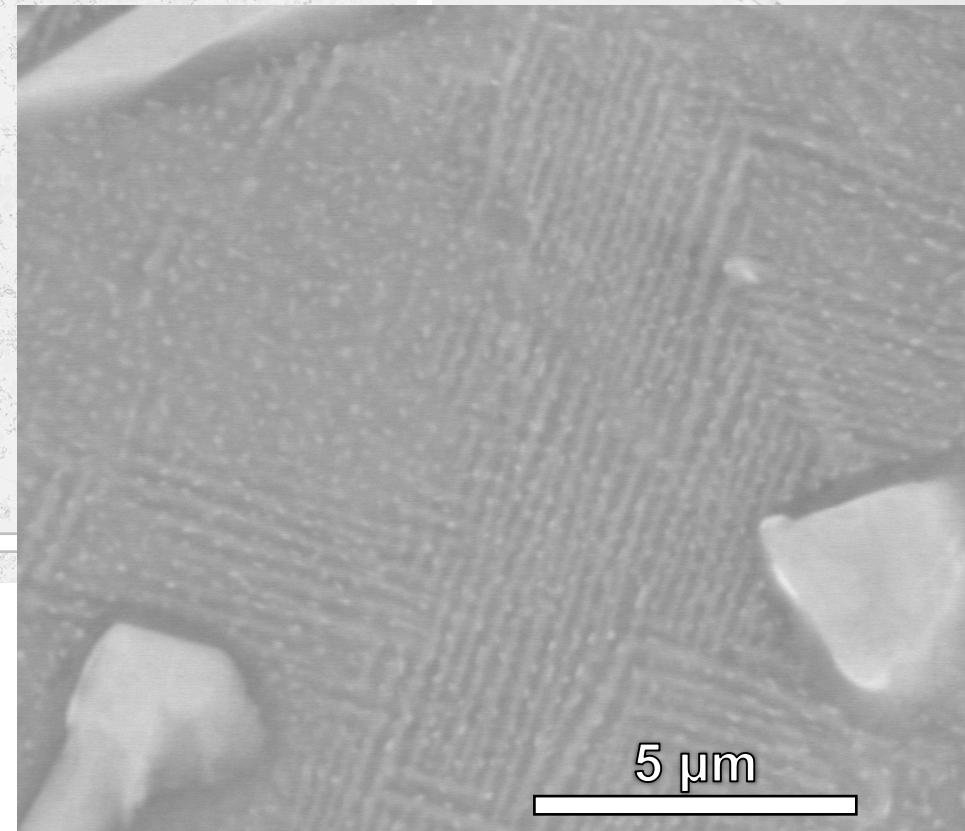
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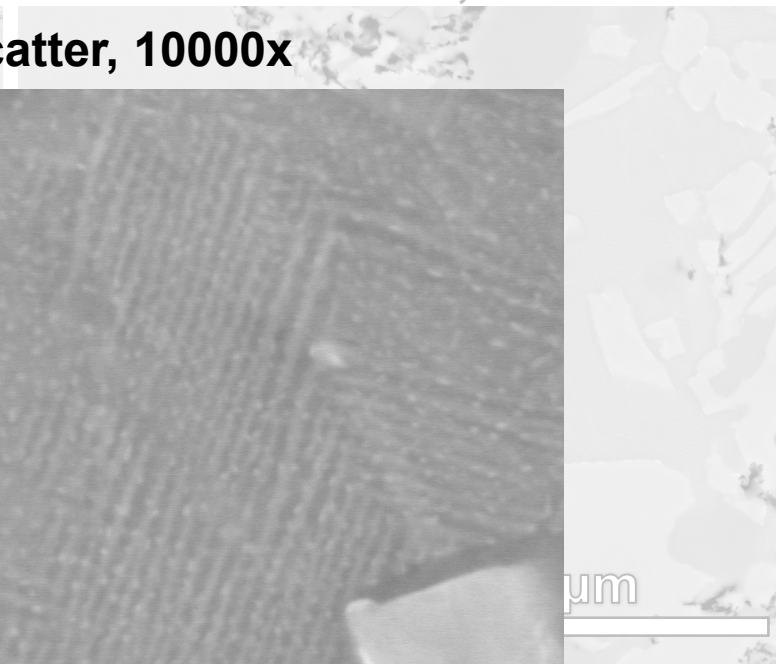
Backscatter, 200x



Backscatter, 10000x



Backscatter, 2000x



$\mu\text{m}$

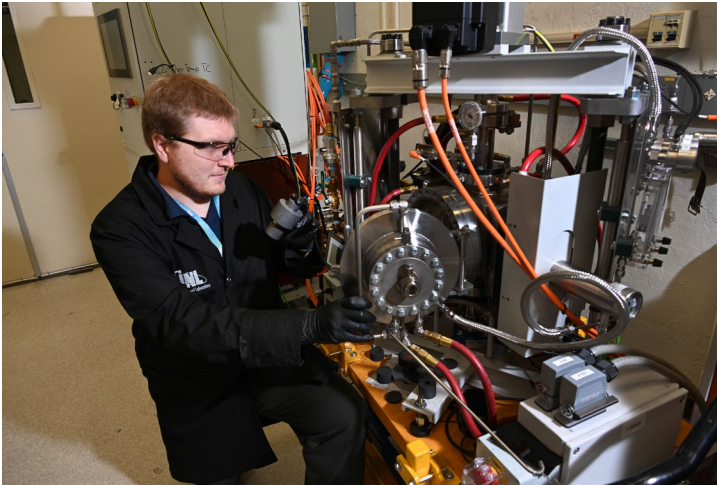
$2 + \sigma$

$\sigma$



# Path Forward: Scale-Up

**DCS-5**  
**~2,500 A, 5 tons**



**INL**  
**Idaho Falls, ID**

**DCS-50**  
**~20,000 A, 50 tons**



■ ■ ■  
**Thermal Technology LLC**  
**Minden, NV**

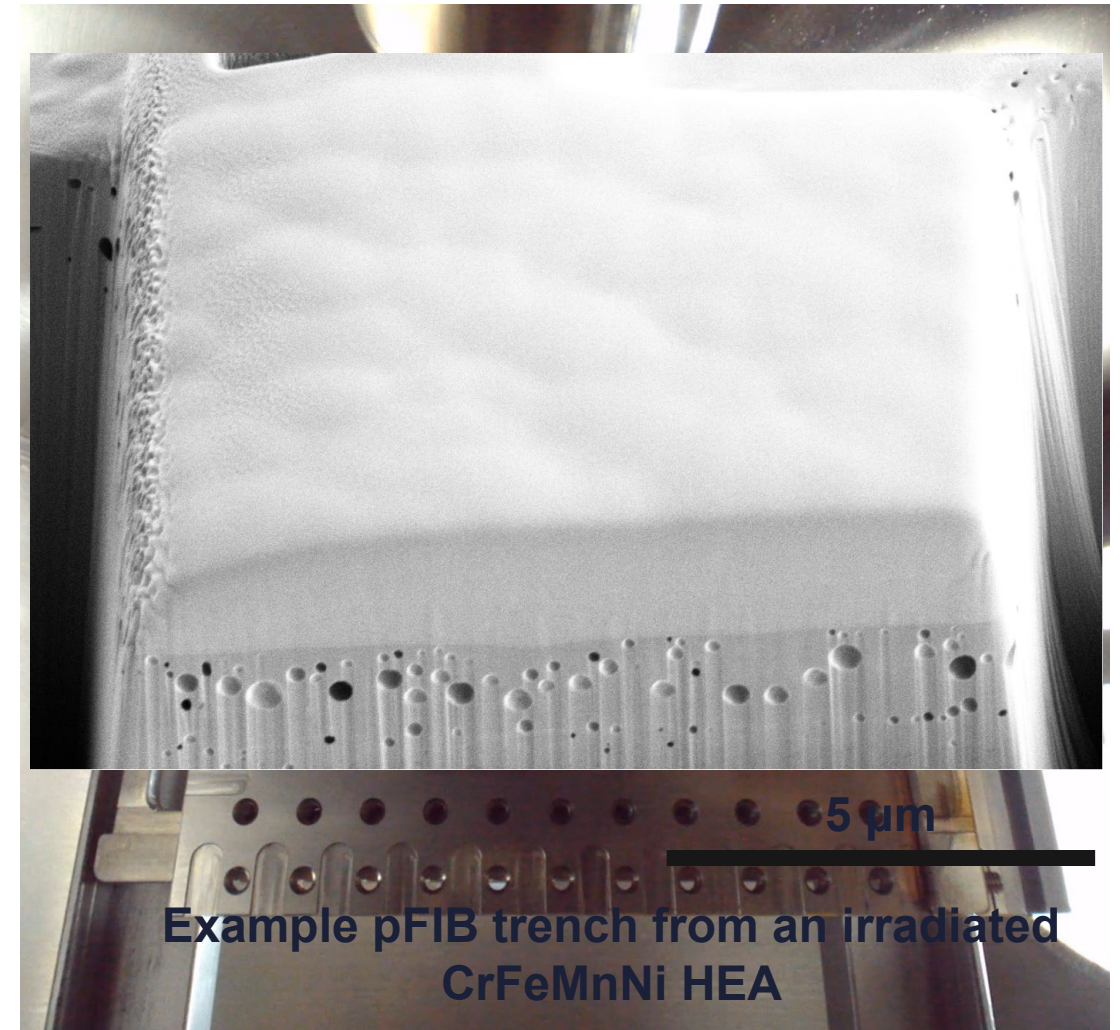
**DCS-800**  
**~150,000 A, 800 tons**



**INL**  
**Idaho Falls, ID**

# Path Forward: High-Throughput Ion Irradiation

- High-throughput ion irradiation
  - UW-Madison, Ion Beam Laboratory
  - 150-mm-travel stage with localized IR laser heating
- High-throughput post-irradiation examination
  - Idaho National Laboratory, IMCL
  - Automated trenching and imaging using the Hydra PFIB



# Conclusions and Acknowledgements

- A new high-throughput synthesis methodology using EFAS has been developed which can produce different samples and compositions in parallel
- Samples produced using this technique:
  - Have negligible levels of unmelted powder
  - Are chemically homogeneous
  - Can be polished/tested/characterized in parallel
- The Parallelized EFAS technique (PEFAS) is fast, energy efficient, and should scale well with larger equipment

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Idaho National Laboratory

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