INL/CON-23-71744-Revision-0

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

Idaho National Laboratory

INL/CON-23-71744-Revision-0

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

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

March 2023

Idaho National Laboratory
Idaho Falls, Idaho 83415

http://www.inl.gov

Prepared for the
U.S. Department of Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

March 22, 2023

Michael Moorehead
Advanced Nuclear
Materials Scientist

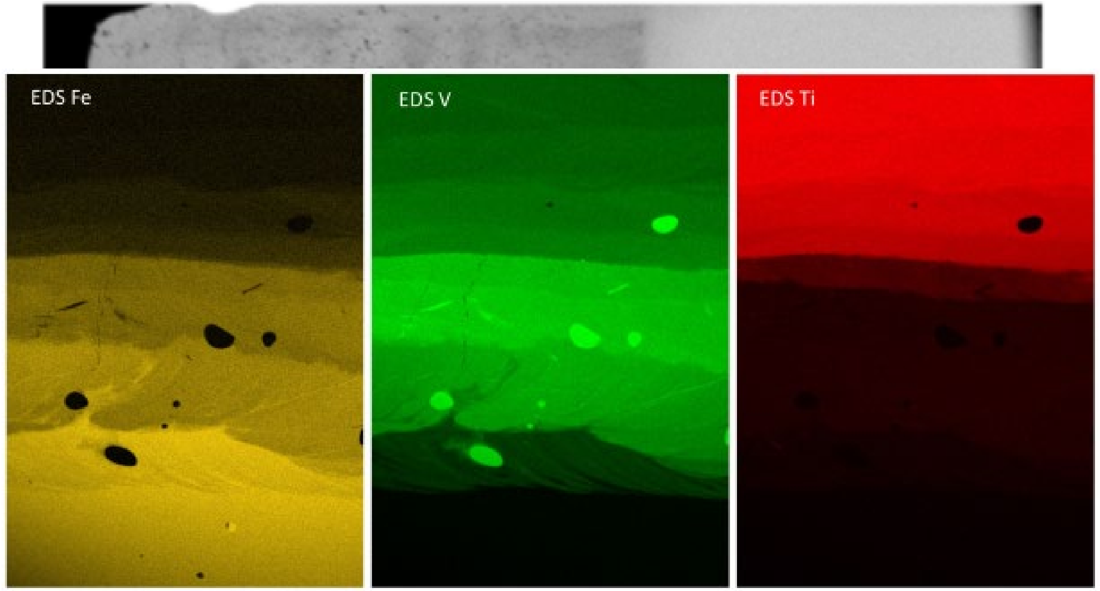
High-Throughput Electric-Field-Assisted Sintering and Characterization Techniques for Materials Discovery

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

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



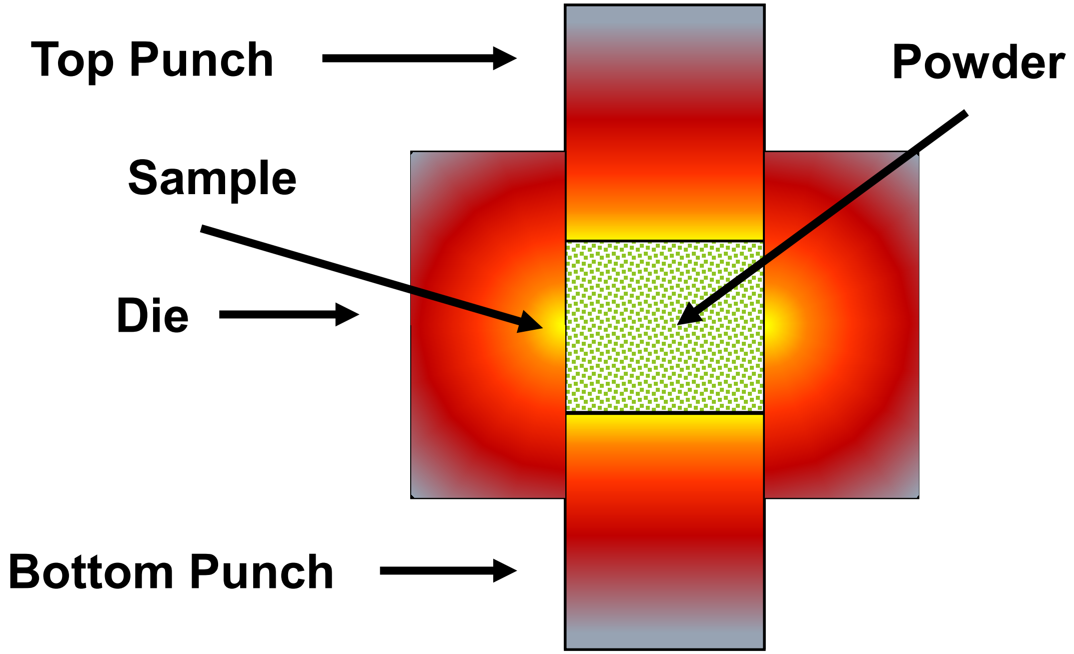
420 martensitic steel to vanadium to Ti-6V-4Al gradient (Reichardt | 2017)

CTF co-deposition schematic (Gebhardt | 2012)

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

Current Material Synthesis via EFAS

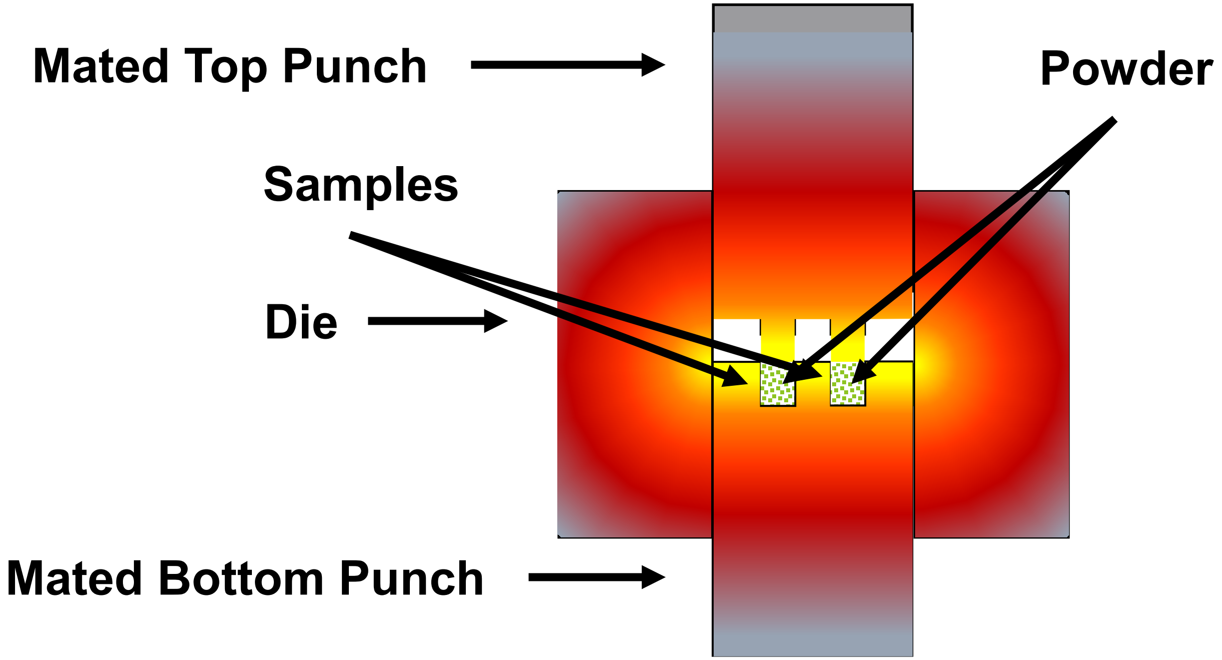
- Single-Sample Production
  - Two flat punches
  - Single powder (mixture)



Current Material Synthesis via EFAS

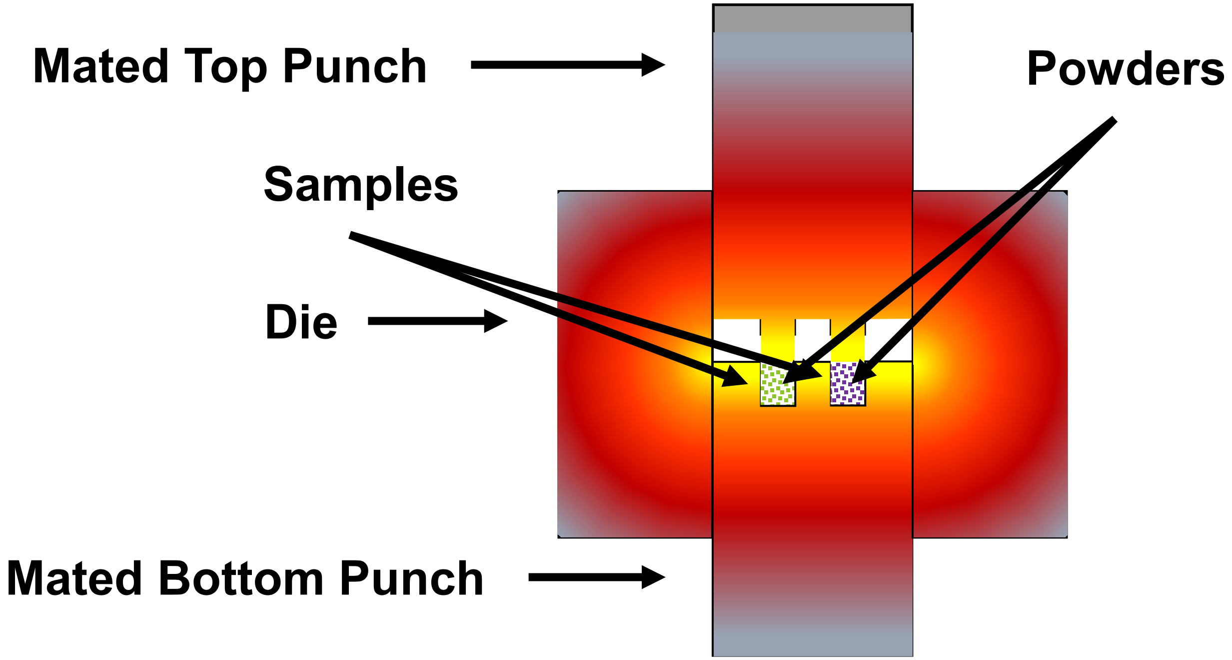
- Single-Sample Production
  - Two flat punches
  - Single powder (mixture)

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



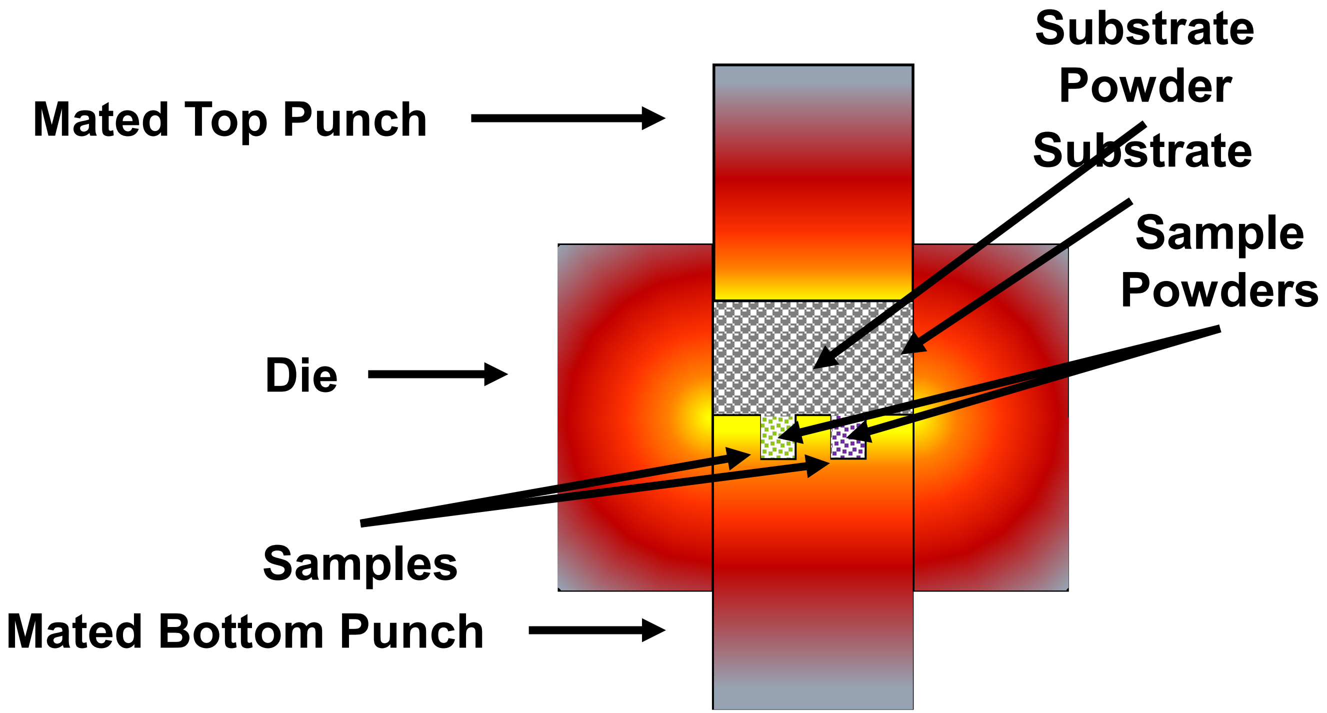
Current Material Synthesis via EFAS

- 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

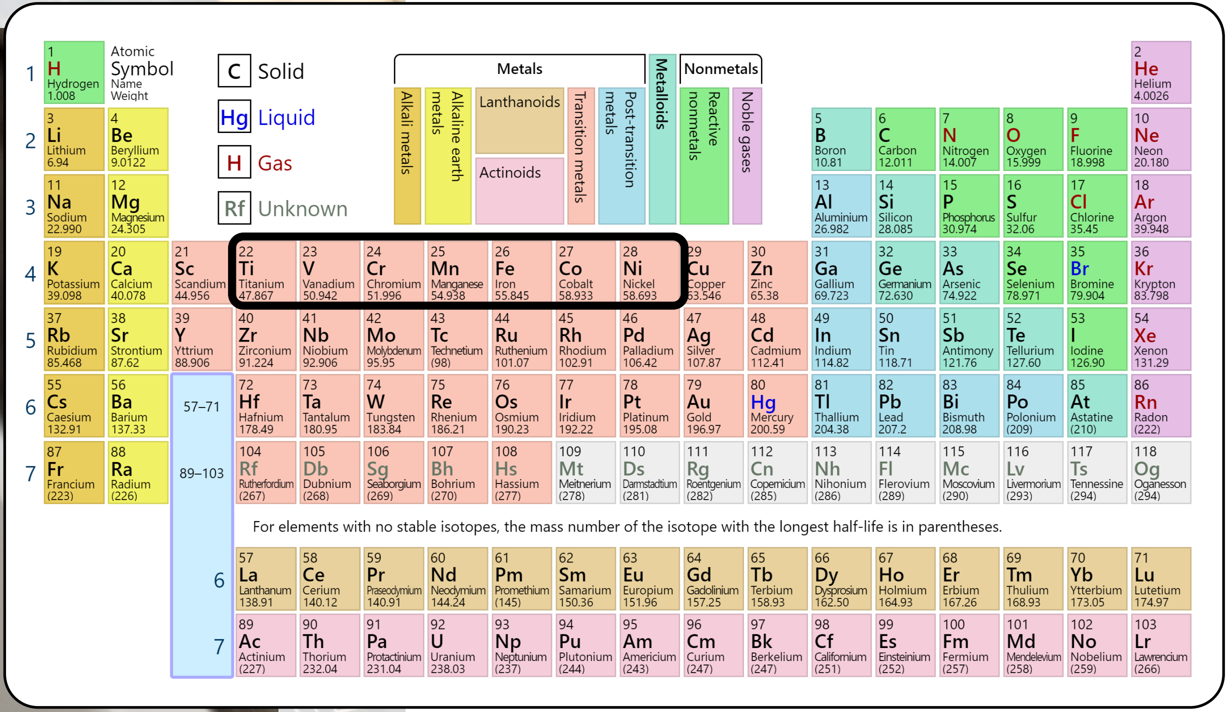


Parallelized EFAS Sample Production

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

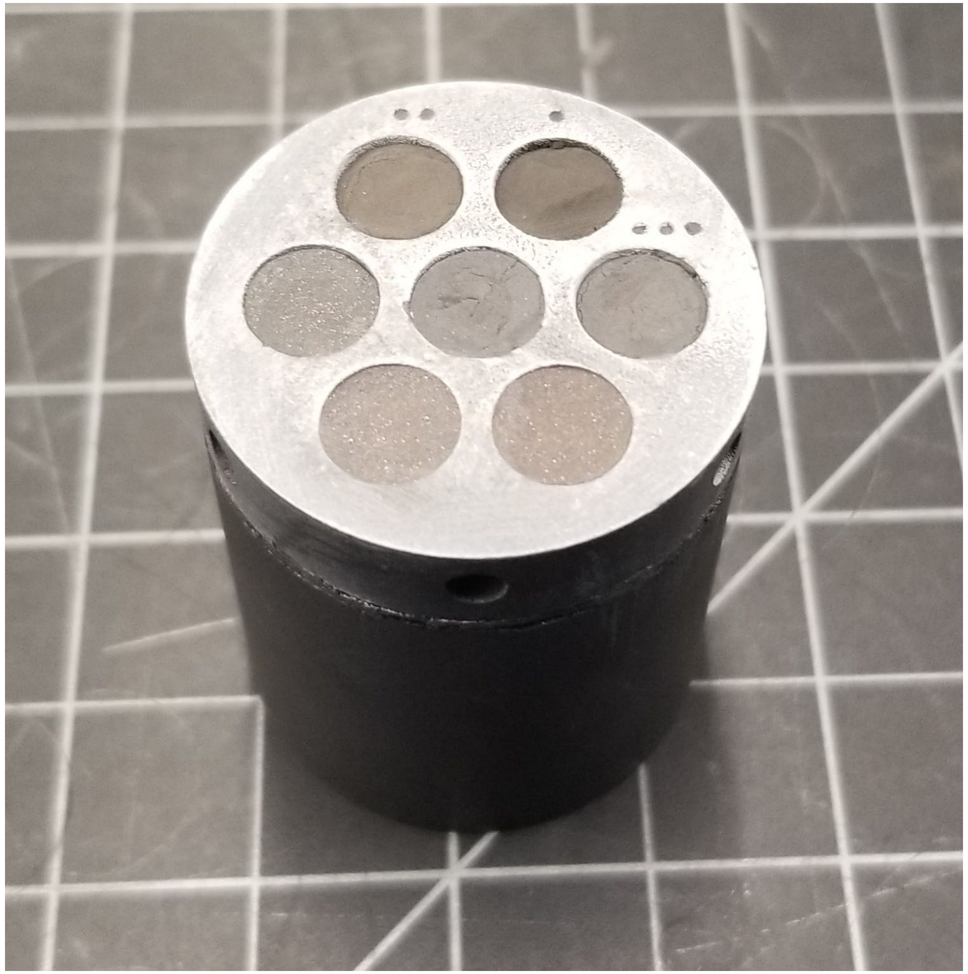


Parallelized EFAS Sample Production



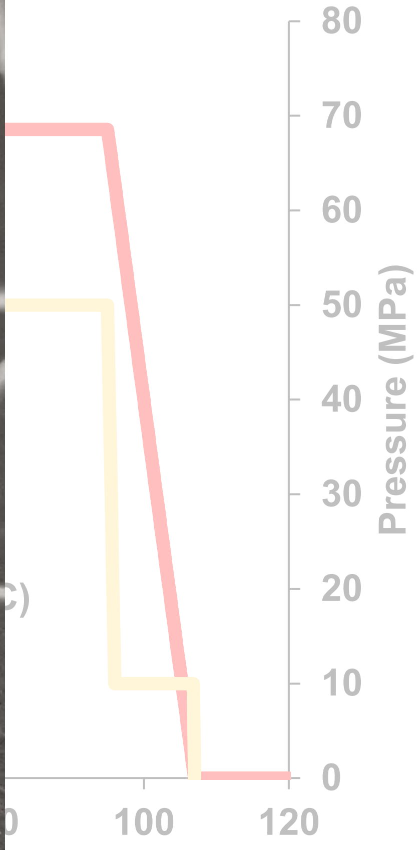
HEA Case Study

- 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

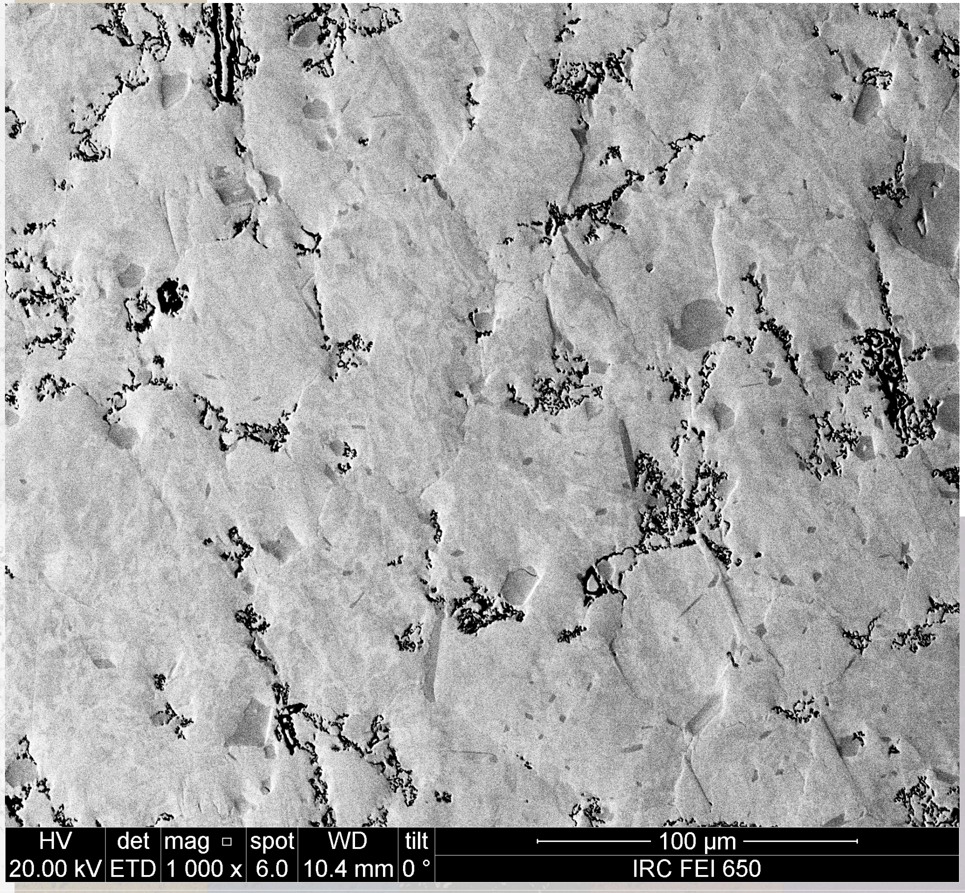
- EFAS pro
  - Press
    MPa/
  - Temp
    - 90
    - 11
    - 12
  - 1-hou
  - Press
    down



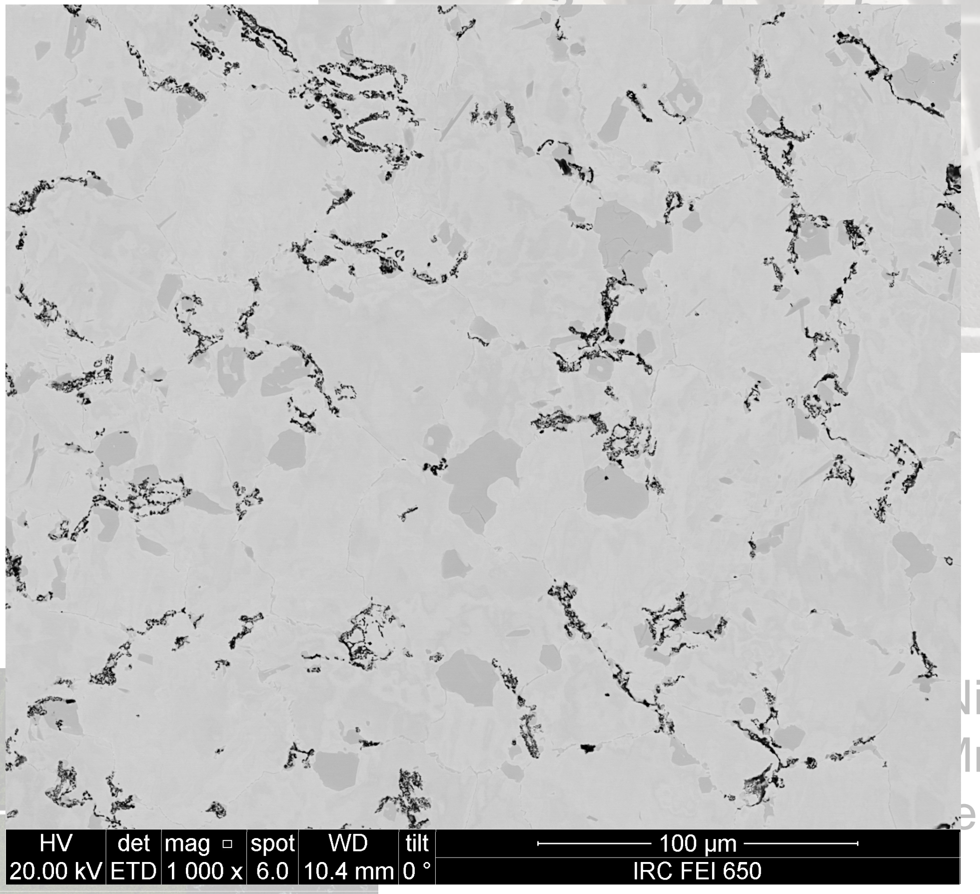
HEA Case Study: SEM/EDS

Equimolar AlCoCrCuFeMnNi

Secondary Electron

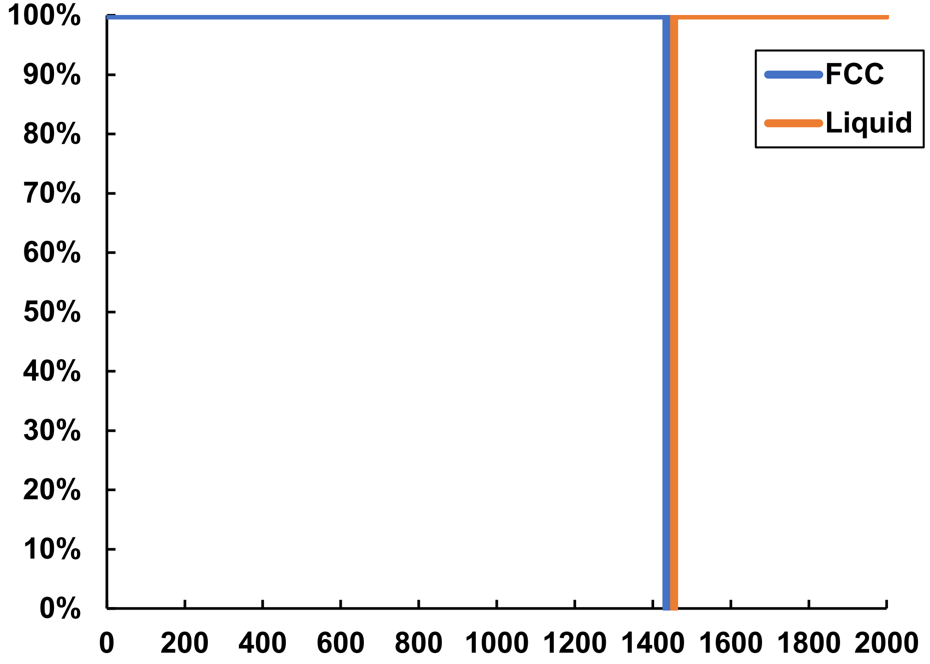


Backscattered Electron

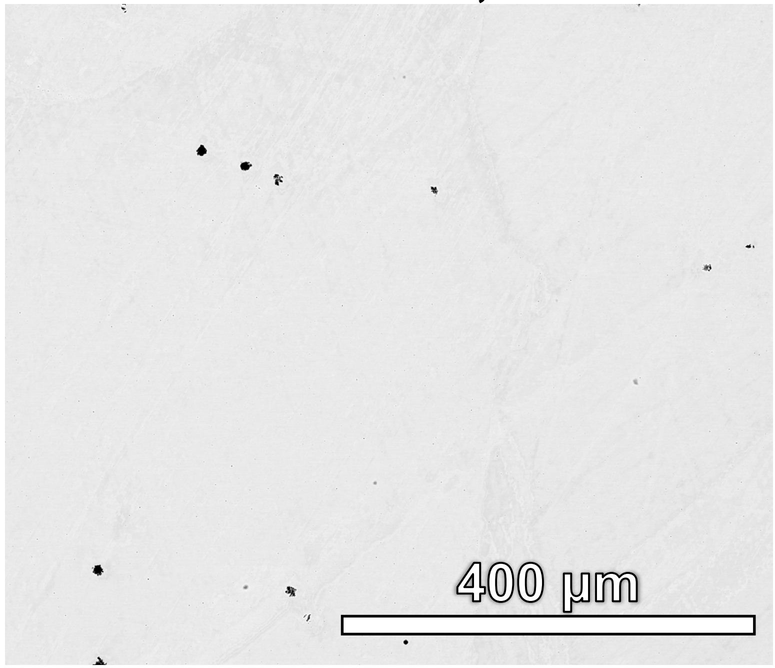


Ni

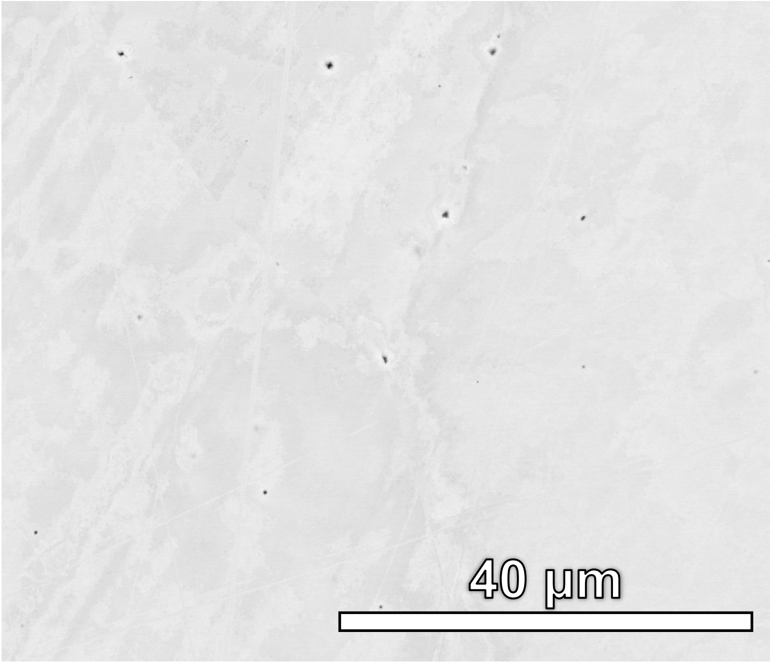
CALPHAD



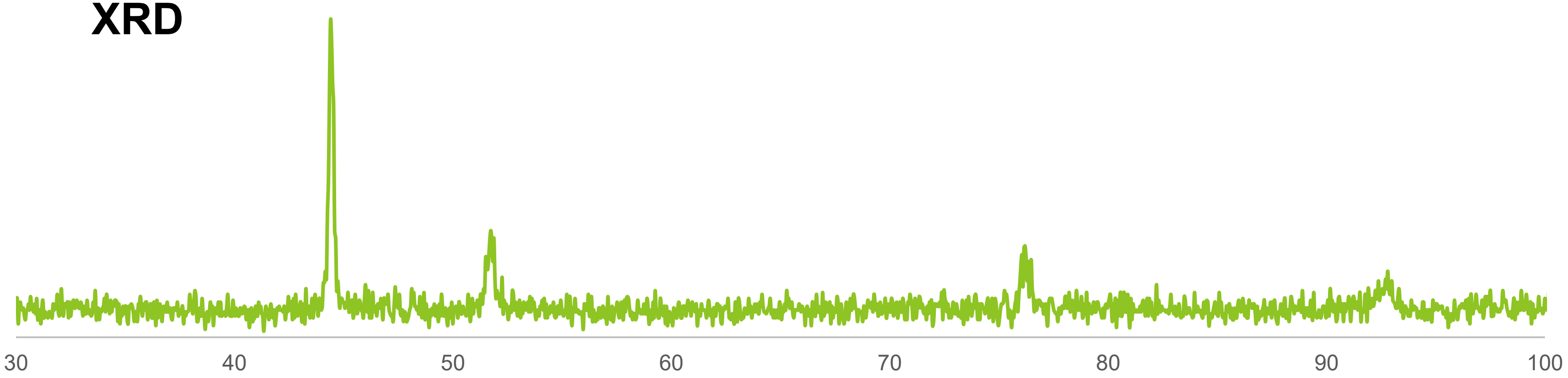
Backscatter, 200x



Backscatter, 2000x



XRD

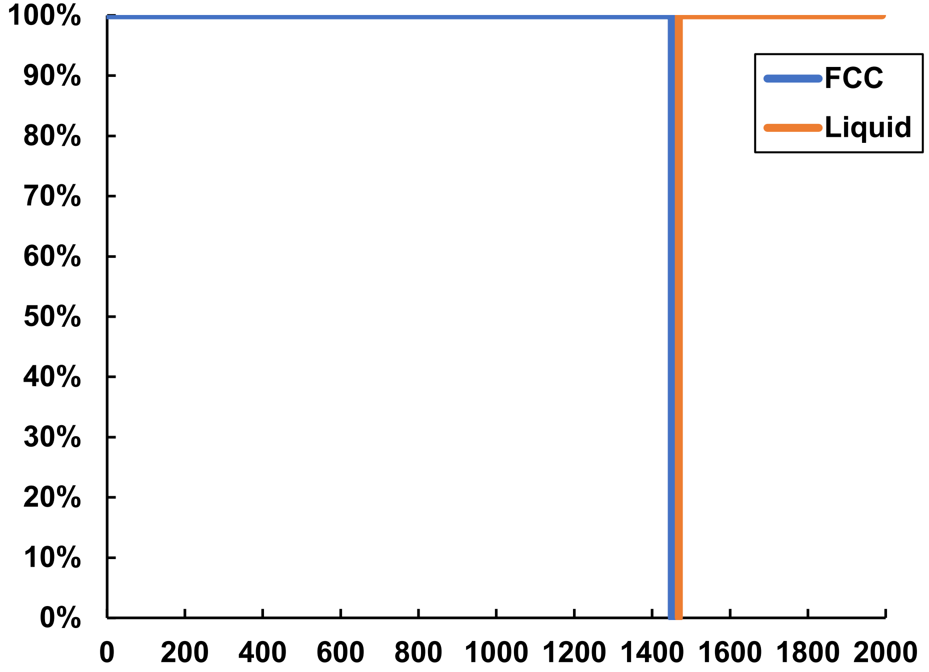


CALPHAD: FCC

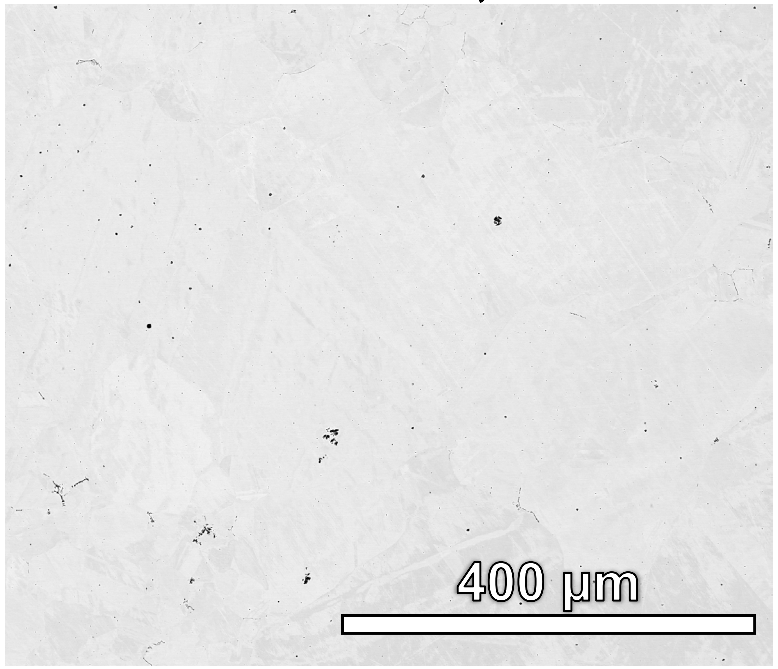
XRD: FCC

NiCo

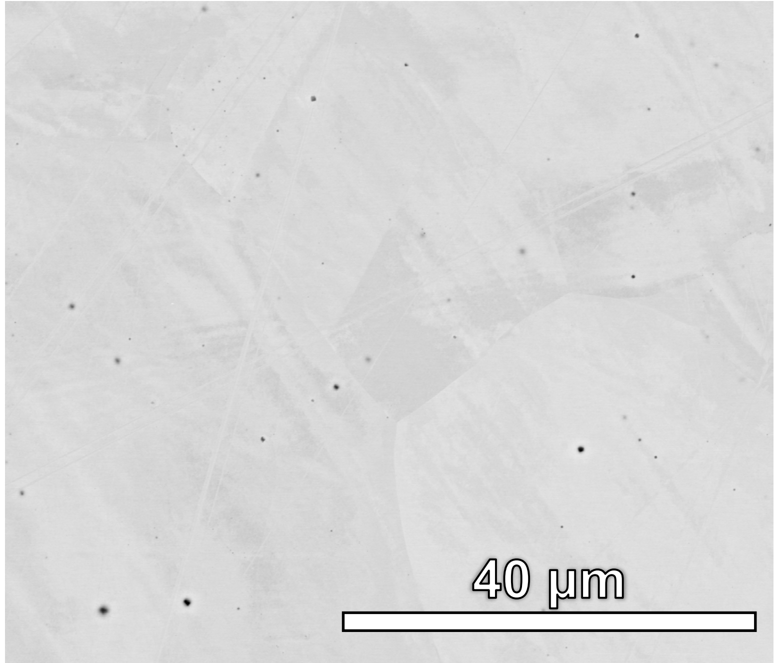
CALPHAD



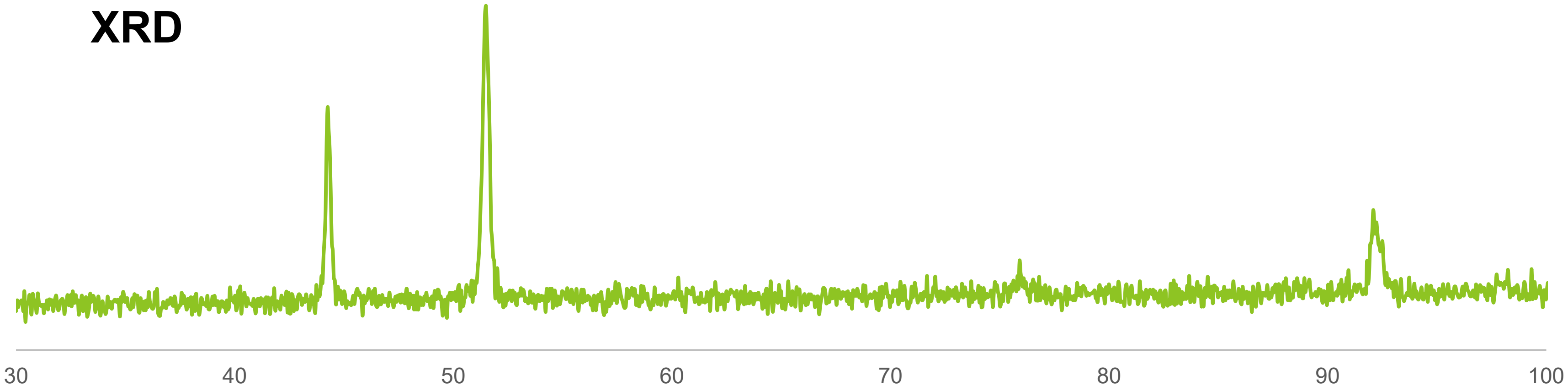
Backscatter, 200x



Backscatter, 2000x



XRD

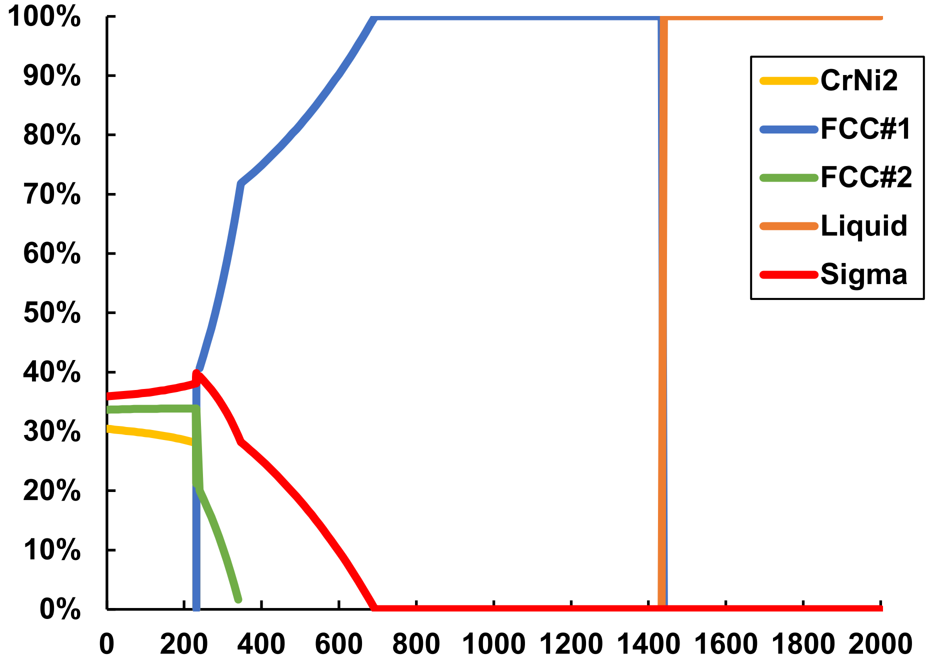


CALPHAD: FCC

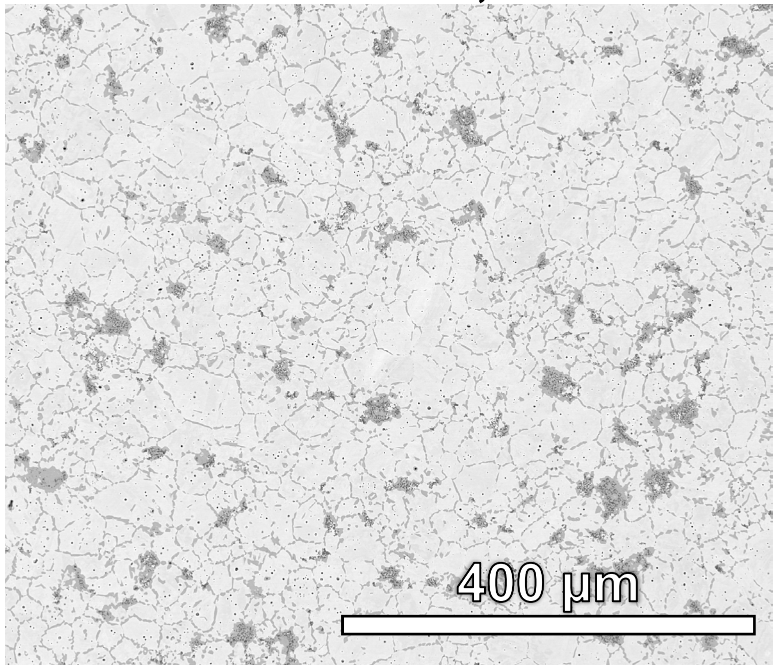
XRD: FCC

NiCoCr

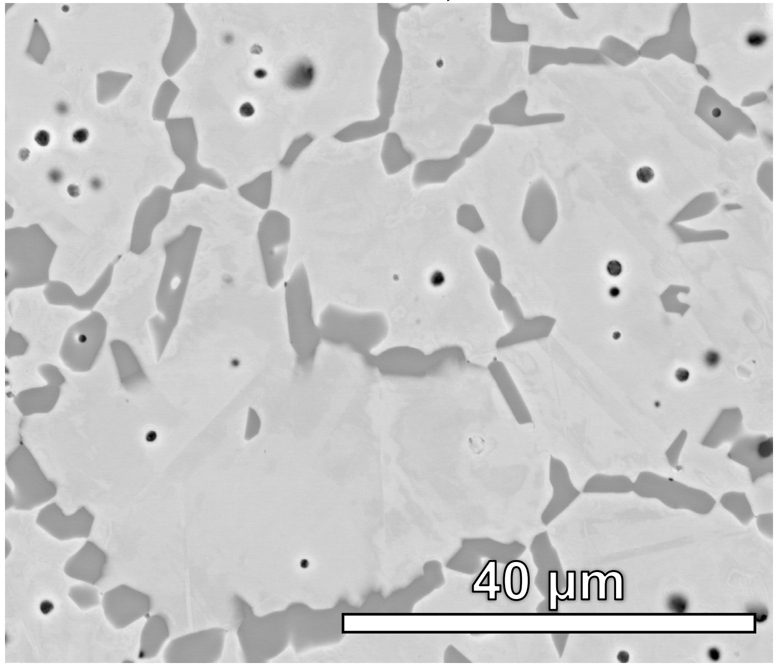
CALPHAD



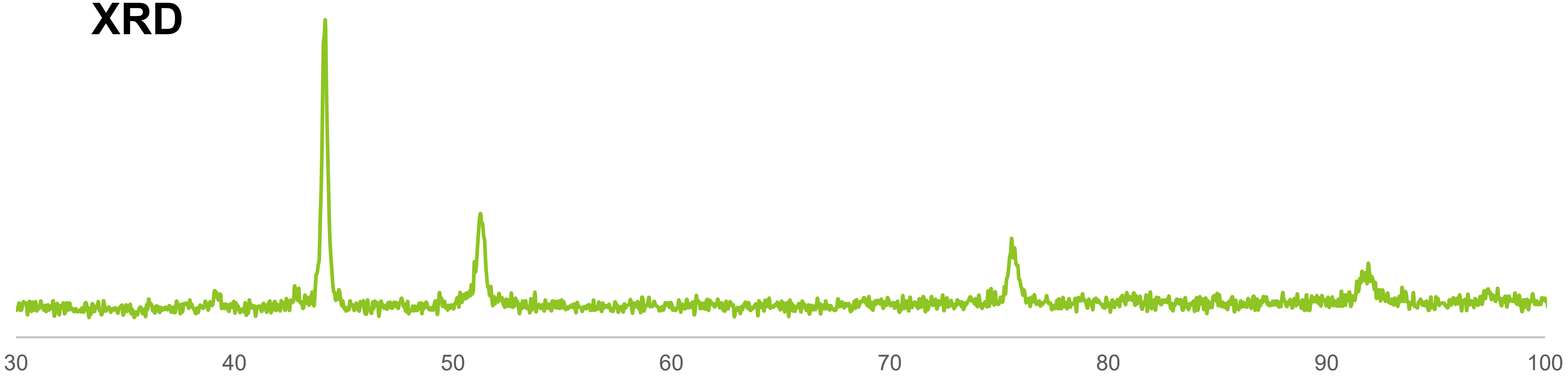
Backscatter, 200x



Backscatter, 2000x



XRD

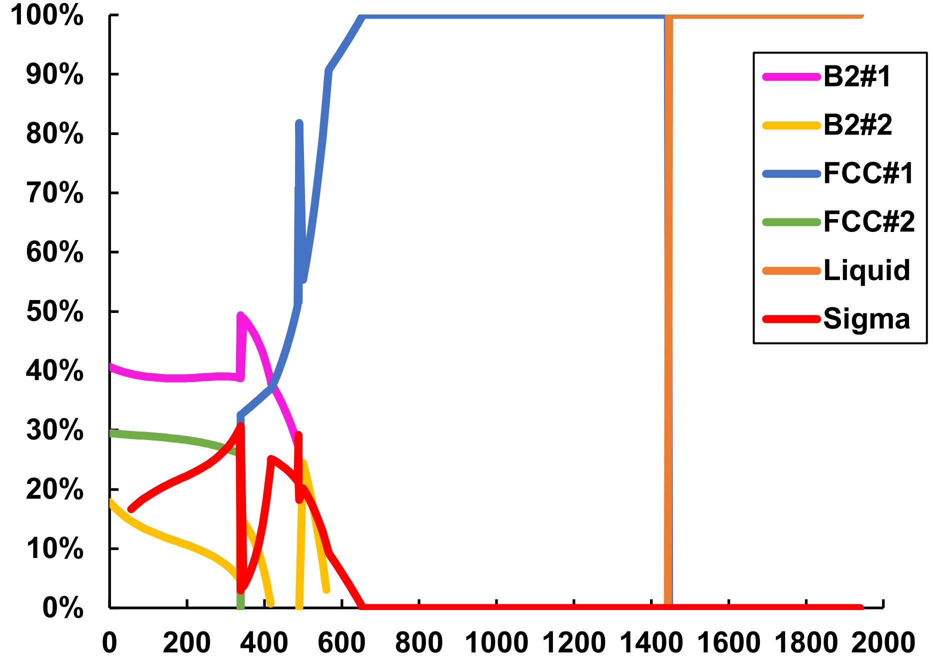


CALPHAD: FCC + σ

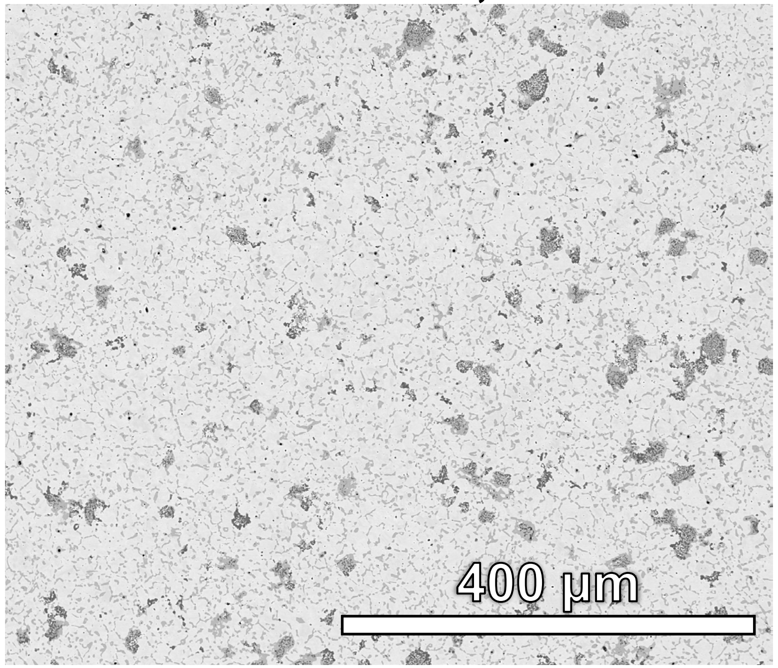
XRD: FCC + σ

NiCoCrFe

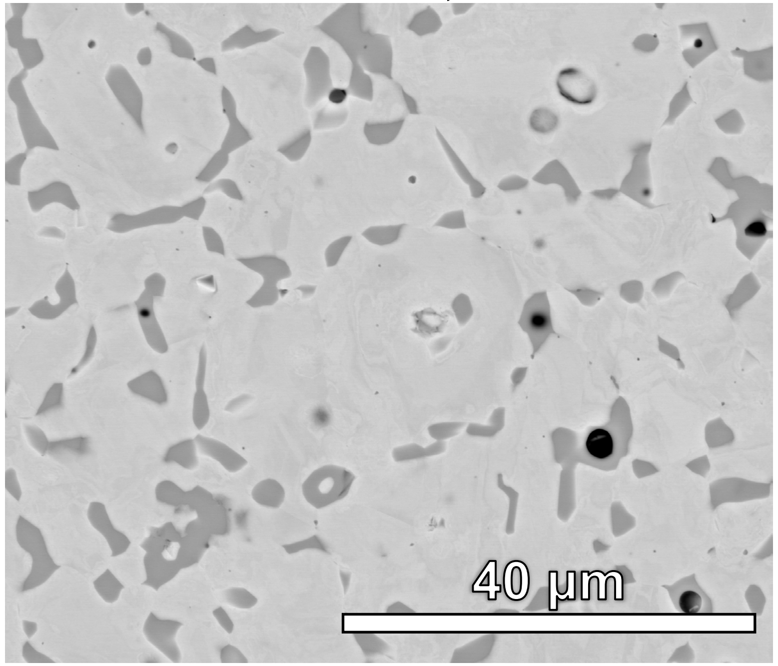
CALPHAD



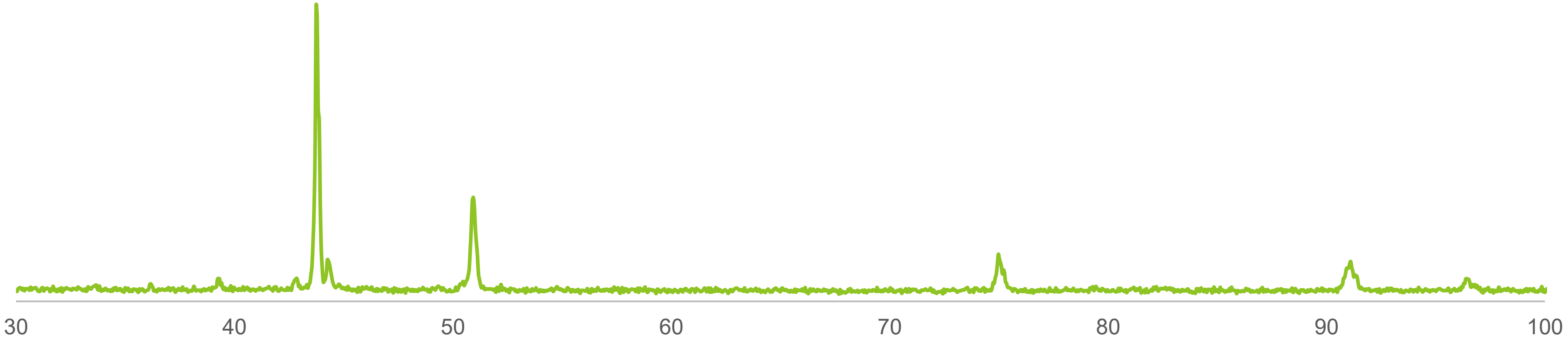
Backscatter, 200x



Backscatter, 2000x



XRD

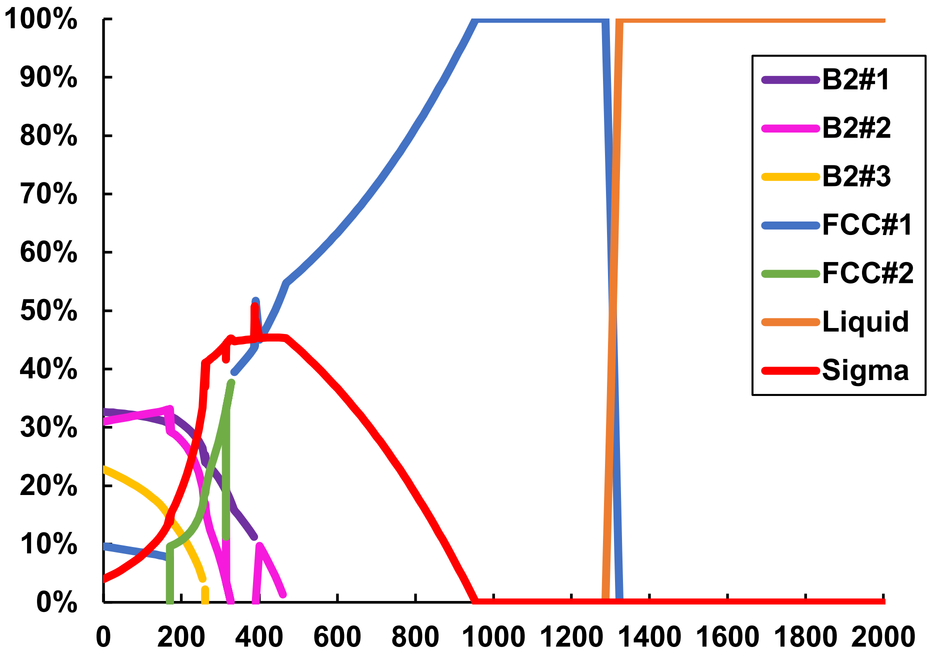


CALPHAD: FCC + σ

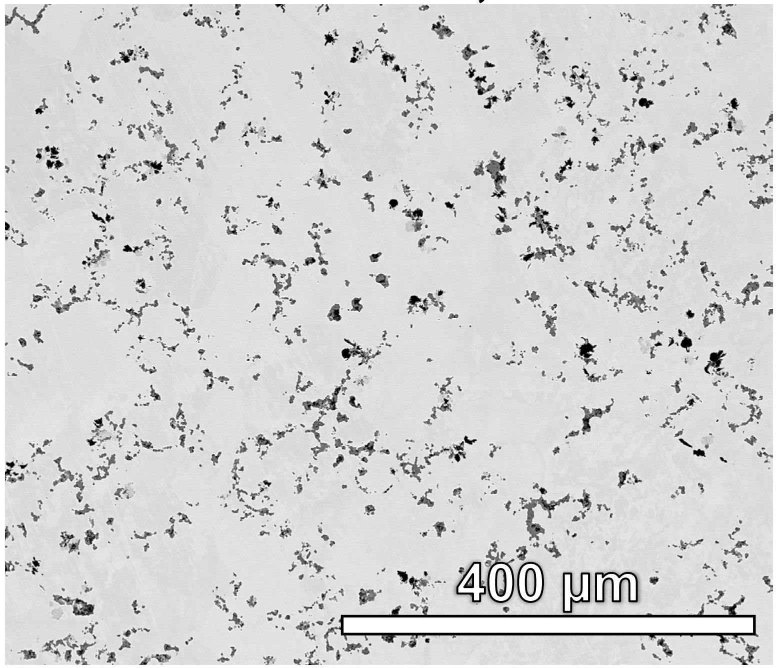
XRD: FCC + σ

NiCoCrFeMn

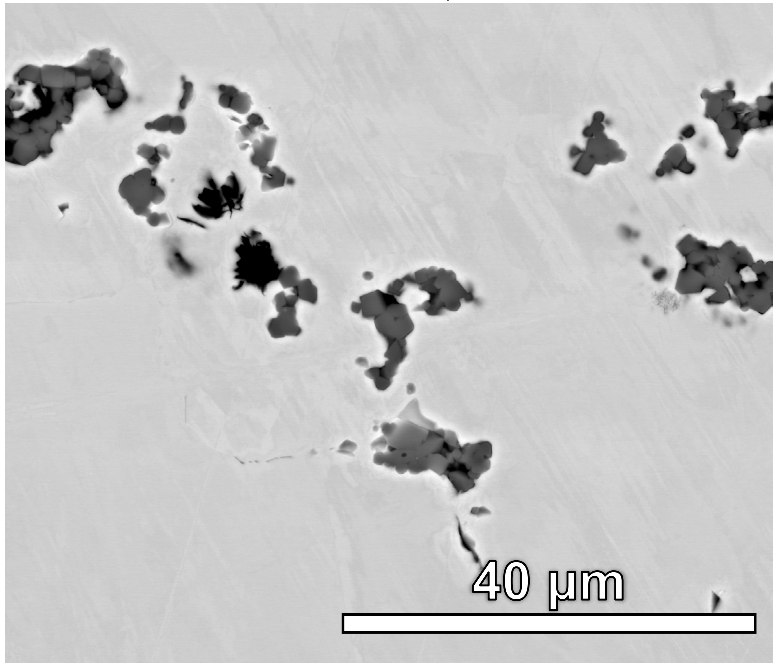
CALPHAD



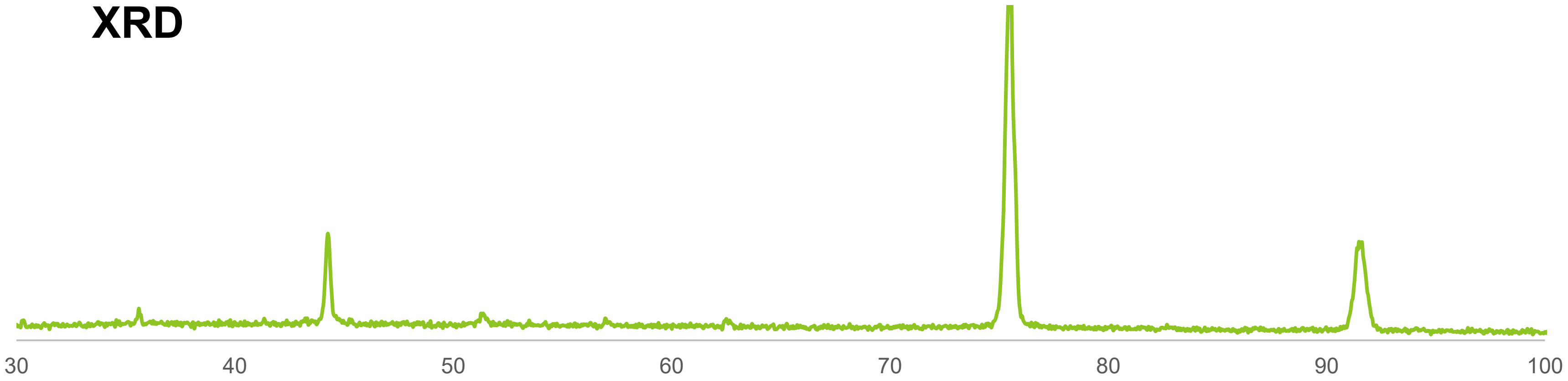
Backscatter, 200x



Backscatter, 2000x



XRD

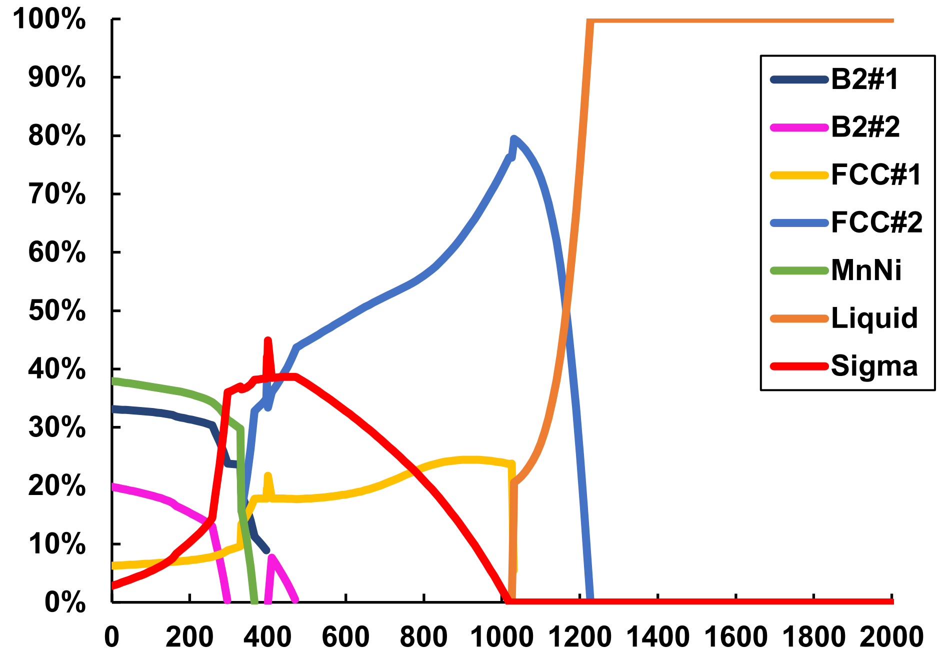


CALPHAD: FCC + σ

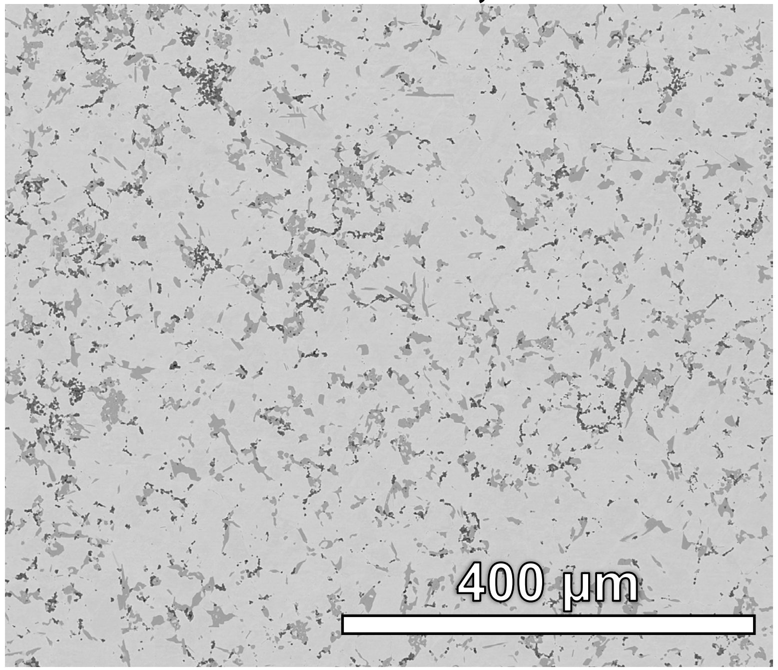
XRD: FCC + σ

NiCoCrFeMnCu

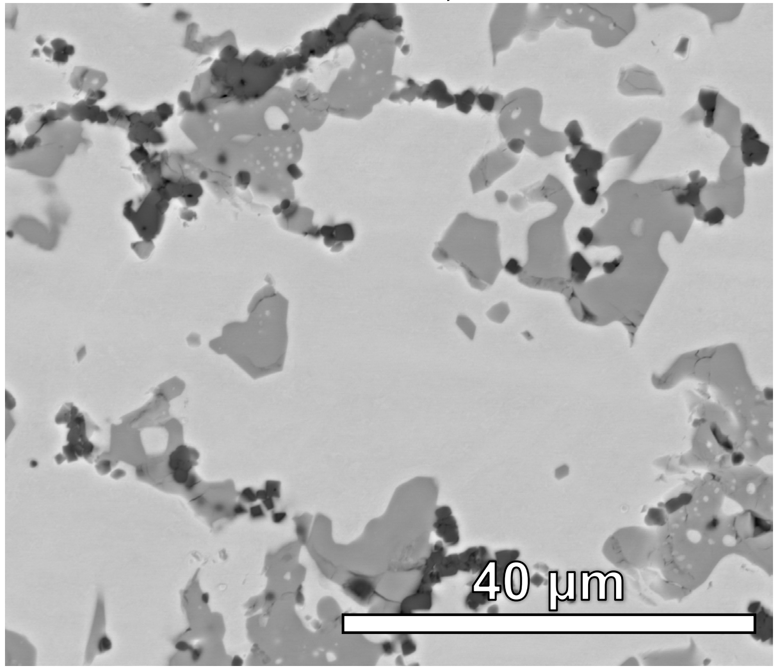
CALPHAD



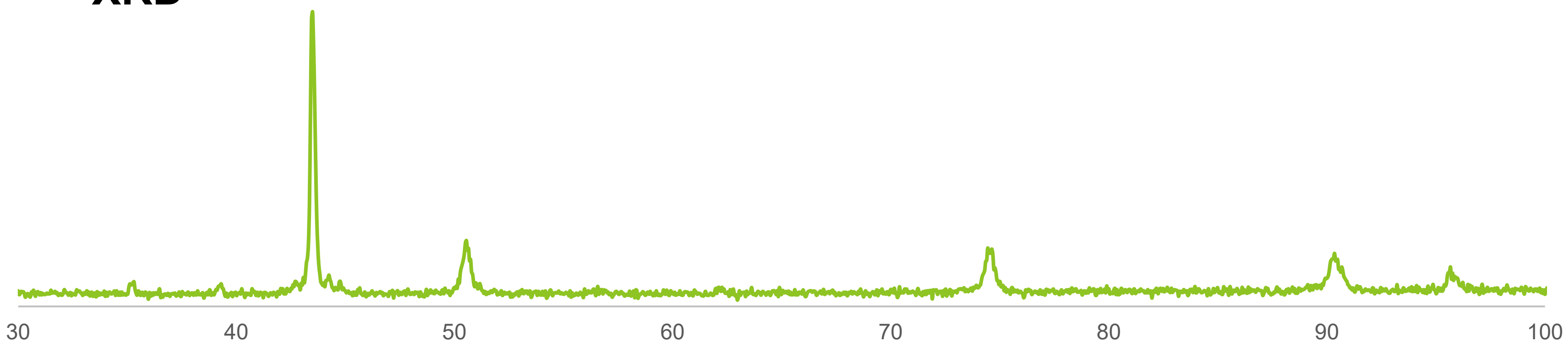
Backscatter, 200x



Backscatter, 2000x



XRD

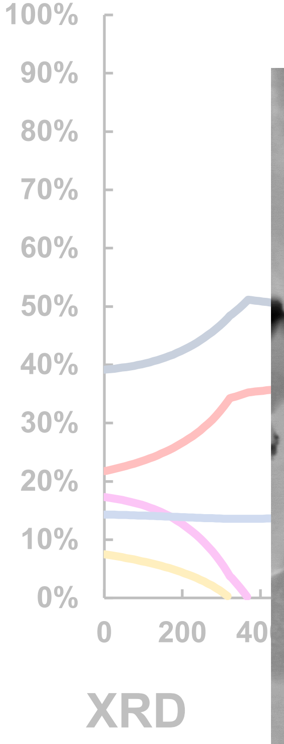


CALPHAD: FCC + FCC + σ

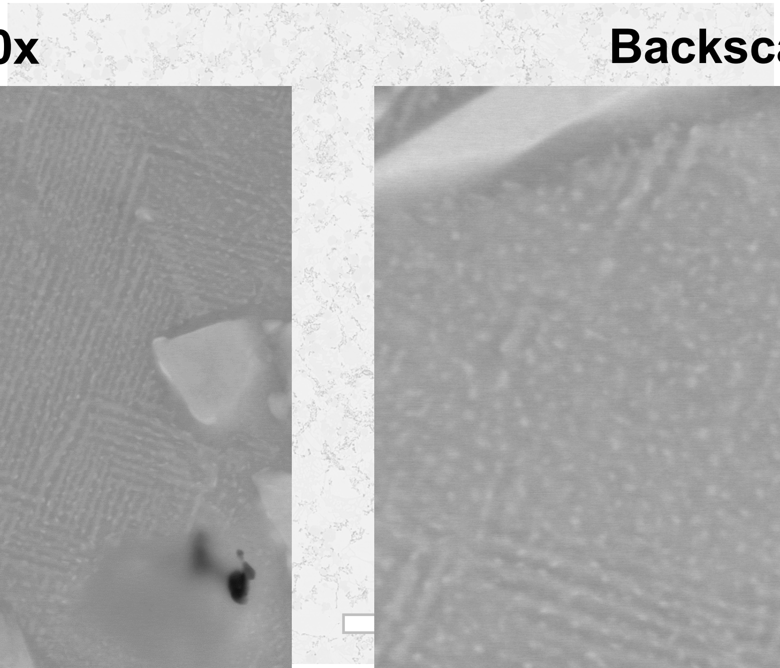
XRD: FCC(x2?) + σ

NiCoCrFeMnCuAl

CALPHAD

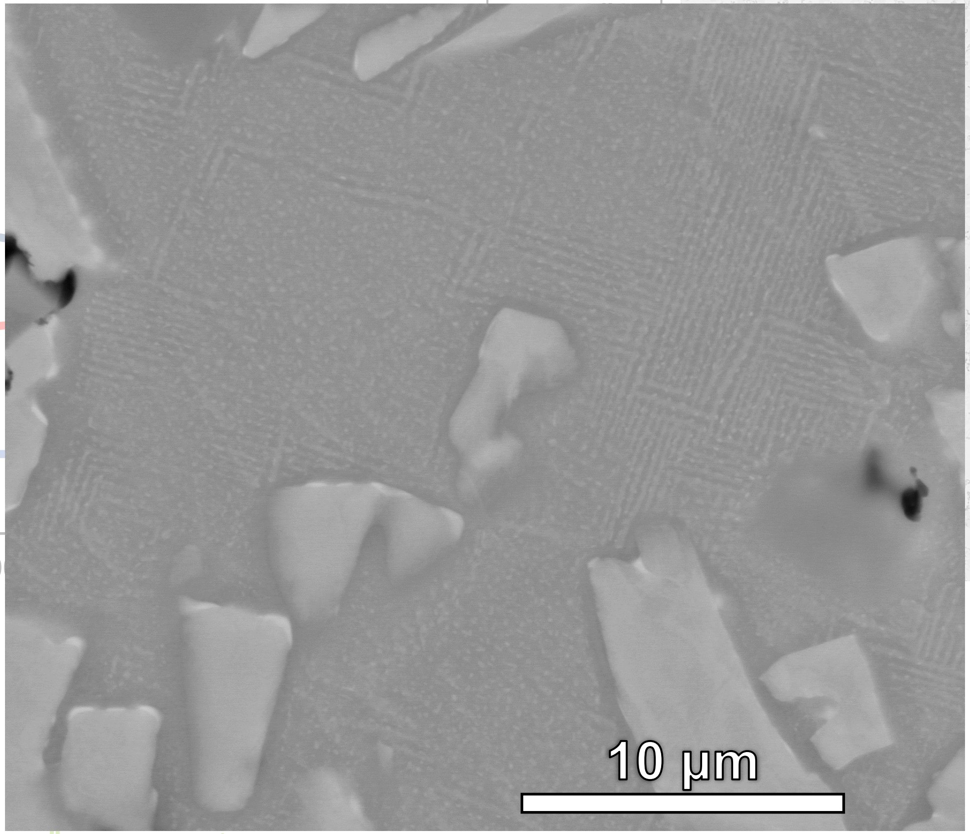


Backscatter, 200x

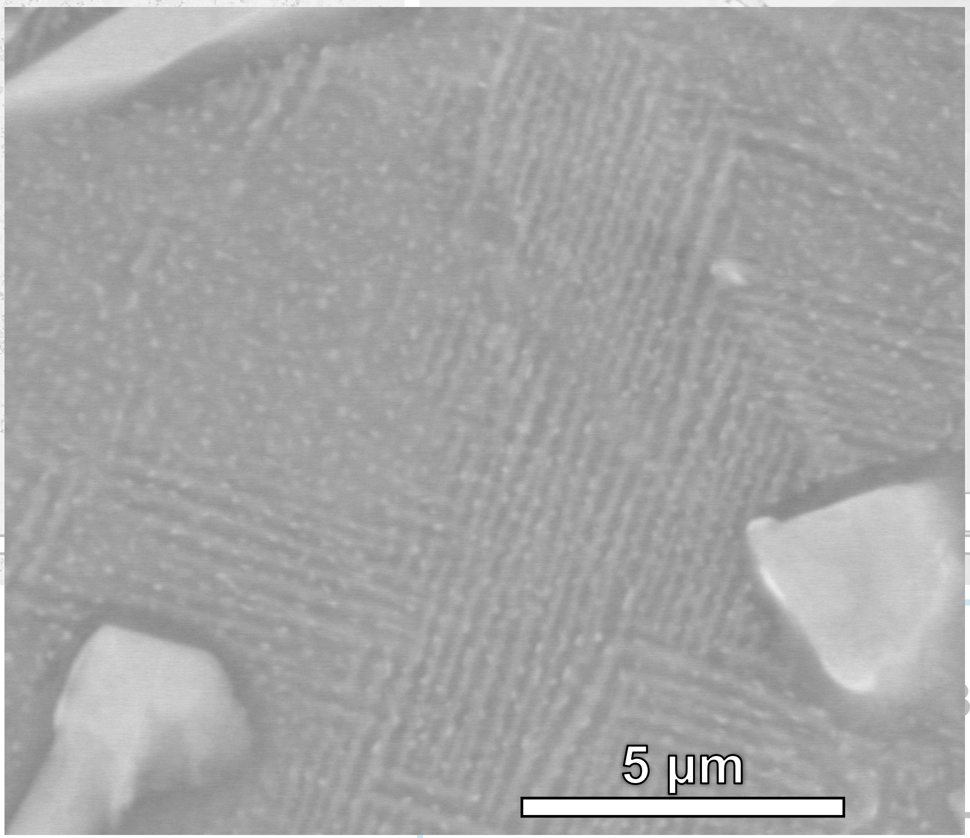


Backscatter, 2000x

Backscatter, 5000x

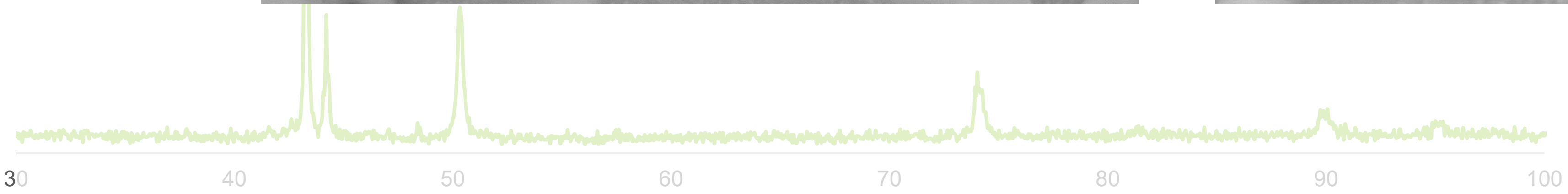


Backscatter, 10000x



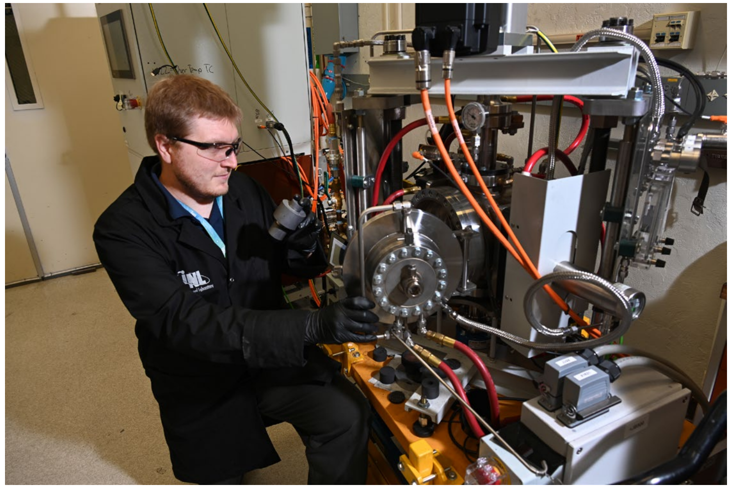
2 + σ

σ



Path Forward: Scale-Up

DCS-5
~2,500 A, 5 tons

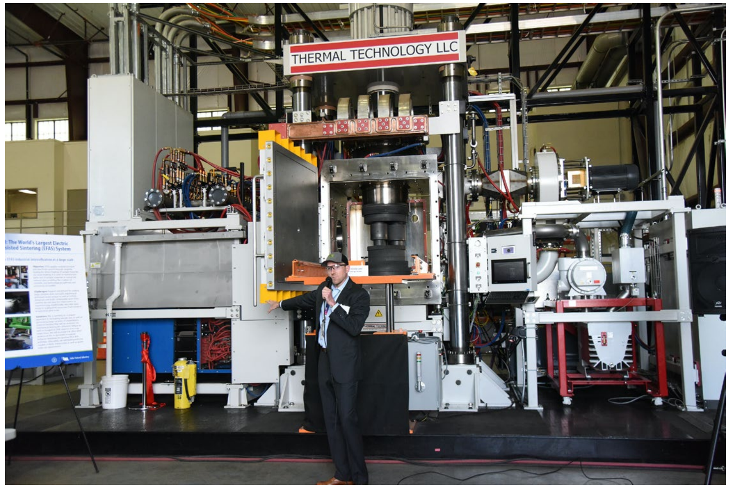


INL
Idaho Falls, ID

DCS-50
~20,000 A, 50 tons

Thermal Technology LLC
Minden, NV

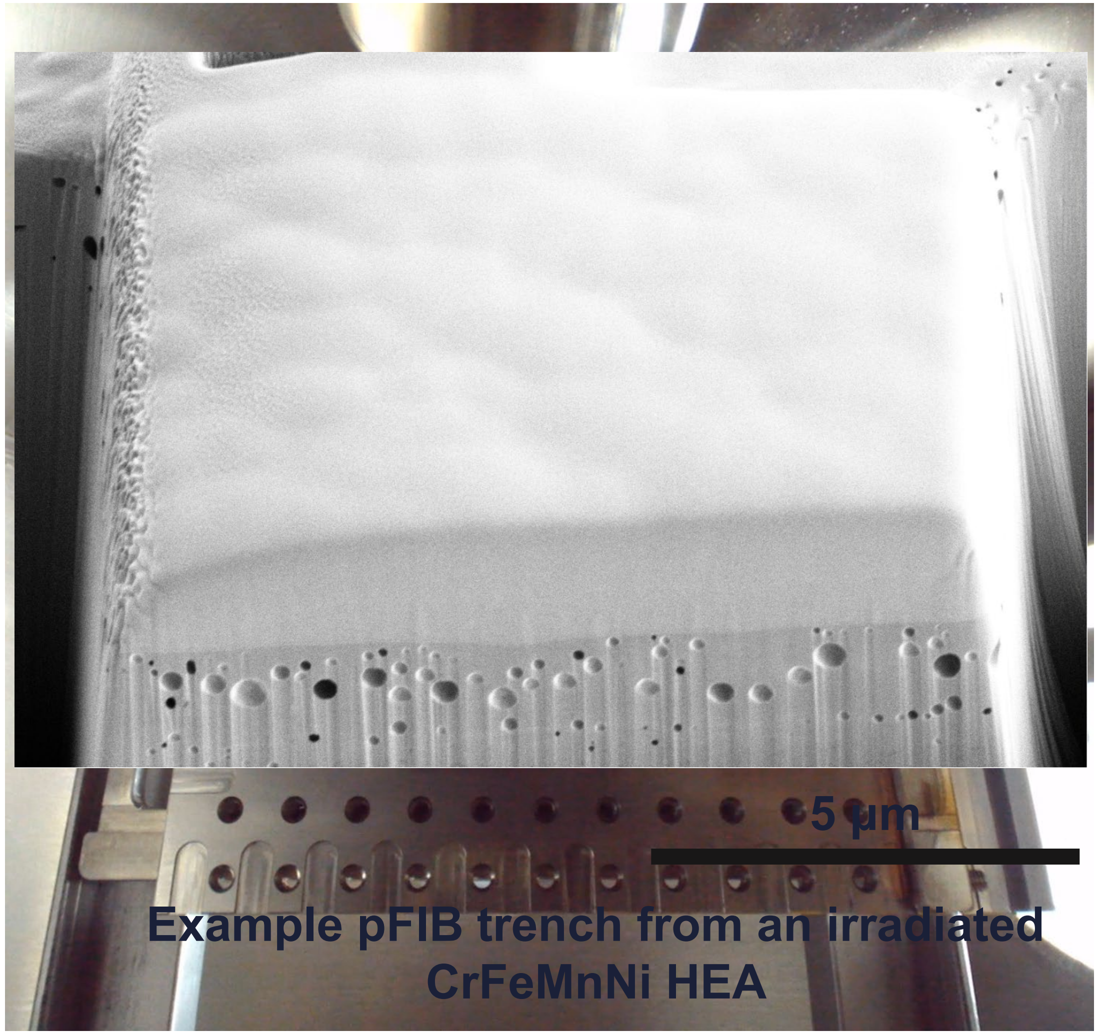
DCS-800
~150,000 A, 800 tons



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



Example pFIB trench from an irradiated CrFeMnNi HEA

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

Funding: Work supported through the INL Laboratory Directed Research& Development (LDRD) Program under DOE Idaho Operations Office Contract DE-AC07-05ID14517

Idaho National Laboratory

Battelle Energy Alliance manages INL for the U.S. Department of Energy's Office of Nuclear Energy. INL is the nation's center for nuclear energy research and development, and also performs research in each of DOE's strategic goal areas: energy, national security, science and the environment.