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Center for Advanced Non-Ferrous Structural Alloys

An Industry/University Cooperative Research Center

Project 33B-L: In-Situ Studies of Strain Rate Effects on Phase Transformation and Microstructural Evolution in Multi-Principal Element Alloys

Spring 2019 Semi-Annual Meeting
Iowa State University, Ames, IA
April 3-5, 2019

Student: John Copley (Mines)

Faculty: Amy Clarke (Mines)

Industrial Mentors: TBD

Other Participants: Francisco Coury (UFSCAR), Jonah Klemm-Toole (Mines), Yaofeng Guo (Mines)

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Project 33B-L: In-Situ Studies of Strain Rate Effects on Phase Transformation and Microstructural Evolution in Multi-Principal Element Alloys

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- Student: John Copley (Mines)
- Advisor(s): Amy Clarke (Mines)

Project Duration
MS: September 2018 to May 2020

- Problem: The effects of strain rate and state and temperature on the TRIP/TWIP behavior exhibited by MPEAs are not well understood.
- Objective: Determine the relationship between alloying, strain rate and strain state effects on the evolution of deformation twins and deformation induced phase changes.
- Benefit: Improved understanding of TRIP/TWIP behavior seen in other materials, alloy design for specific applications, especially blast resistance.

Recent Progress
- Thermo-Calc Modelling of TRIP likely regions of CoCrNi ternary
- In-situ diffraction during dynamic testing at the Advanced Photon Source (APS) at Argonne National Lab

Metrics

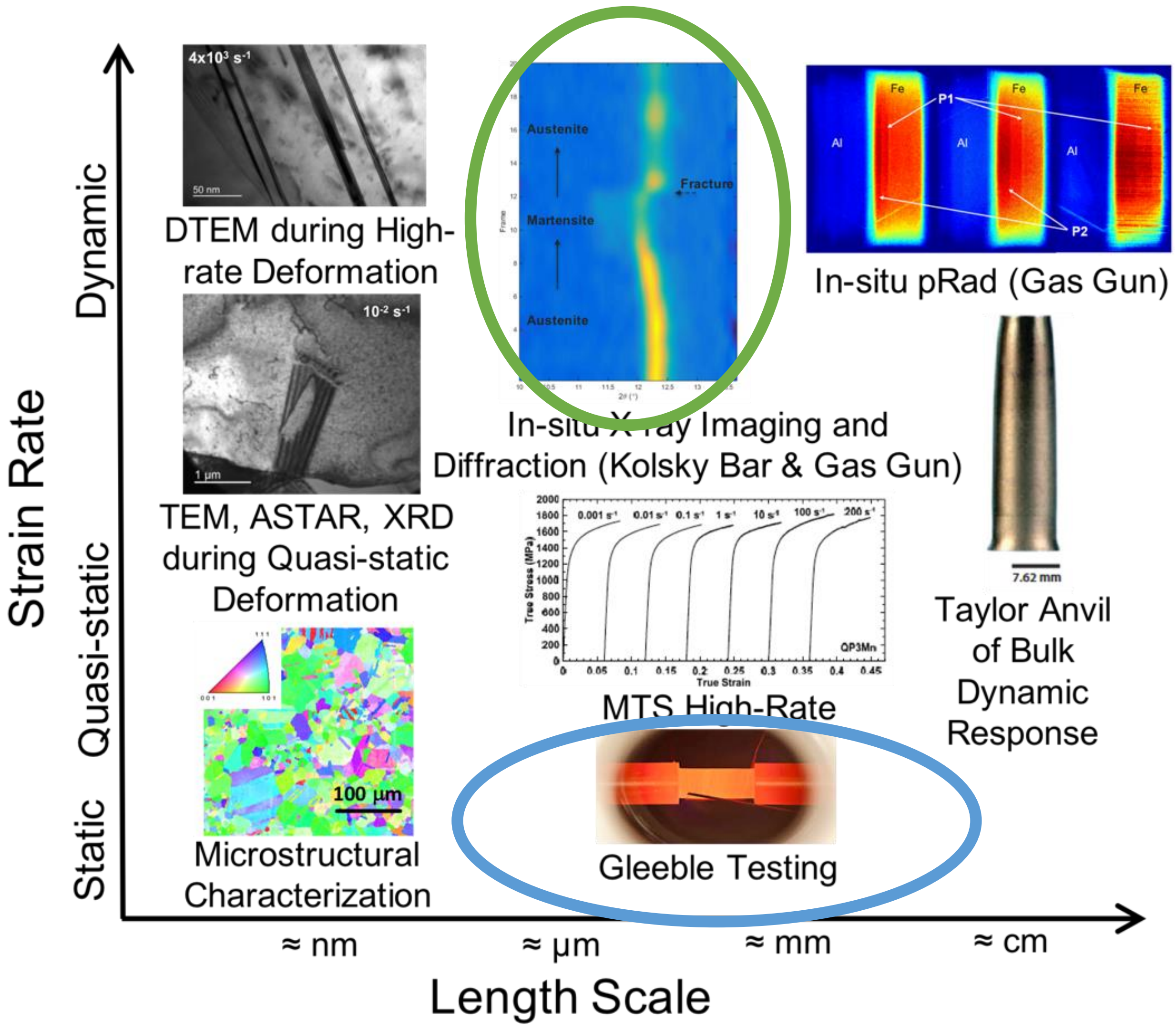
Description	% Complete	Status
1. Literature review	40%	●
2. Quasi-Static Testing	15%	●
3. Dynamic Testing	25%	●
4. Multi-scale in-situ imaging and diffraction	25%	●

Industrial Relevance

- Understanding of TRIP/TWIP of MPEAs during high rate deformation
  - New strategies to design deformation mechanisms
  - Drive development of alloys for blast-resistance and performance in extreme environments

- Fundamental understanding of TRIP/TWIP
  - Applications to more commonly used Advanced High Strength Steels and some Ti alloys

Project Vision

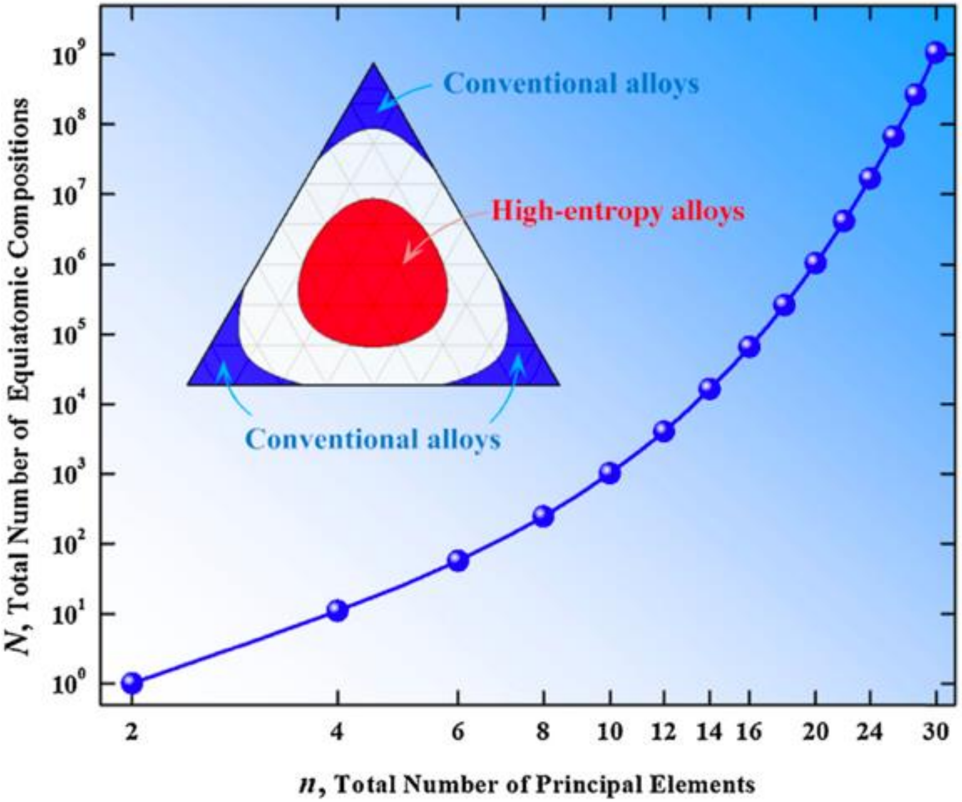


State-of-the-art, multi-scale microstructural characterization with electrons, x-rays, and protons of TRIP/TWIP in MPEAs for blast resistance

Figure courtesy of Dr. Amy Clarke

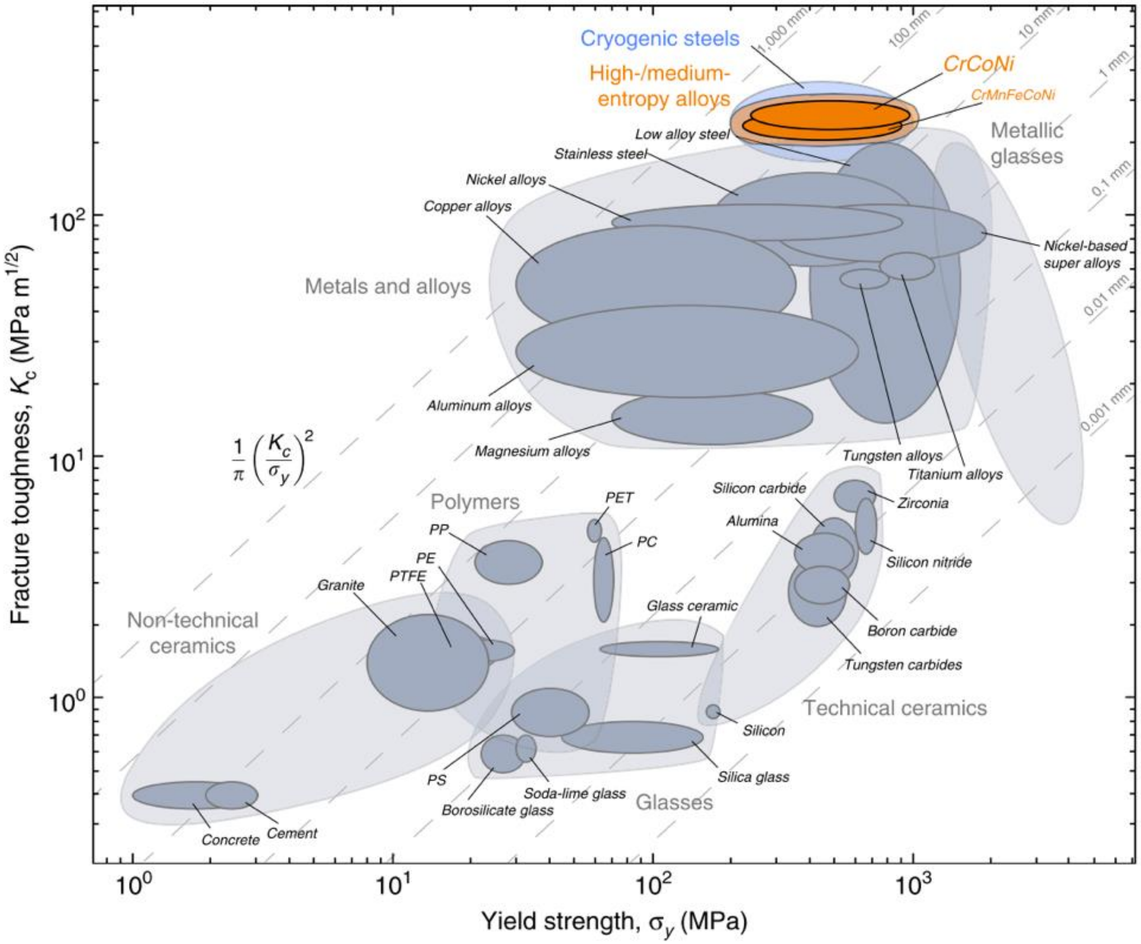
MPEAs

- No definable main element
  - Equiatomic, or
  - Several (>2) components present in very high concentrations
- Almost infinite combinations
- Commonly referred to as High Entropy Alloys (HEAs) and/or Complex Concentrated Alloys (CCAs)



Y.F. Ye, Q. Wang, J. Lu, C.T. Liu, Y.Yang. Materials Today, 2016, 19(6):349-362

Project Background: MPEAs



B. Gludovatz, et al., Nature Communications, 2016, 7:10602

- Multi-Principal Element
  - Broader definition than HEAs
    - Strength and toughness do not scale with entropy
  - CoCrNi Family
    - Fails HEA criteria
    - Toughest known CCAs

Twinning and Transformation Induced Plasticity

- Deformation accommodated by change in local atomic stacking
- Increased work hardening rates
  - Burgers vectors are not conserved at twin or phase interfaces
  - The "Dynamic" Hall-Petch Effect
  - High work hardening rates delay instability
- Delayed Instability
  - Increased UTS, elongation
  - Improved toughness

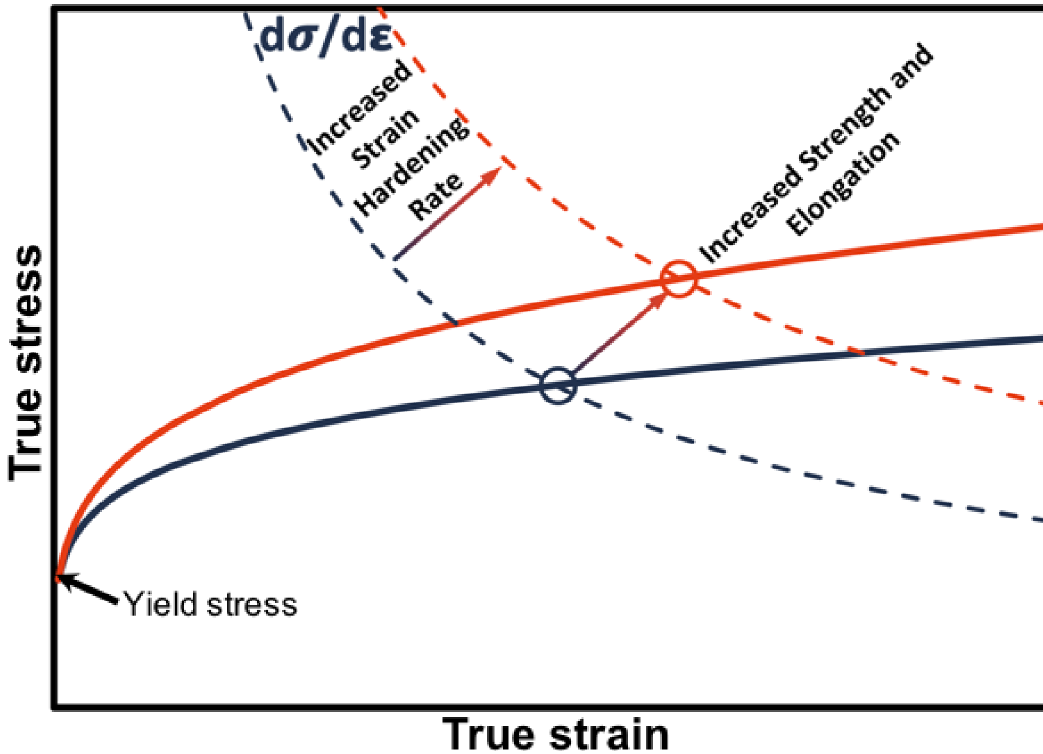
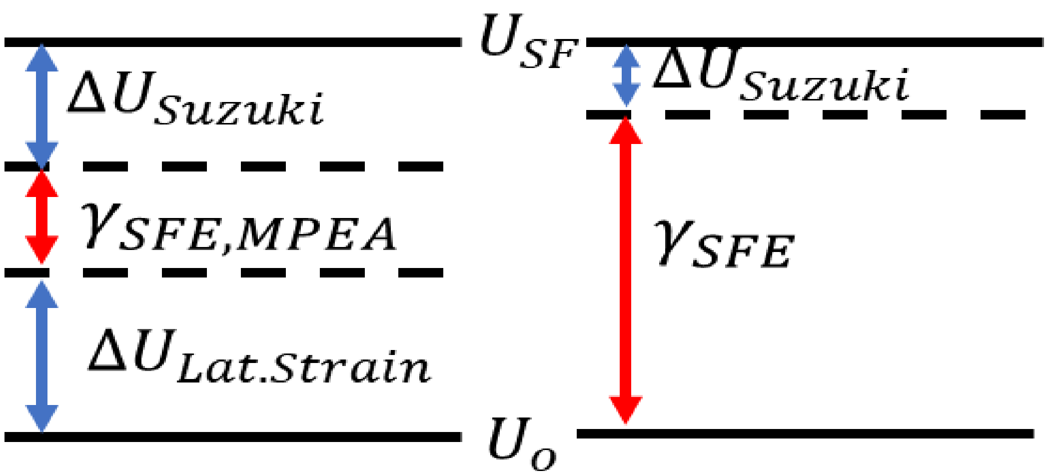


Figure courtesy of Dr. Kester Clarke

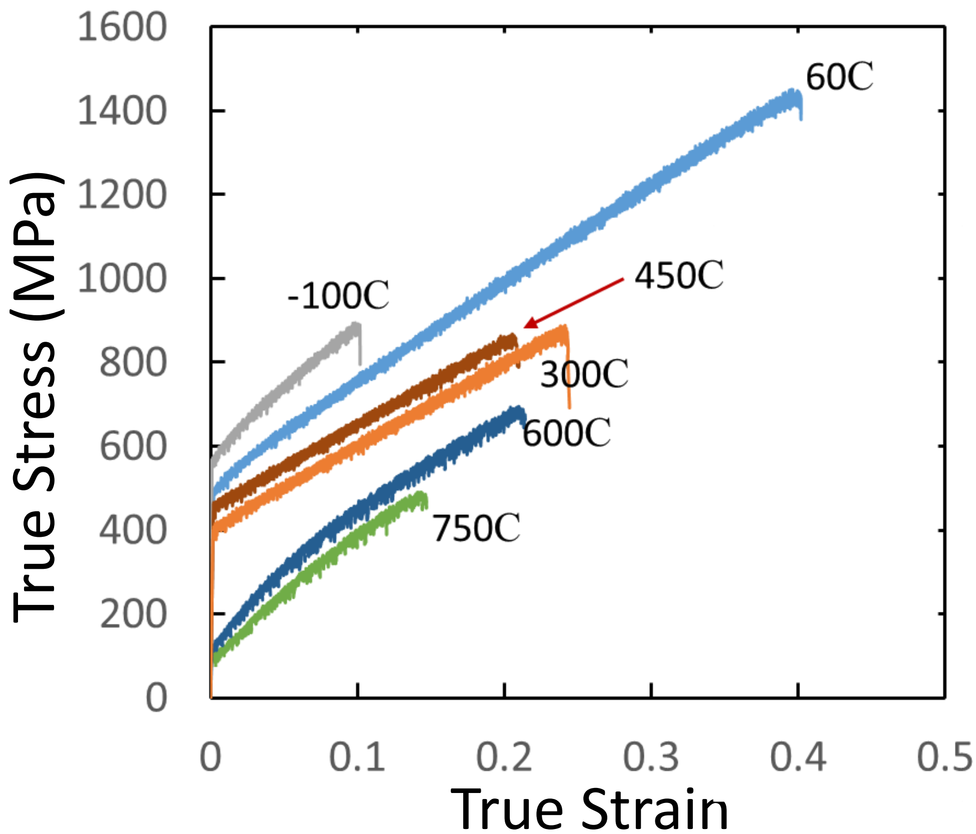
Why are MPEAs Good Candidates for TRIP/TWIP

- Shown to occur in some MPEAs
  - CoCrNi, FeCoCrNi, FeMnCoCr, FeMnCoCrNi
- High occurrence of twins is expected (low SFE)
  - Suzuki Interaction
  - Lattice Distortion



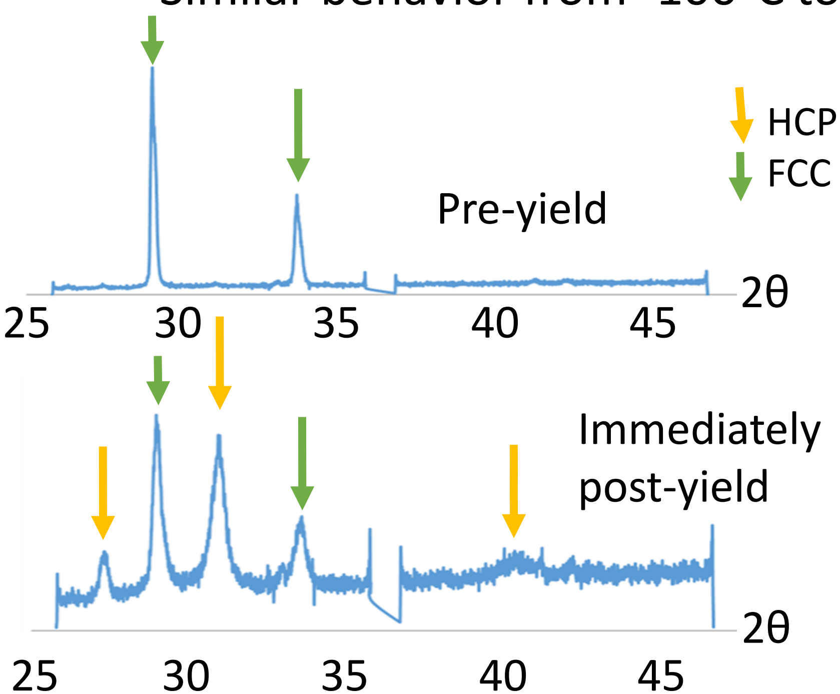
Project Review:

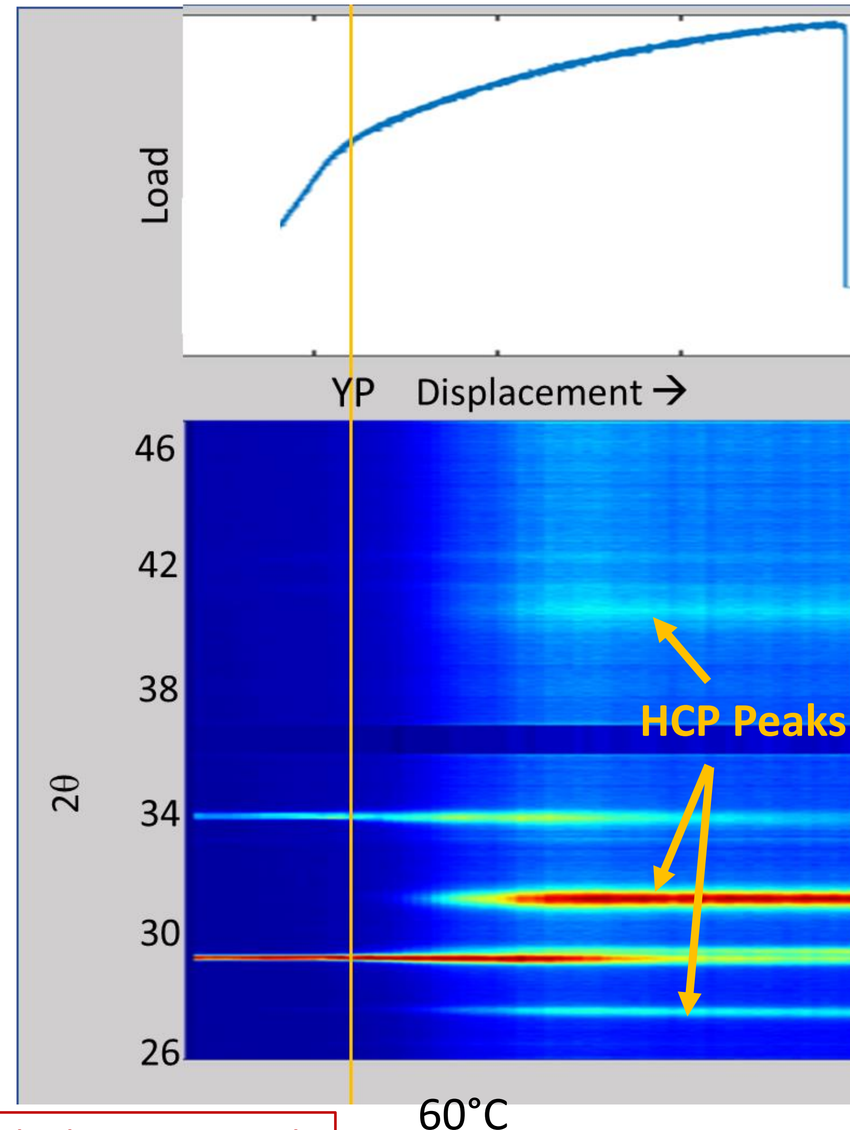
- Quasi-Static Thermo-Mechanical Testing ($Co_{55}Cr_{40}Ni_5$)
  - Gleeble thermo-mechanical simulator during synchrotron x-ray diffraction
  - Spray formed samples
    - Porosity led to premature failure



Project Review:

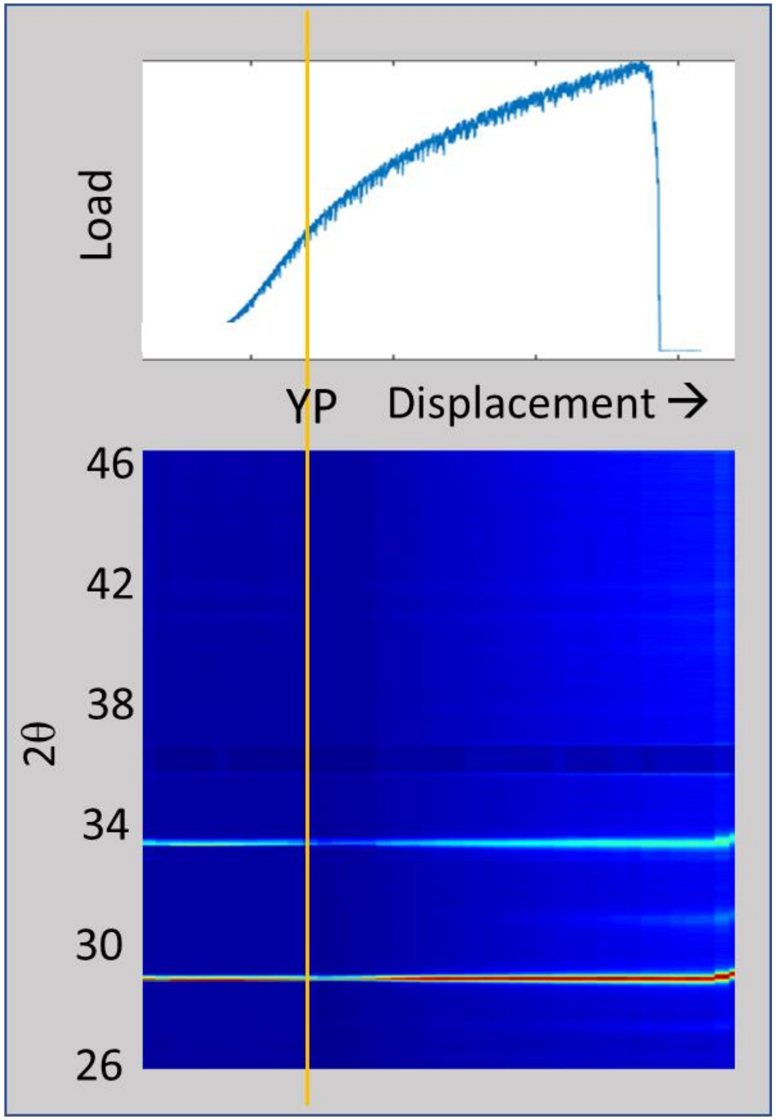
- Low Temperature Deformation ($Co_{55}Cr_{40}Ni_5$):
  - Transformation from FCC→HCP as strain increases
  - Onset of transformation at or near yielding
  - Similar behavior from -100°C to 450°C





Project Review

- Low Temperature Deformation ($Co_{55}Cr_{40}Ni_5$):
  - No evidence of transformation induced plasticity
  - Transformation suppressed at elevated temperatures
    - Some evidence of transformation to HCP after failure of sample—diffusional transformation
  - Similar behavior for tests conducted between 600°C and 900°C



600°C

Recent Progress: Thermo-Calc Modelling of T_0

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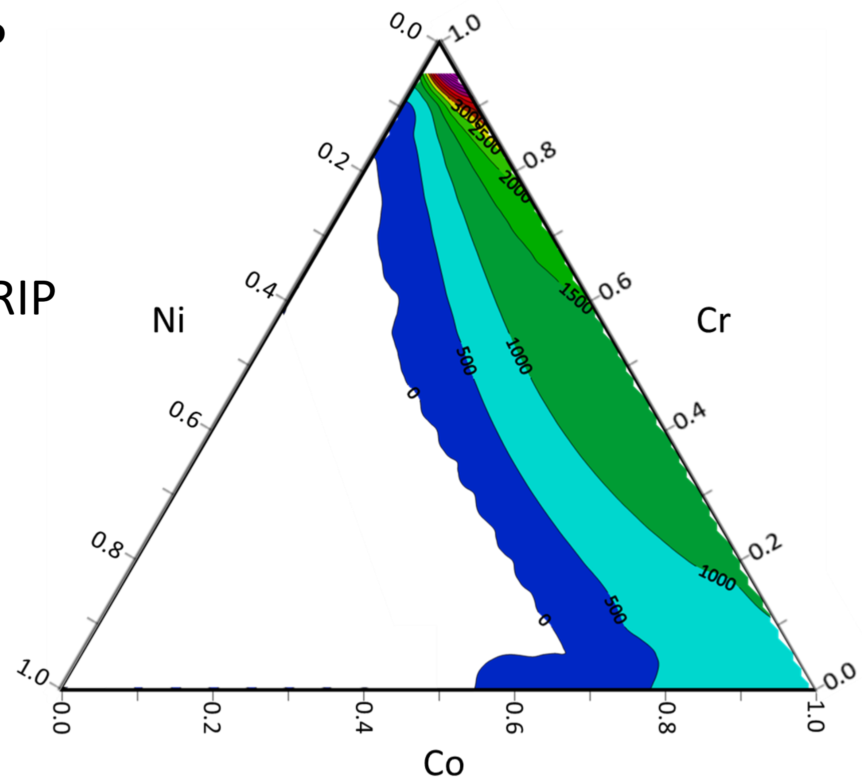
- T_0: The diffusionless transformation temperature
  - Free energy and composition of HCP and FCC phases are the same
  - Diffusionless transformations (i.e. TRIP) must occur below T_0
  - High T_0 indicates the possibility of TRIP behavior at elevated temperatures
- In CoCrNi
  - T_0 is a strong function of cobalt content → low cobalt alloys had no predicted T_0
  - T_0 is highest with large chromium concentrations



Recent Progress: Thermo-Calc Modelling for Heat Treatments

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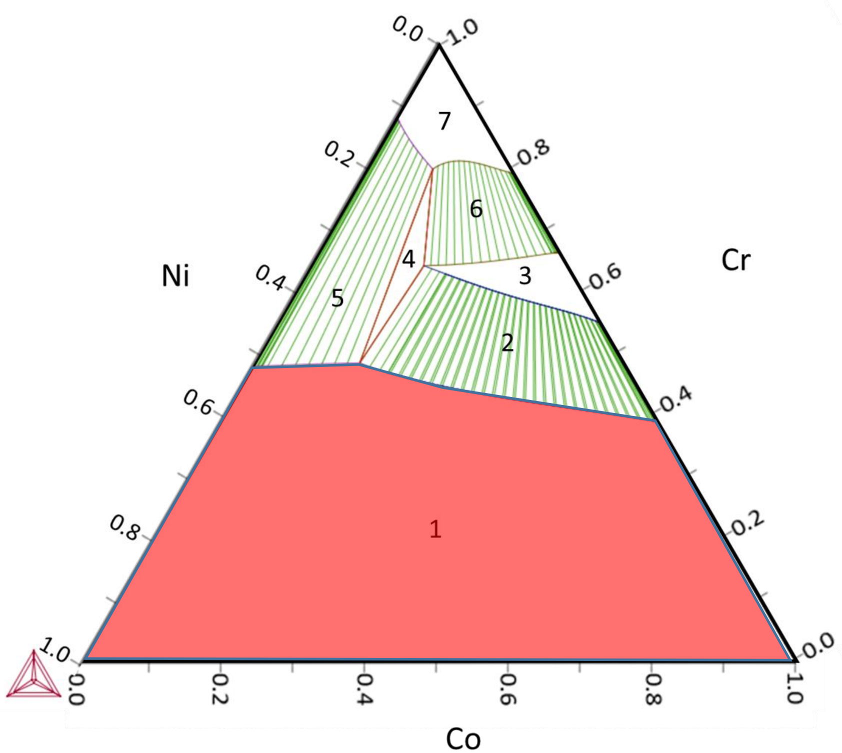
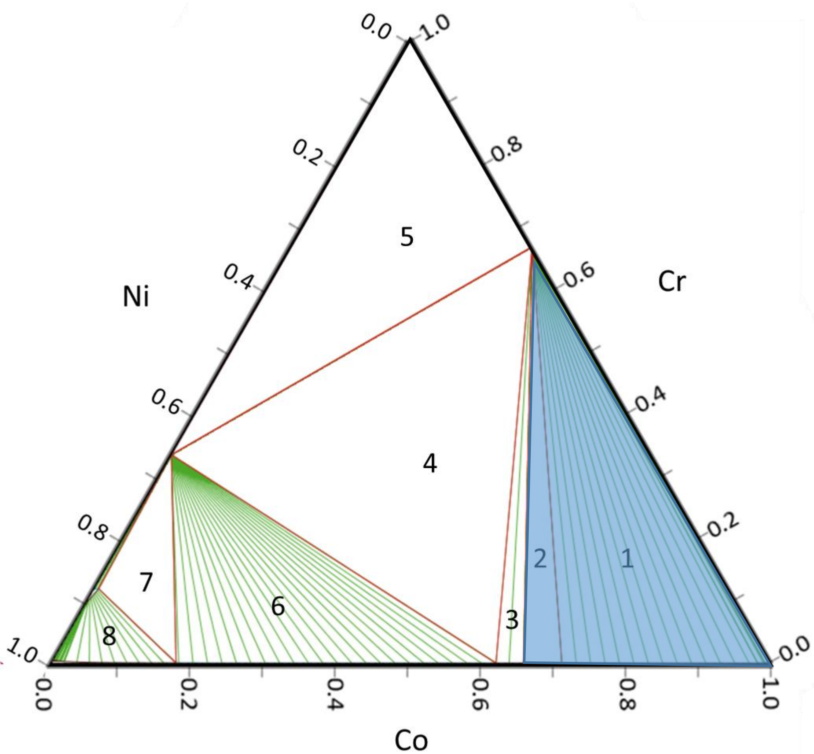


1: FCC_L12
2: FCC_L12+Sigma
3: Sigma
4: FCC_L12+BCC_B2+Sigma
5: FCC_L12+BCC_B2
6: BCC_B2+Sigma
7:BCC_B2

Isothermal section of CoCrNi ternary at 1100°C

- MPEAs are susceptible to formation of brittle intermetallics
  - Want to design an alloy and heat treatment capable of achieving a single phase, FCC solid solution

Recent Progress: Thermo-Calc Modelling for TRIP regions



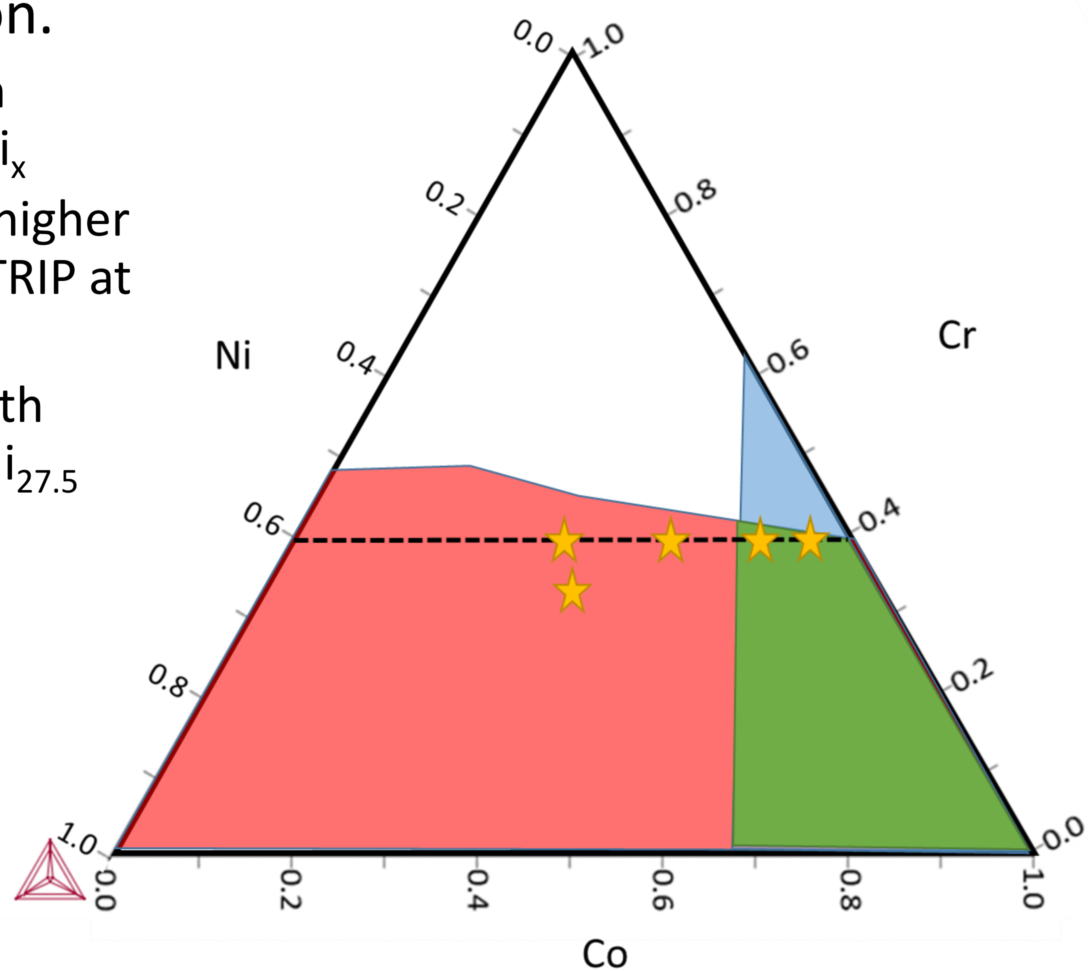
1: HCP_A3+Sigma
2: HCP_A3+FCC_L12+Sigma
3: FCC_L12+Sigma
4: $CrNi_2$+FCC_L12+Sigma
5: $CrNi_2$+BCC_B2+Sigma
6: $CrNi_2$+FCC_L12
7: $CrNi_2$+FCC_L12+FCC-L12#2
8: FCC_L12+FCC_L12#2

Isothermal section of CoCrNi ternary at 25°C

- For TRIP behavior, there must be an energy saving from transforming.
  - Must transform to the stable or metastable phase
  - Likely regions for FCC→HCP TRIP will be near stable HCP phase regions

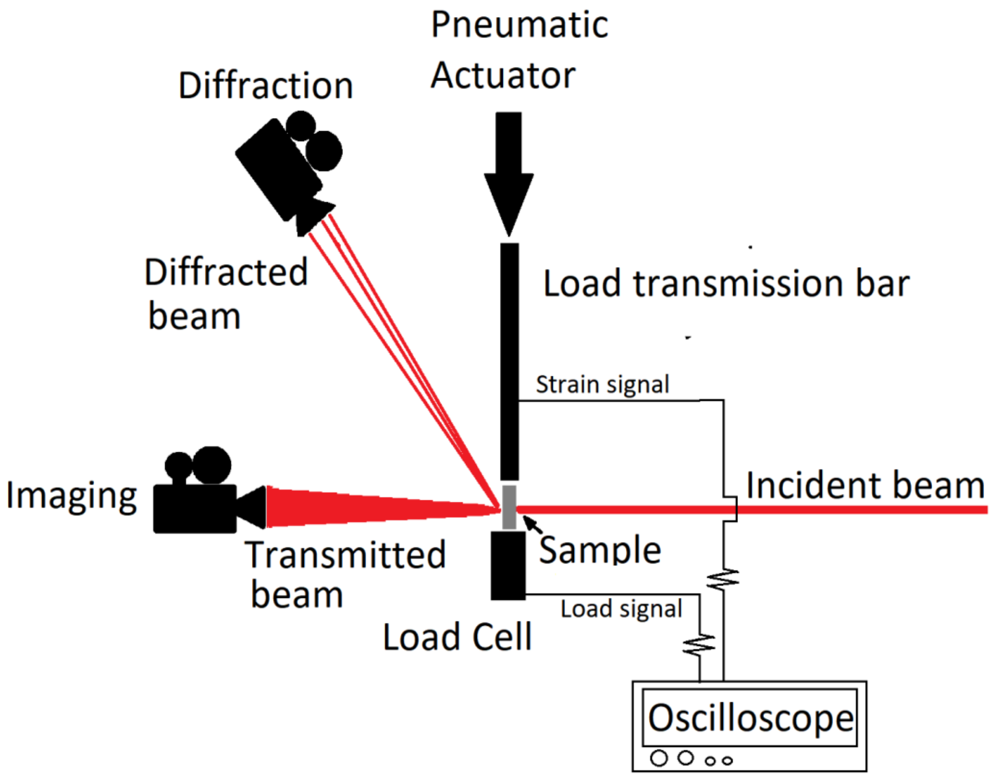
Alloy Design Space

- TRIP capable alloys should be in or near the green region.
  - Alloys for study have been selected along $Co_{60-x}Cr_{40}Ni_x$
  - High chromium alloys for higher T_0 values→ possibility of TRIP at high temperatures
  - Also nears the high strength and toughness $Co_{27.5}Cr_{45}Ni_{27.5}$ predicted by Dr. Francisco Coury's EARS model
  - Alloys selected:
    - $Co_{55}Cr_{40}Ni_5$
    - $Co_{50}Cr_{40}Ni_{10}$
    - $Co_{40}Cr_{40}Ni_{20}$
    - $Co_{30}Cr_{40}Ni_{30}$
    - $Co_{33.3}Cr_{33.3}Ni_{33.3}$



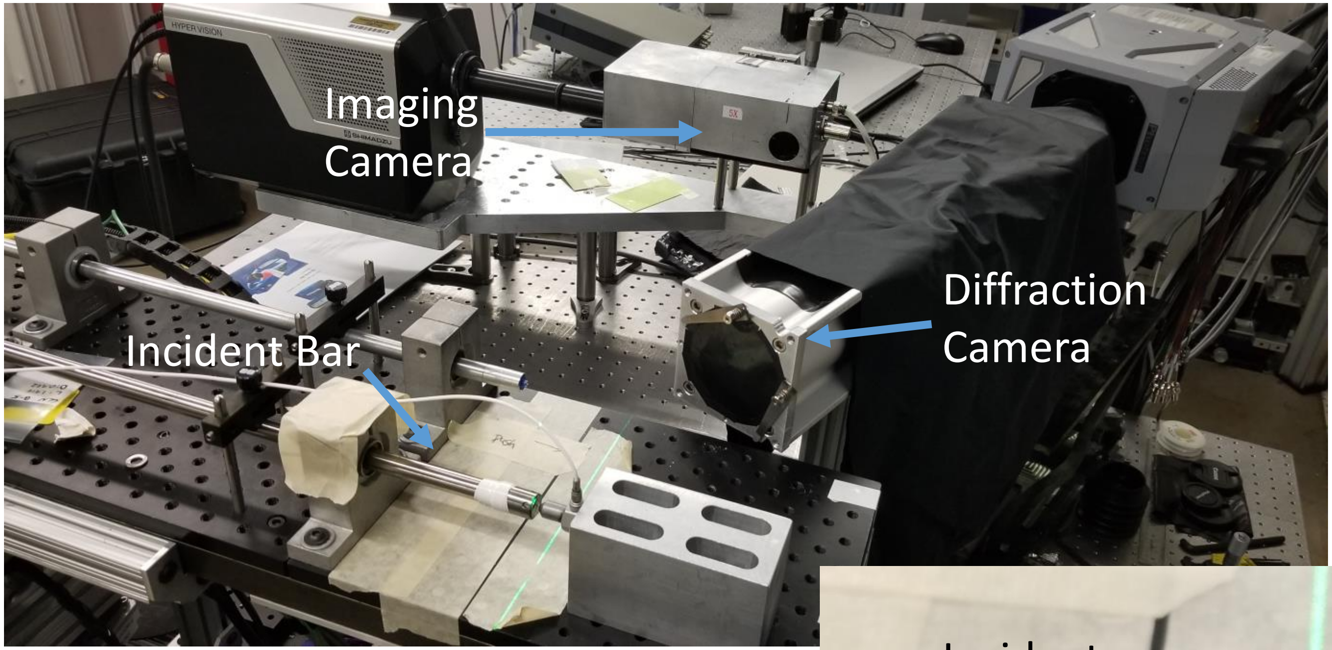
In-situ Dynamic Testing at APS

- In-situ Kolsky Bar, X-ray imaging and diffraction experiments
- Alloys tested:
  - $Co_{50}Cr_{40}Ni_{10}$
  - $Co_{40}Cr_{40}Ni_{20}$
  - $Co_{30}Cr_{40}Ni_{30}$
  - $Co_{33.3}Cr_{33.3}Ni_{33.3}$
- Loading in both tension and compression
  - Compression testing undertaken at two strain rates

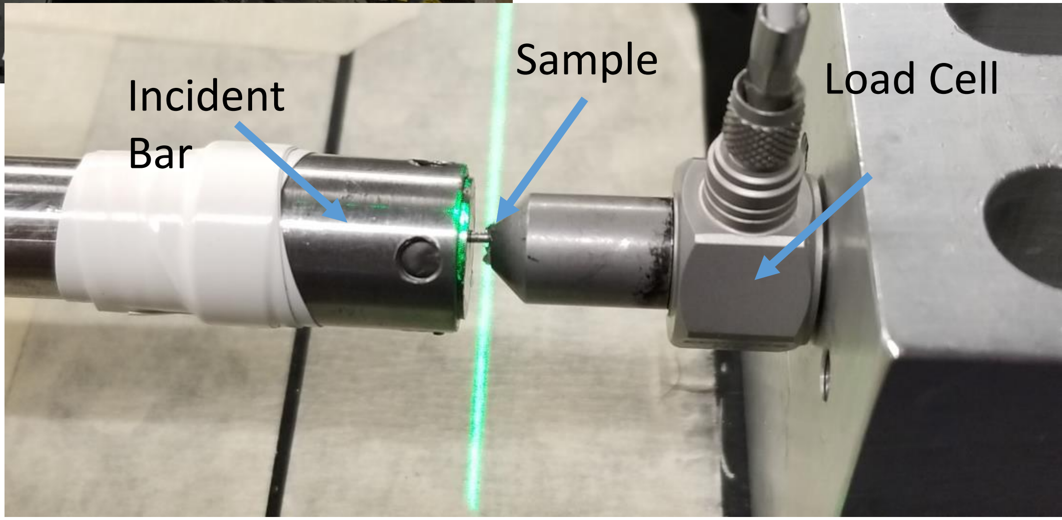


Thanks to Ben Ellyson for the figure

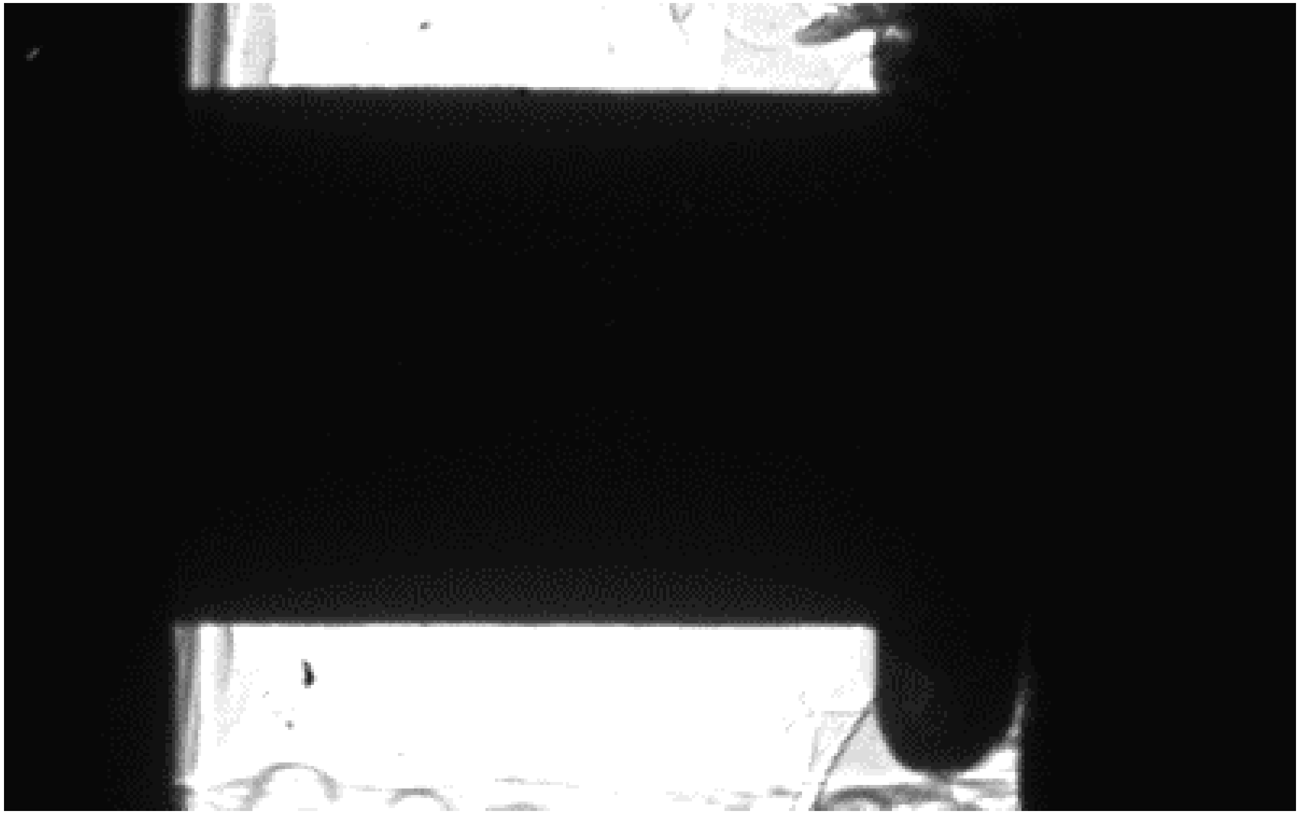
APS Setup


Imaging
Camera
Diffraction
Camera
Incident Bar


5mm


Incident
Bar
Sample
Load Cell

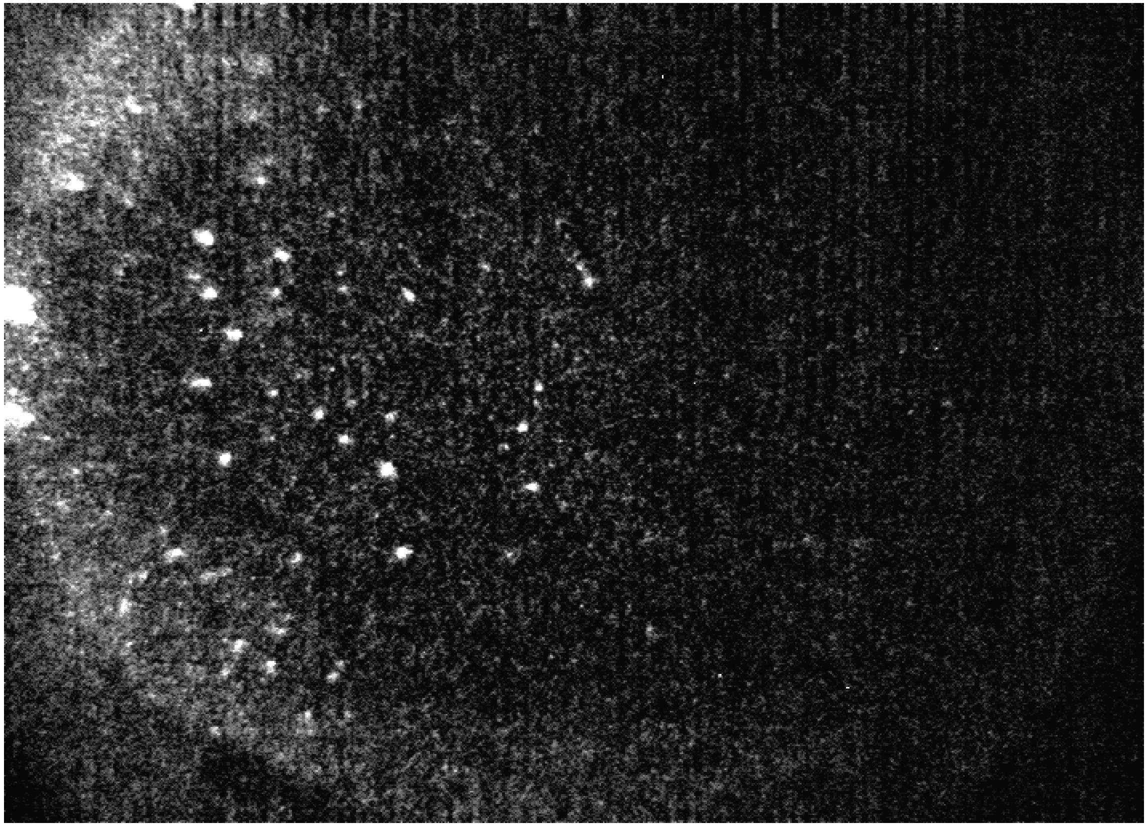
Preliminary APS Results



Dynamic Compression of Equiatomic CoCrNi MPEA

0.5mm

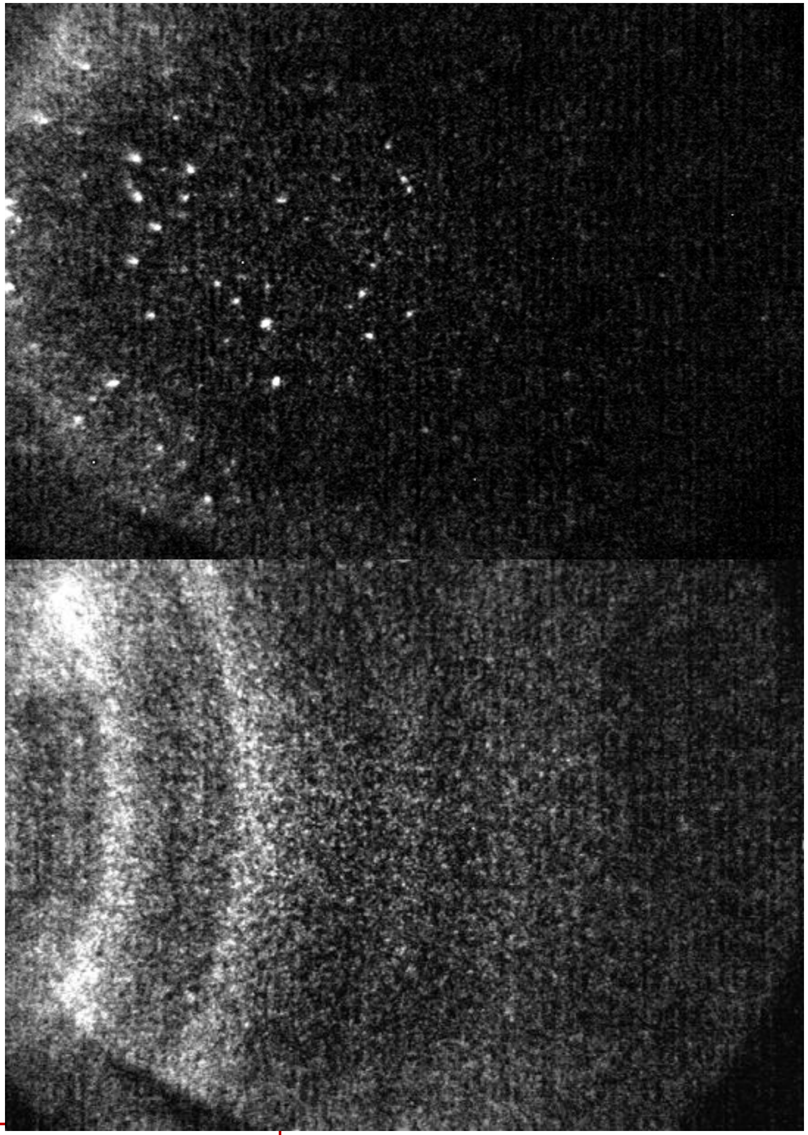
APS Diffraction Data



- Example diffraction data from CoCrNi MPEA during dynamic testing at APS

Diffraction Results

- Initial observation of the data suggests refinement in crystallite domain size
  - Few scattered points evolved into full, continuous rings
  - Either development of dislocation cells or twins
    - Both $Co_{27.5}Cr_{45}Ni_{27.5}$ and the equiatomic CoCrNi have been shown to have significant deformation twinning

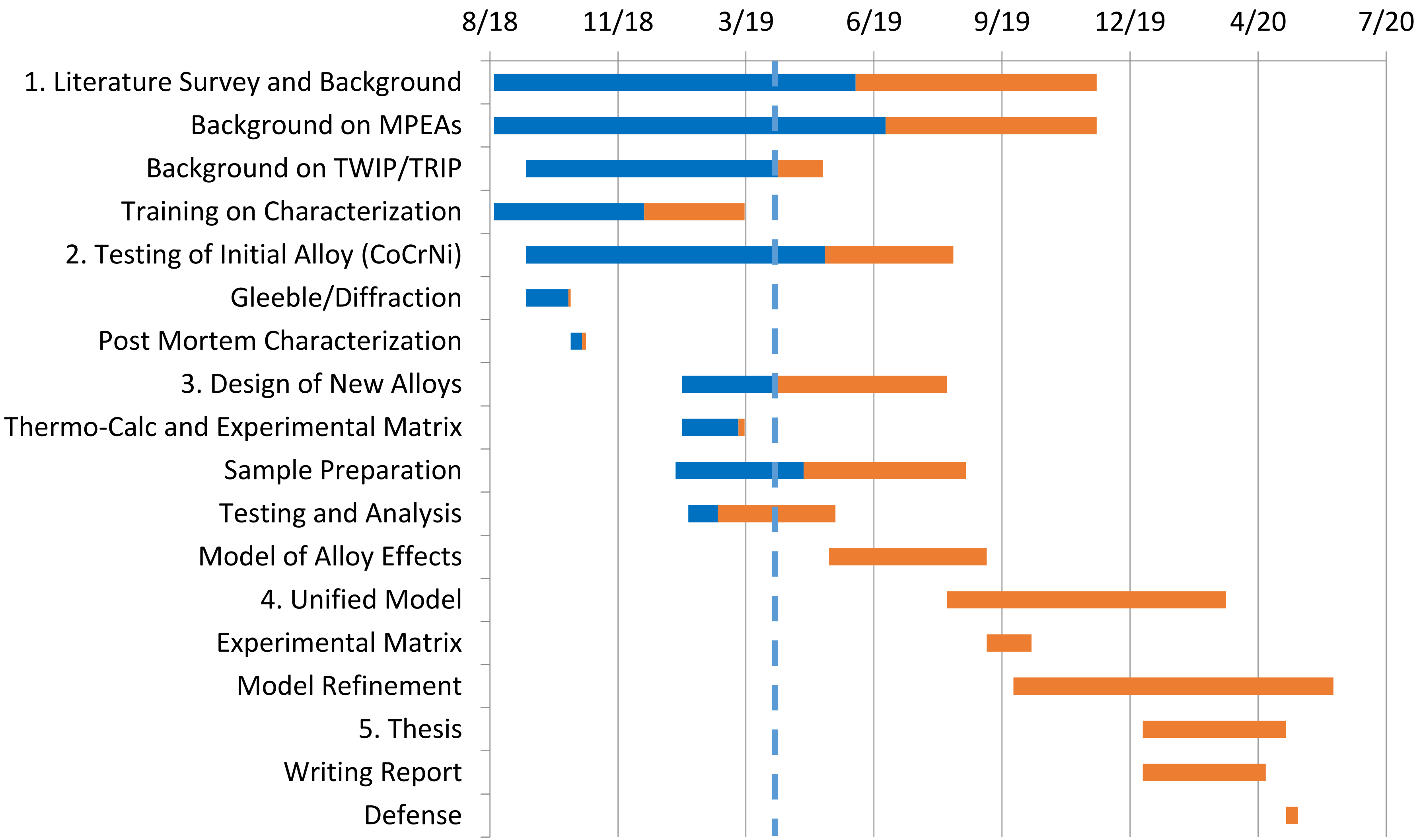


Future Work

- APS Data and Samples
  - Post-mortem microstructural analysis
    - EBSD and XRD
  - Processing of stress-strain data
  - Processing of diffraction data
    - Matlab program HisPoD
      - Integrate to get 1D diffraction pattern
  - Additional time at APS has been requested

- Additional Mechanical Testing
  - Strain rates of 10^{-2}-10^{2} s^{-1} achievable at CSM
  - Arc-melt and roll samples for larger size specimens

Gantt Chart


8/18
11/18
3/19
6/19
9/19
12/19
4/20
7/20
1. Literature Survey and Background
Background on MPEAs
Background on TWIP/TRIP
Training on Characterization
2. Testing of Initial Alloy (CoCrNi)
Gleeble/Diffraction
Post Mortem Characterization
3. Design of New Alloys
Thermo-Calc and Experimental Matrix
Sample Preparation
Testing and Analysis
Model of Alloy Effects
4. Unified Model
Experimental Matrix
Model Refinement
5. Thesis
Writing Report
Defense

Thank you!

John Copley
jacopley@mines.edu

Project 33B-L: In-Situ Studies of Strain Rate Effects on Phase Transformation and Microstructural Evolution in Multi-Principal Element Alloys

Student: *John Copley*

Faculty: *Amy Clarke*

Goals

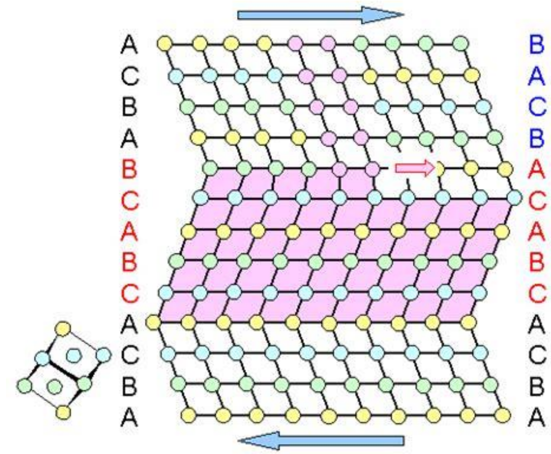
- Modeling of deformation behavior as it pertains to microstructural changes in MPEAs

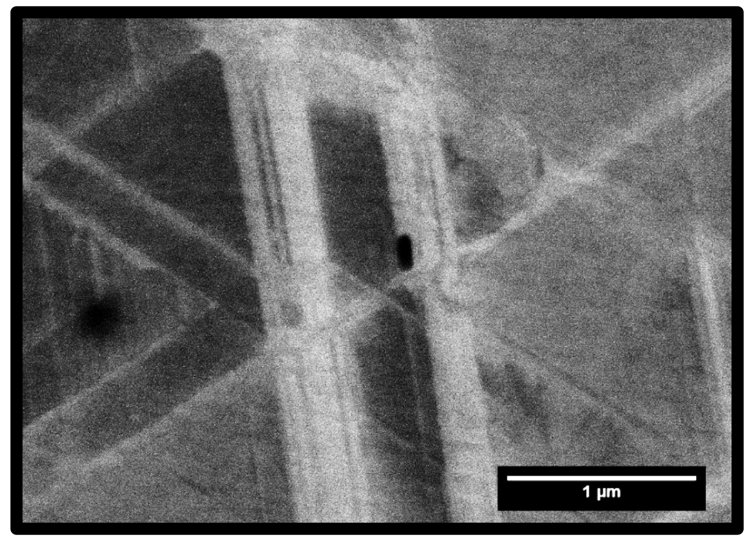
Significance and Impact

- The TRIP/TWIP behavior seen in some MPEAs results in high work hardening behavior, resulting in an increased ductility, toughness and blast resistance.

Research Details

- In-situ diffraction tests will show microstructural evolution (twins or HCP phase in a FCC matrix) which can be compared to the strain rate, strain state and alloy composition to allow alloy design for specific applications





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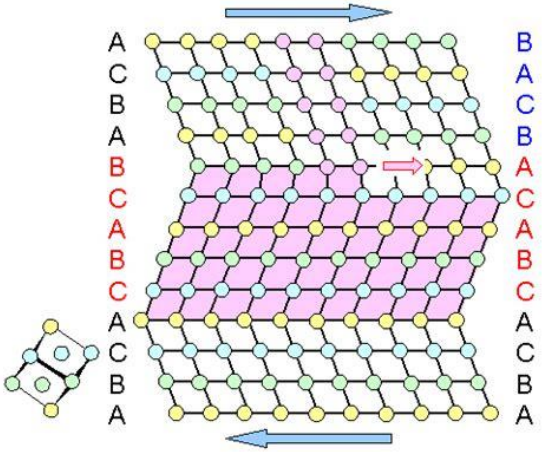

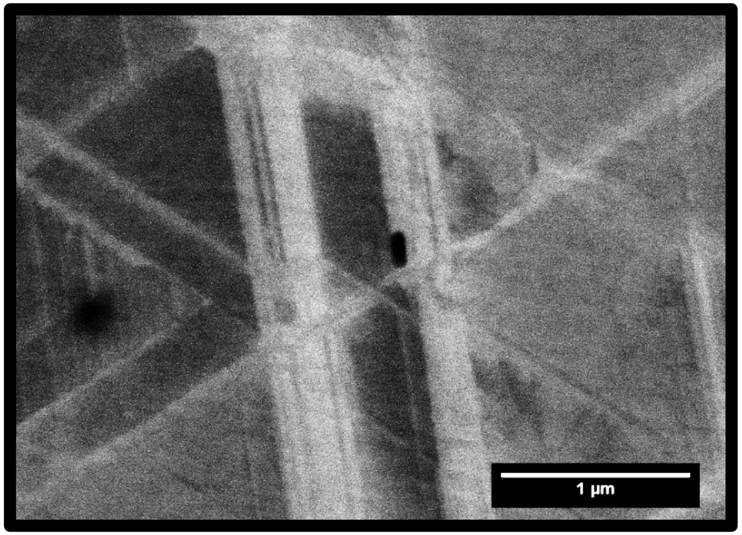

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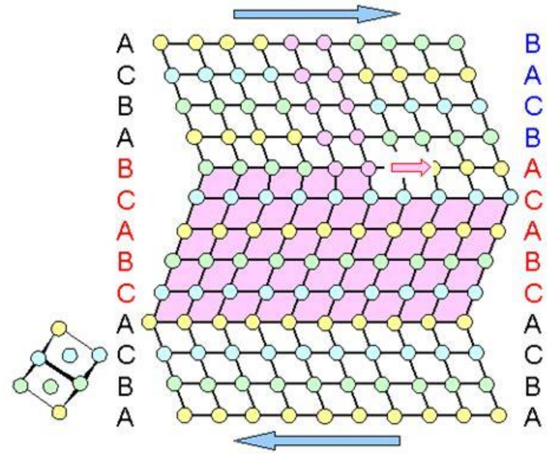
Project Duration: *Sept. 2018 – May 2020*

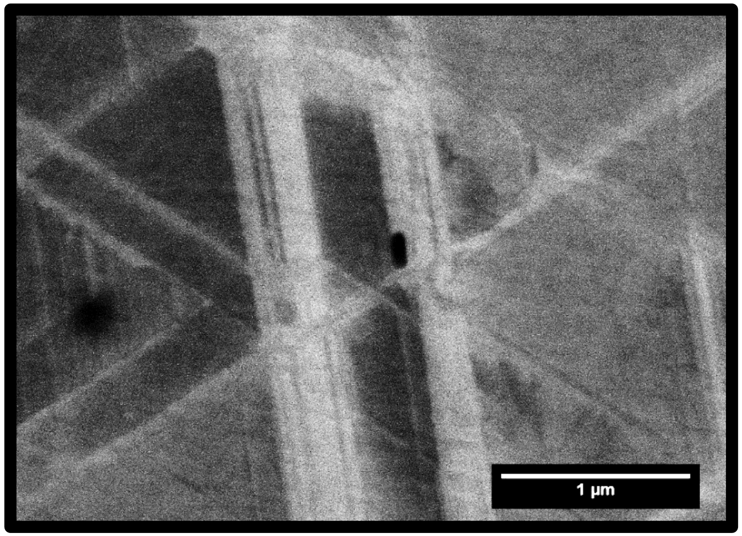


<u>Program Goal</u>

- Modeling of deformation behavior as it pertains to microstructural changes in MPEAs

<u>Approach</u>

- In-situ diffraction tests will show microstructural evolution (twins or HCP phase in a FCC matrix) which can be compared to the strain rate, strain state and alloy composition to allow alloy design for specific applications



<u>Benefits</u>

- The TRIP/TWIP behavior seen in some MPEAs results in high work hardening behavior, resulting in an increased ductility, toughness and blast resistance.