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

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

Project Duration
MS: September 2018 to Summer 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

- Material production and characterization of effects of processing (rolling, heat treatment) on grain size and phase.
- Determination of methods for better sample preparation for in-situ testing at the Advanced Photon Source

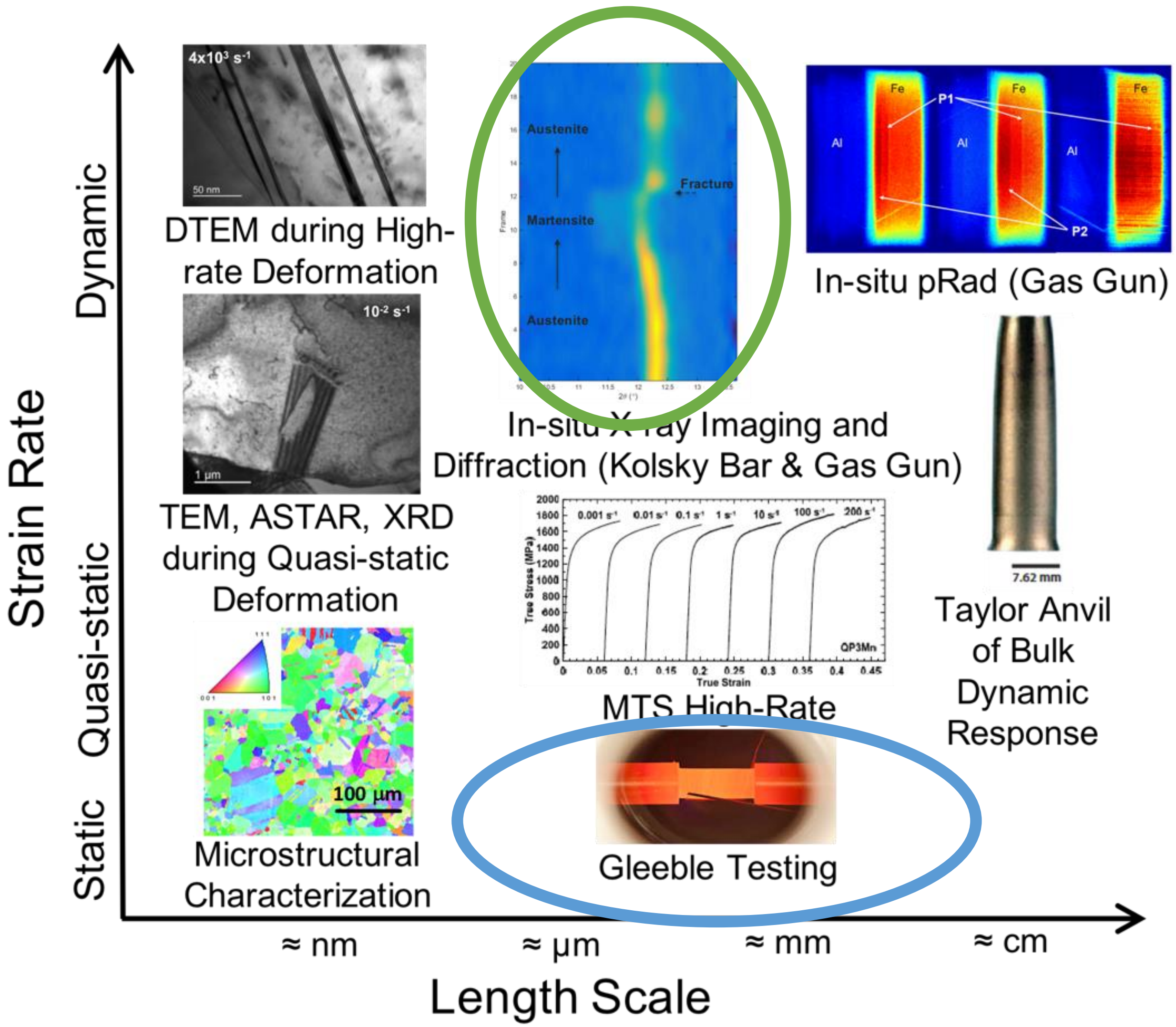
Metrics		
Description	**% Complete**	**Status**
1. Literature review	75%	●
2. Quasi-Static Testing	30%	●
3. Dynamic Testing	25%	●
4. Multi-scale in-situ imaging and diffraction	35%	●

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

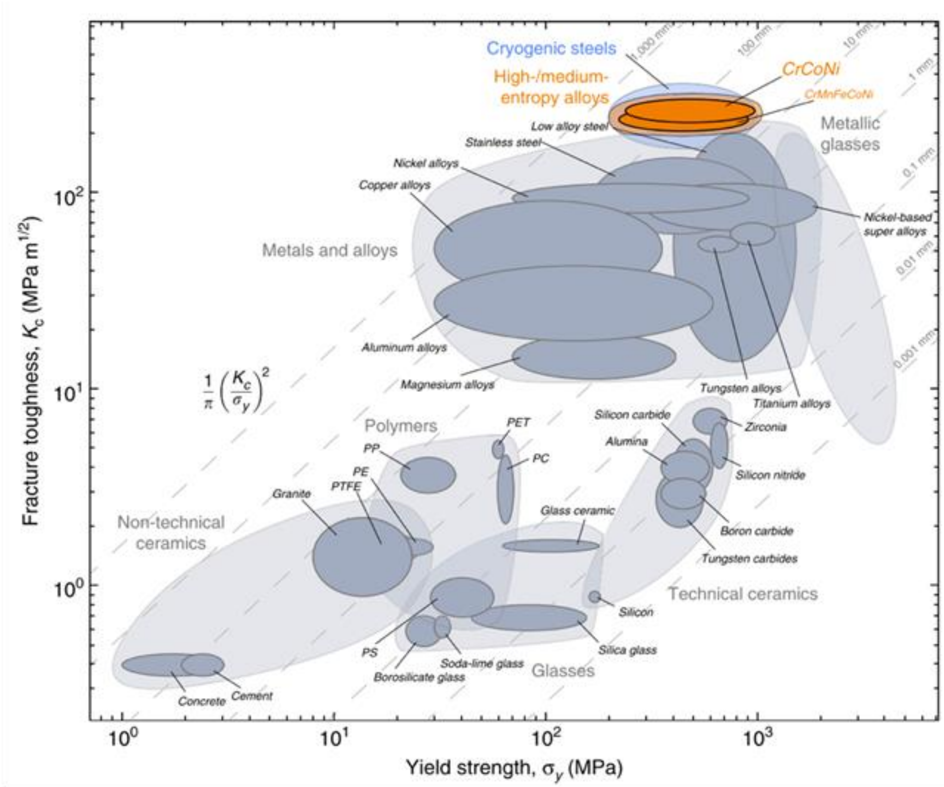


State-of-the-art, multi-scale microstructural characterization with electrons, x-rays, and protons of TRIP/TWIP in MPEAs for blast resistance

Figure courtesy of Dr. Amy Clarke

MPEAs

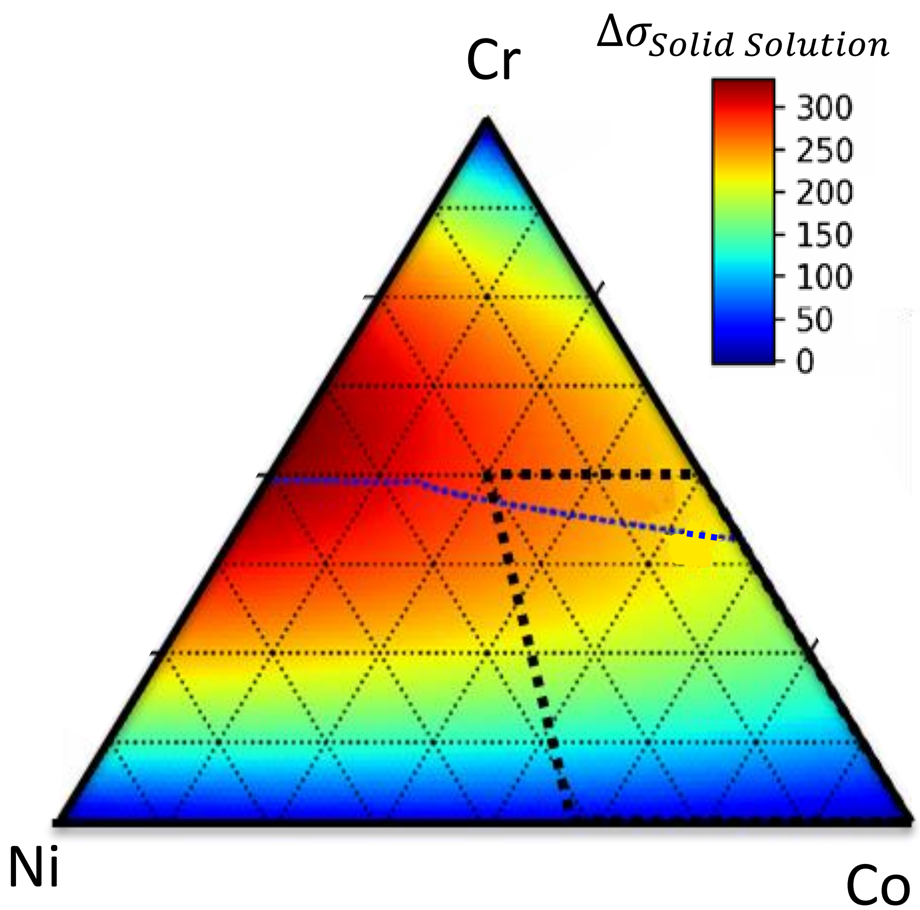
- No definable main element
  - Equiatomic, or
  - Several (>2) components present in very high concentrations
- Almost infinite combinations
- MPEA vs HEA
  - Broader definition than HEAs
    - Strength and toughness do not scale with entropy
  - CoCrNi Family
    - Fails HEA criteria
    - Toughest known CCAs



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

MPEAs and Solid Solution Strengthening

- Initial HEA/MPEA studies:
  - Equiatomic compositions
  - Solid solid solution strengthening
- Effective Atomic Radii for Strengthening (EARS)
  - Atoms in solution do not have the radii conventionally used to predict strength
  - EARS radii act as better strength predictors
  - Shows that optimal properties are not correlated with maximum entropy



Center Proprietary – Terms of CANFSA Membership Agreement Apply

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

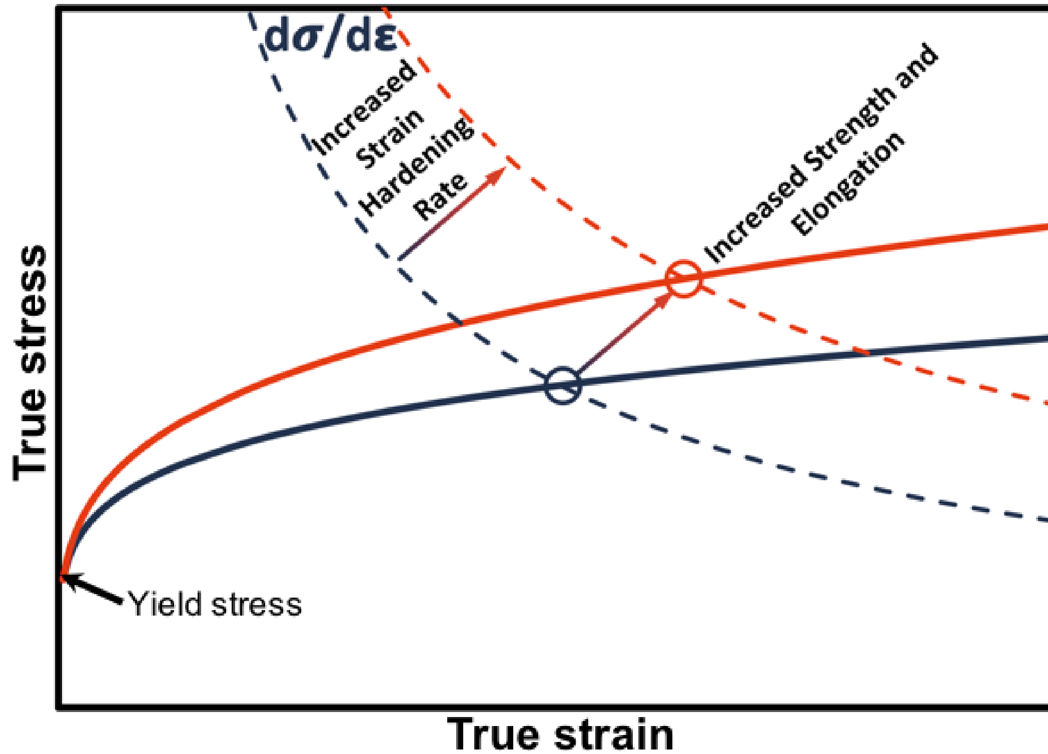
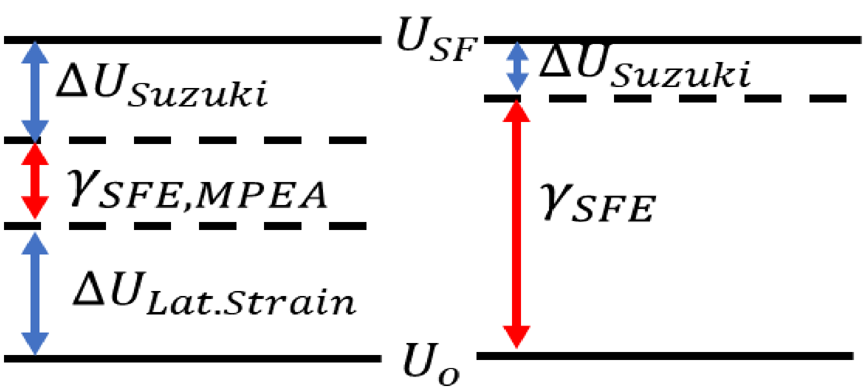
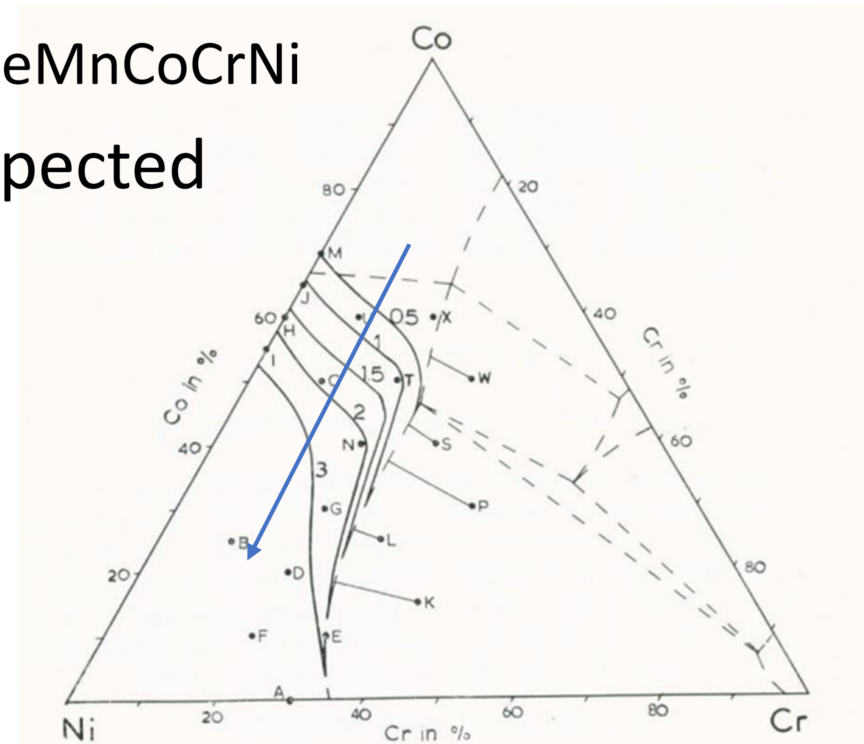


Figure courtesy of Dr. Kester Clarke

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



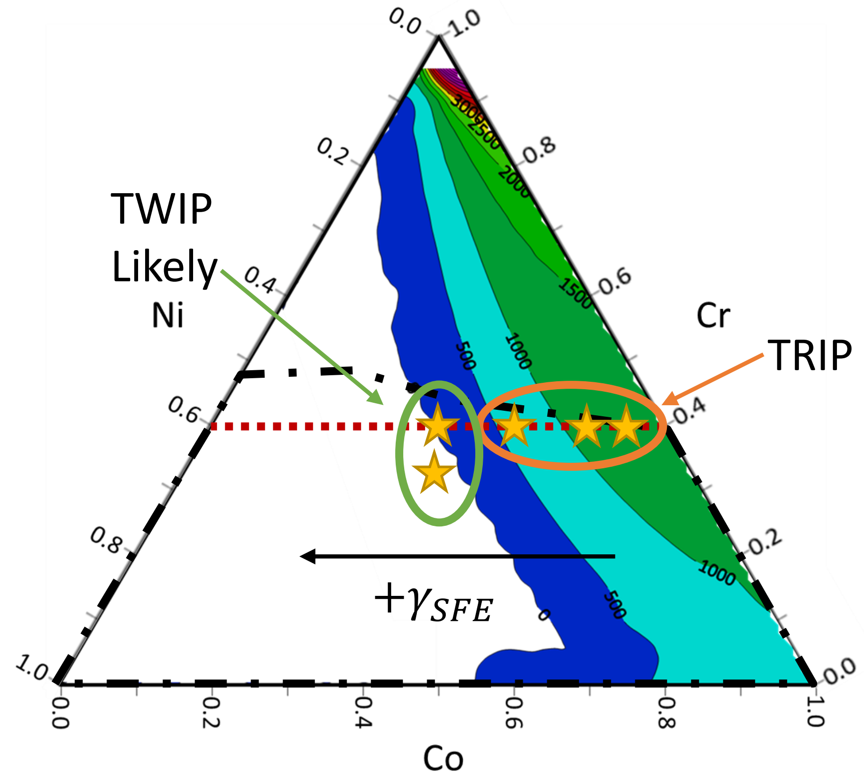


Isopleths in γ_{SFE} for CoCrNi Ternary Ranging from $9 * 10^{-7} - 5.6 * 10^{-6} J/cm^2$ (0.5-3Gb)

From: E.H. Koster et al. Stacking Fault Energies of Ni-Co-Cr Alloys, The Philosophical Magazine, Series 8, Vol 10 (1964)

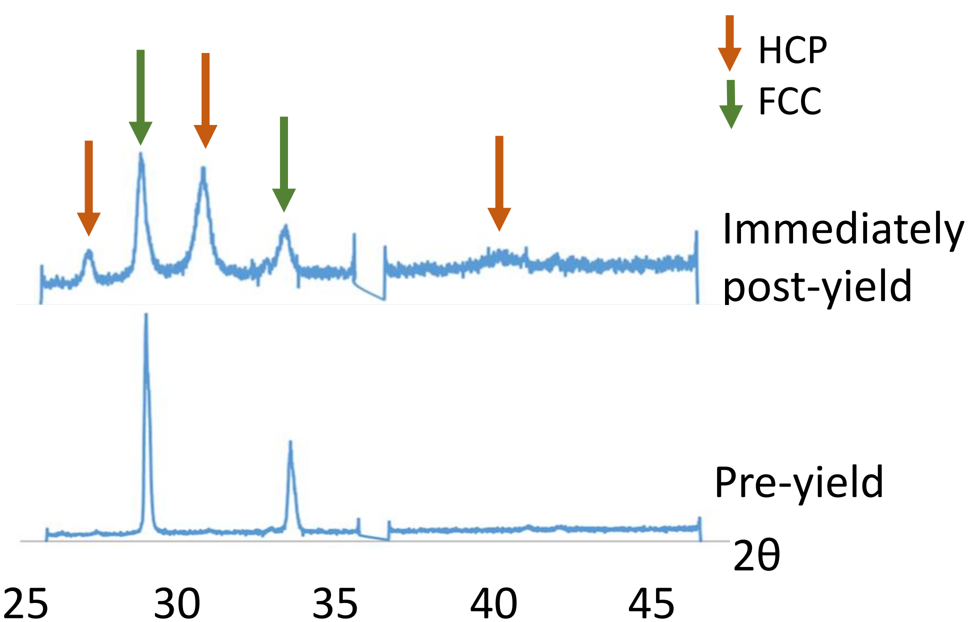
Alloy Design for TRIP and TWIP

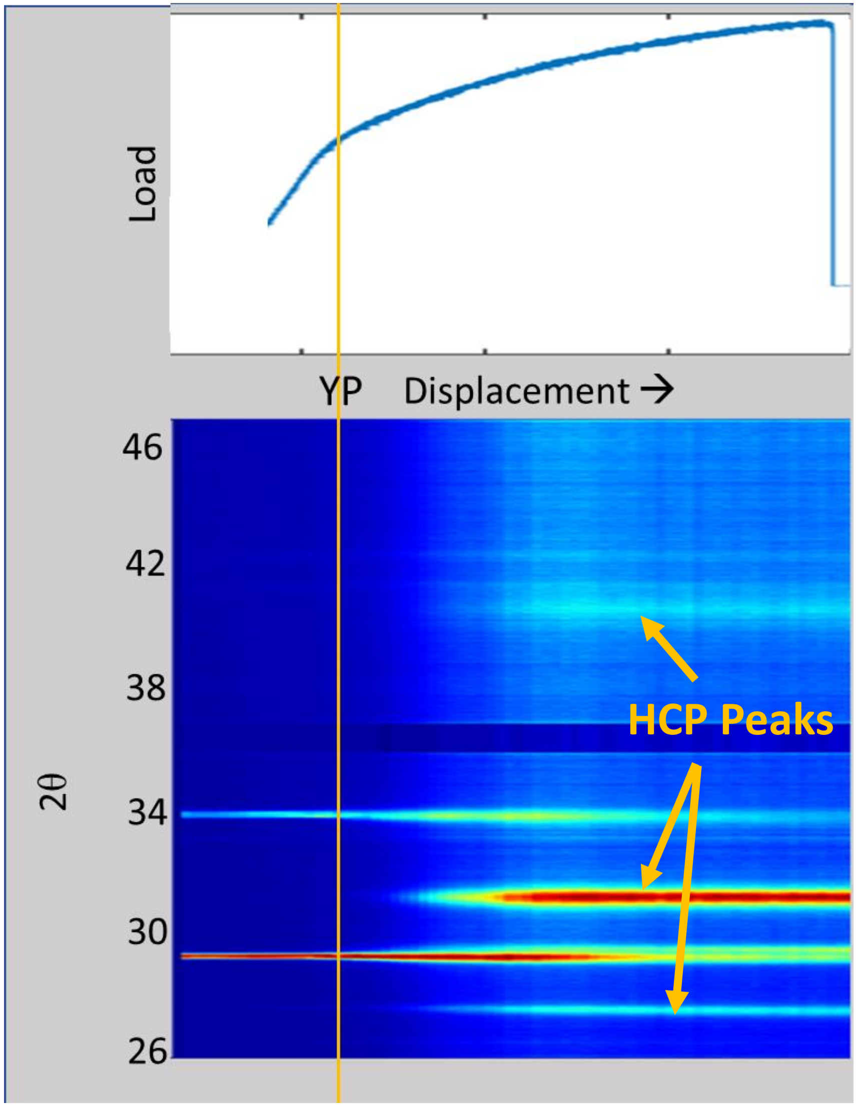
- T_0: The diffusionless transformation temperature
  - Free energy and composition of HCP and FCC phases are the same
  - High T_0 indicates the possibility of TRIP behavior at elevated temperatures
- Alloy Selection
  - High chromium alloys for higher T_0 values→ possibility of TRIP at high temperatures
    - Constrained by FCC single phase region (at 1100°C, black dashed line)
      - Surrounded by σ-phase
  - Alloys for study have been selected along $Co_xCr_{40}Ni_{60-x}$ (dotted red line)
    - Values of X=0.55, 0.50, 0.40, 0.30



Project Review: Evidence of TRIP in $Co_{55}Cr_{40}Ni_5$ at 60°C

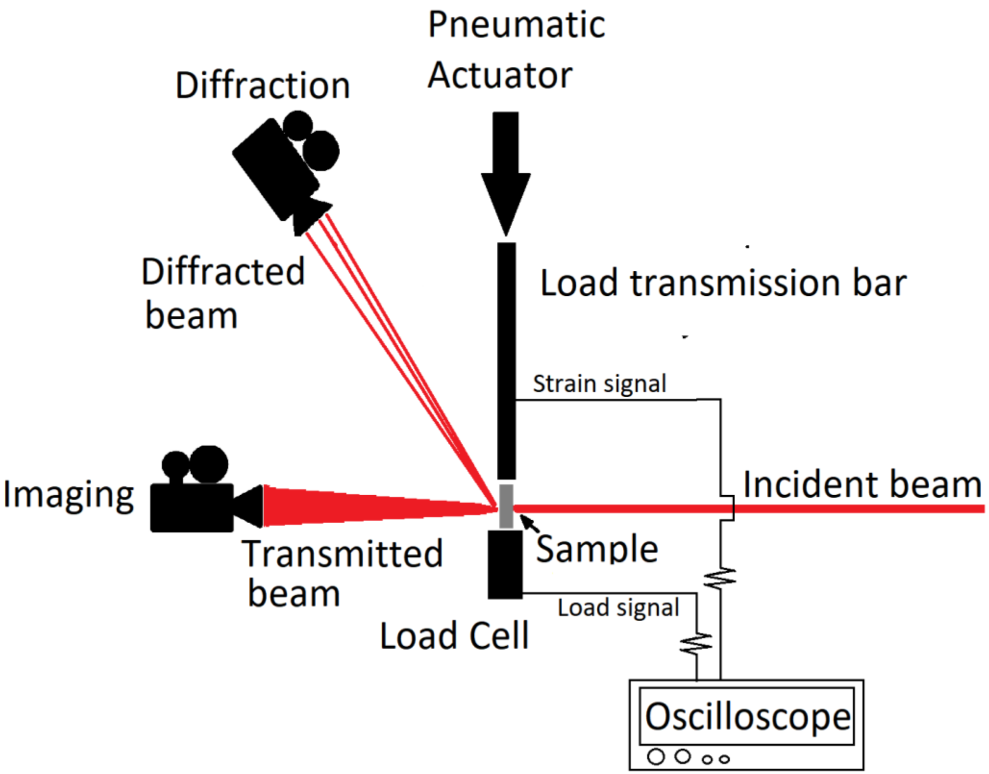
- Low Temperature Deformation ($Co_{55}Cr_{40}Ni_5$):
  - Transformation from FCC→HCP as strain increases
  - Onset of transformation at or near yielding
  - Similar behavior from -100°C to 450°C





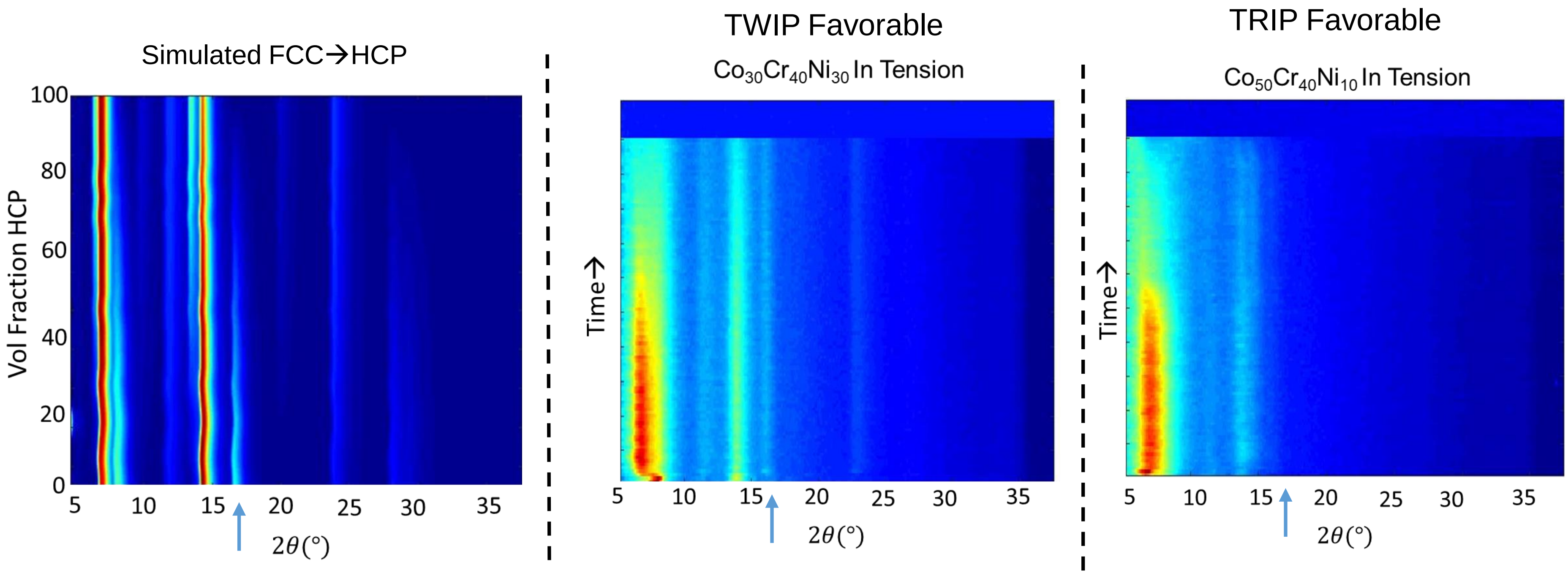
In-situ Dynamic Testing at APS

- In-situ Kolsky Bar, X-ray imaging and diffraction experiments
  - $\dot{\varepsilon} \sim 10^3\ s^{-1}$
- 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



Thanks to Ben Ellyson for the figure

Project Review: APS Simulation vs Experimental Results



- No apparent change in FCC (200) 1st Harmonic peak intensity
  - Expected for Co30
  - Peak is difficult to discern in Co50, but transformation may have occurred
    - High intensity from 2nd harmonic washes out 1st harmonic peaks

Project Review: Sample Geometry Effects

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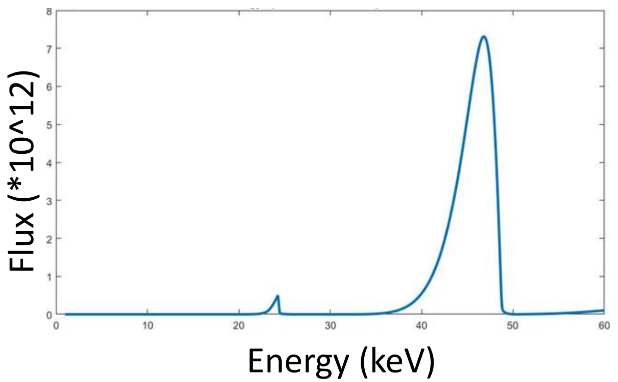
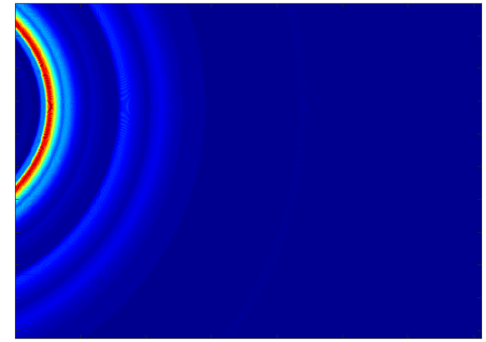
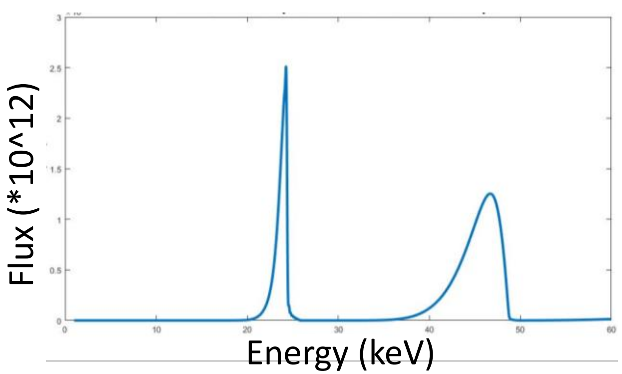
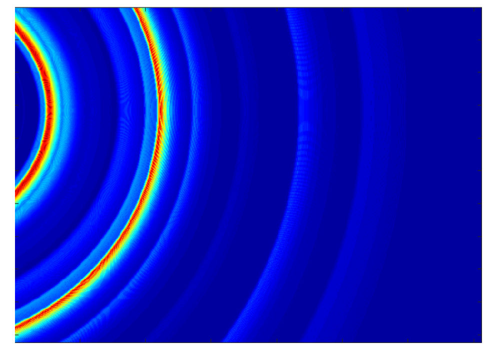
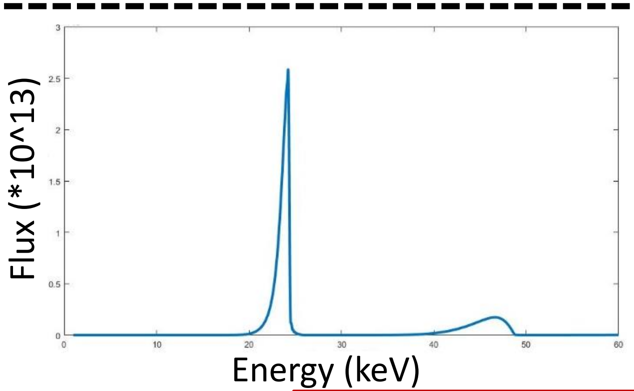
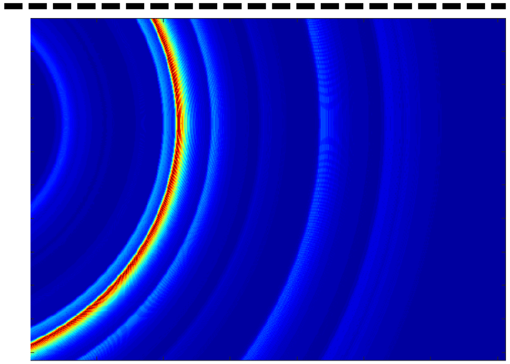
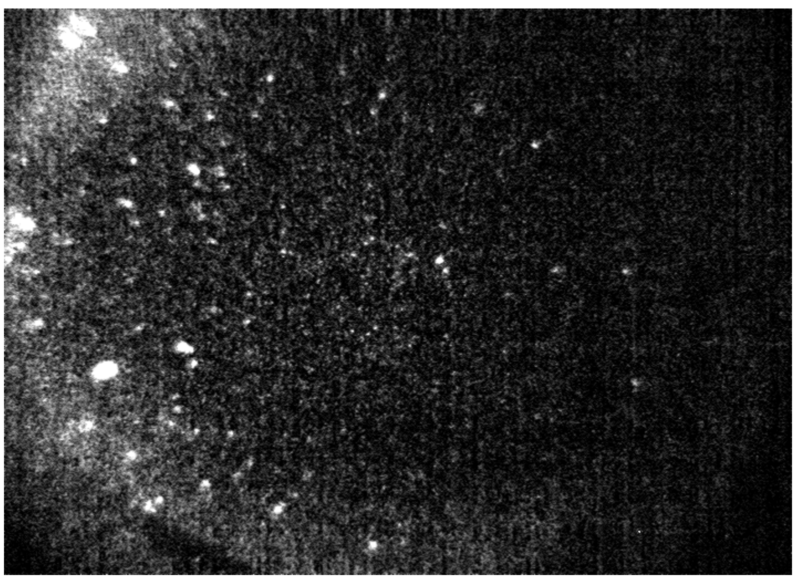
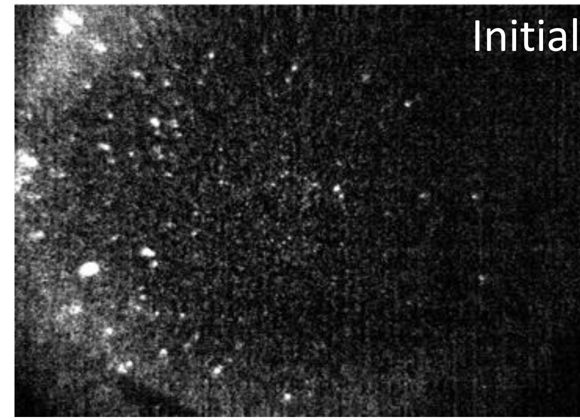
Thickness	Modified Energy Spectrum	Ratio of $I_{1st}:I_{2nd}$	
$500\mu m$		1:10 Tilting of the sample improved ratio by having some thinner sections	
$250\mu m$		2:1	
$100\mu m$		10:1	

Project Review: Twinning Seen by Domain Refinement

- Evolution of full rings
  - Crystallite refinement → twinning → TWIP
  - Seen in all alloys and strain rates tested at APS



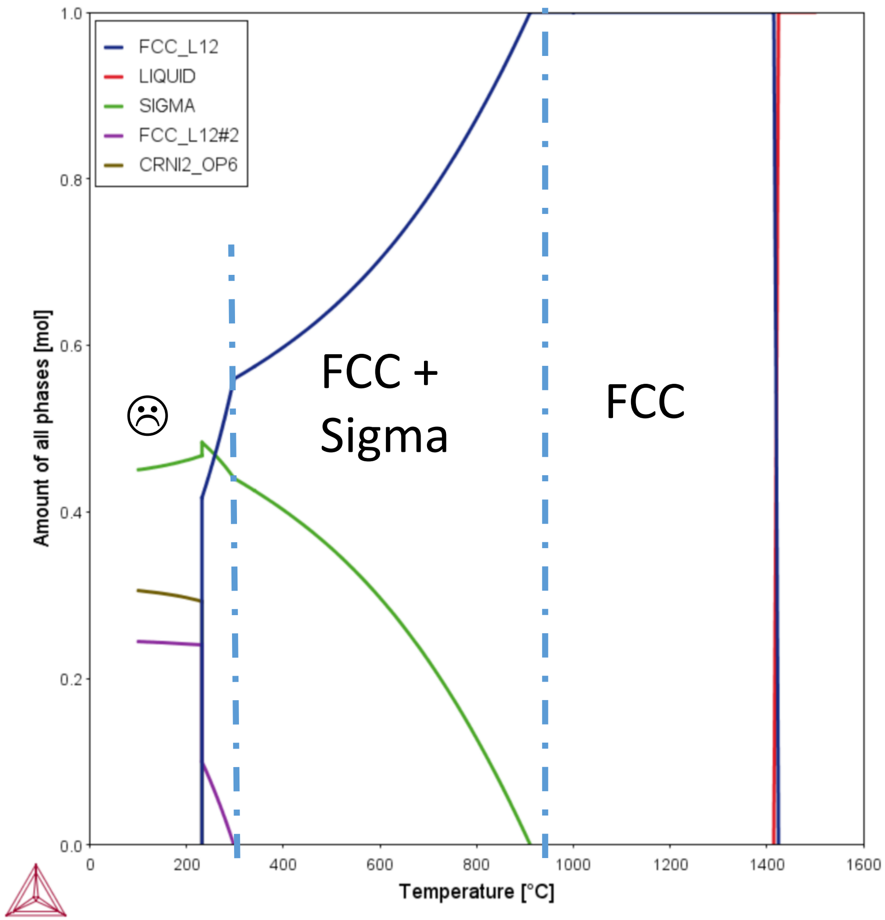


Recent Work

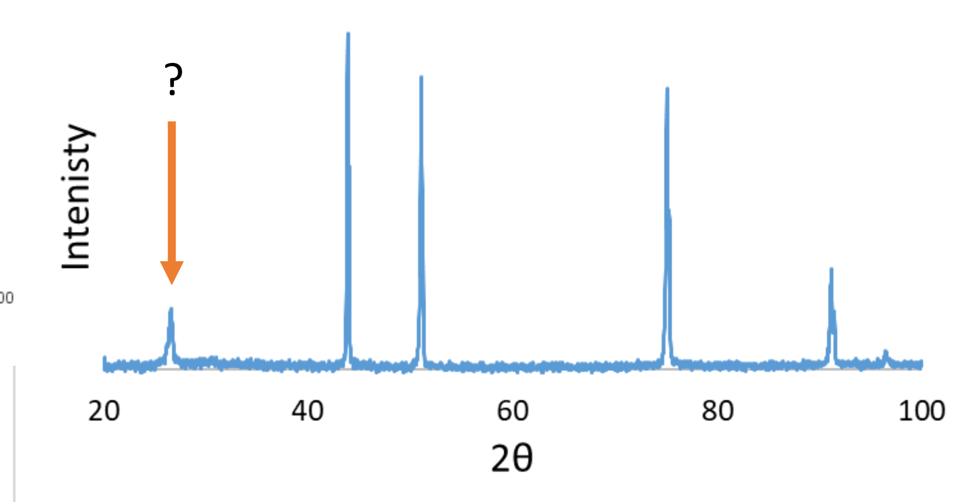
- Making material → making material into samples
  - Cast material (from Dr. Coury Co55, Co40)
  - Arc melted material (all other alloys)
    - Needs to be rolled
    - 50g limit

- Recent Findings
  - Cr_2O_3 particles in cast material
  - Multi-phase microstructures in low Co content alloys

Multi-Phase Buttons ☹

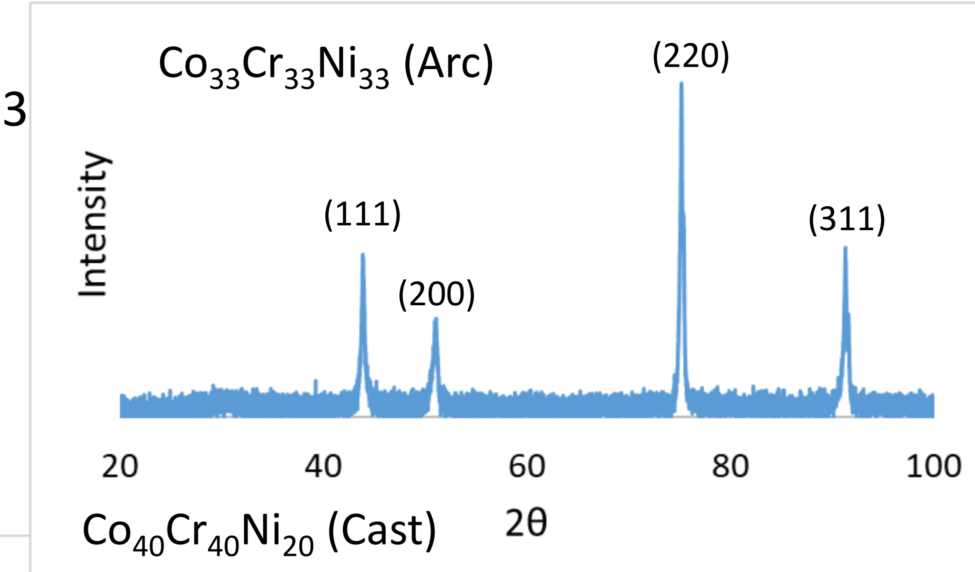


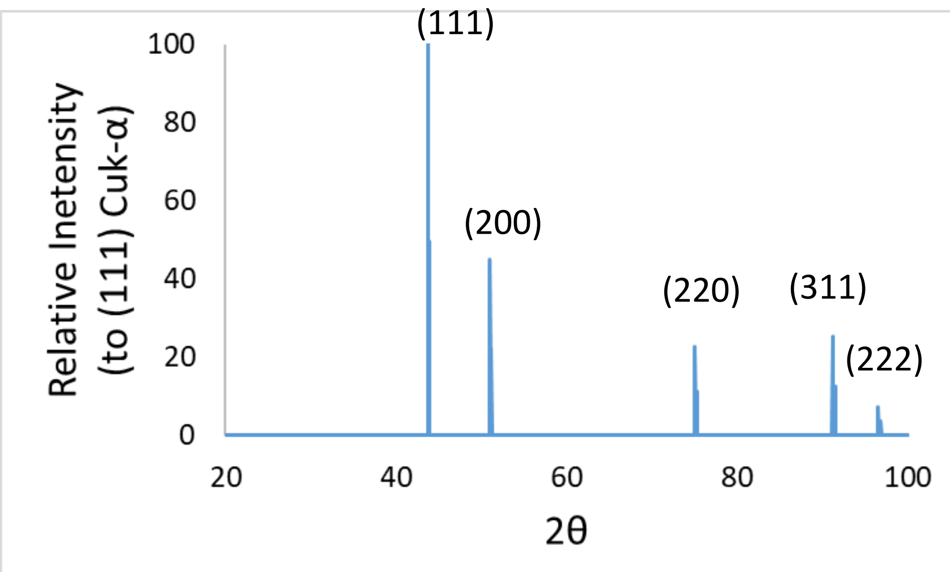
- Preparation:
  - Cold rolling to 75%
  - Furnace 1150°C for 2 hours
    - Needed to prevent σ-phase
  - Polish → XRD
- Observed for Co30, CoCrNi
- Buttons did not fully transform back to FCC

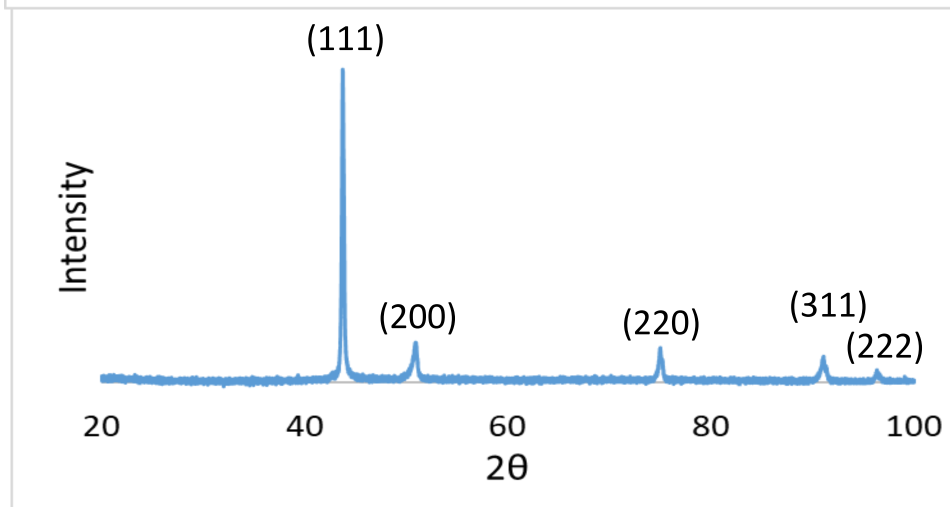


Single Phase Achieved

- 2 hours @ 1200°C
  - Additional heat treatment for Co33
  - Initial heat treat for Co40
- Both appear textured
  - Co33→Rolling ≈ 70%
  - Co40,55→Rolled only 20%, but as cast

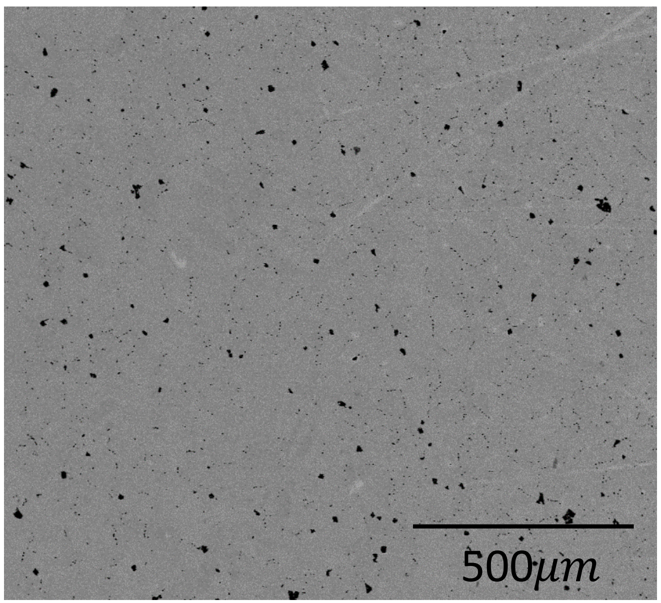


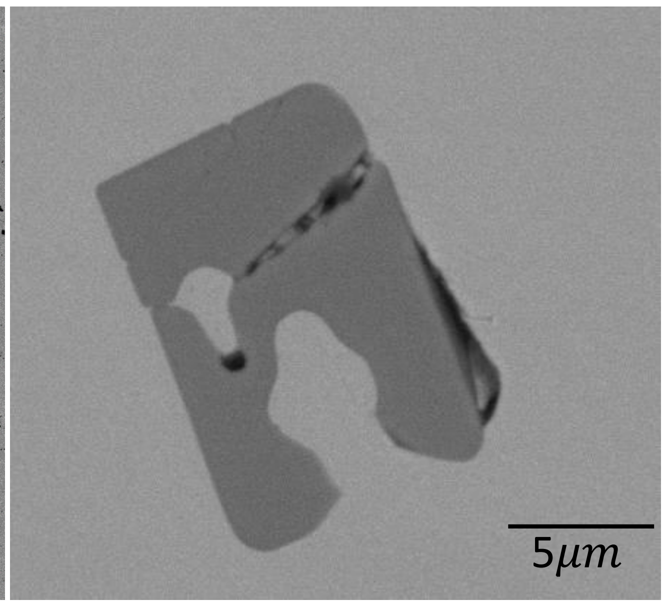


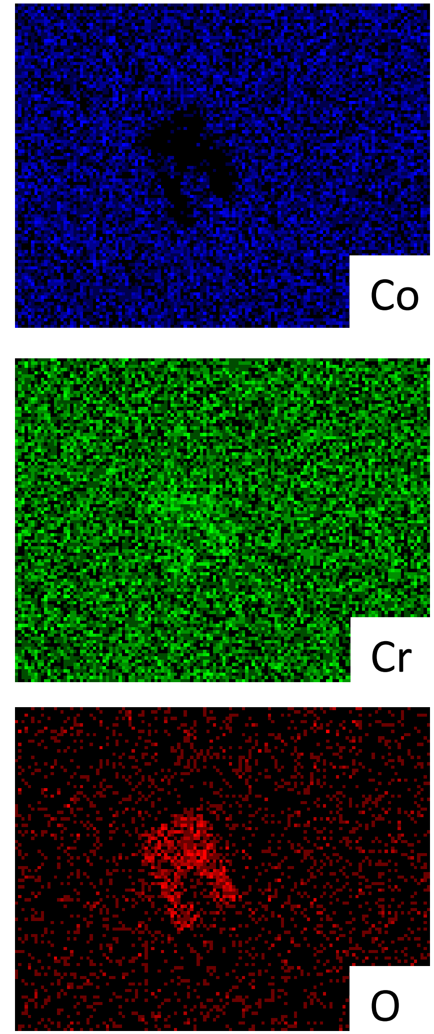


Chromium Oxides in Cast Material

- Co40 and Co55 ingots have Cr rich oxides
  - Potentially detrimental to mechanical properties
    - Reduced rolling tolerance from 75% to 25%

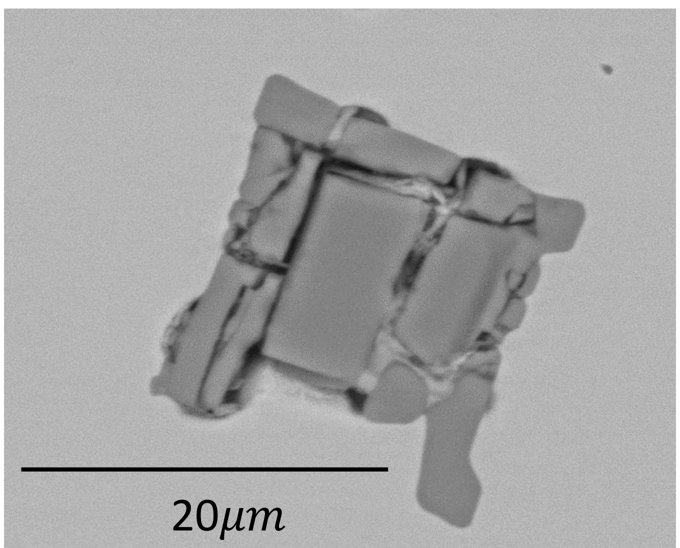


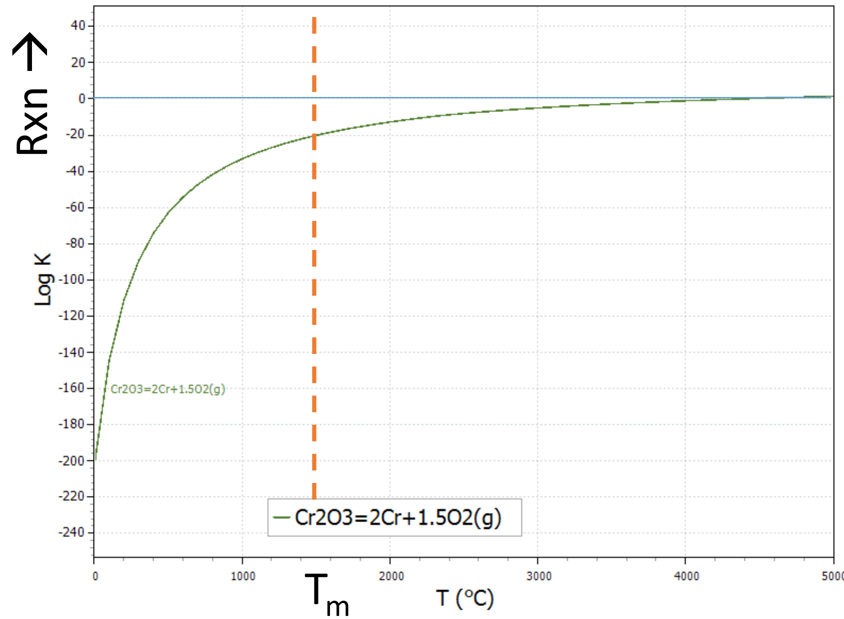




Removing Oxides?

- $2Cr + 3/2 O_2 \rightarrow Cr_2O_3$
  - Favorable beyond melting point of alloy—in air
  - Cannot be dissolved
- May be able to re-melt in vacuum to drive reaction leftwards





Plot of reaction constant vs temperature for the dissociation of Cr_2O_3 (green). The blue line shows the point at which the reaction becomes favorable, the orange dashed line shows the approximate melting point of the alloy.

TRIP and TWIP Observed in CoCrNi

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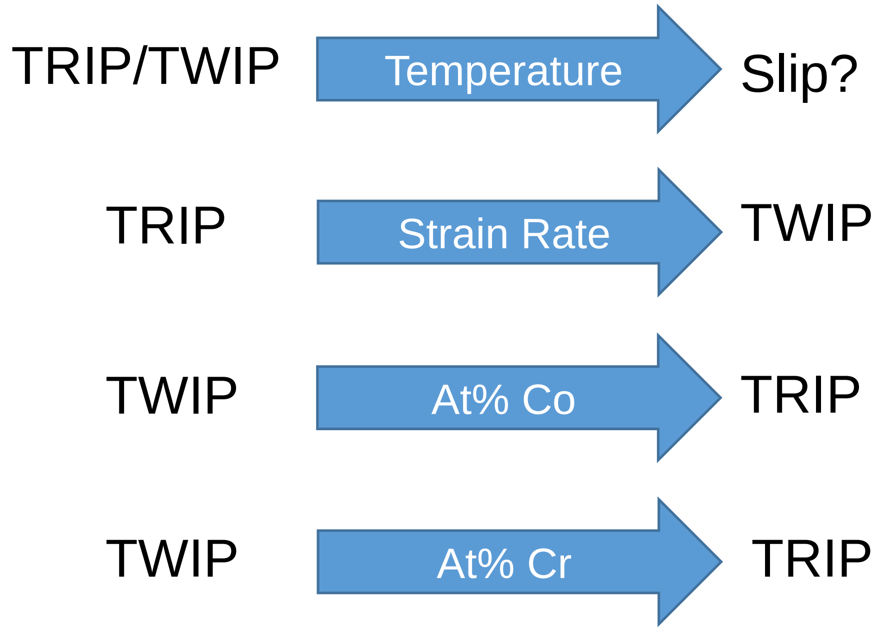
- TRIP behavior seen:
  - At low strain rates $(10^{-2}s^{-1})$
  - Temperatures from -100°C to 450°C
  - In $Co_{55}Cr_{40}Ni_5$, predicted to TRIP by Thermo-Calc modelling
- TWIP behavior seen:
  - At high strain rates $(10^{3}s^{-1})$
  - Across a range of CoCrNi alloys



Alloy composition in CoCrNi can be controlled to activate specific deformation mechanisms given knowledge of use conditions.

Continuing Work

- Developing methods of material processing
  - Aiming for single phase, fine-grained material for mechanical testing
- Upcoming testing at CHESS and APS (Early 2020)
  - For APS new sample geometries and improved heat treatment methods have been designed
- Mechanical testing at Mines
  - Strain rates $10^{-2} - 10^{2} s^{-1}$
  - Interrupted testing
  - Post-mortem XRD and EBSD to observe microstructural evolution

Progress

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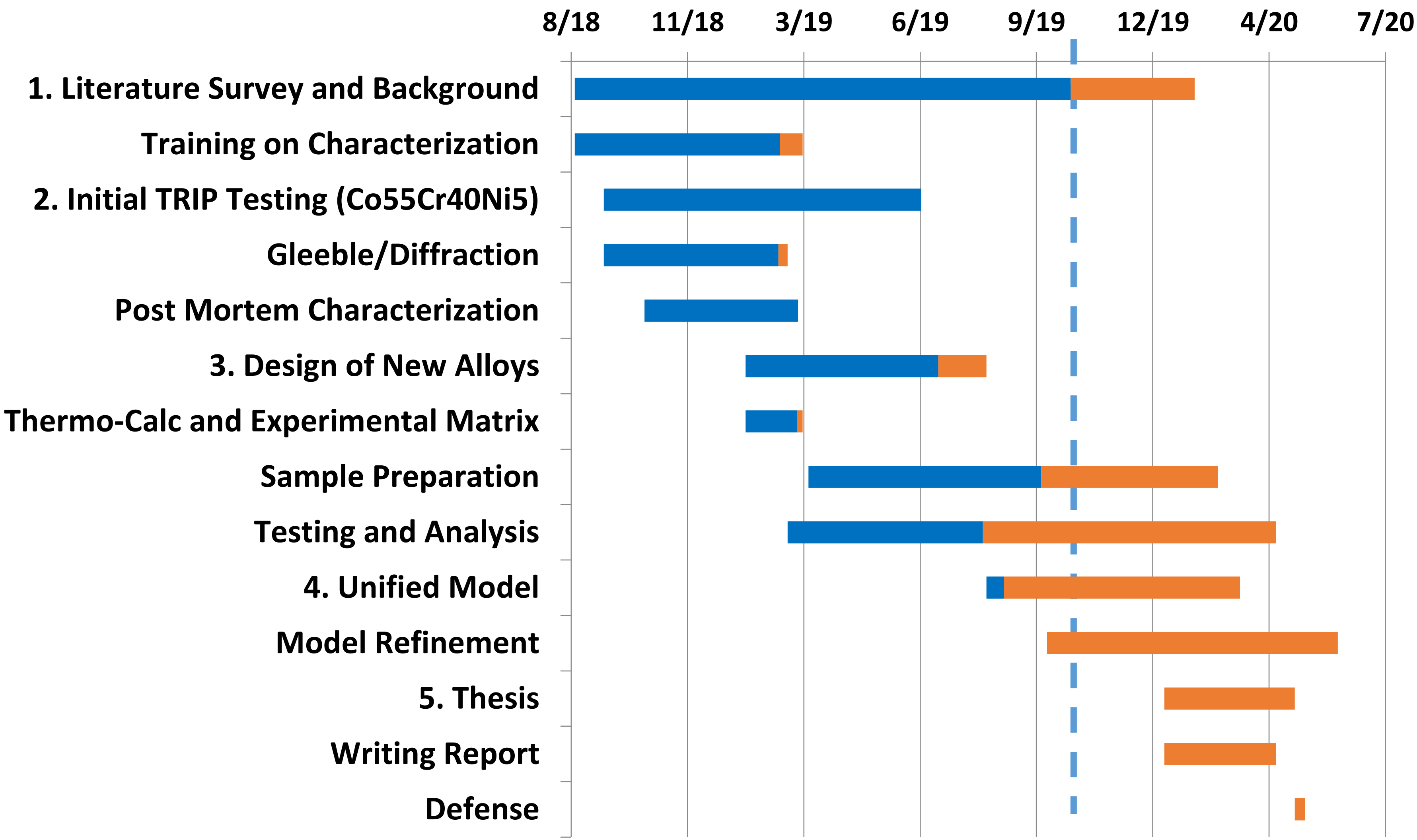
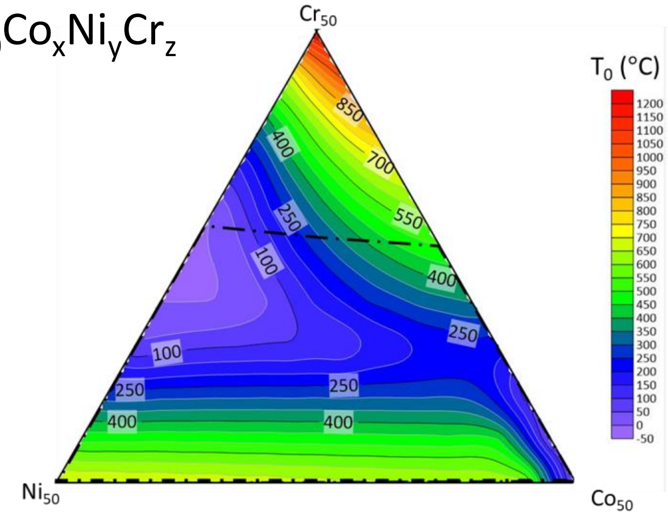


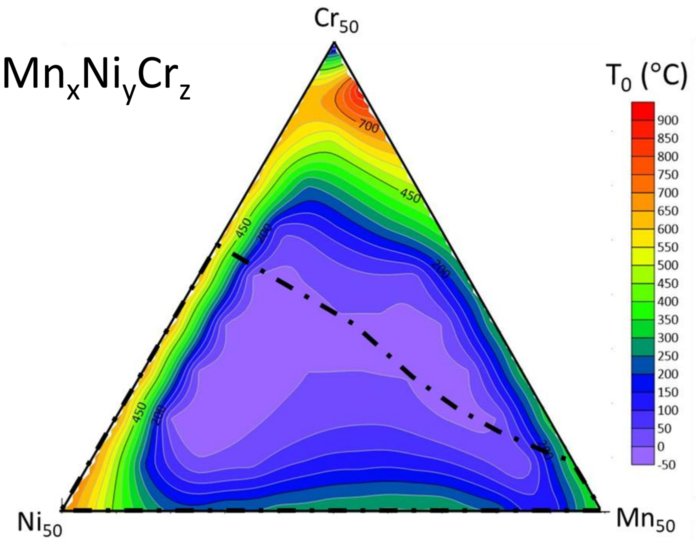
Challenges and Opportunities

- Challenges:
  - Making material is difficult
    - Arc melted buttons require rolling → texture evolution
    - Spray forming → Porous material
    - Casting → large Cr-oxide inclusions
- Opportunities
  - Understanding of designing TRIP and TWIP into CoCrNi can be extended to other alloy systems

$Fe_{50}Co_xNi_yCr_z$



$Fe_{50}Mn_xNi_yCr_z$



Thank you!

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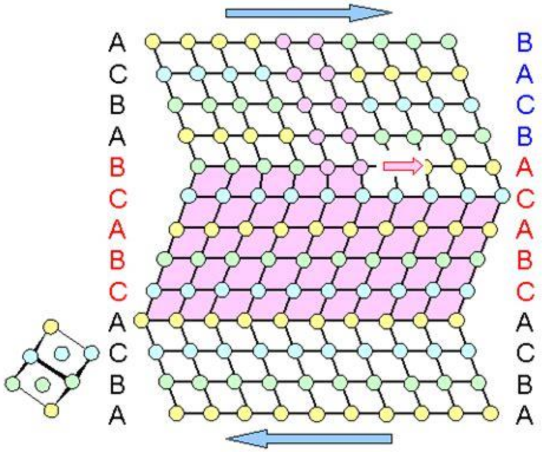


Goals

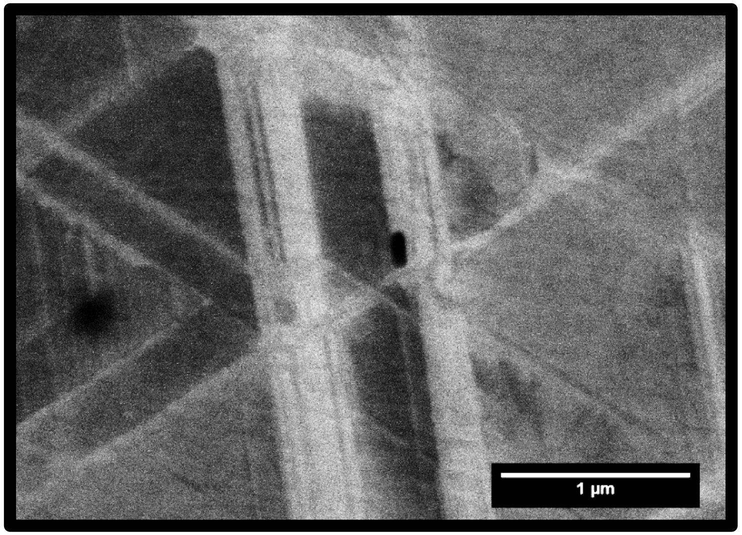
- Modeling of deformation behavior as it pertains to microstructural changes in MPEAs

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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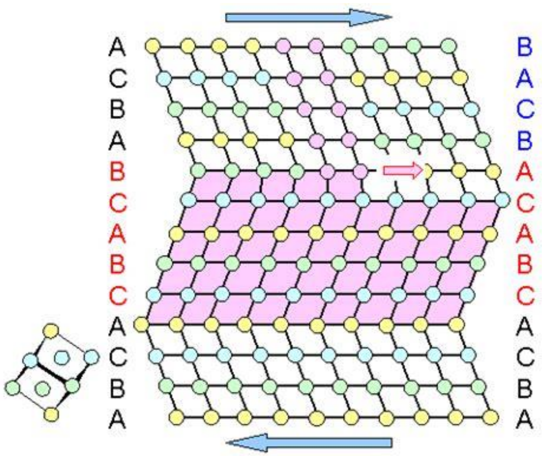

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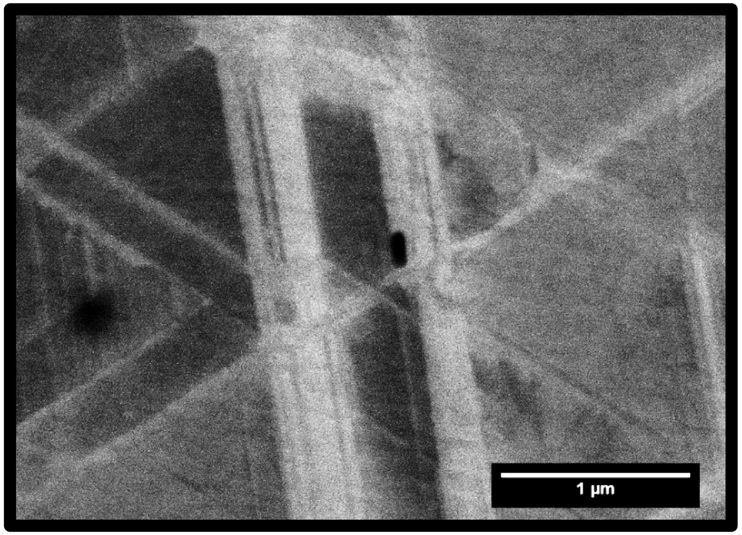

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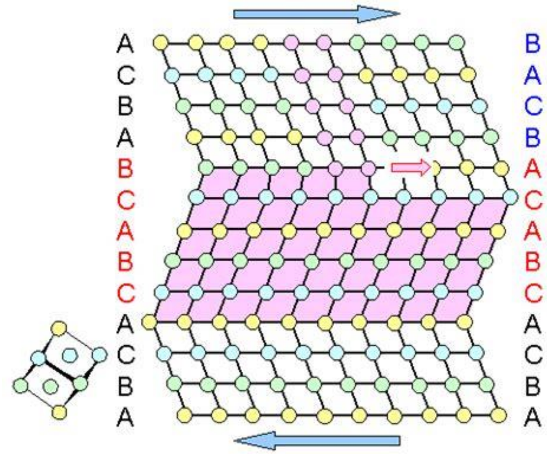
Project Duration: *Sept. 2018 – May 2020*

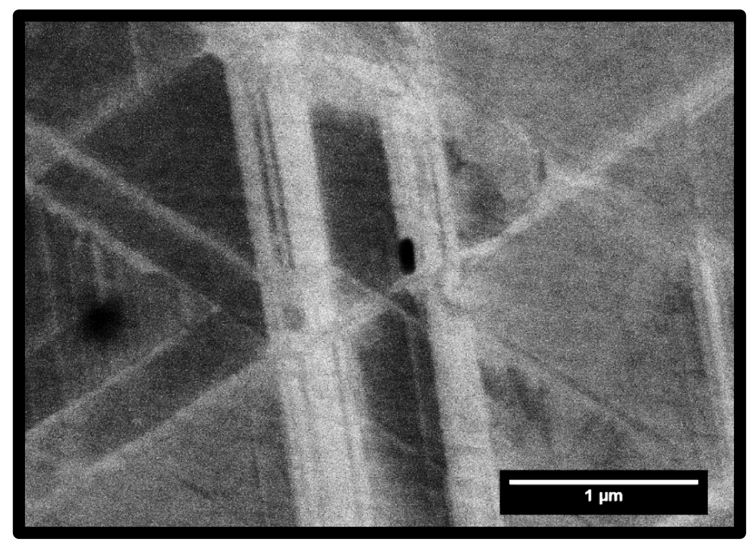


Program Goal

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

Approach

- 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



Benefits

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