

# Effective Exploration of New 760°C Capability Steels for Coal Energy

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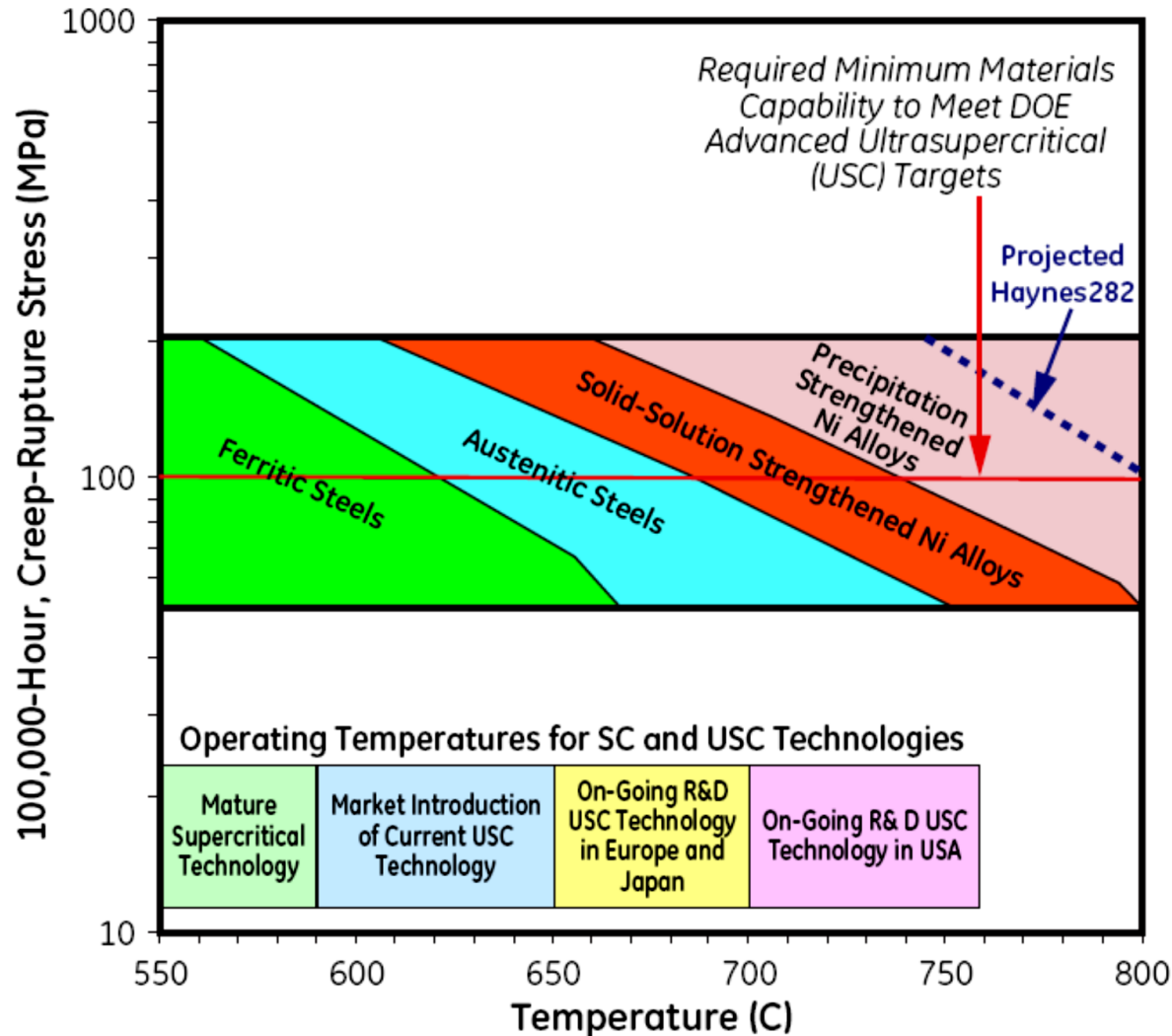


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May 19-23, 2014 in Pittsburgh, PA

# Outline

1. Background and Project Objectives
2. Technical Approach
3. Summary of Progress
4. Future Work

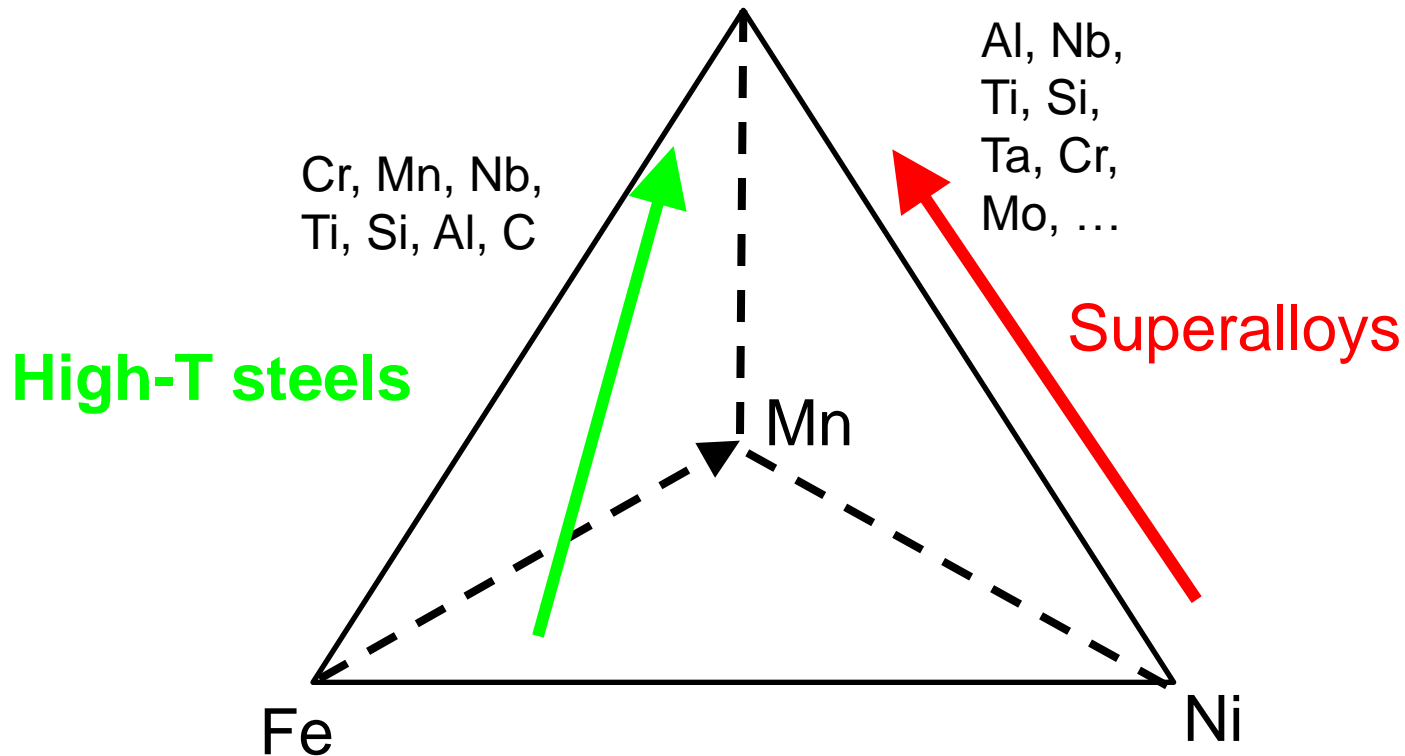
# 1. Background and Project Objectives



- Martensite strengthening no longer workable at 760 °C.
- New strengthening mechanism is sought.

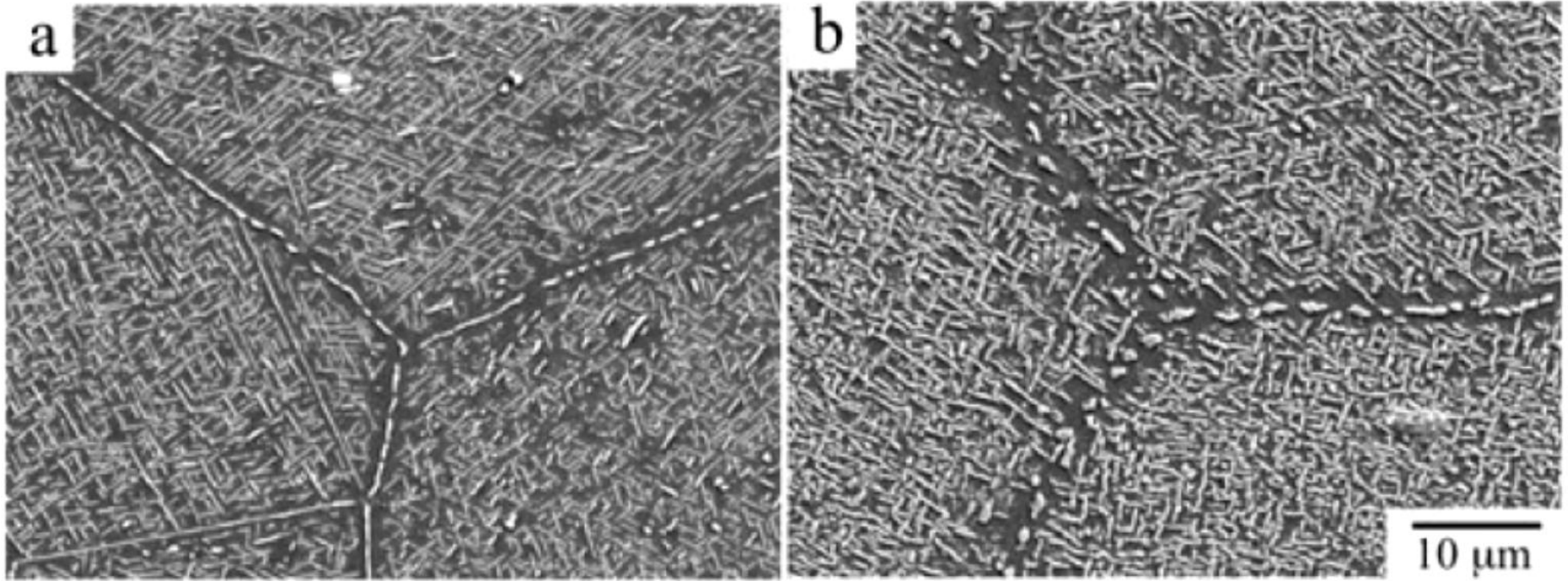
L. Jiang, 2011

# 1. Background and Project Objectives



- Identification of new strengthening phases through high-throughput exploration together with computational thermodynamics.
- Cost-effective steels for AUSC clean coal systems.

# 1. Background and Project Objectives

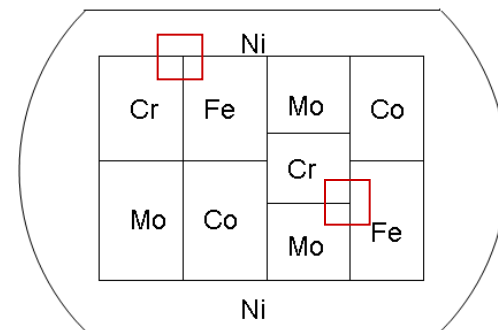
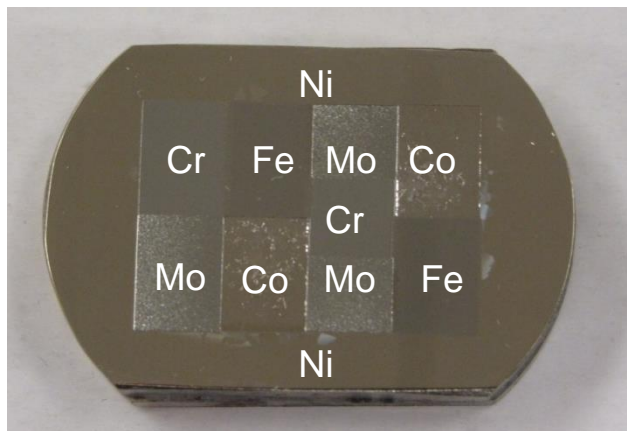
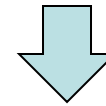
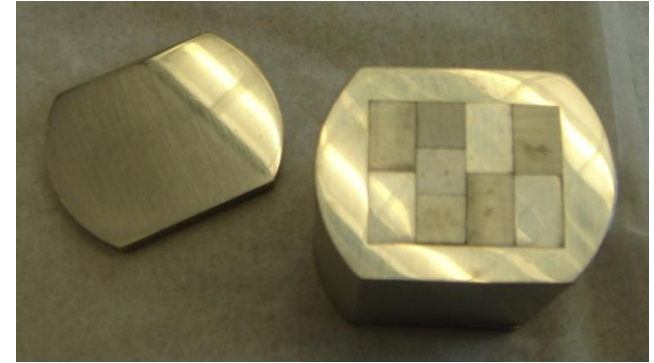
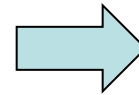
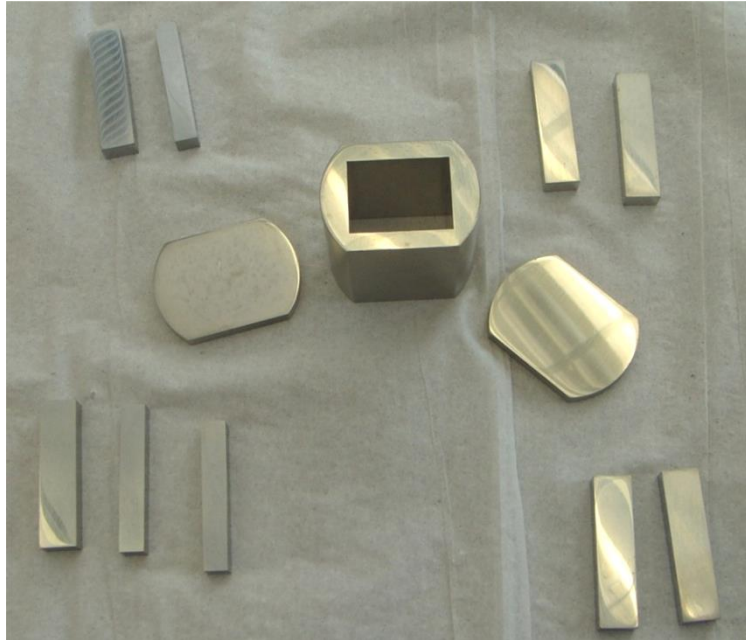


*Finely dispersed Laves phase in a Fe-20Cr-30Ni-2Nb (at.%) steel after a creep test at 700°C and 120 MPa: (a) boron-doped steel, and (b) boron-free steel (Takeyama et al.)*

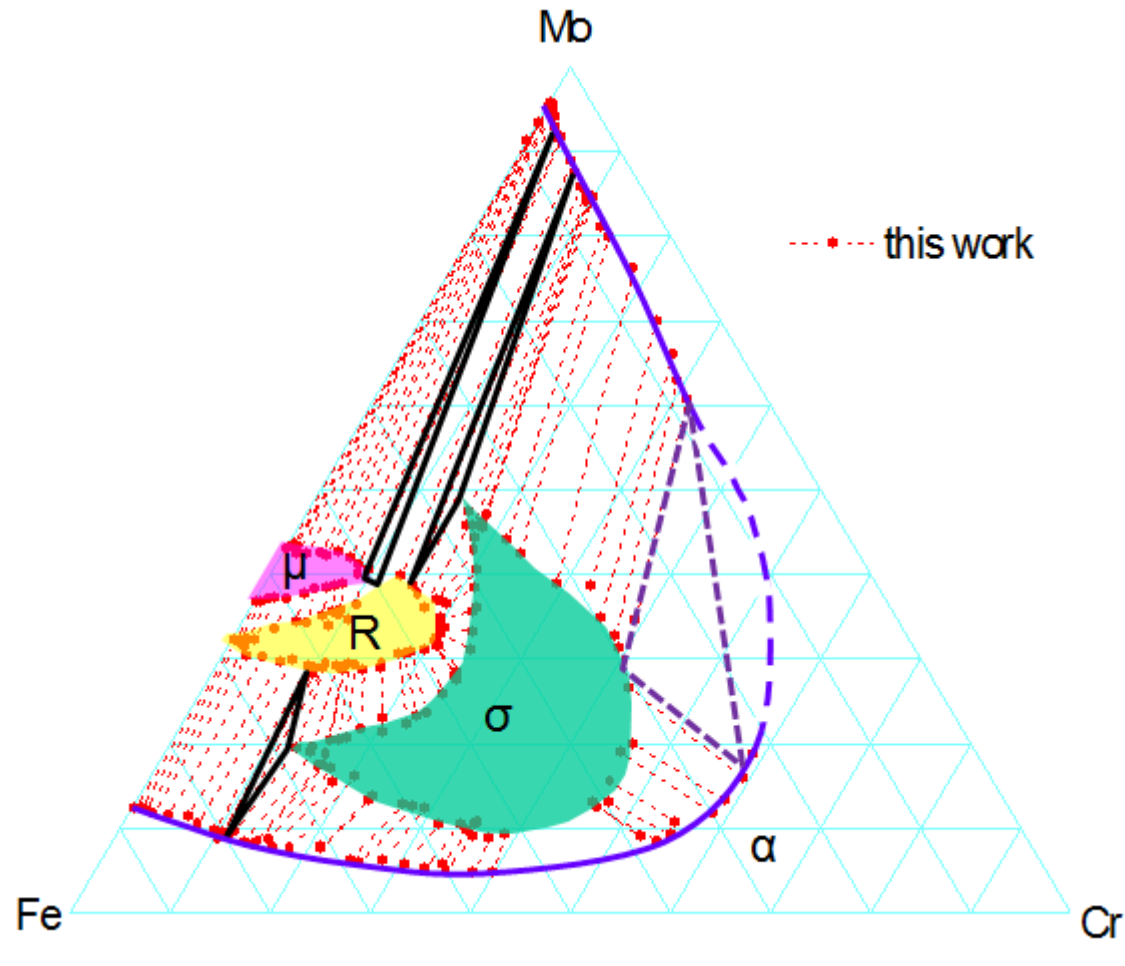
- Laves phase has demonstrated good properties.
- Sluggish precipitation kinetics.
- Grain boundary precipitates key to good creep strength.
- High enough Cr for hot corrosion resistance.

# 2. Technical Approach

## Diffusion-multiple approach

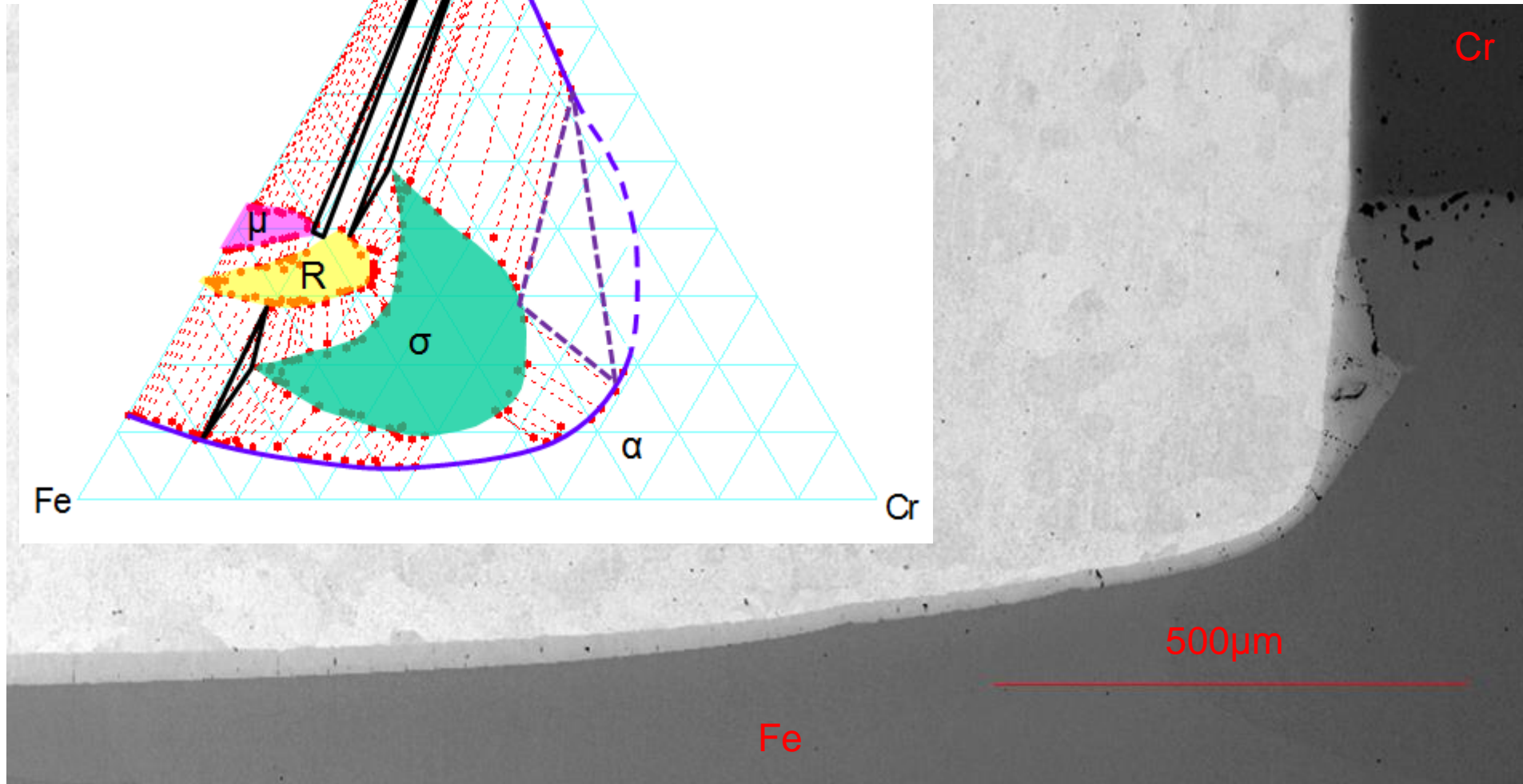


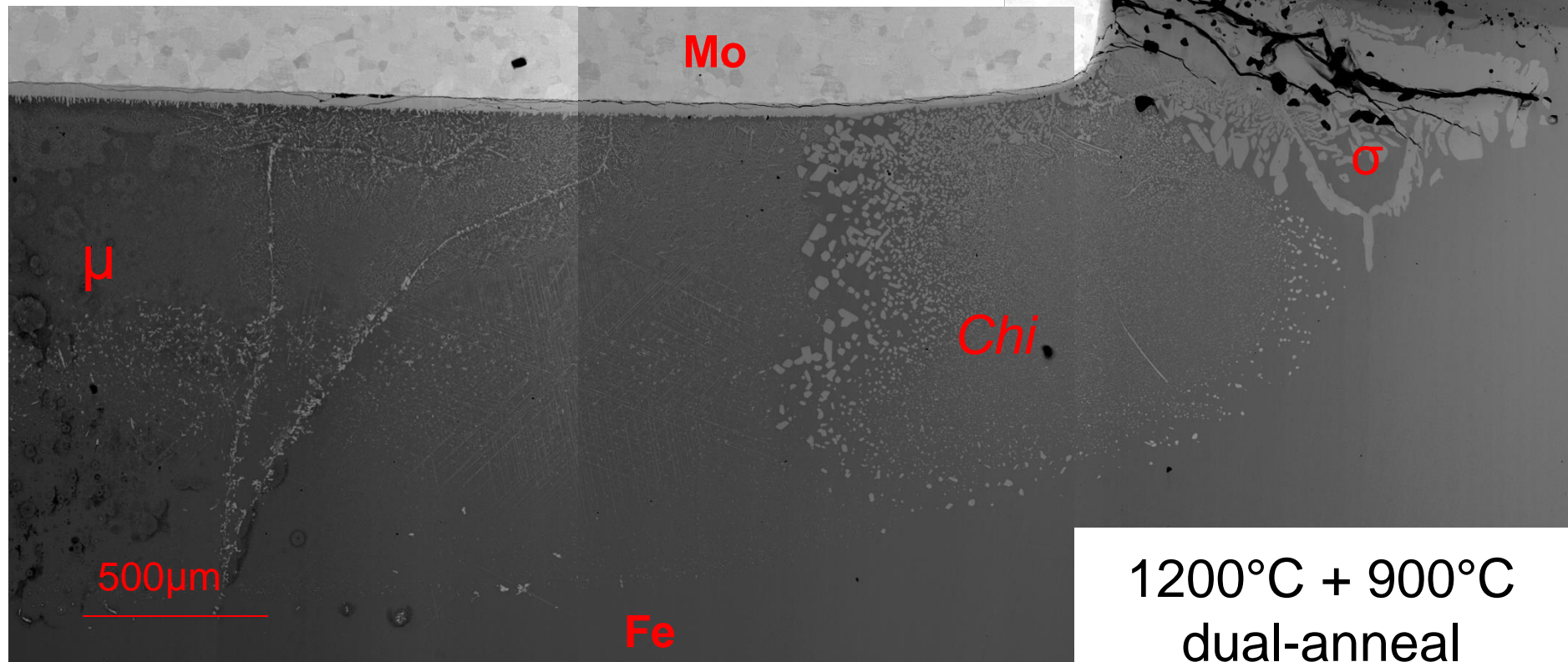
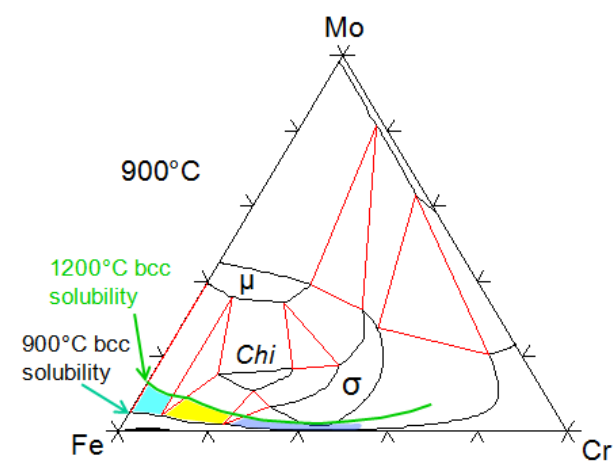
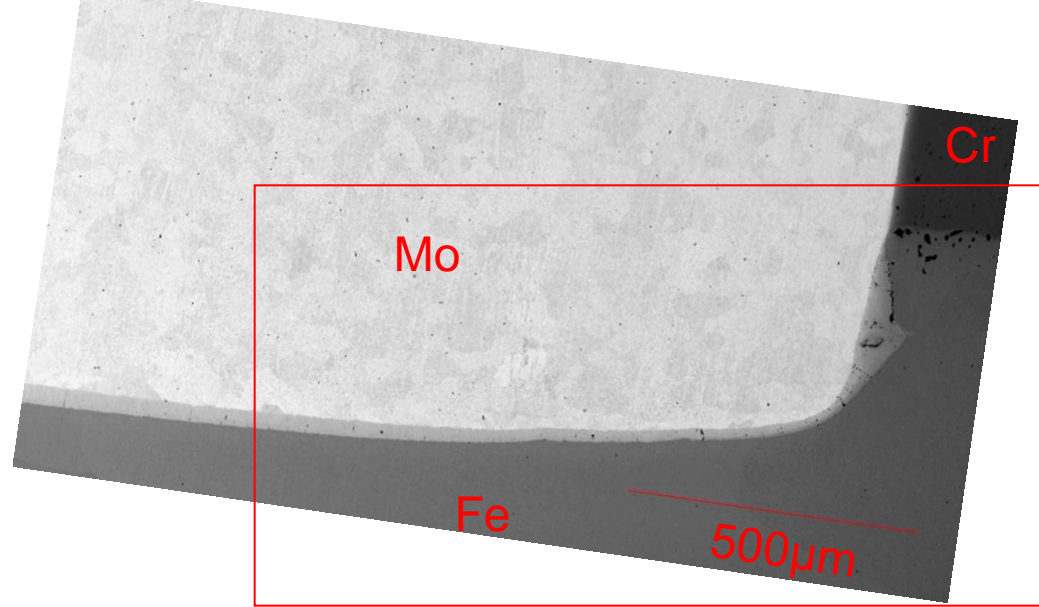
## 2. Technical Approach



al diffusion multiple

ngle-anneal Fe-Cr-Mo



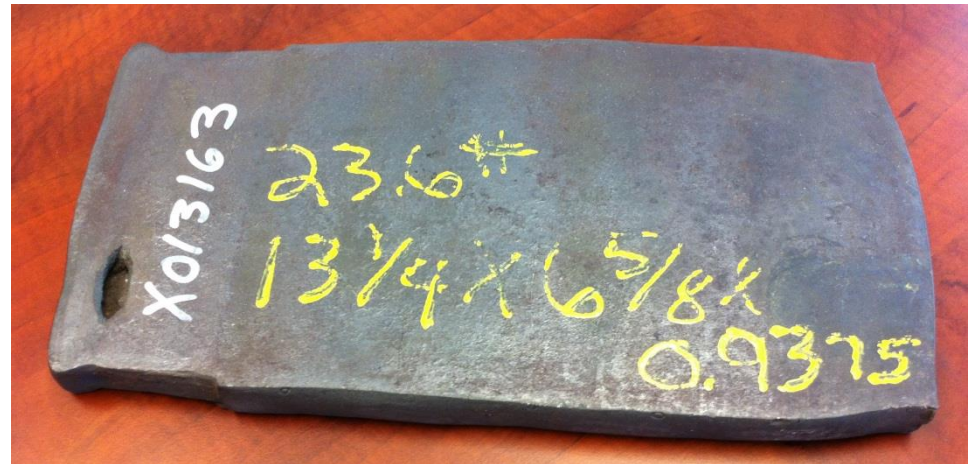
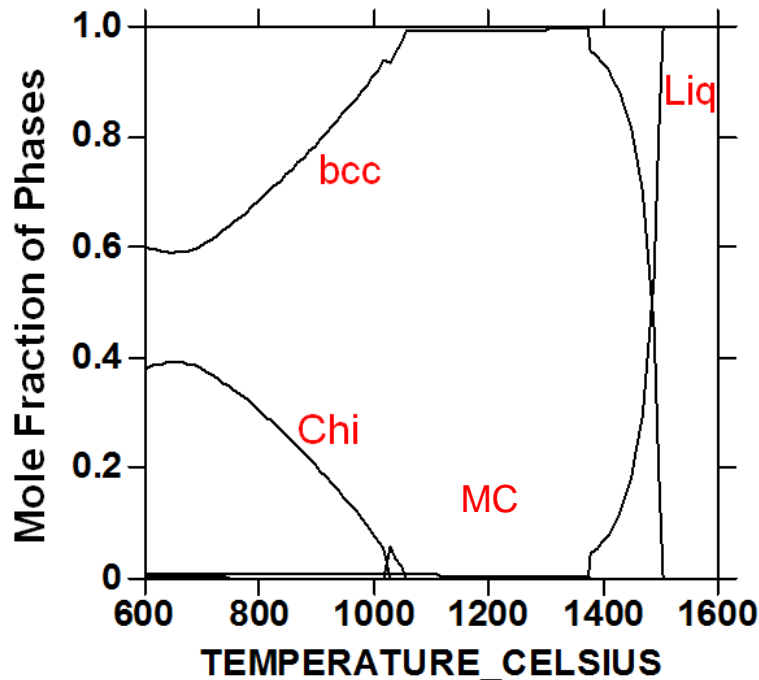


1200°C + 900°C  
dual-anneal



# 3. Summary of Progress

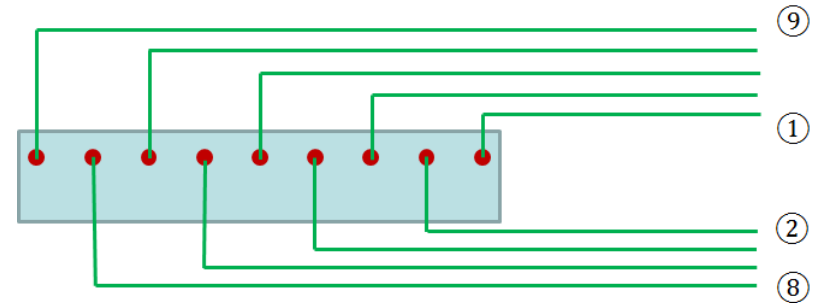
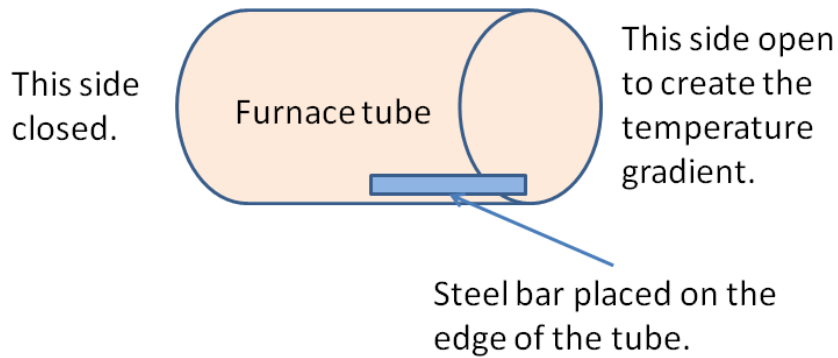
- Designed a Chi-phase strengthened steel using Thermo-Calc
- Induced MC carbide for grain boundary pinning during solution annealing
- Induction melted the alloy (24 lbs) and processed to 1" plate
- Performed systematic study of solution annealing and precipitation annealing
- Excellent oxidation resistance was observed.



- 24 lb cast ingot
- Hammer forged to 1" thick plate

# 3. Summary of Progress

## Gradient Temperature Heat Treatment to quickly find the optimum precipitation H/T

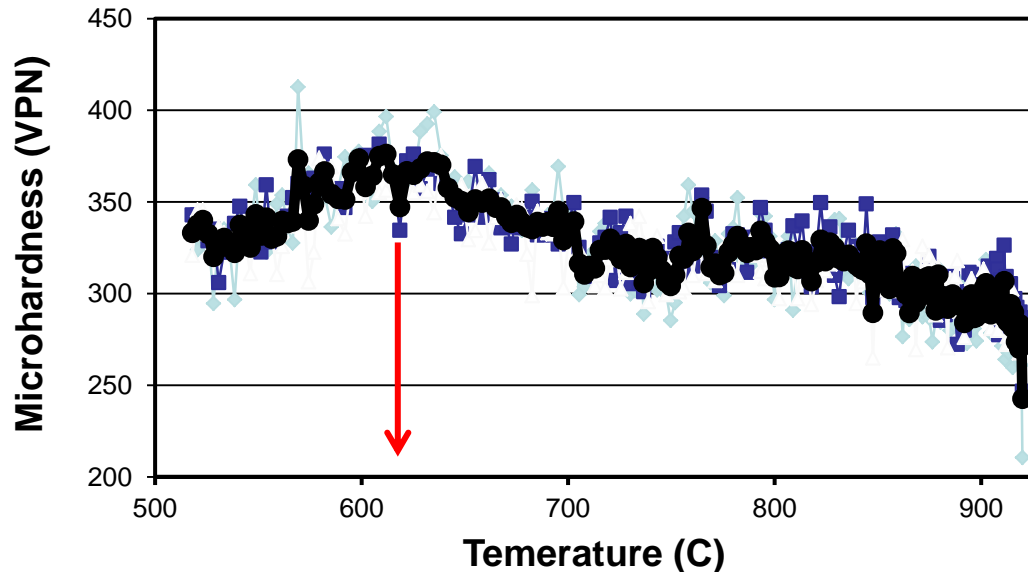
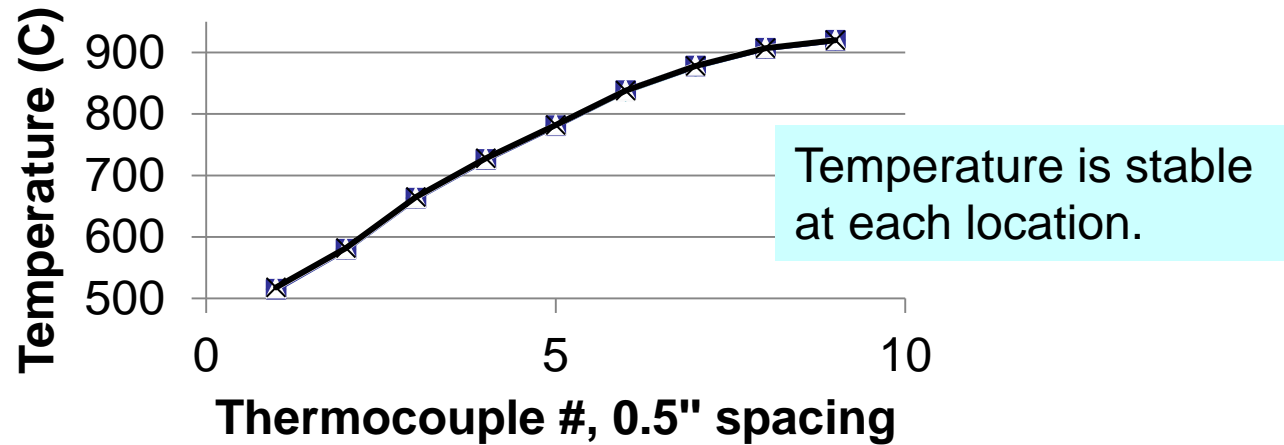


9 type-K thermocouples were attached to the steel bar at an equal spacing.

The steel bar was placed to an open end tube furnace at 1000°C (center) for 10 hrs.

# 3. Summary of Progress

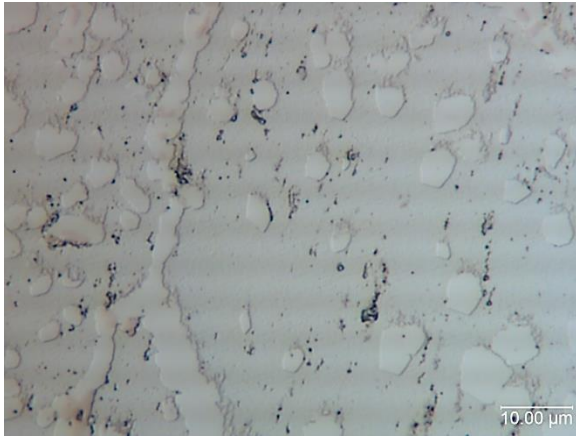
## Gradient Temperature H/T and microhardness



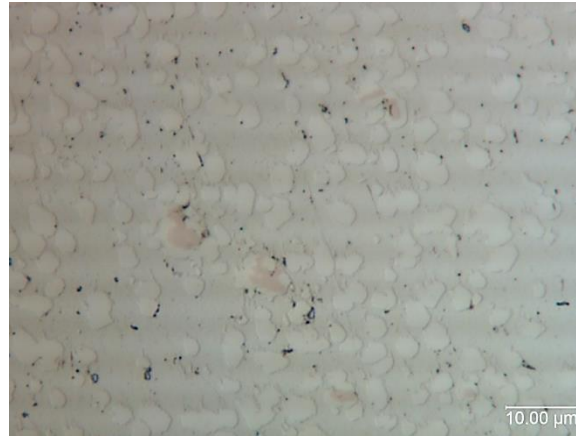
Microhardness as a function of T shows a peak hardness at ~620 °C.

# 3. Summary of Progress

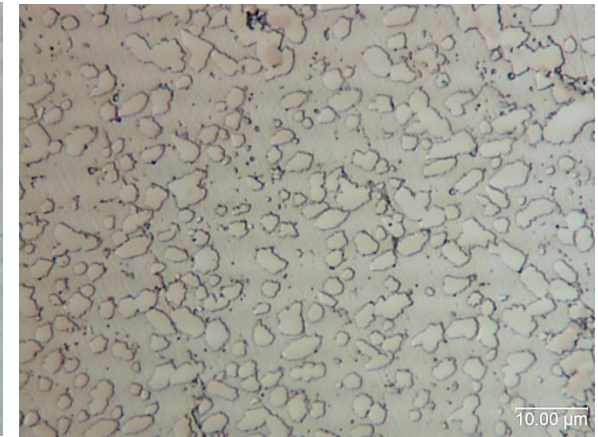
## Microstructure at different locations (T)



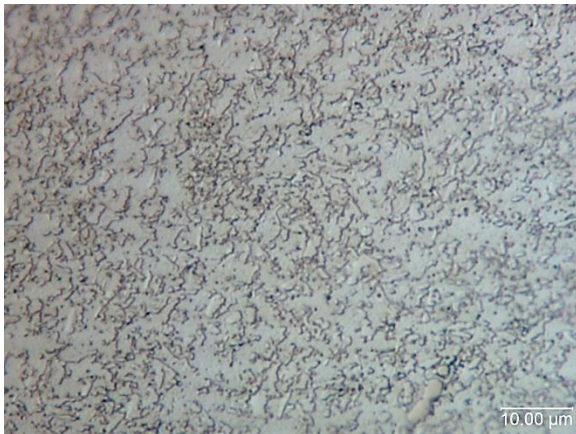
T = 907 °C



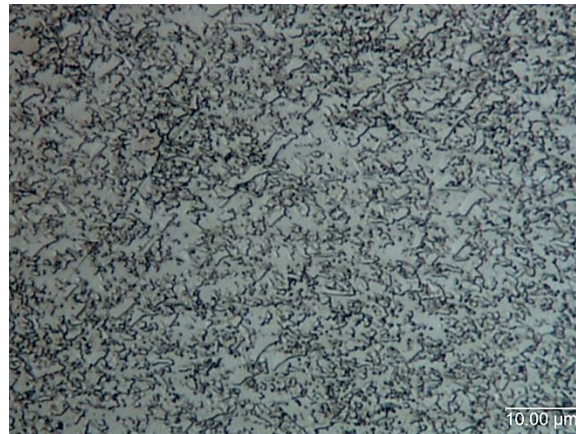
T = 878 °C



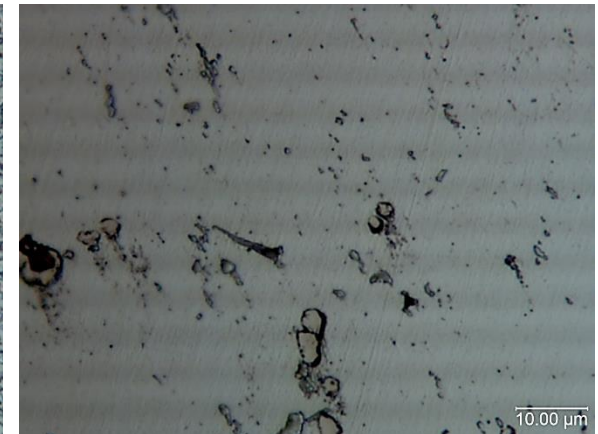
T = 838 °C



T = 728 °C



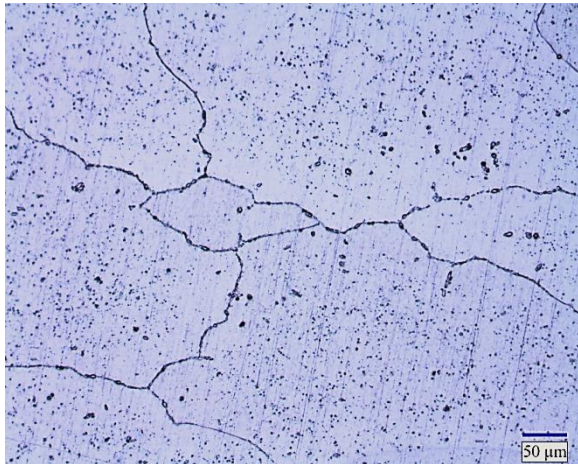
T = 665 °C



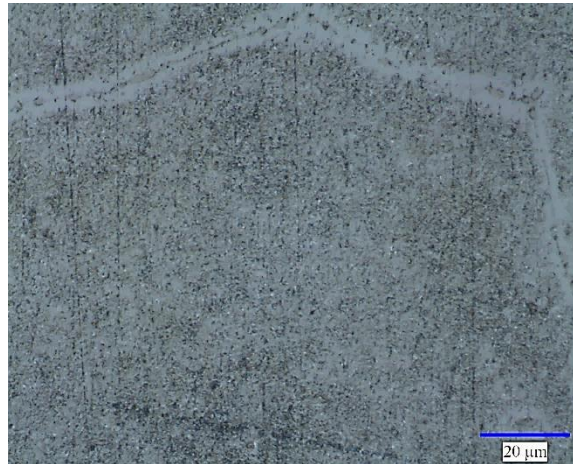
T = 582 °C

### 3. Summary of Progress

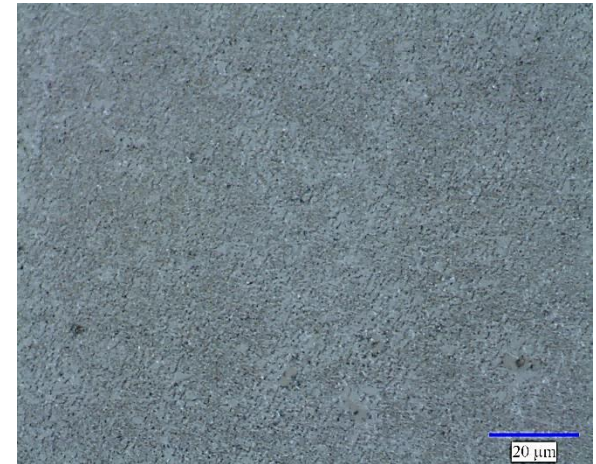
## Microstructure after 620 °C precipitation H/T



1200 °C 8 hr Solution H/T  
Microhardness (VPN): 237



Solutionization + 620 °C 18 hr  
Microhardness (VPN): 349

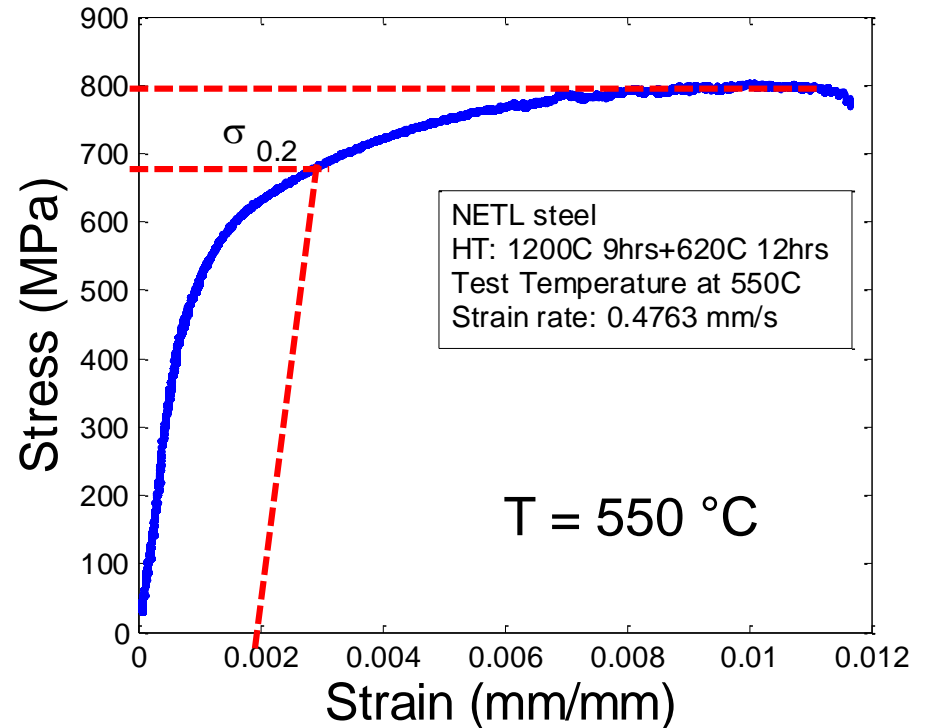


Solutionization+ 620 °C 75 hr  
Microhardness (VPN): 335

Optimum precipitation annealing condition is identified from high-throughput experiment and confirmed with individual samples.

# 3. Summary of Progress

## Tensile test at 550 °C

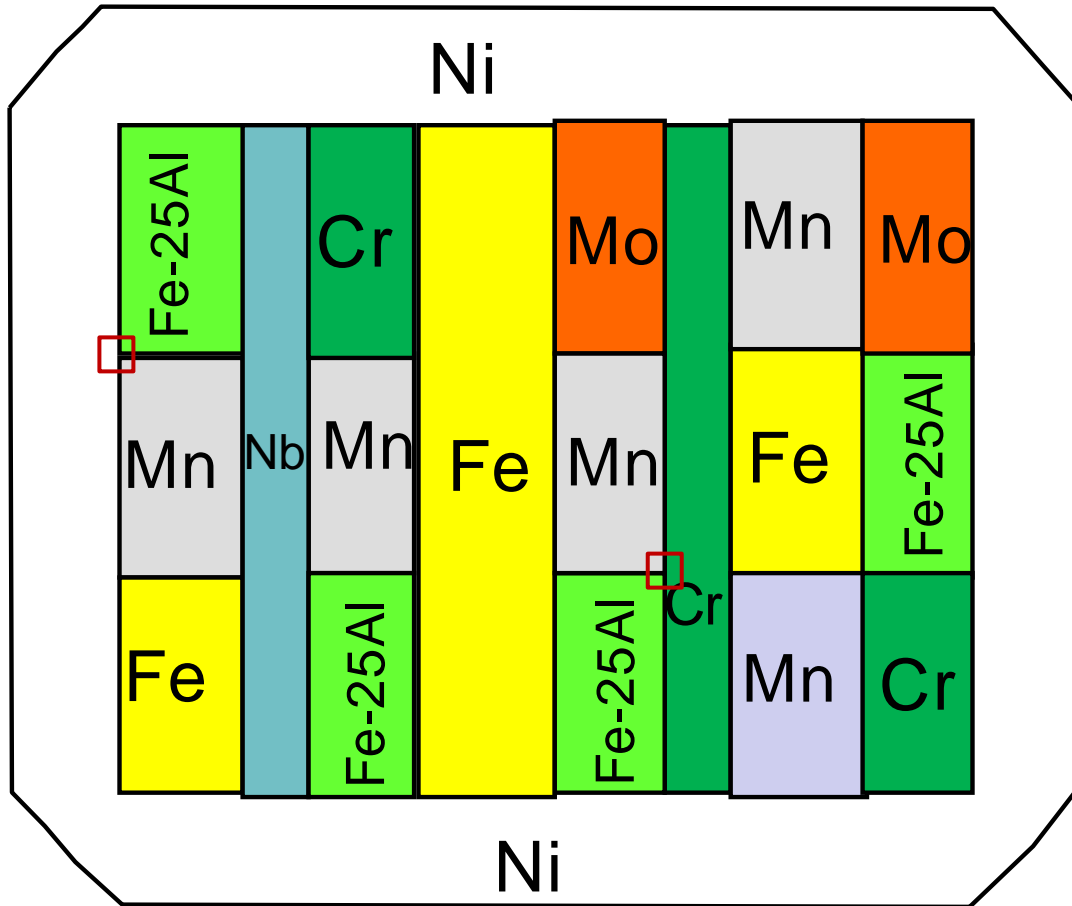


$$\sigma_{0.2} \approx 680 \text{ MPa (at 550 °C)}$$

### Current alloy too brittle:

- Need to reduce Chi phase volume fraction
- Need to engineering grain boundaries

### 3. Summary of Progress

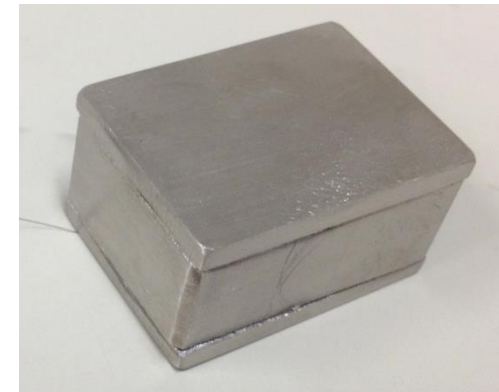
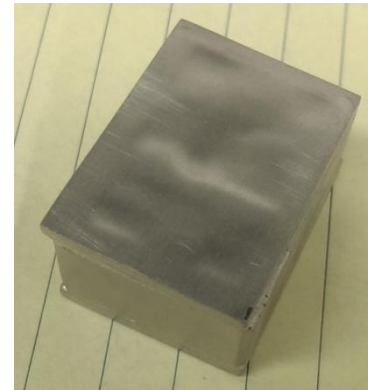
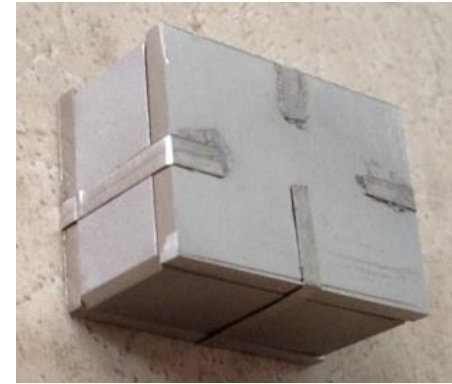
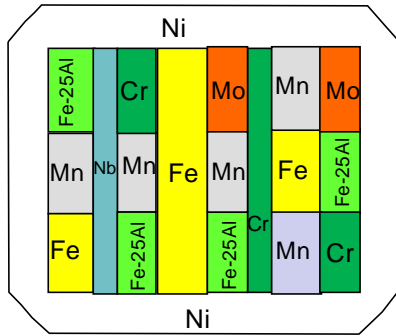


Fe-Mn-Cr  
 Fe-Mn-Al  
 Fe-Mn-Ni  
 Fe-Mn-Mo  
 Fe-Mn-Nb  
 Fe-Ni-Al  
 Fe-Ni-Mo  
 Fe-Ni-Nb  
 Fe-Cr-Ni  
 Fe-Cr-Nb  
 Ni-Mn-Cr  
 Ni-Mn-Mo  
 Ni-Mn-Nb  
 Ni-Mo-Nb  
 Ni-Cr-Nb

Fe-Mn-Ni-Al  
 Fe-Mn-Nb-Al  
 Fe-Mn-Cr-Al  
 Fe-Ni-Mo-Al  
 Fe-Ni-Cr-Al  
 Fe-Ni-Nb-Al.

- Four such diffusion multiples are made for different temperature treatments
- Looking for high Mn and high Cr compositions with fine stable precipitates

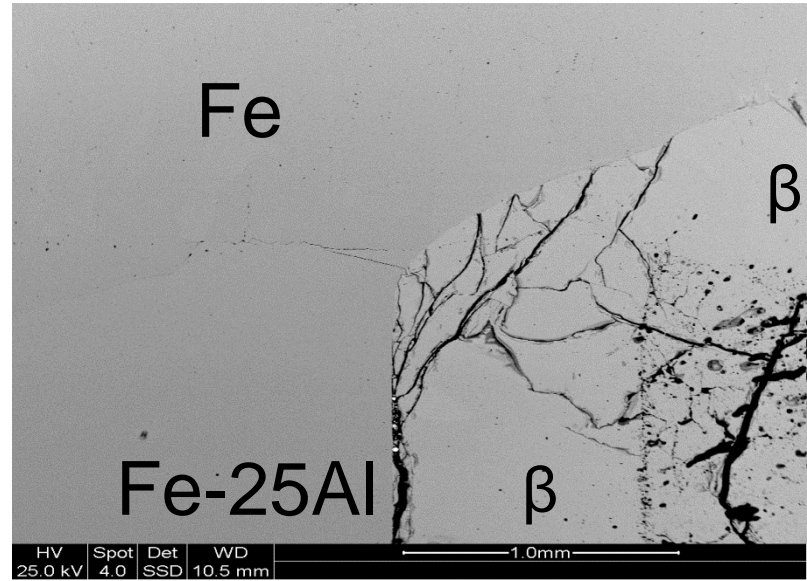
# 3. Summary of Progress



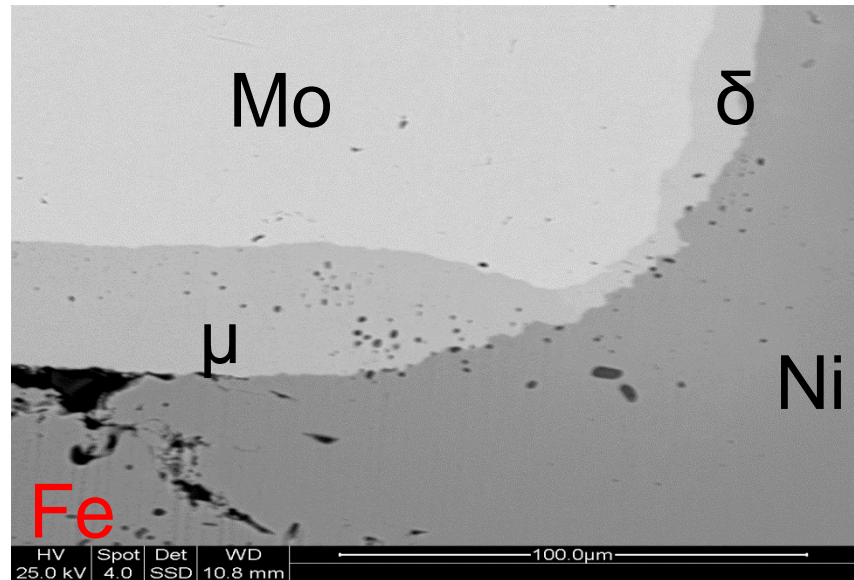
The hot pressed pure Mn pieces provided by a vendor was porous and caused problems during HIP and annealing



# 3. Summary of Progress

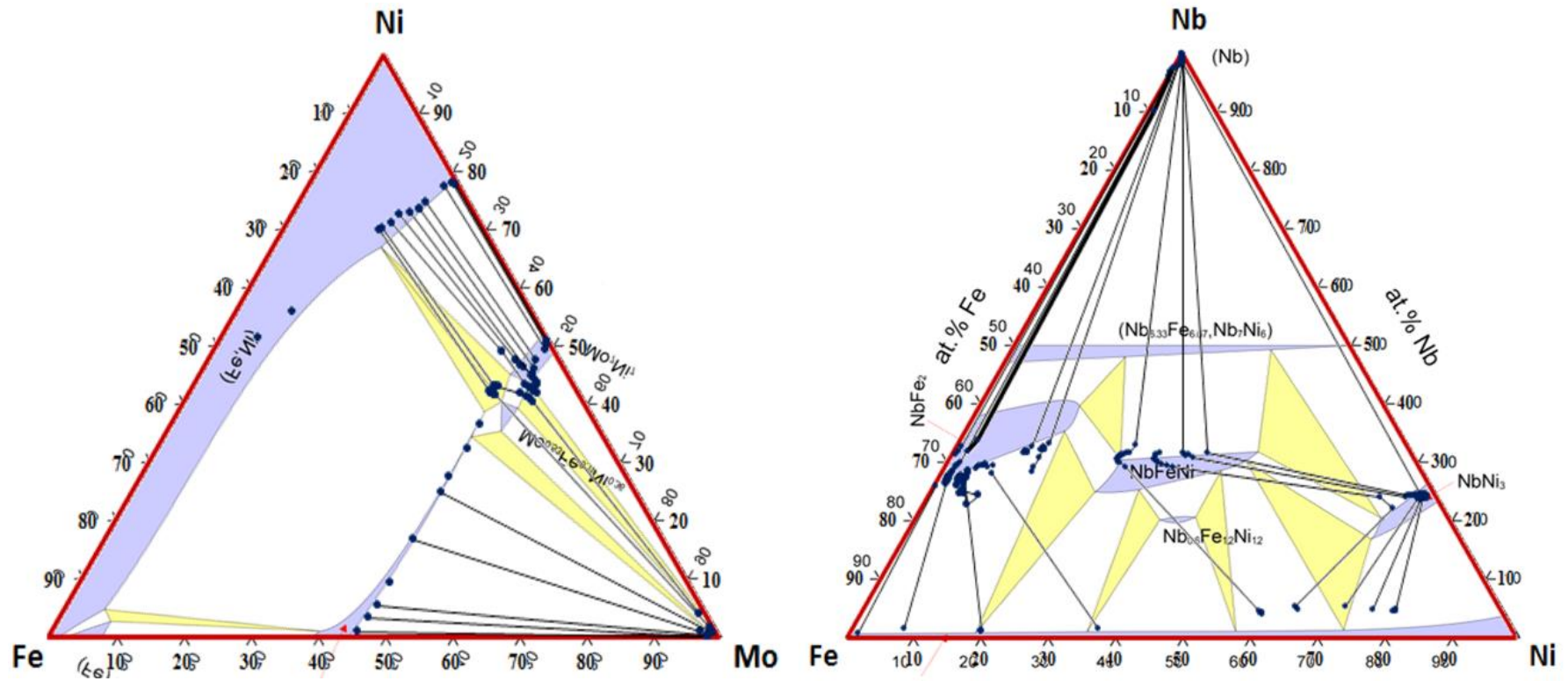


Mn



### 3. Summary of Progress

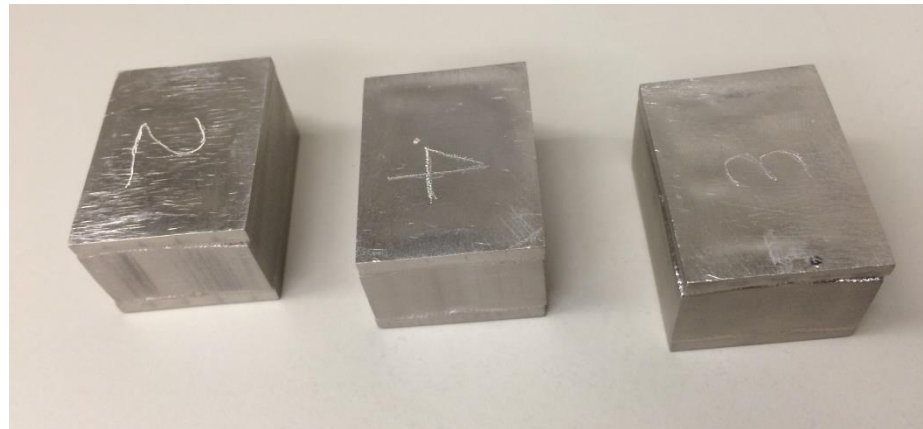
# Preliminary phase diagram data



### 3. Summary of Progress



After EB welding



After HIP

- New diffusion multiples have been made with high-quality Mn
- Potentially useful precipitates for high Mn, high Cr, austenitic steels will be screened

# 3. Summary of Progress

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## Brief Summary of Fe-Cr-Mo ferritic Chi steel

- Fine stable Chi-phase precipitates observed in a dual-anneal diffusion multiple was confirmed by a cast ferritic steel
- Fe-Cr-Mo Chi-phase strengthened steel was ductile at high temperature (hammer-forged from round ingot to flat plate)
- High-throughput gradient temperature tests are very effective in identifying the optimum precipitation annealing temperature
- The Chi-strengthened steel has good strength at high temperature (e.g.,  $\sigma_{0.2} \approx 680$  MPa at 550 °C)
- Current alloy too brittle:
  - Need to reduce Chi phase volume fraction
  - Need to engineering grain boundaries
- A new steel is designed and will be made soon

## 3. Summary of Progress

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### **Brief Summary of Fe-Mn-Cr-Al-Ni-Mo-Nb diffusion multiples**

- First set of Fe-Mn-Cr-Al-Ni-Mo-Nb diffusion multiple failed (melted) completely due to a furnace thermocouple failure
- A make-up diffusion multiple was only partially successful (Mn porosity problem) and is being analyzed for phase diagram information related to high Mn and high Cr (or Al) austenitic compositions from useful regions of the diffusion multiple
- A new set of diffusion multiples have been made and are being analyzed

## 4. Future Work

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- Cast and test a new Chi-strengthened ferritic steel with lower Chi volume fraction and engineered grain boundary chemistry
- Complete dual-anneal diffusion multiples containing (Fe-Mn-Cr-Al-Ni-Mo-Nb)
- Generate large amount of data and useful information to help identify new steels, especially high Mn austenitic steels;
- Design high Mn austenitic steels for casting and property tests.

## 4. Future Work

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We have gone through the entire processes of making diffusion multiples, identifying precipitate phases, designing alloys, induction melting alloys, performing high-throughput gradient temperature tests for precipitation condition optimization, and performing property testing

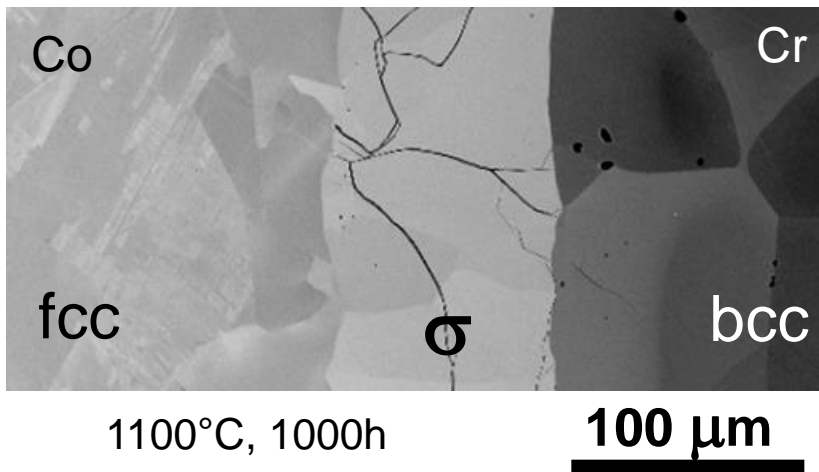
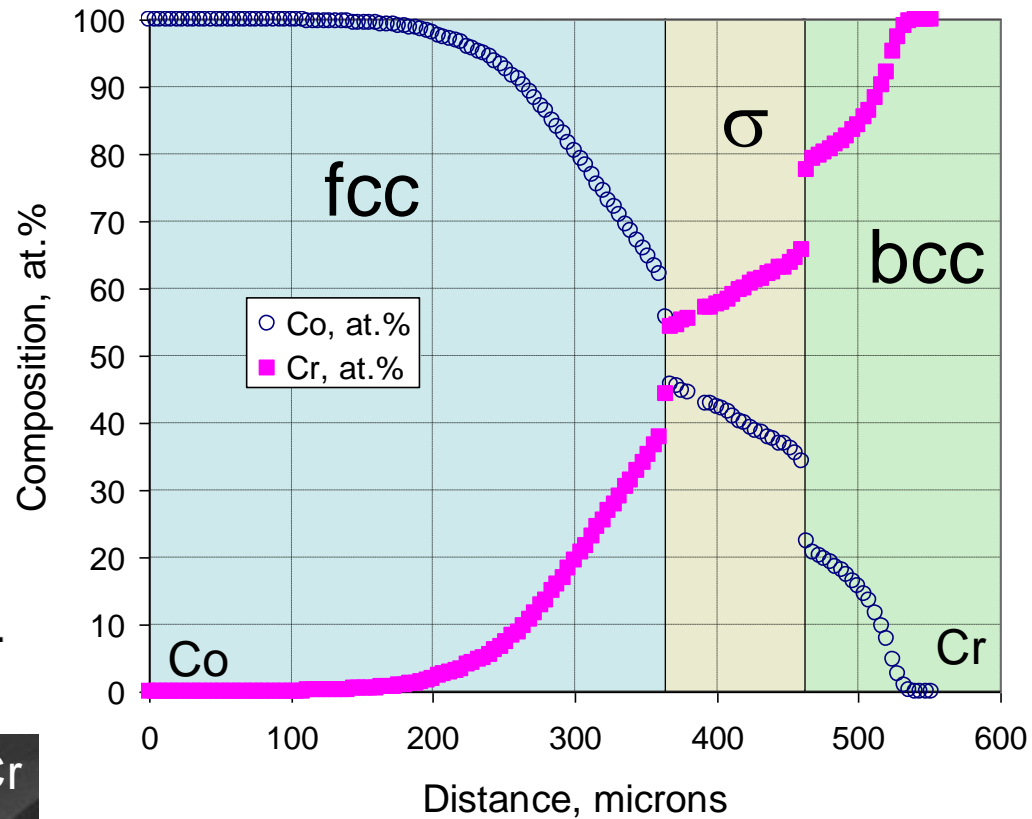
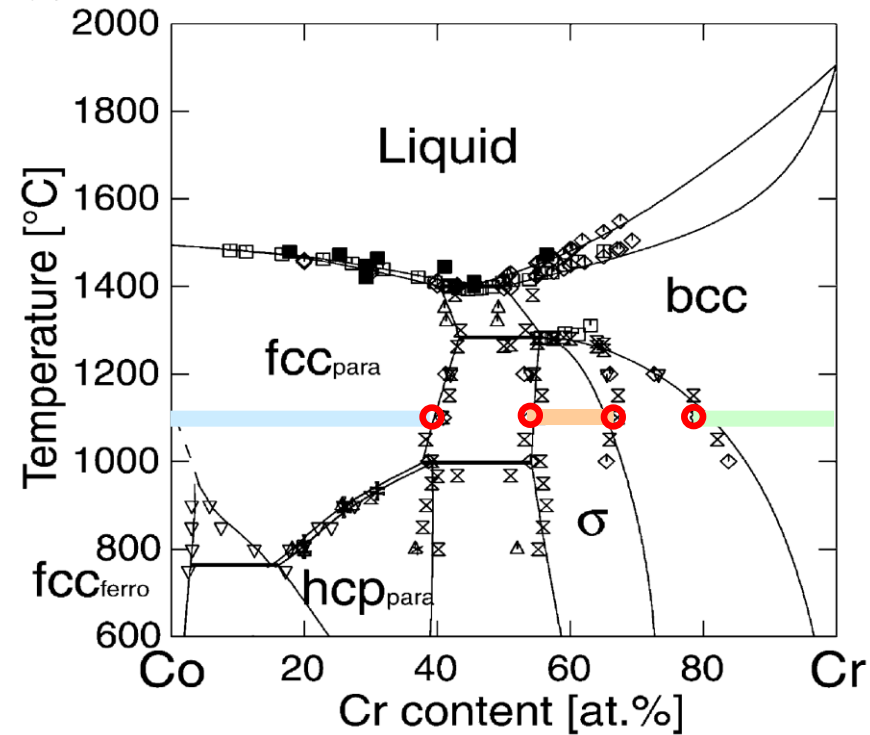
We will be able to move quickly to accomplish the proposed work as soon as we get the new diffusion multiples annealed.

**Thank you!**



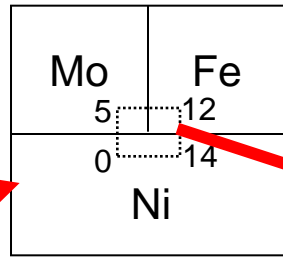


# 2. Technical Approach

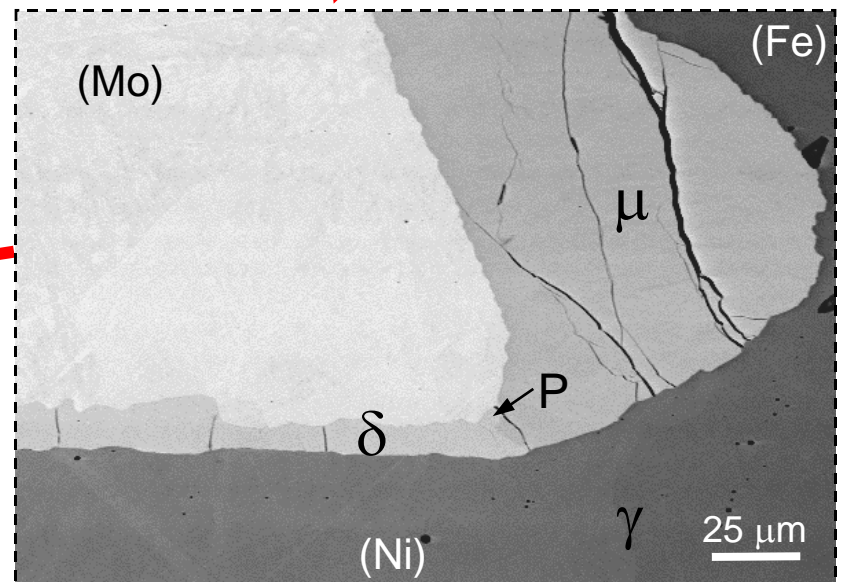
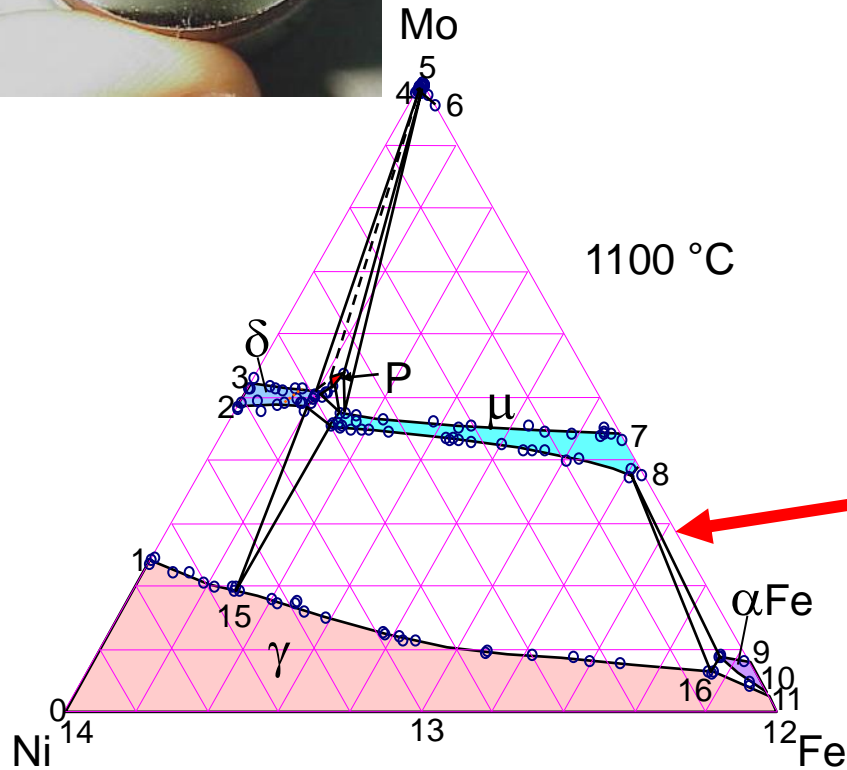
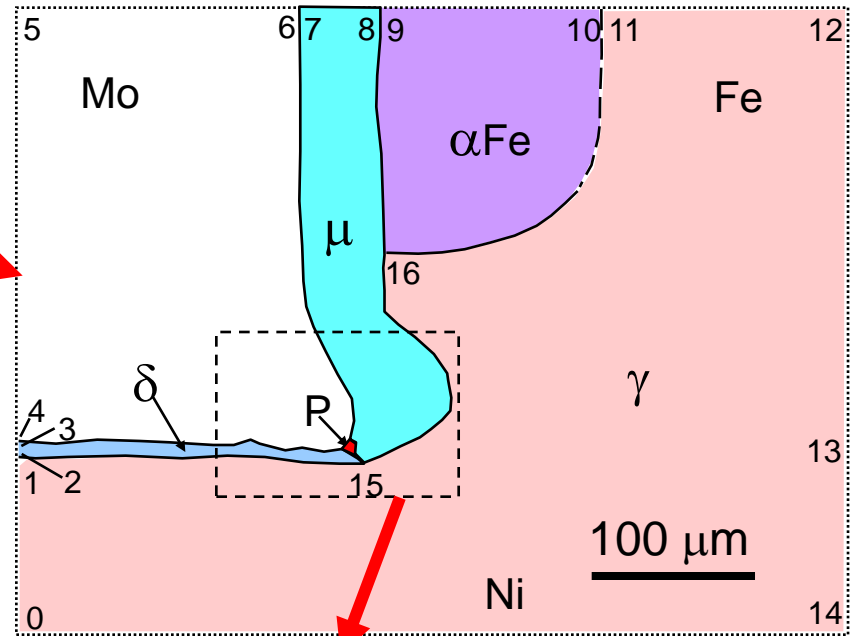


- Local equilibrium at phase interfaces defines the tie-lines
- Interdiffusion creates all single-phase compositions

# 2. Technical Approach



1100°C 1500 hrs



# High-Throughput Experimental Tools

## 1200°C single-anneal Fe-Cr-Mo

