

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

**Changdong Wei, William Clark, J.-C. Zhao**

*Department of Materials Science and Engineering  
The Ohio State University*

**DOE/UCR Award FE0008960**



NETL Managers: **Charles Miller** and **Vito Cedro**

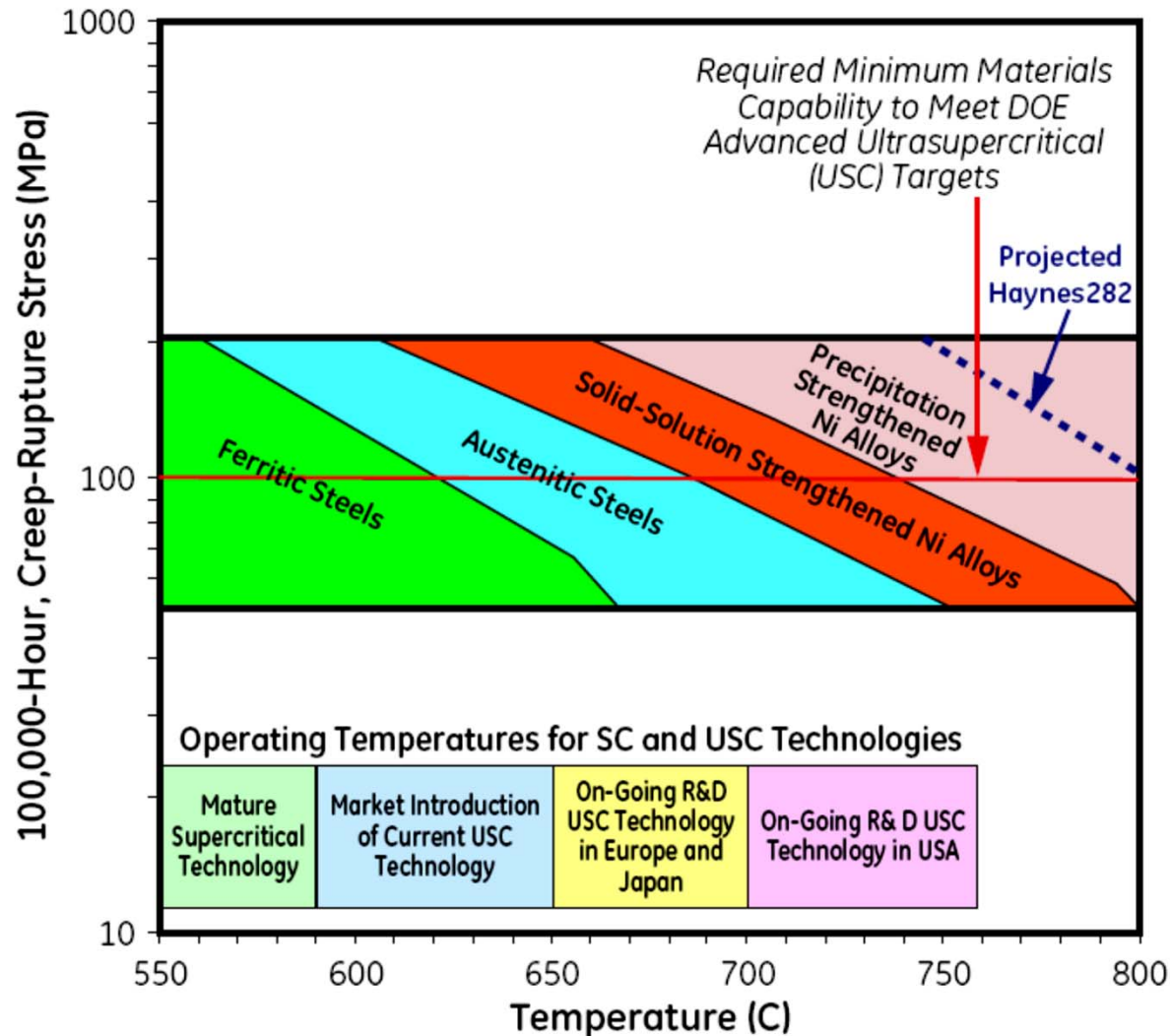


2015 NETL's Crosscutting Research Review Meeting  
April 27-30, 2015 in Pittsburgh, PA

# Outline

1. Background and Project Objectives
2. Technical Approach
3. Ferritic Steels
4. Austenitic Steels
5. Summary & Future Work

# 1. Background and Project Objectives

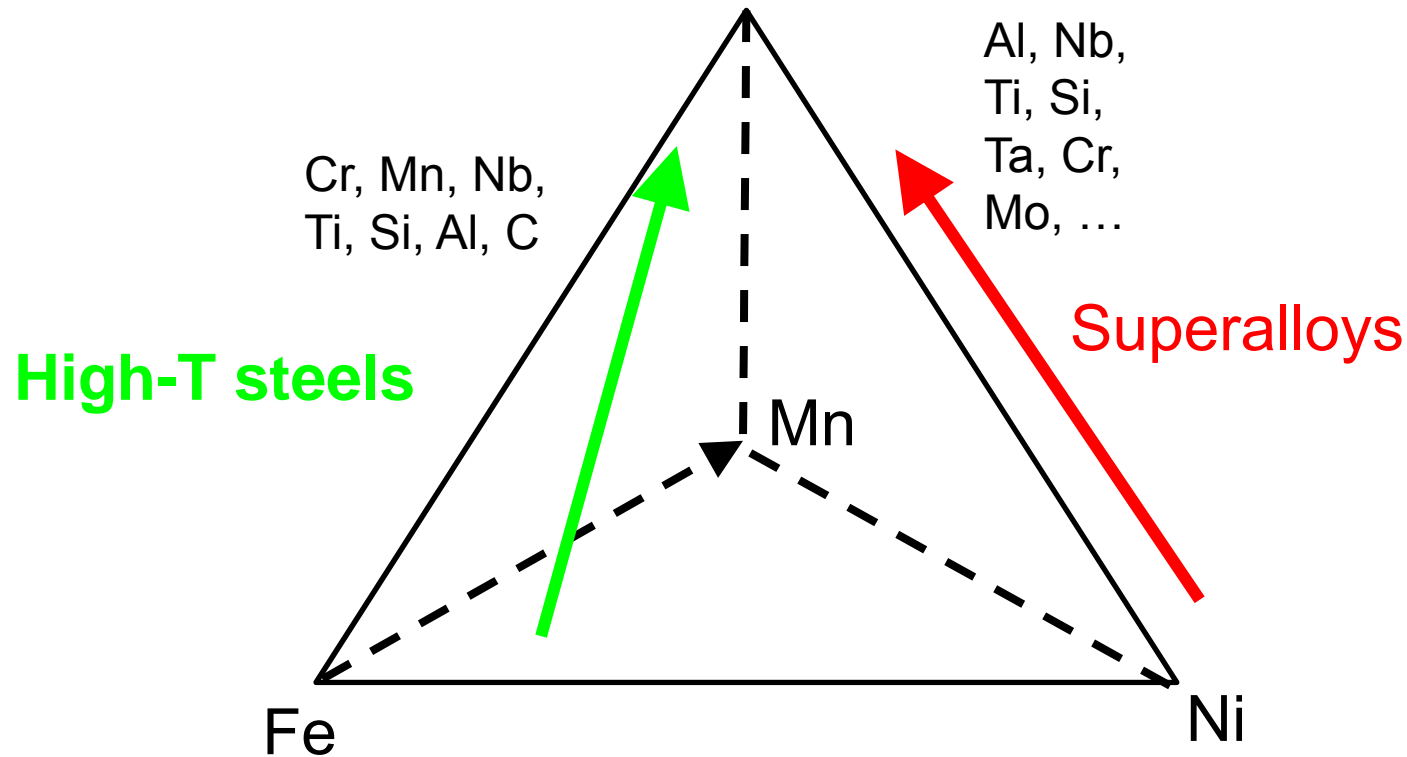


L. Jiang, 2011

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

# 1. Background and Project Objectives

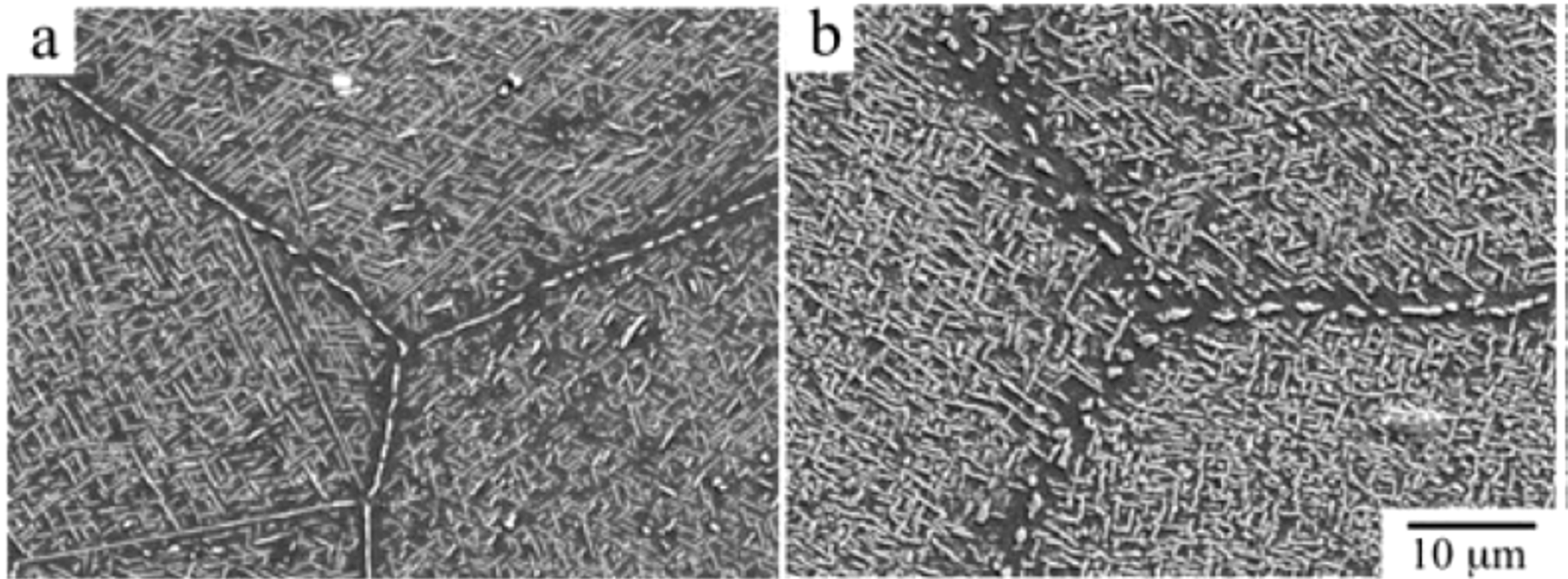
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- Identification of new strengthening phases through high-throughput exploration together with computational thermodynamics.
- Cost-effective steels for AUSC clean coal systems.

# 1. Background and Project Objective

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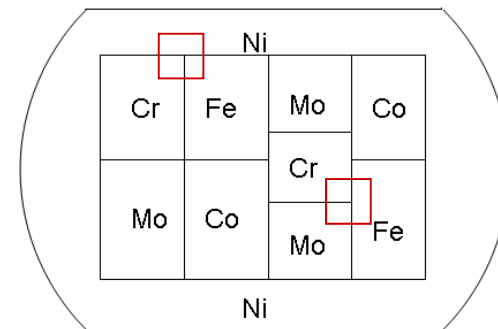
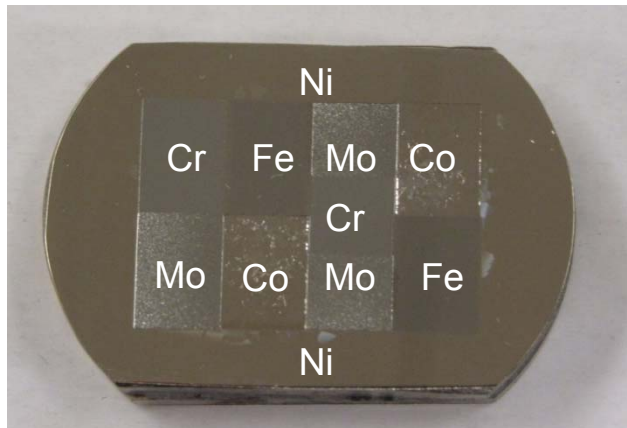
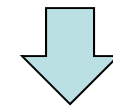
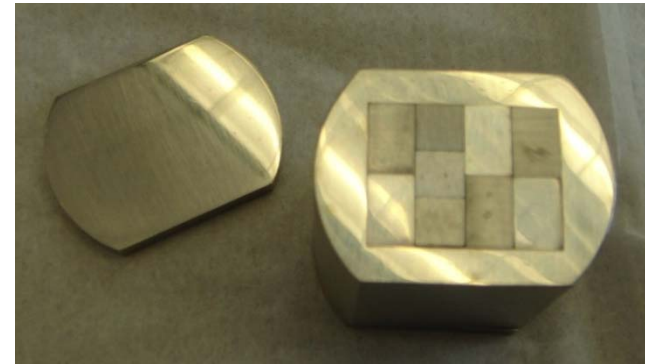
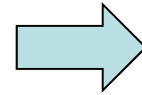
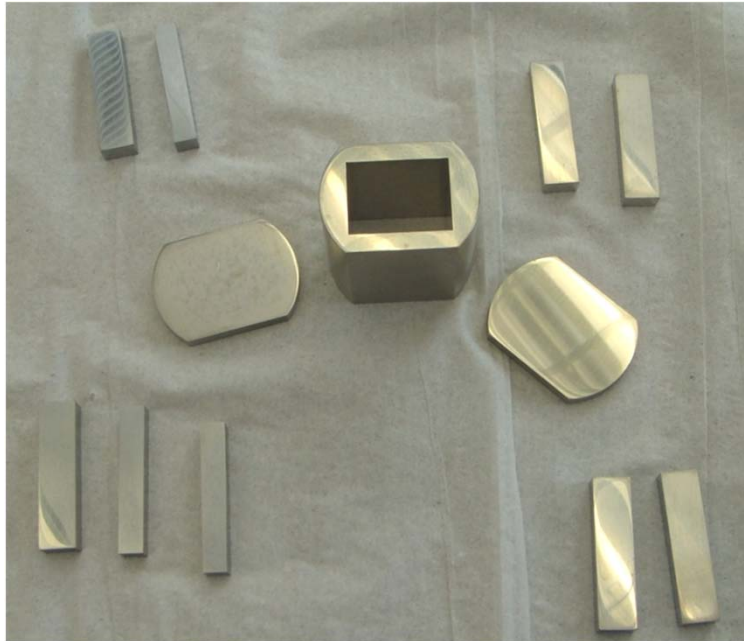


*Finely dispersed Laves phase in a Fe-20Cr-30Ni-2Nb (at.%) steel after a creep test at 700°C and 120 MPa: (a) boron-doped steel, and (b) boron-free steel (Takeyama et al.)*

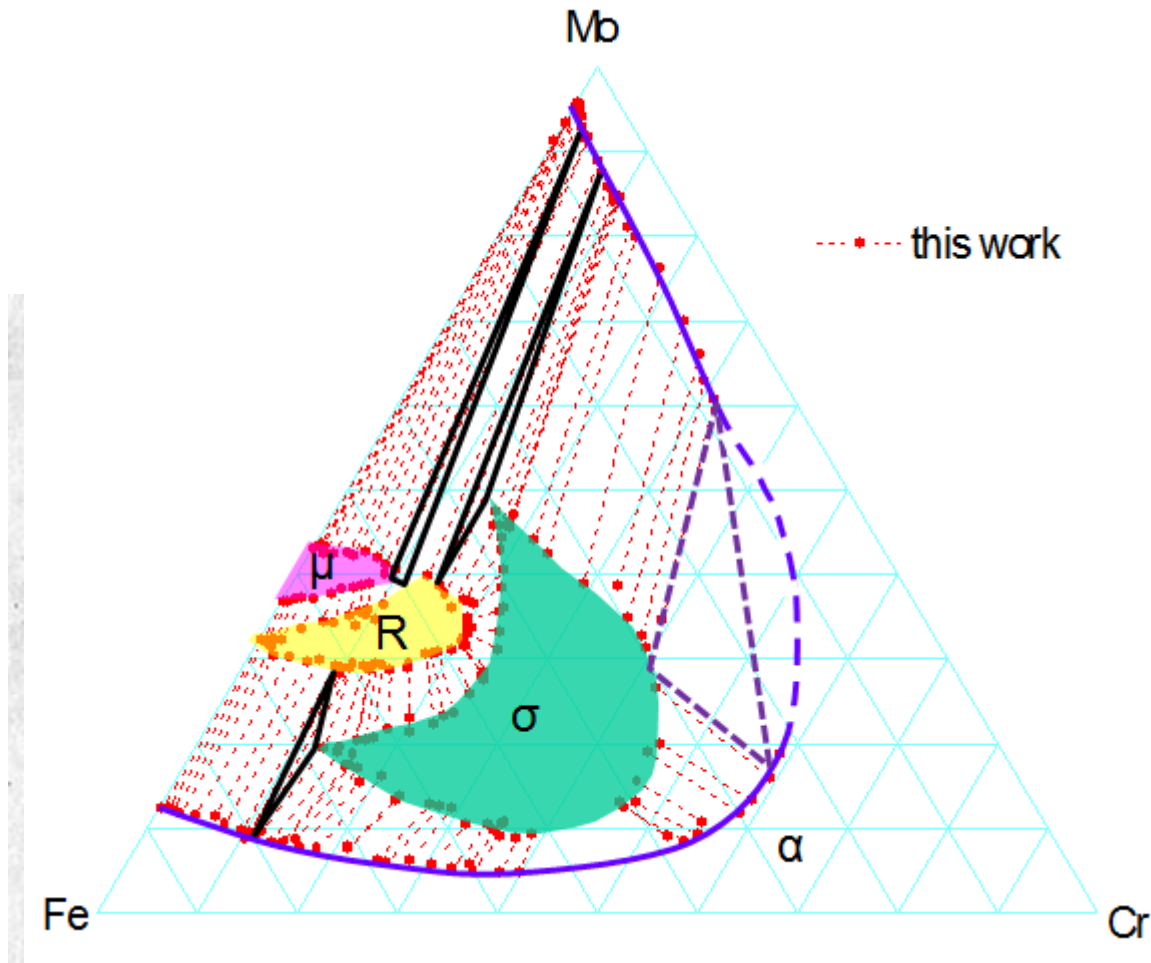
- Laves phase has demonstrated good properties.
- Sluggish precipitation kinetics.
- Grain boundary precipitates key to good creep strength.
- High enough Cr for hot corrosion resistance.

## 2. Technical Approaches

### Diffusion-multiple approach

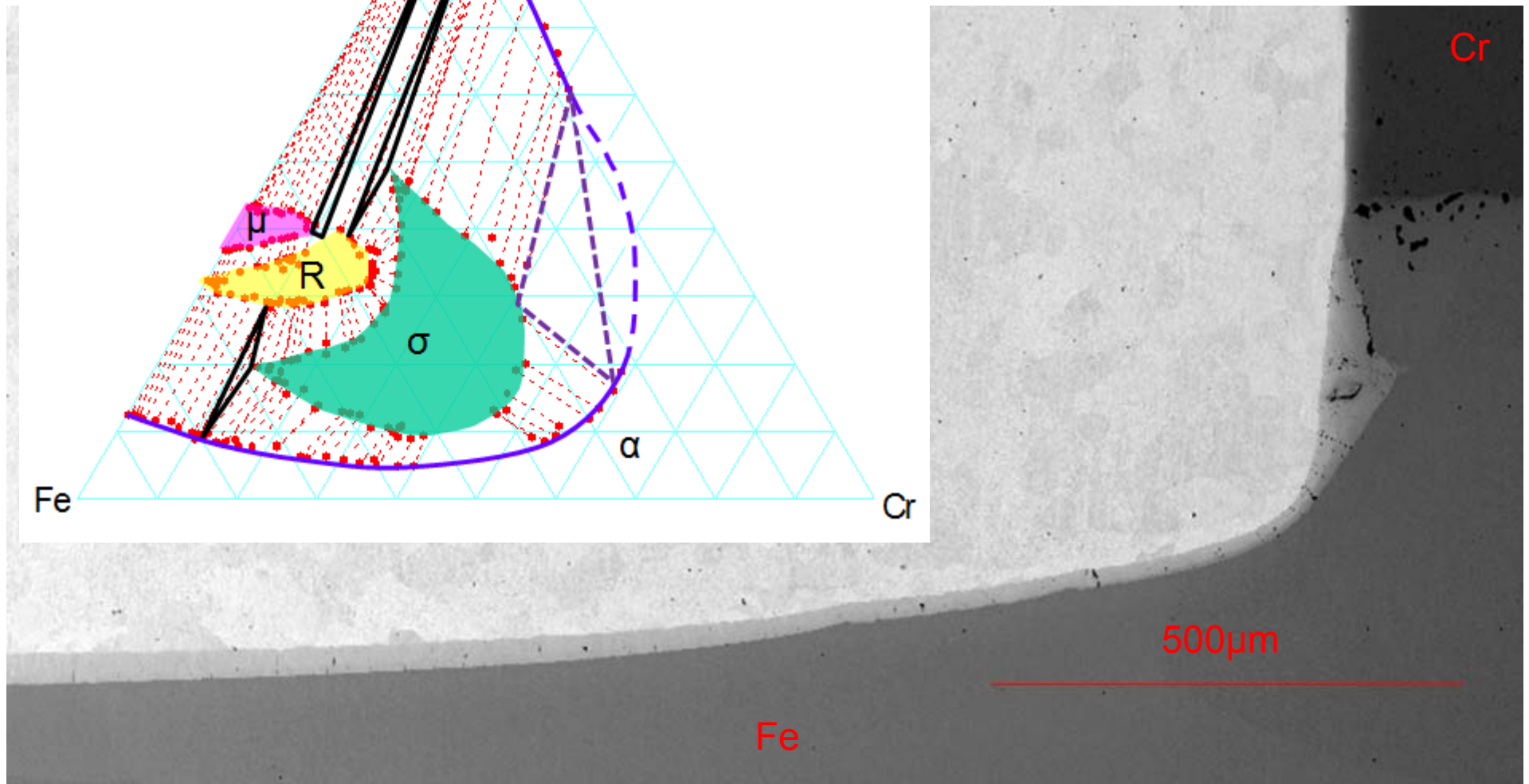


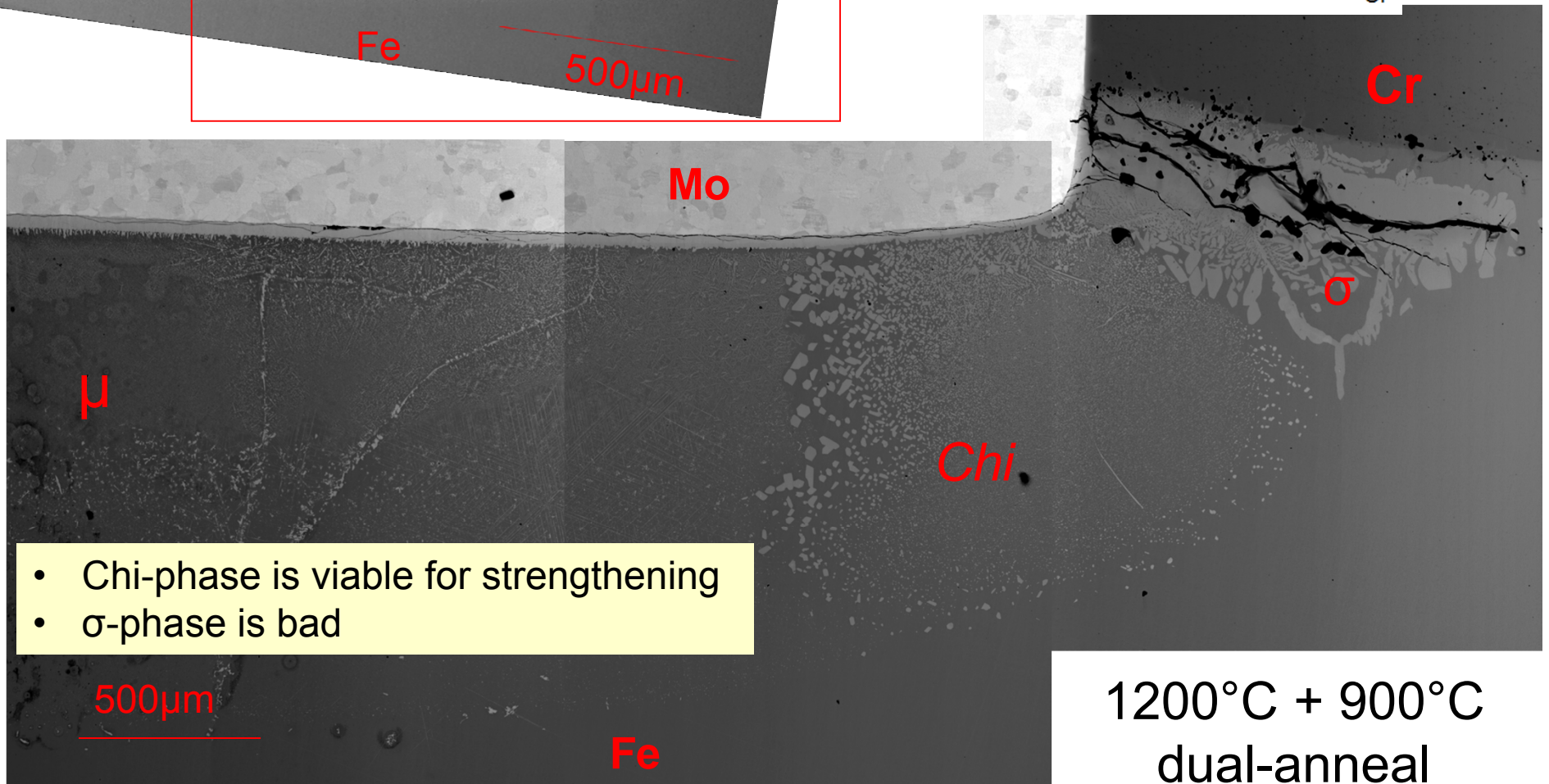
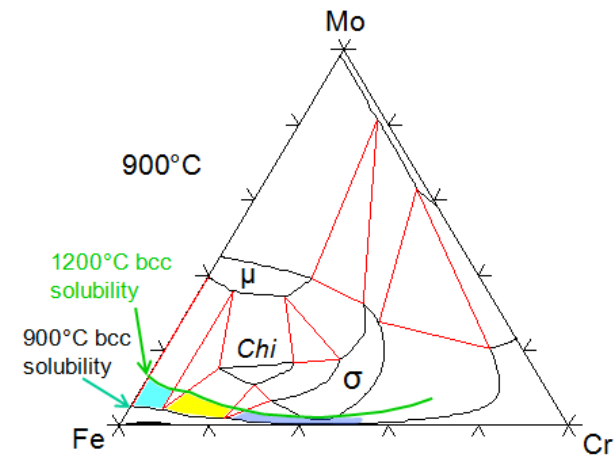
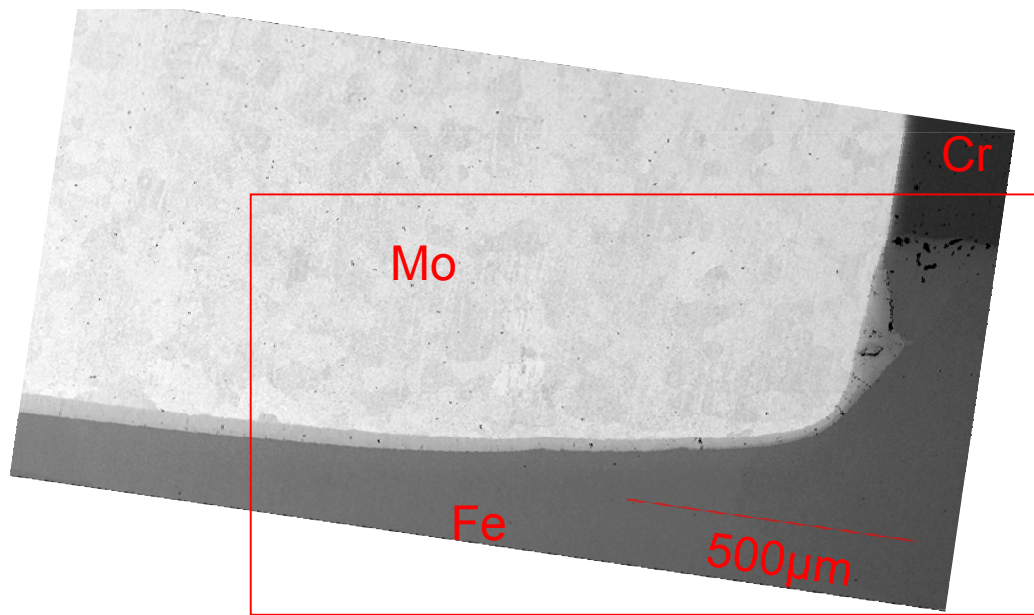
## 2. Technical Approaches



al diffusion multiple

ngle-anneal Fe-Cr-Mo





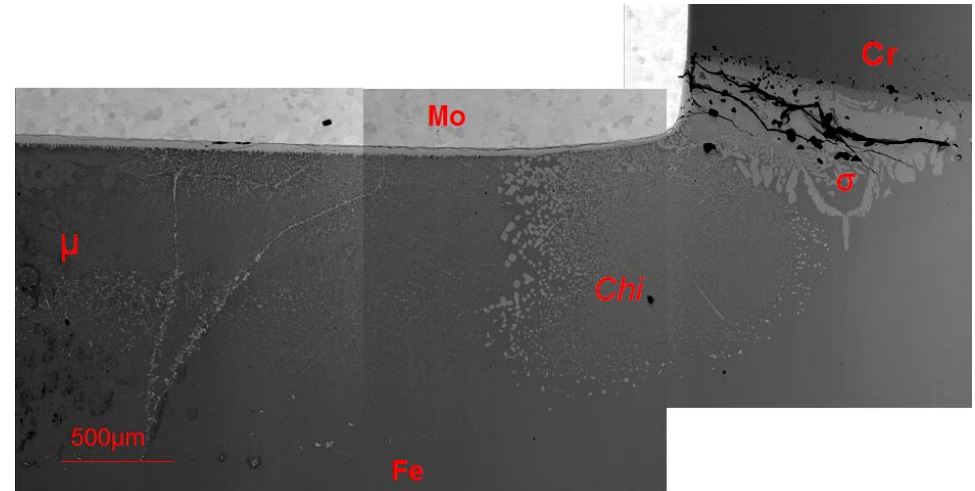
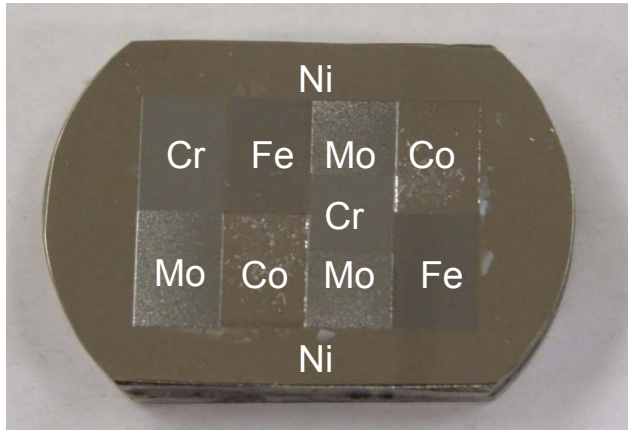
- Chi-phase is viable for strengthening
- $\sigma$ -phase is bad

1200°C + 900°C  
dual-anneal

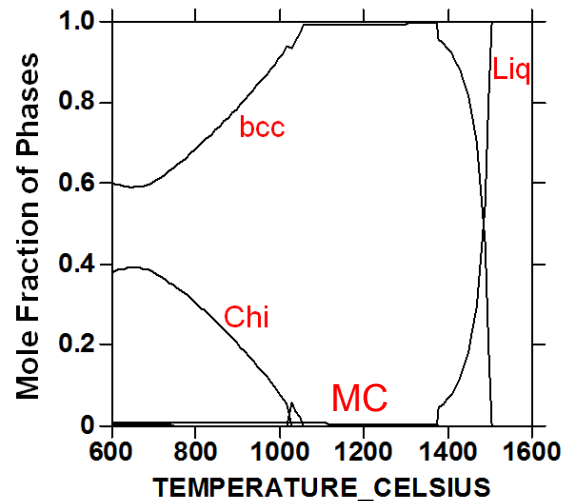


## 2. Technical Approaches

Dual-Anneal Diffusion Multiple → Viable Strengthening Phase



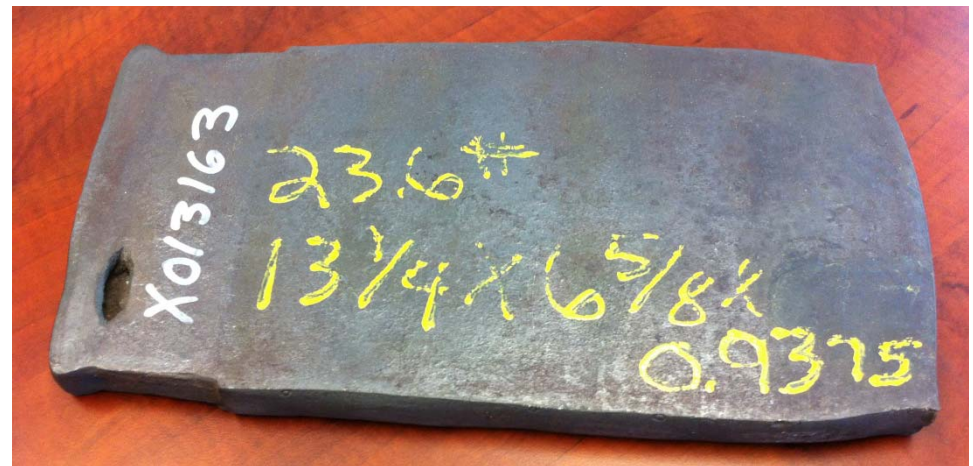
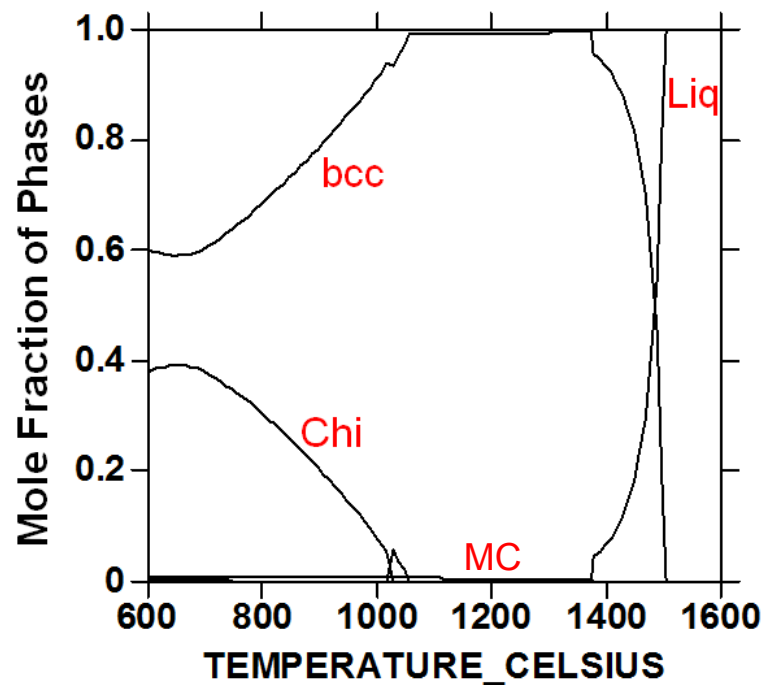
Computational Thermodynamics → Design of Alloys for Property Balance



- Multicomponent alloys
- Volume fractions of phases
- Grain boundary phases
- Oxidation resistance consideration
- Cost and other requirements

### 3. Ferritic Steels

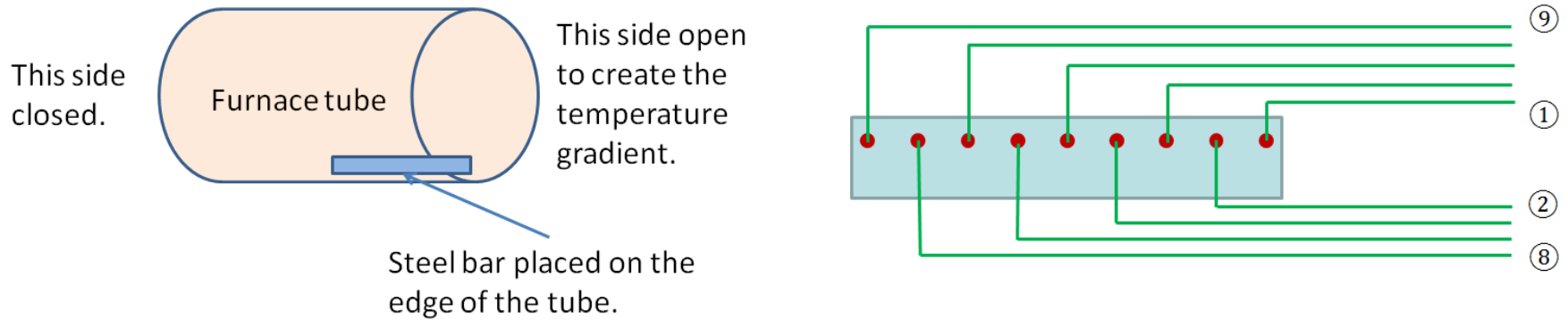
- Designed a Chi-phase strengthened steel using Thermo-Calc
- Induced MC carbide for grain boundary pinning during solution annealing
- Induction melted the alloy (24 lbs) and processed to 1" plate
- Performed systematic study of solution annealing and precipitation annealing



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

# 3. Ferritic Steels

Gradient Temperature Heat Treatment to quickly find the optimum precipitation heat treatment

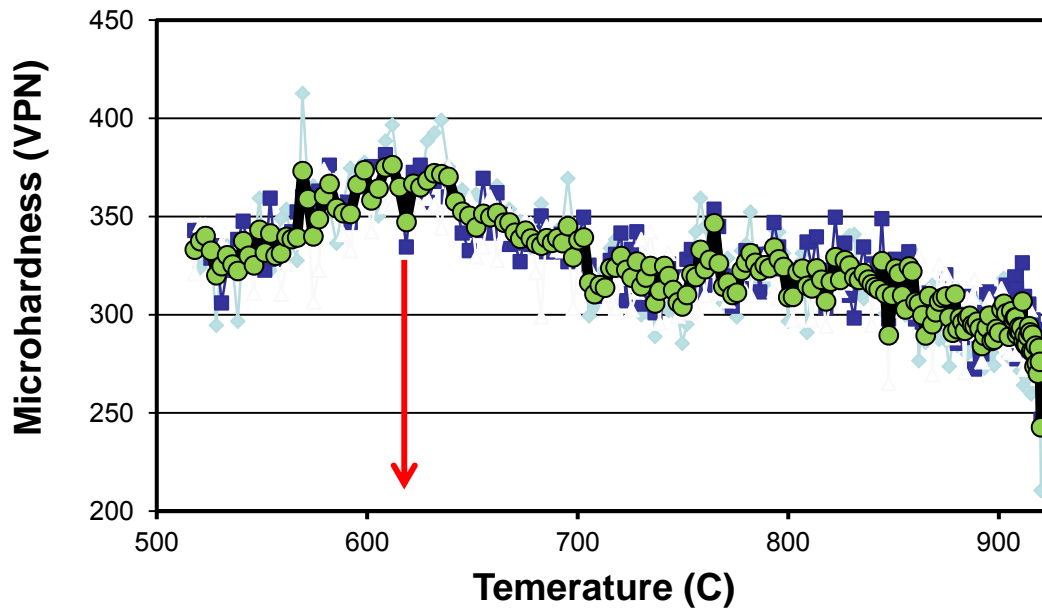
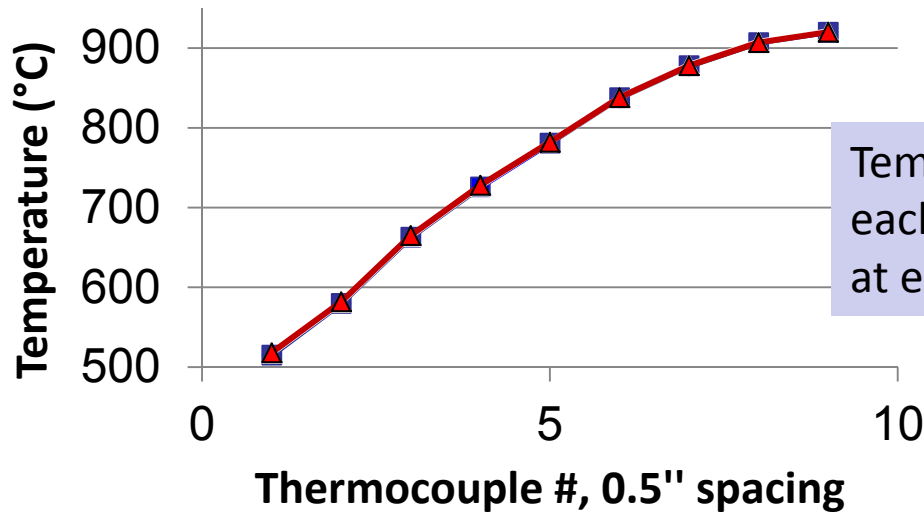


9 type-K thermocouples were attached to the steel bar at an equal spacing.

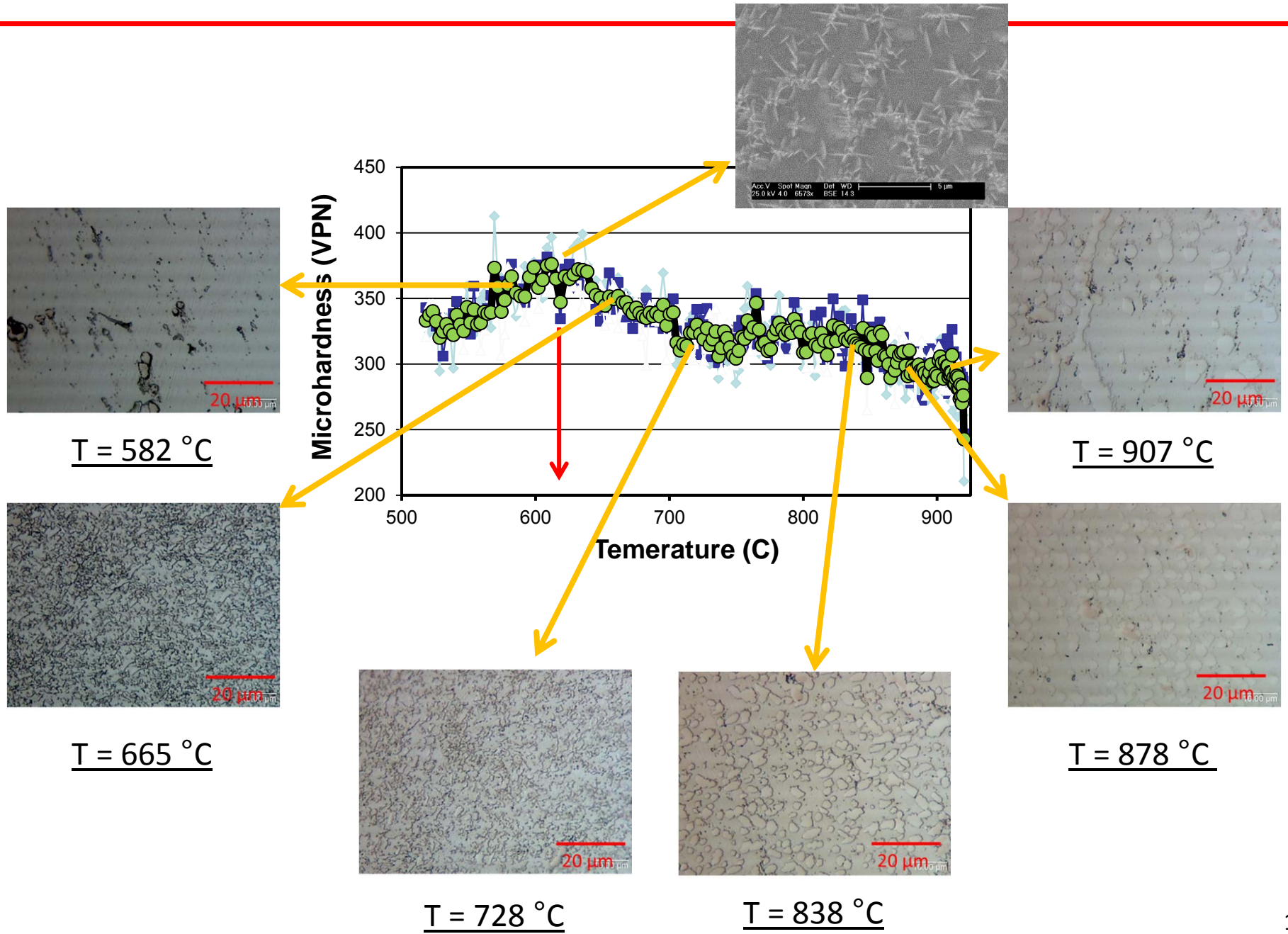


The steel bar was placed to an open end tube furnace at 1000°C (center) for 10 hrs.

### 3. Ferritic Steels



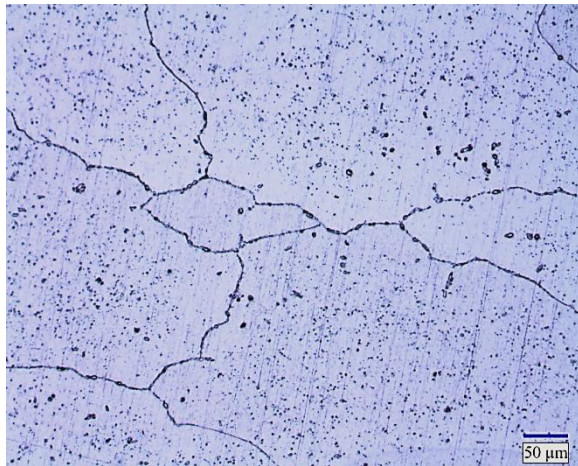
# 3. Ferritic Steels



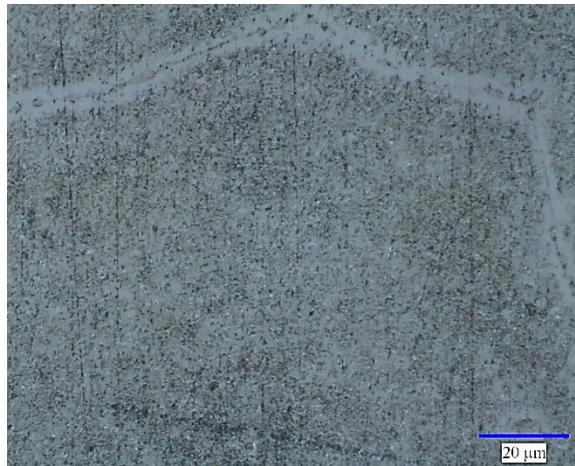
# 3. Ferritic Steels

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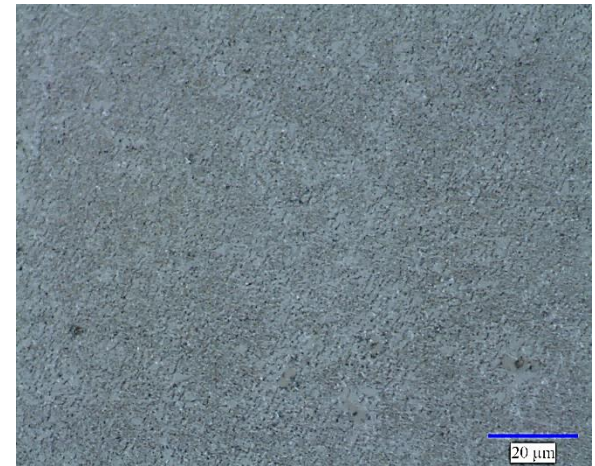
## Gradient Temperature Heat Treatment (GTHT)



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



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



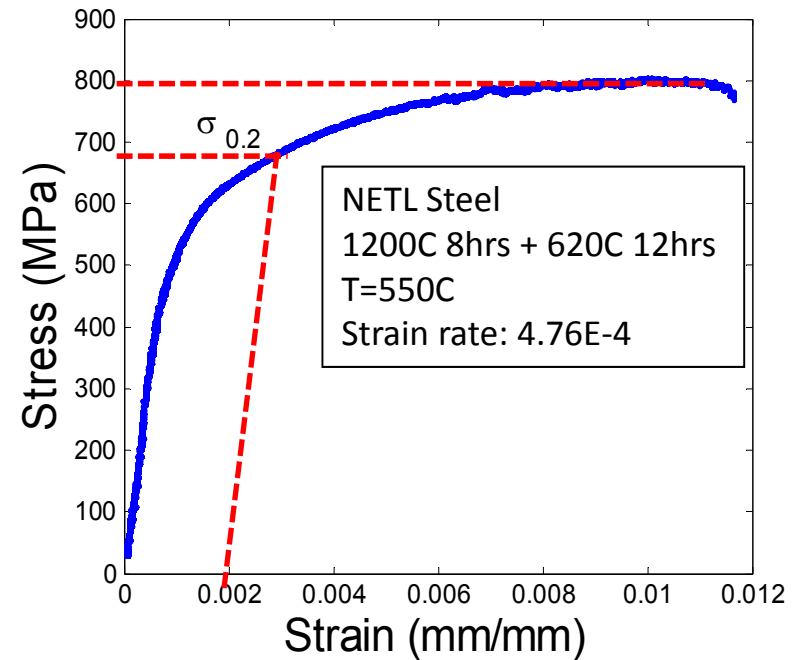
Solutionization+ 620 °C 75 hr  
Microhardness (VPN): 335

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

### 3. Ferritic Steels

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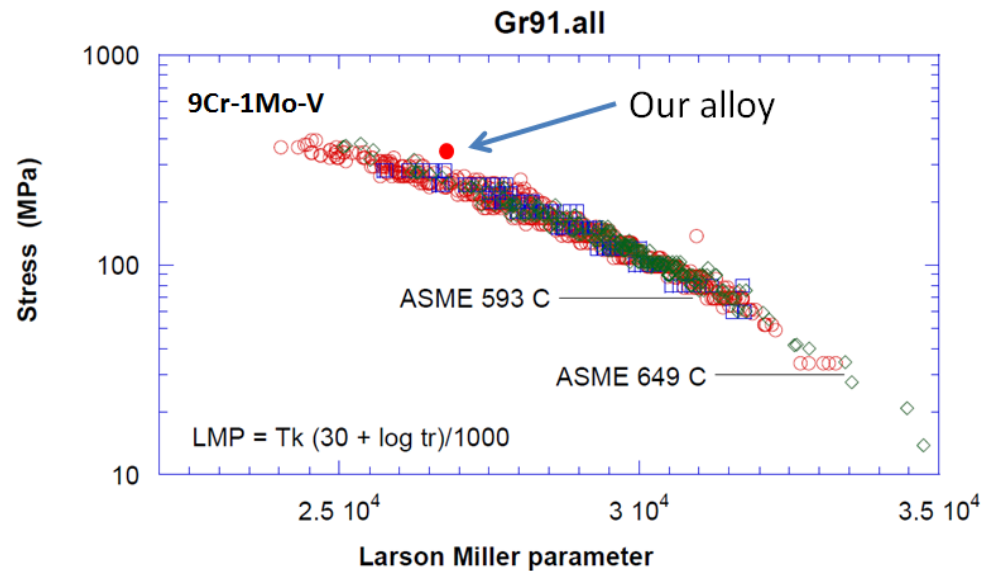
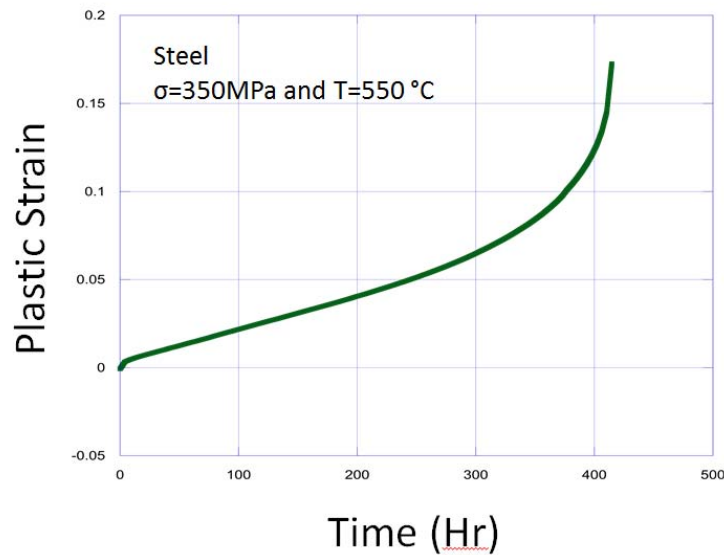
## Tensile test at 550 °C



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

### 3. Ferritic Steels

## Creep test at 550 °C, 350MPa



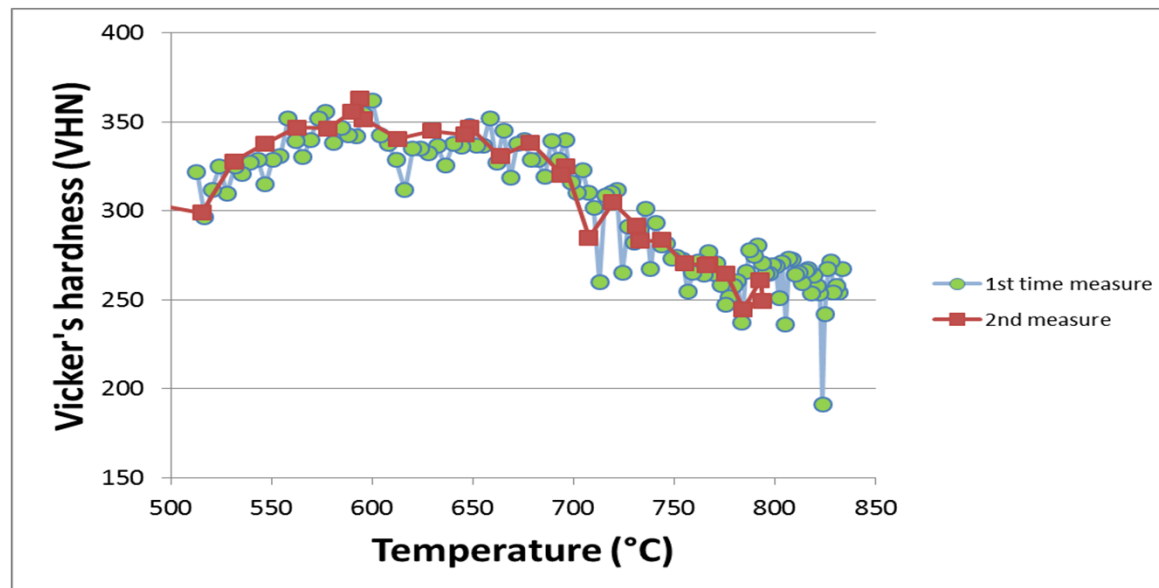
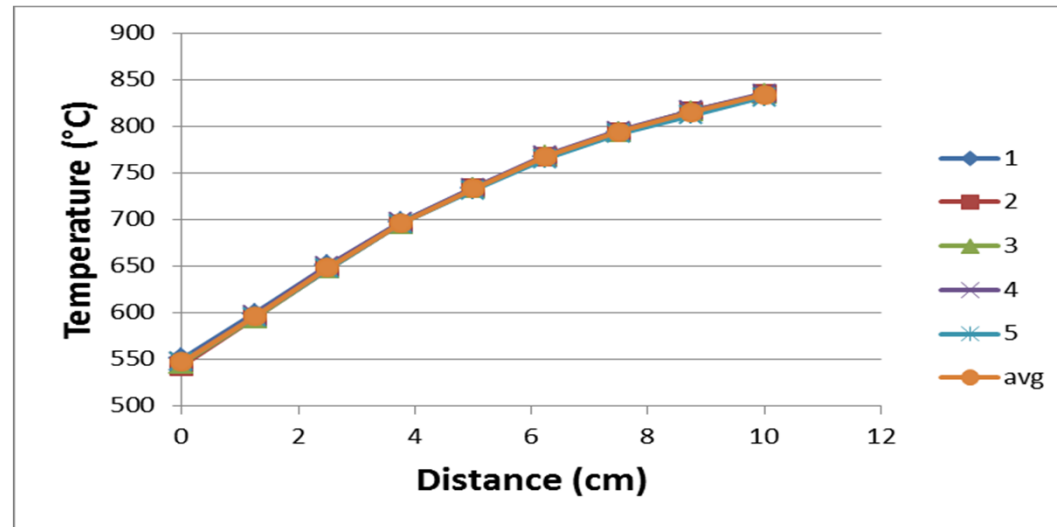
Calculated Larson-Miller parameter and compared with high temperature ferritic steel Fe-9Cr-1Mo-V in literature.

Data from Michael Gold at <http://web.ornl.gov/~webworks/cppr/y2001/rpt/119036.pdf>



# 3. Ferritic Steels

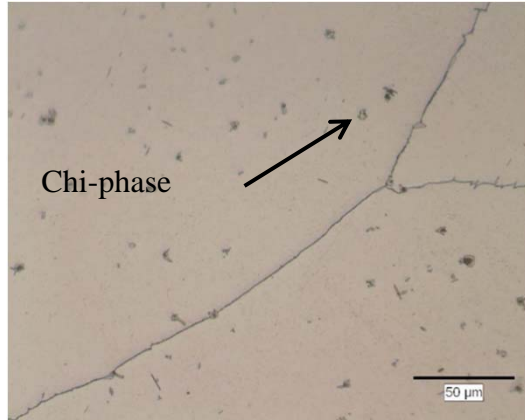
## Gradient Temperature Heat Treatment for ferritic steel #2



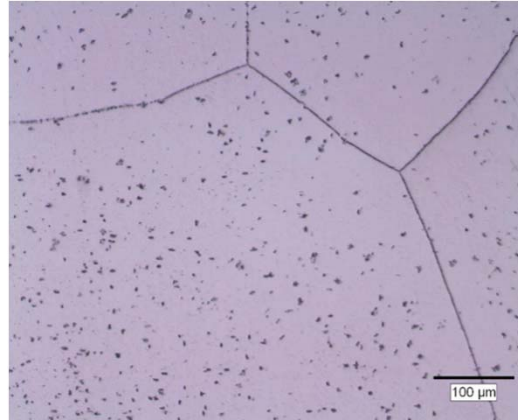
# 3. Ferritic Steels

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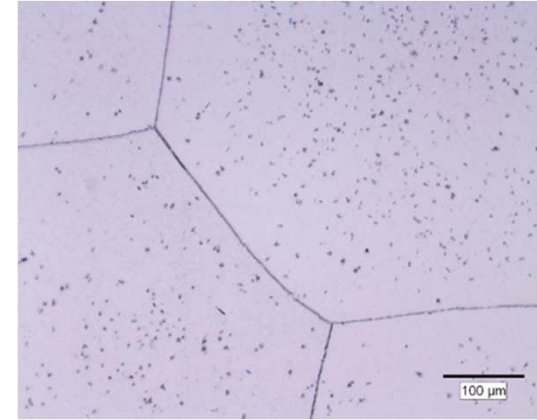
Optical images (after etch) at different locations



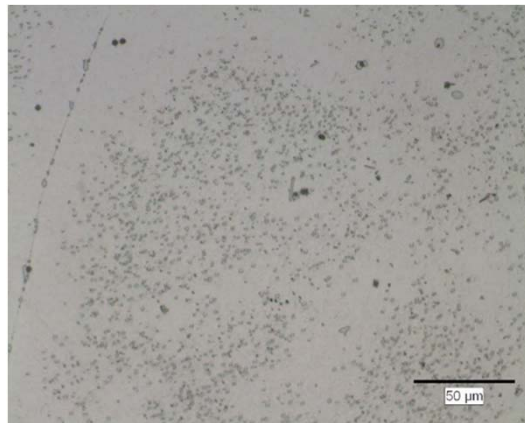
T = 733 ° C



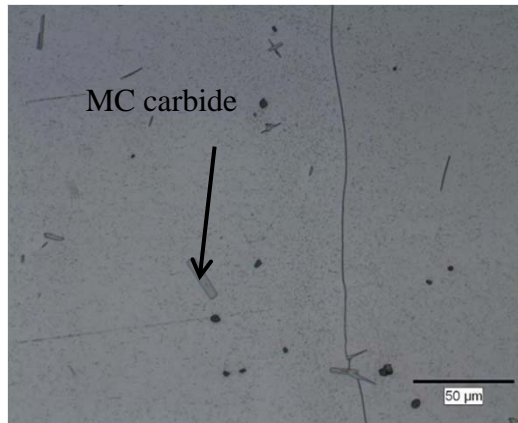
T = 715 ° C



T = 689 ° C



T = 654 ° C



T = 611 ° C

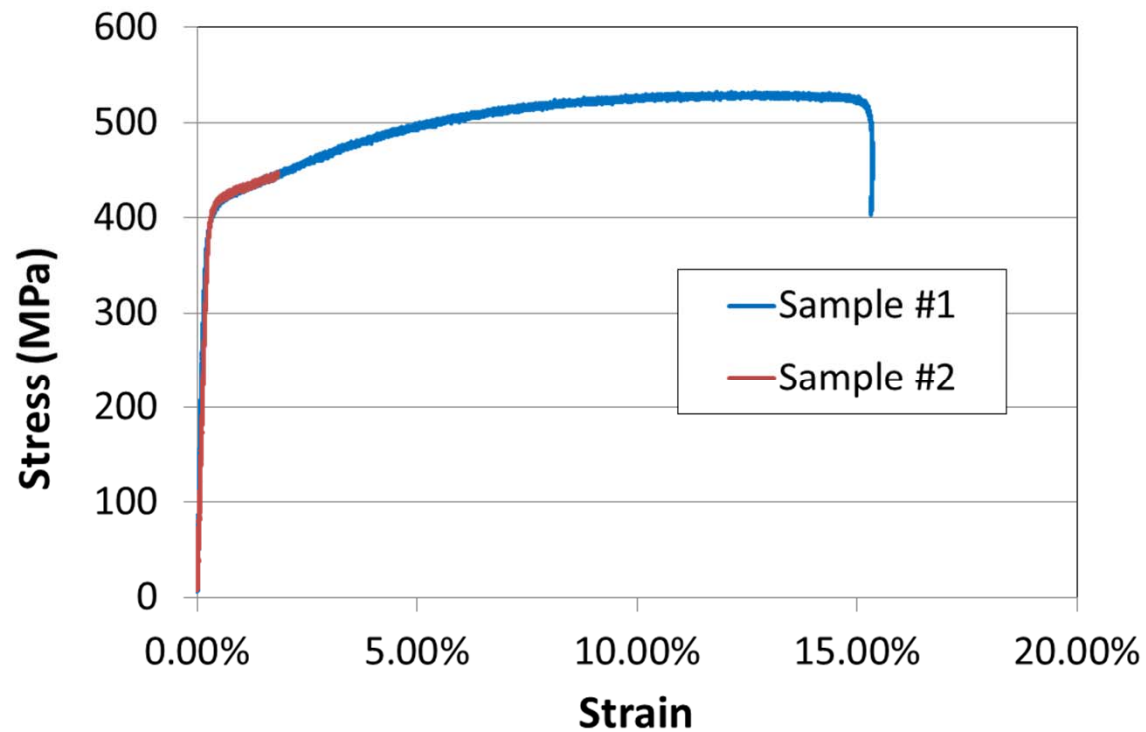


T = 566 ° C

### 3. Ferritic Steels

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#### Tensile tests of “as-received” ferritic steel #2

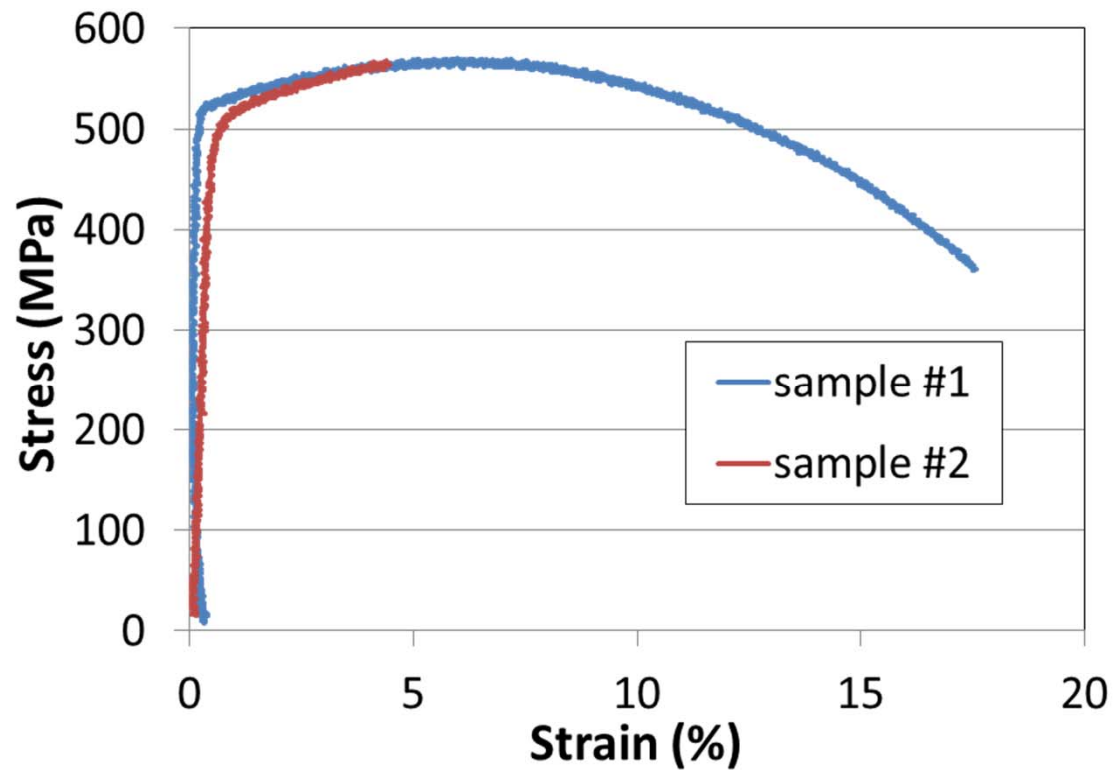


Induction melting + hammer forging (no additional heat treatment)

### 3. Ferritic Steels

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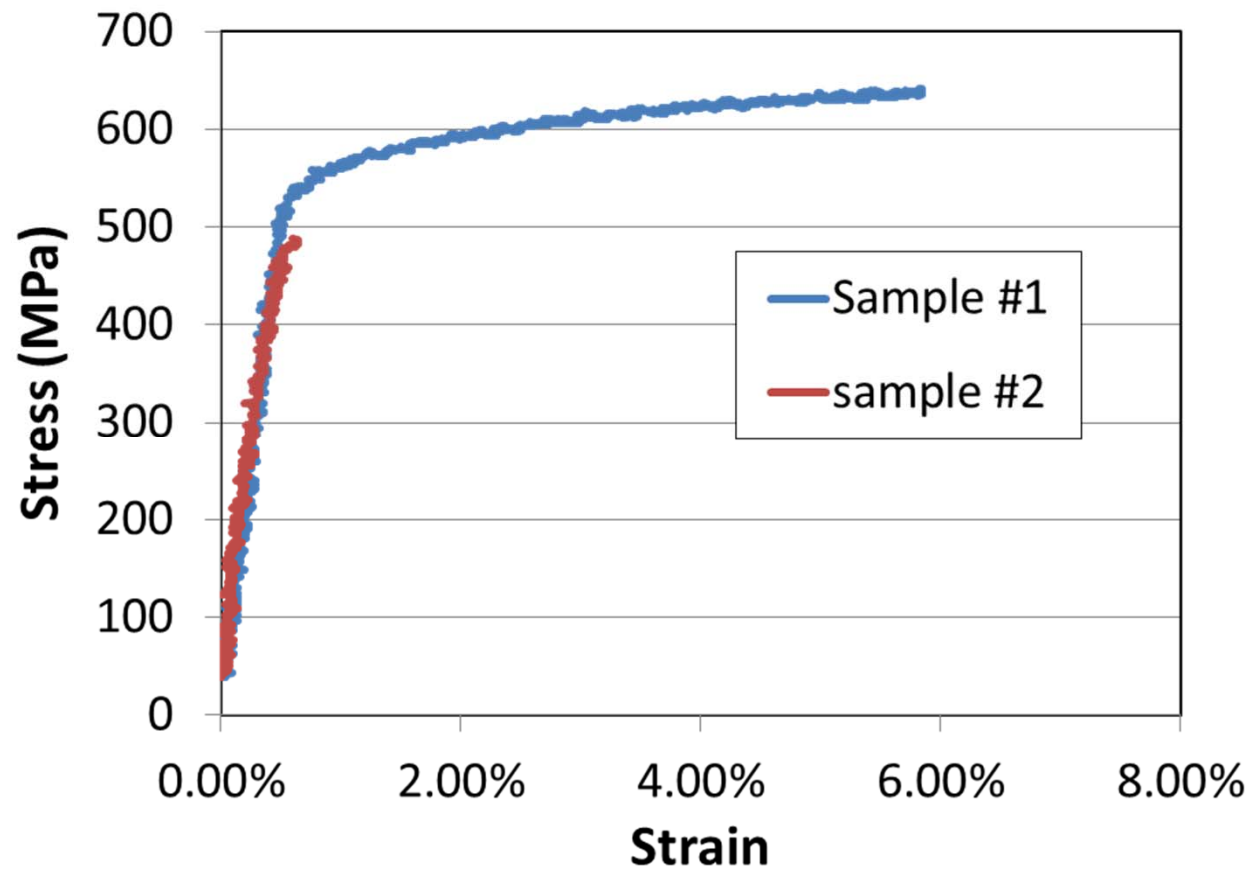
Tensile tests of steel #2 after 1200 °C for 8 h heat treatment



### 3. Ferritic Steels

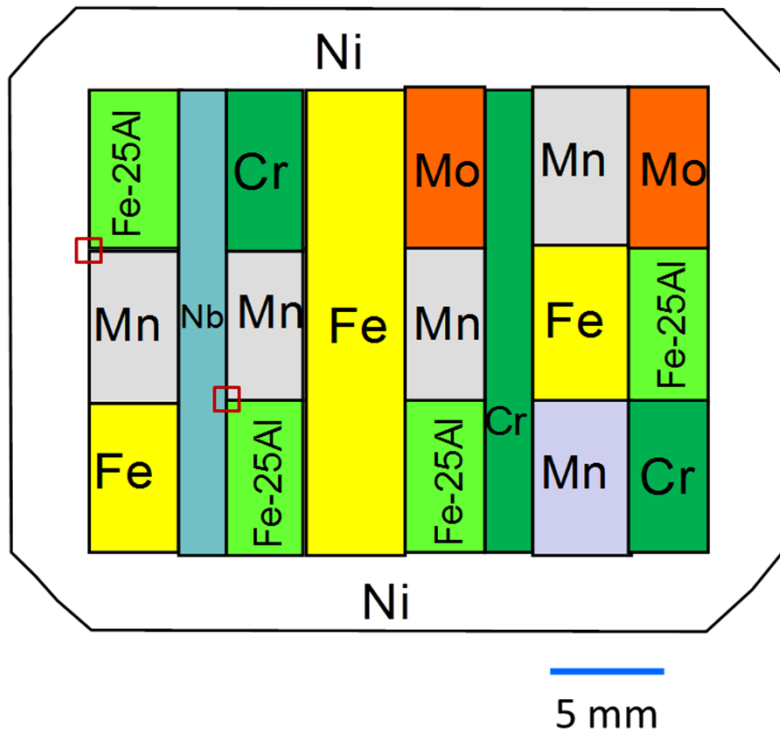
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Tensile tests after heat treatment at  
1200 °C 8 hr + 600 °C 12 hr



# 4. Austenitic Steels

## Diffusion multiple



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

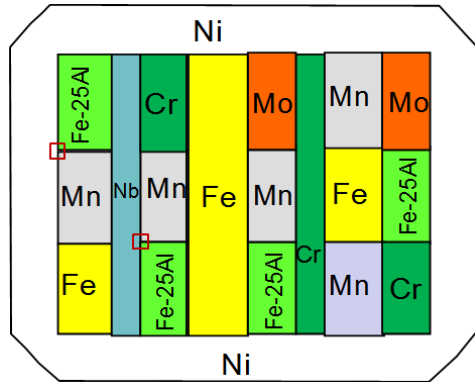
15 ternary systems +  
6 quaternary systems

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

# 4. Austenitic Steels

## Diffusion-multiple manufacturing process



Design



Grind and assemble



EB welding



Mounting/polishing

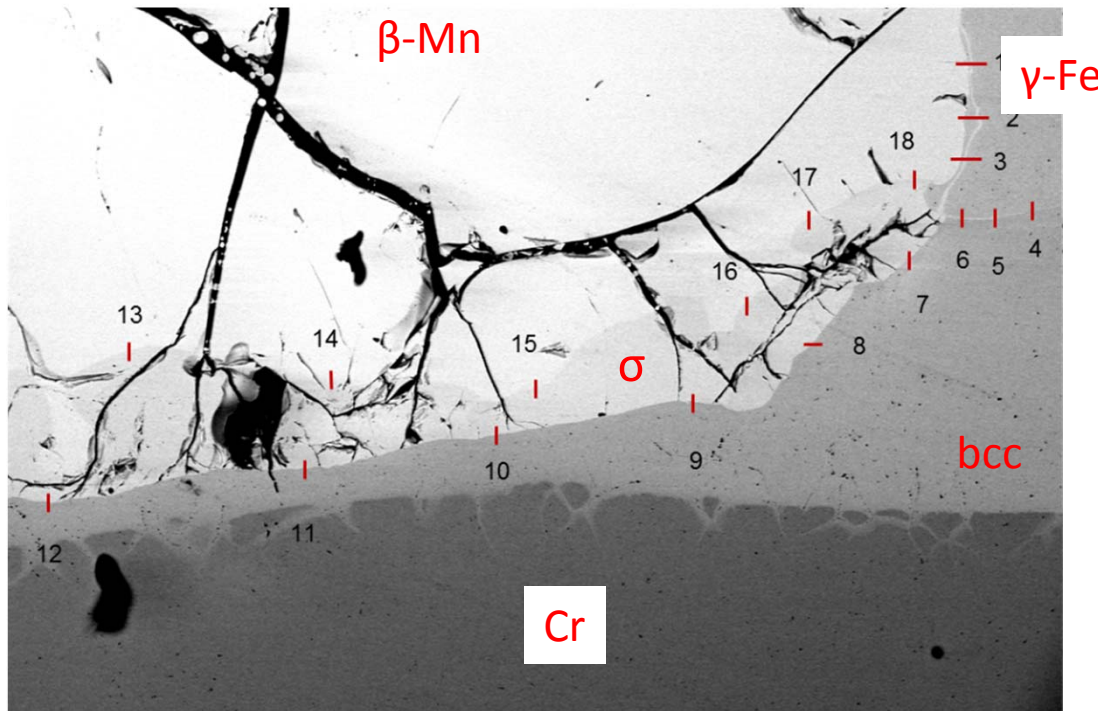


Encapsulation & annealing



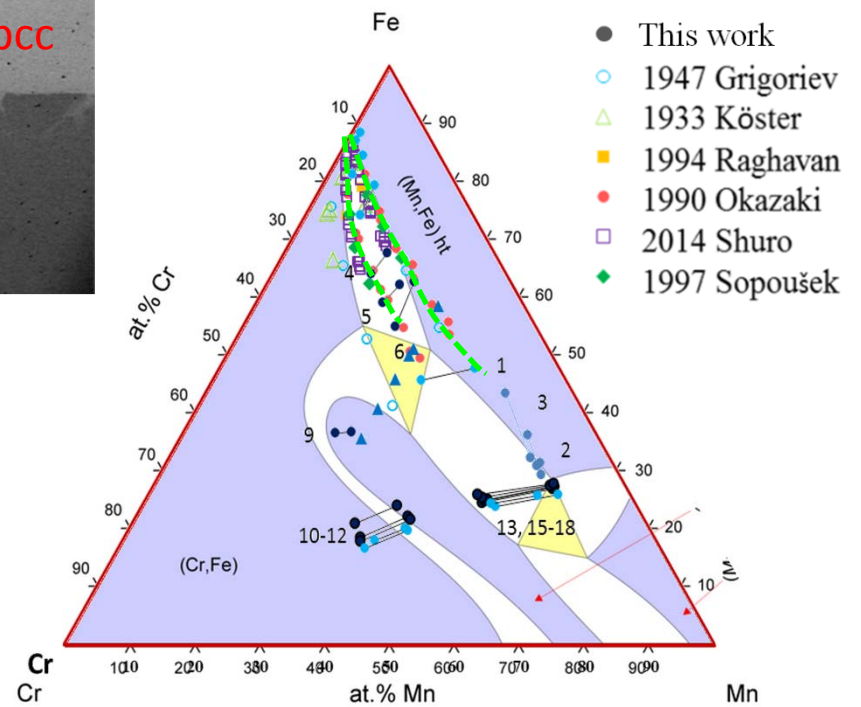
Hot-isostatic pressing

# 4. Austenitic Steels



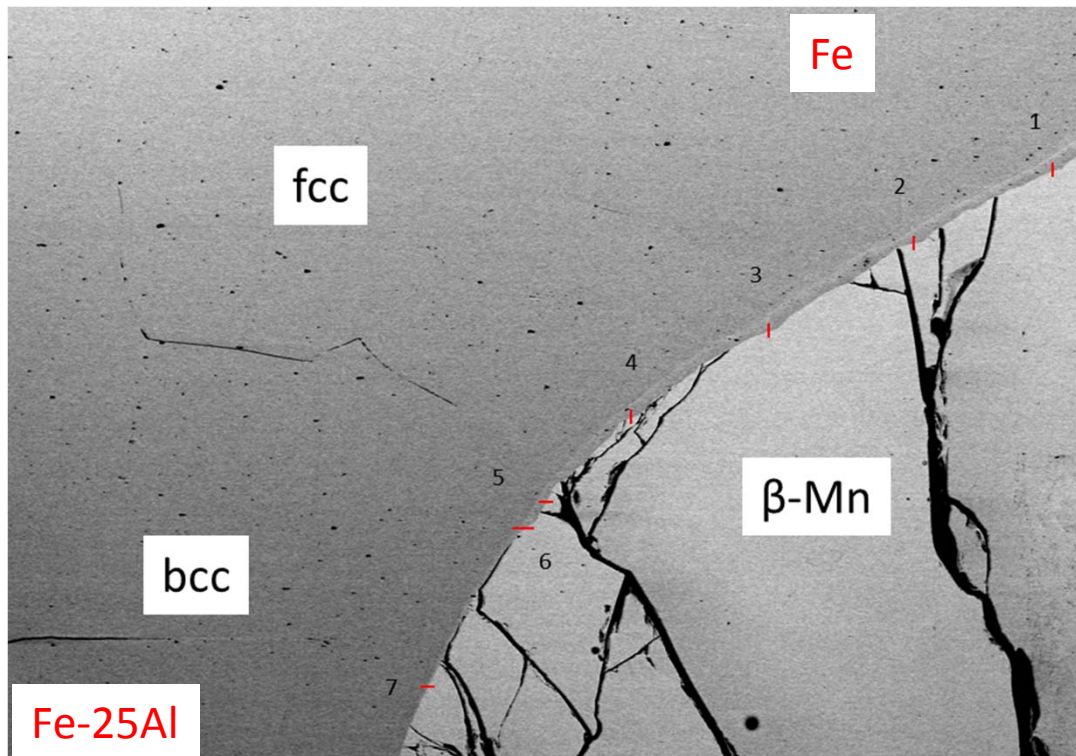
Fe-Mn-Cr (1000 °C 500 hrs)

Phase equilibria:  
Fe-Mn-Cr

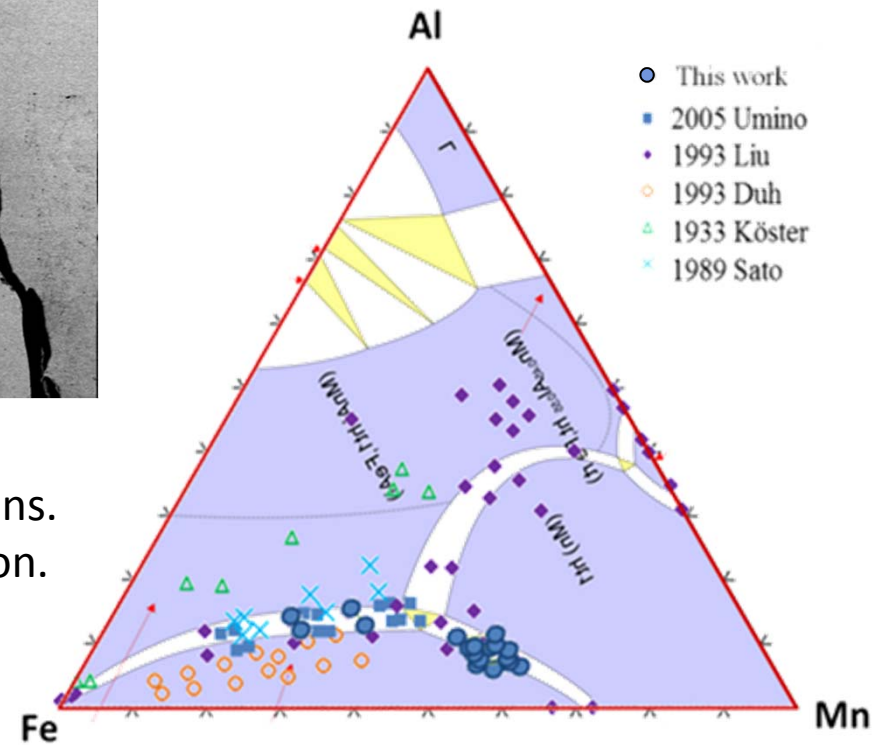




# 4. Austenitic Steels

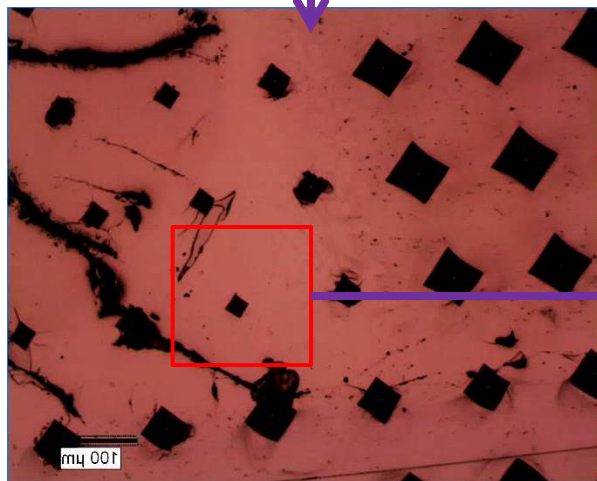
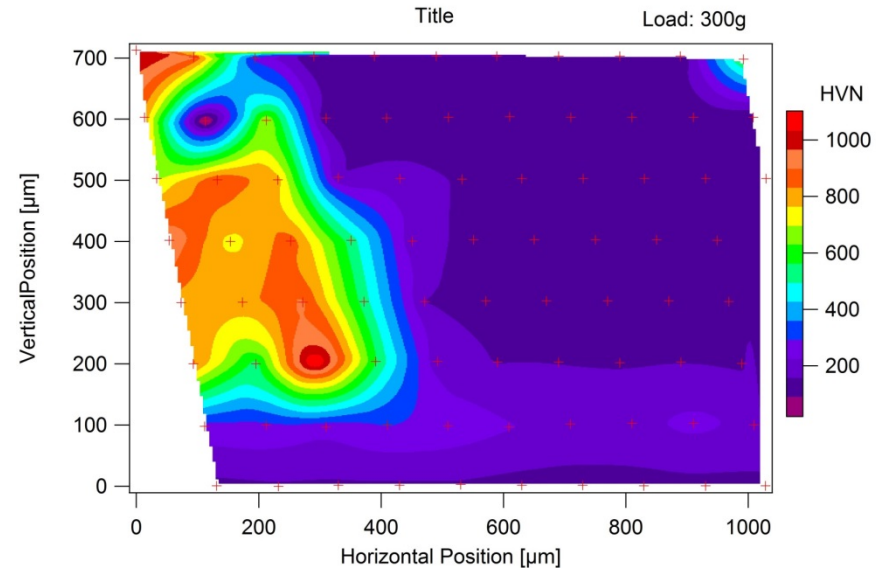
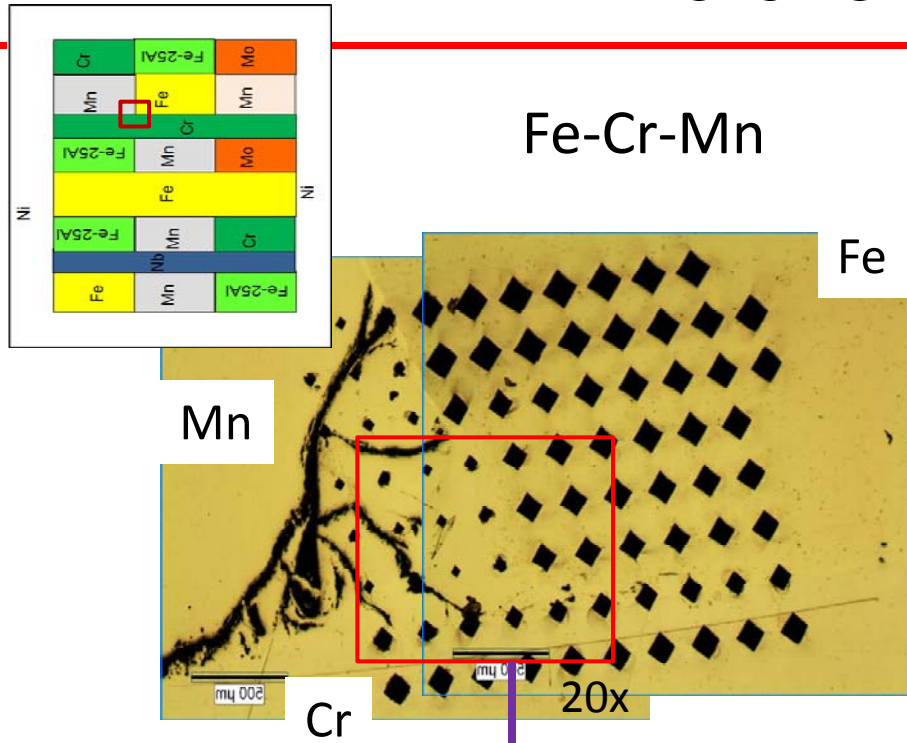


Fe-Mn-FeAl ternary junction

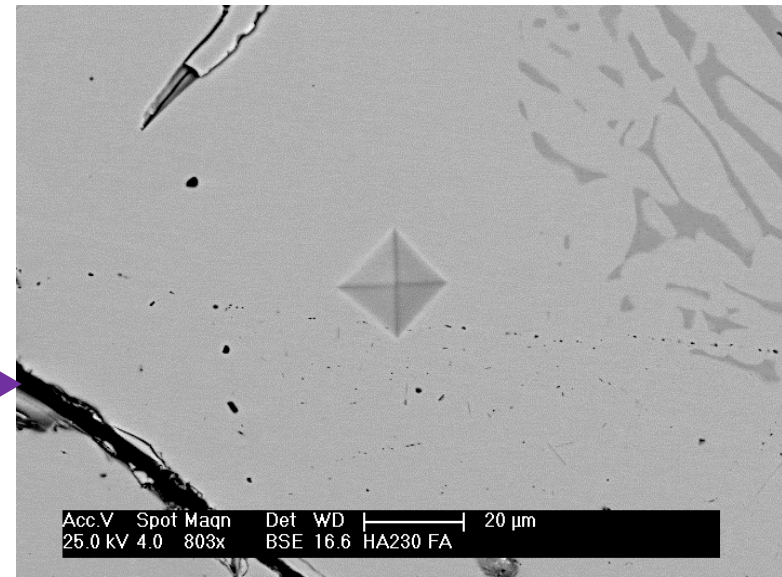


- Efficiently establish the phase-equilibria relations.
- High fidelity thermodynamic database validation.
- Guidance to the design of high-Mn austenitic steels.

# Microhardness map

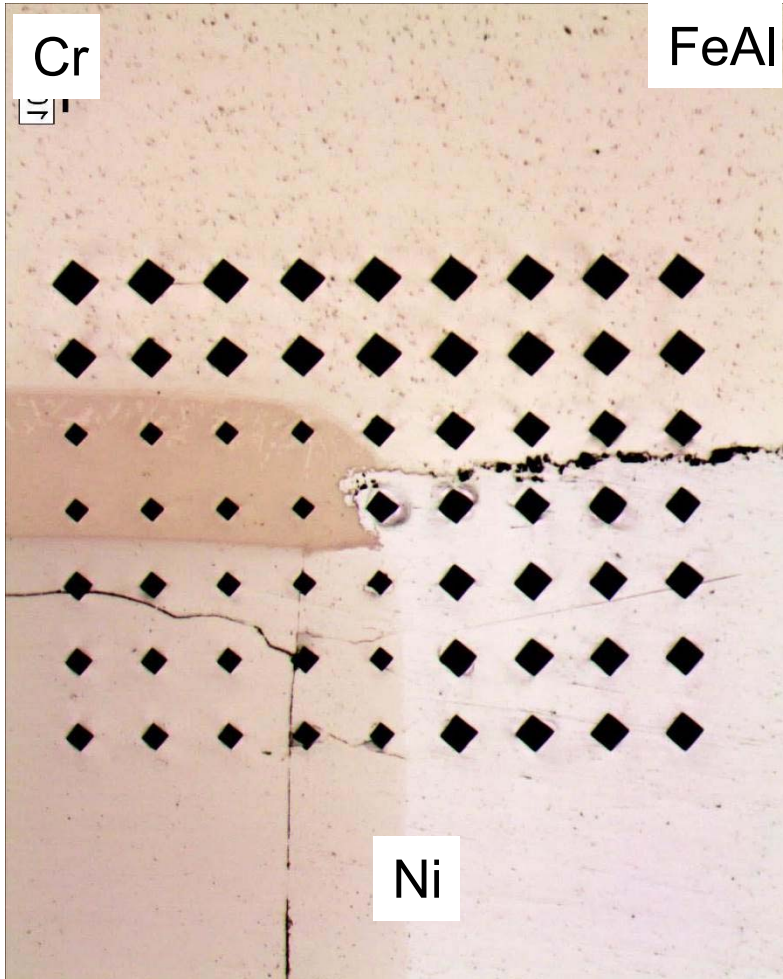
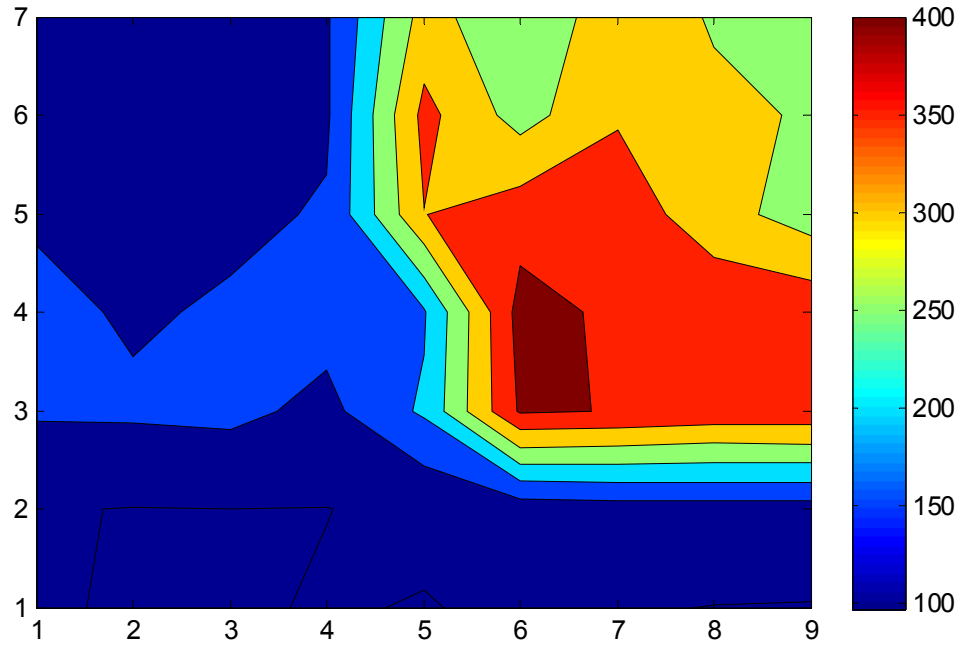


SEM

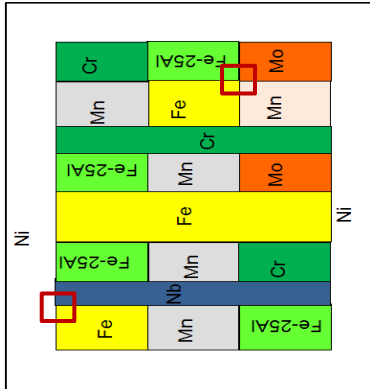


	Cr	Fe-25Al	Mo
Mn	Fe	Mn	
Fe-25Al	Mn	Cr	Mo
Ni	Fe		Ni
Fe-25Al	Mn	Cr	
Fe	Nb	Mn	Fe-25Al

# FeAl-Ni-Cr



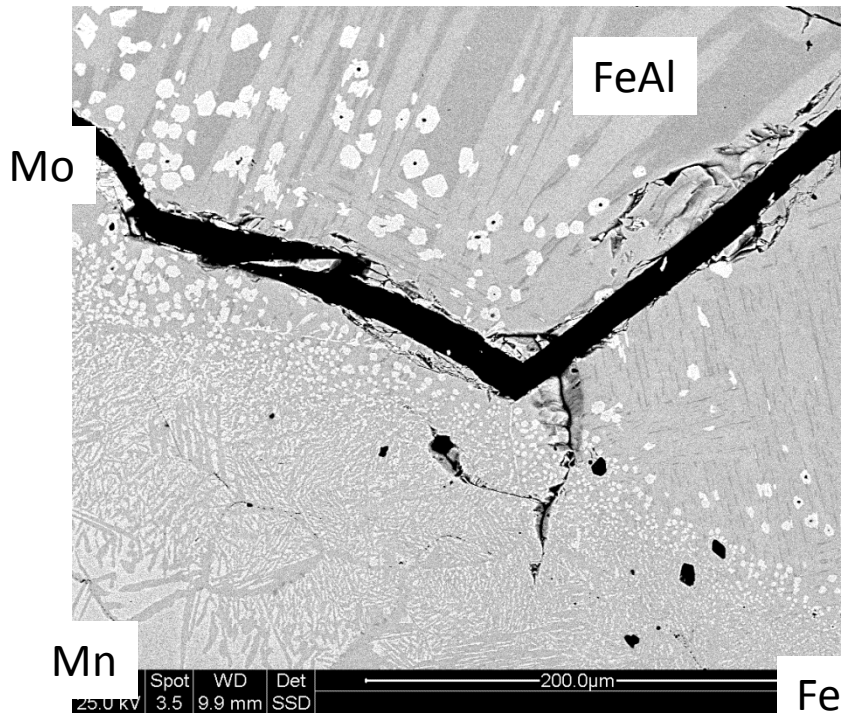
# 4. Austenitic Steels



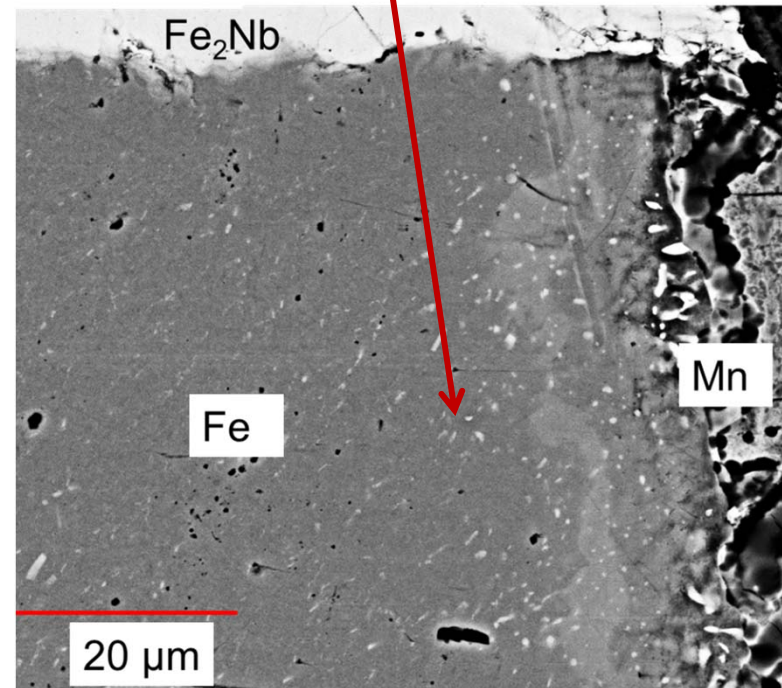
Dual-Anneal Diffusion multiple: precipitates screening

Annealed at 1000°C for 500 hrs+760°C for 200 hrs

**Fine Fe<sub>2</sub>Nb laves-phase precipitates**



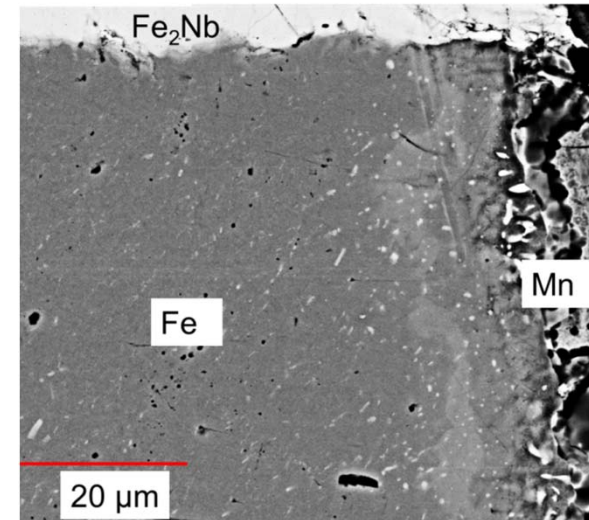
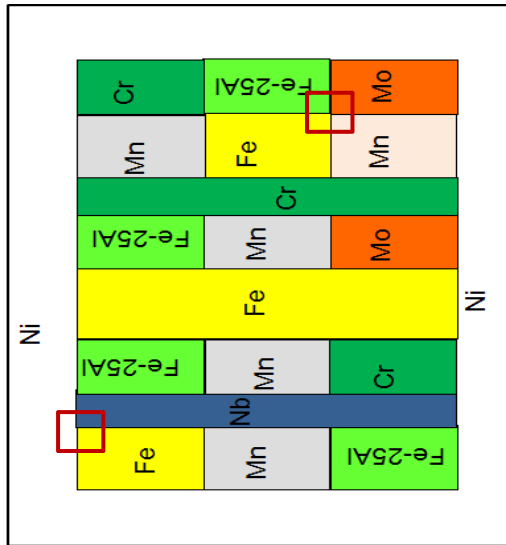
FeAl-Mo-Mn-Fe quaternary system



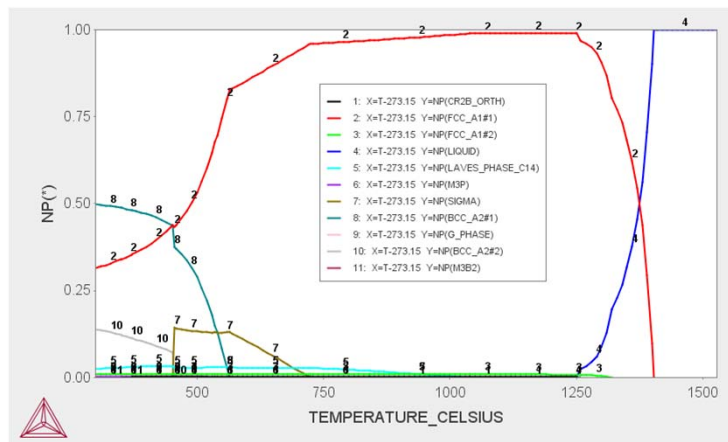
Fe-Mn-Nb ternary system

# 4. Austenitic Steels

Dual-Anneal Diffusion Multiple → Viable Strengthening Phase



Computational Thermodynamics → Design of Alloys for Property Balance



- Multicomponent alloys
- Volume fractions of phases
- Grain boundary phases
- Oxidation resistance consideration
- Cost and other requirements

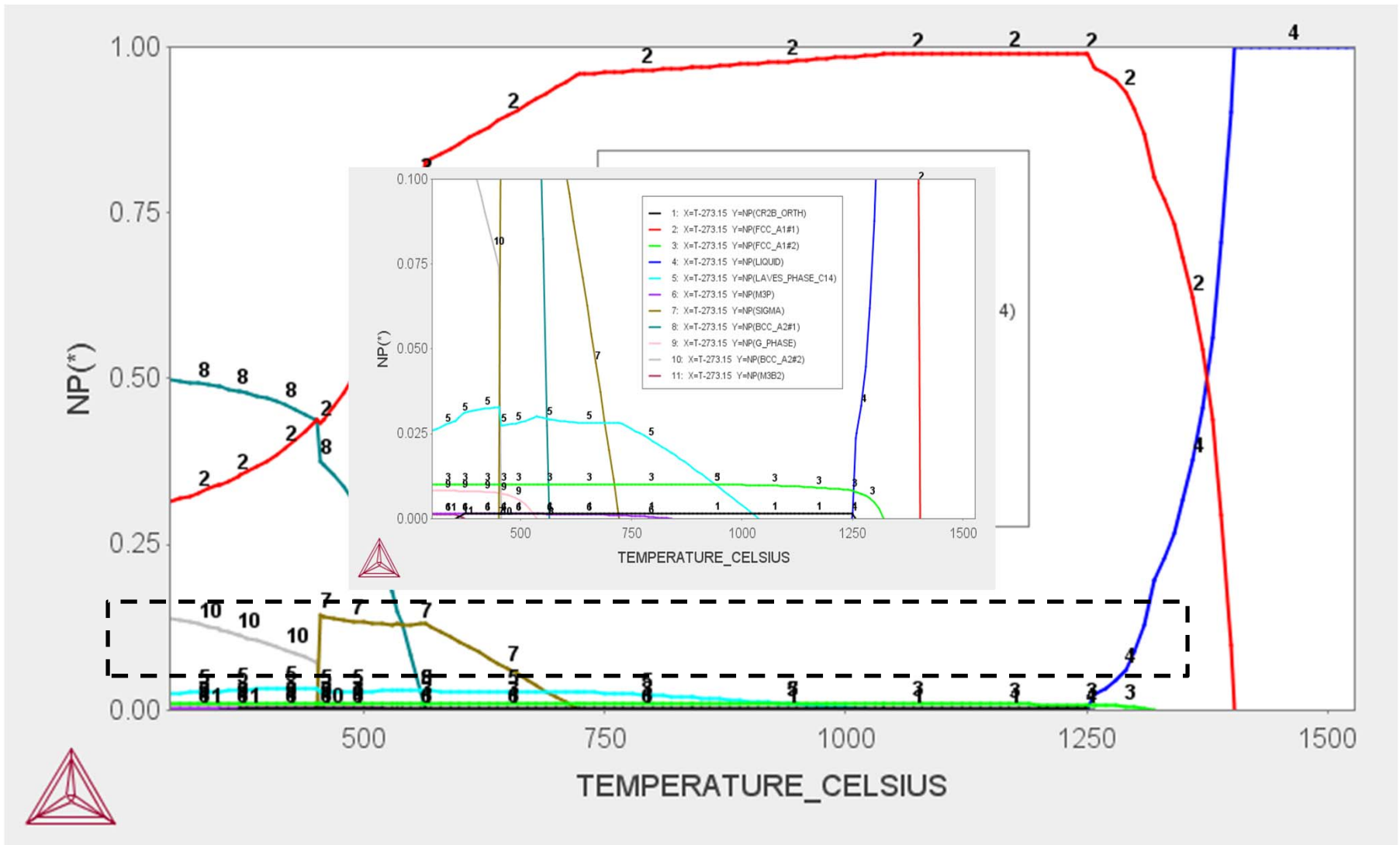
## 4. Austenitic Steels

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### Design of Laves-strengthened Mn-containing austenitic steels

- Target use temperature: 760°C.
- Bcc/fcc transition temperature < 760°C; sigma phase start temperature < 760°C.
- Enough Cr (or Al) for a good oxidation resistance at target temperature.
- Optimal Nb content
- Additional of Mo or W for creep resistance enhancement
- C, B, P for grain boundary engineering during casting and forging
- TCFE7 database was used in Thermo-Calc for the thermodynamics calculation. JMatPro was also employed to check certain calculations

# 4. Austenitic Steels



## 5. Summary & Future Work

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### **Brief Summary of Fe-Cr-Mo Chi-strengthened ferritic 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 alloys too brittle:
  - Need to further reduce Chi phase volume fraction
  - Need to engineering grain boundaries
- More work is needed to figure out the cause of brittleness



## 5. Summary & Future Work

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

- Fe-Mn-Cr-Al-Ni-Mo-Nb diffusion multiples made to effectively explore potential strengthening phases in Mn-containing austenitic steels
- Significant amount of phase diagram information related to high Mn and high Cr (or Al) austenitic compositions was obtained from the diffusion multiples
- Fe<sub>2</sub>Nb-based Laves phase was identified as a viable strengthening phase in Mn-containing austenitic steels
- A new multicomponent Mn-containing and Laves phase strengthened steel is designed and made
- Precipitation kinetic study and property testing are underway

**Thank you!**

