Task Number 5.0
Simulate and Manufacture Large-Scale Ingots and Summary of Ni-Alloy Development

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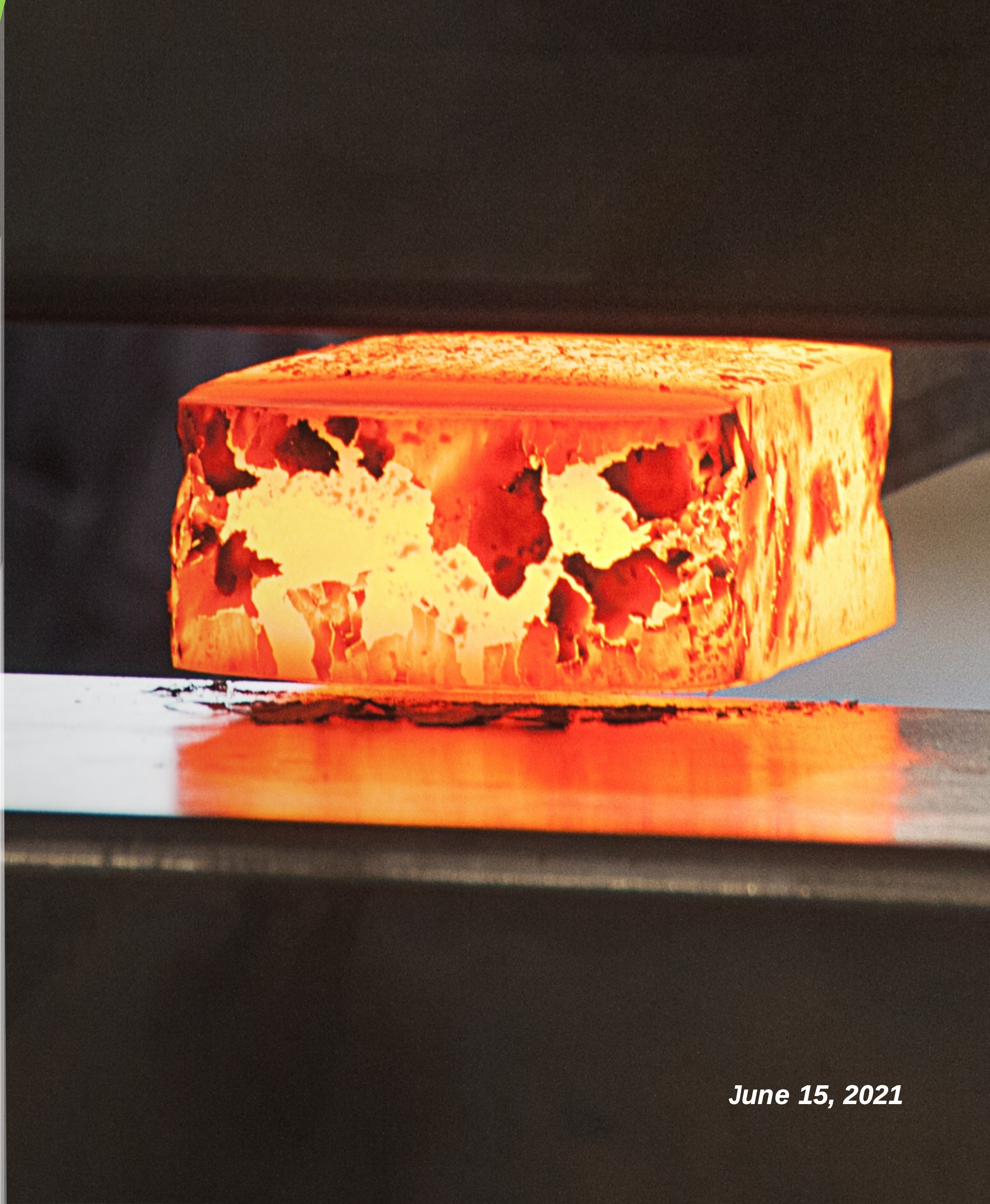

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Acknowledgment and Disclaimer

Task 5: Simulate and Manufacture Large-Scale Ingots

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Objective

Task 5: Simulate and Manufacture Large-Scale Ingots

Research Objective

- Optimize the industrial melting processes used for advanced alloys by (1) induction melting (IM), (2) Vacuum Induction Melting (VIM), (3) Vacuum Arc Remelting (VAR), and (4) Electro-Slag-Remelting (ESR).
  - ESR is used in mission critical applications. The goal is to optimize melt parameters and alloy/slag compositions to maximize ingot quality, leading to improved performance & life.
  - Another goal is to improve the cast versions of wrought alloys for large scale, complex components.
- Support commercial sector by troubleshooting complex industrial manufacturing processes using NETL’s Melt / Fabrication Laboratory for advanced alloys in NETL HPM Program.
- Expand the supply chain by providing *scale-up* ready advanced alloys for the commercial sector, thereby increasing likelihood of transfer to U.S. industry.
- Provide material/processing insights on manufacture of cutting-edge superalloys (pre-competitive).

Alignment with High-Performance Materials Program Goals

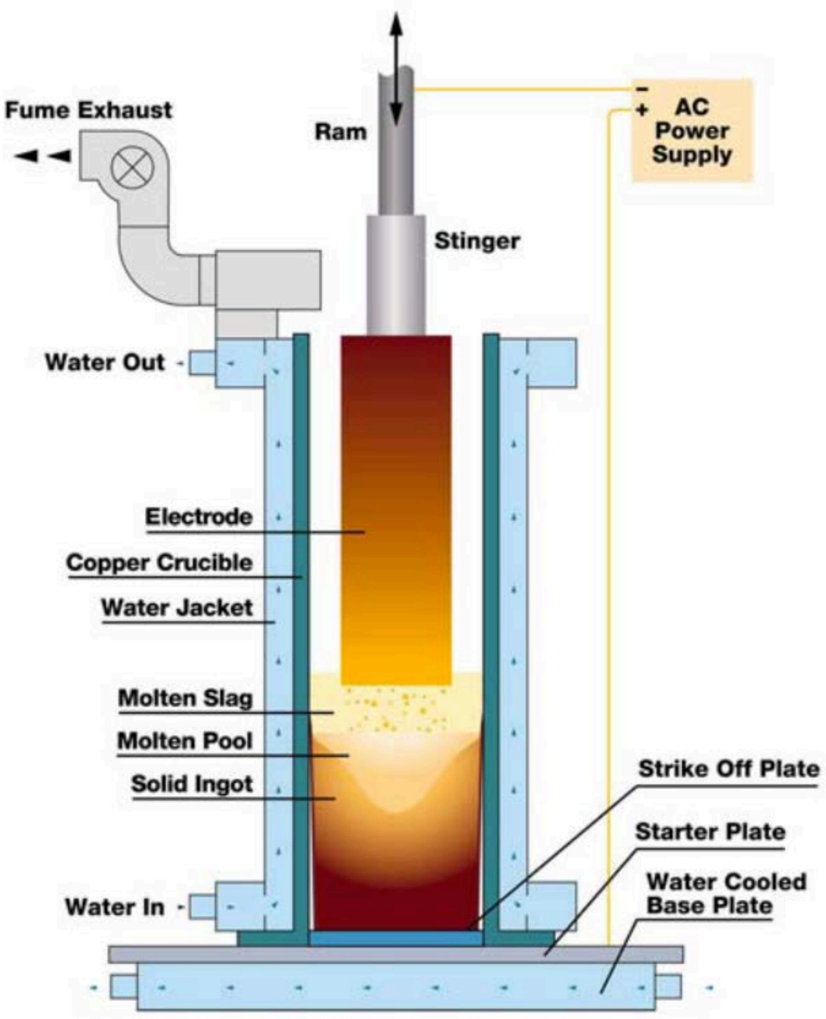
- Supports ***domestic advanced alloy supply chain*** through improved melt practice & develops advanced affordable alloys for use in ***next generation Natural Gas Combined Cycle plants & other Hydrogen economy systems***.

Background

Task 5: Simulate and Manufacture Large-Scale Ingots

ESR Melt Introduction:

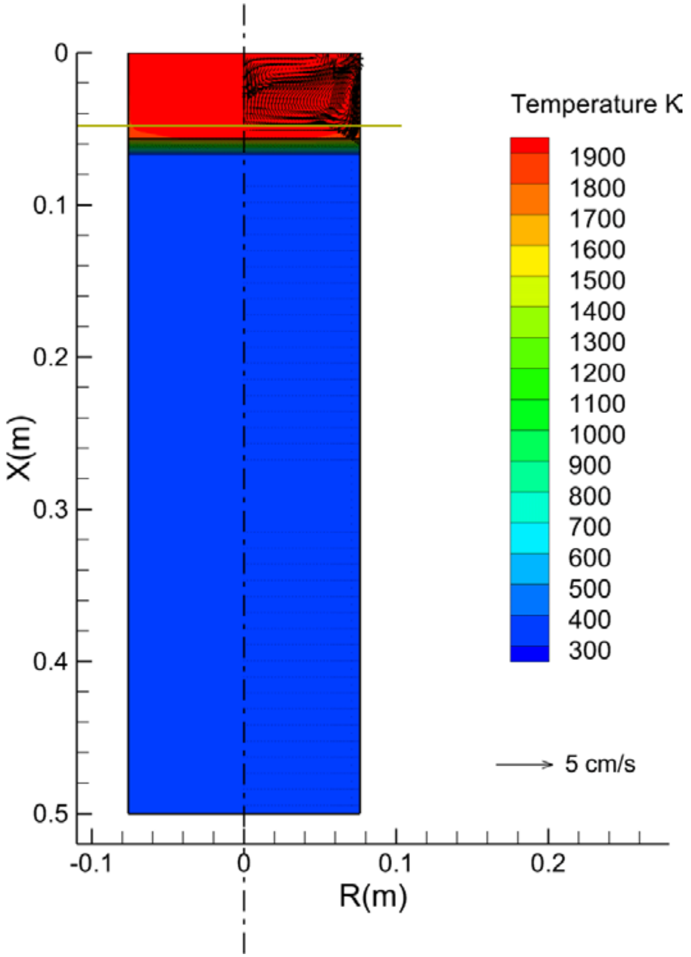
- ESR is a widely used process to produce materials in which cleanliness & quality is critical.
- A consumable electrode is cast using VIM and placed in a water-cooled crucible that contains a slag.
- Electrical current passes from the electrode through the slag to the bottom of the crucible.
- Liquid metal droplets travel from the bottom of the electrode to the crucible where the ESR ingot forms.
- The droplets are superheated, and reactions occur leading to the removal of tramp elements.

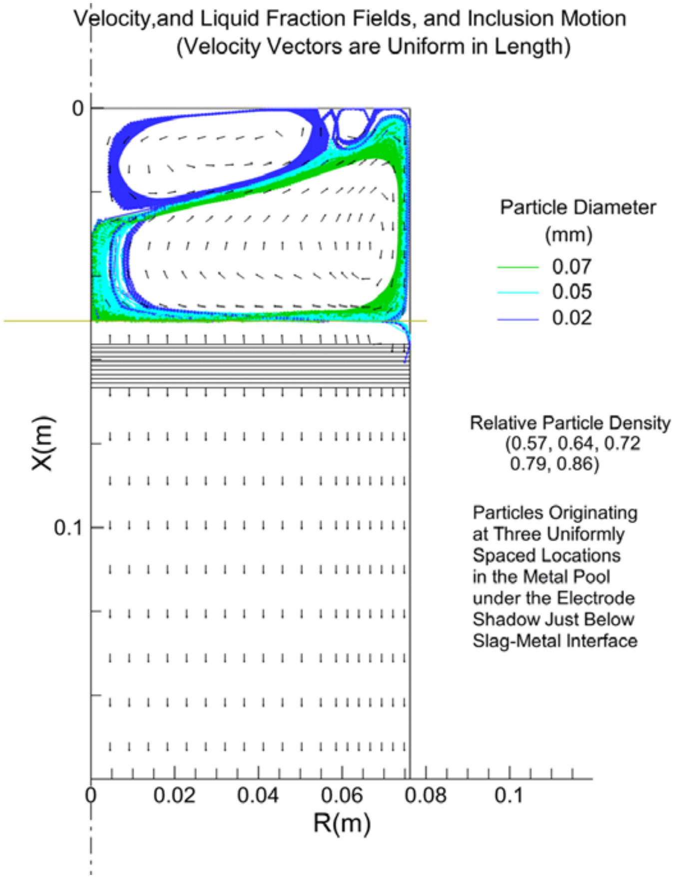


ESR Melting: Simulation and Experiments

Progress and Accomplishments: Process Optimization

- Combine CFD (MeltFlow) & CALPHAD (JMatPro, Thermo-Calc) combined to guide experiments & validate results.
- Modeled and demonstrated that minor alloy additions improved efficiency during ESR melt processing of various alloys.
- Modeling was used to identify melting conditions that result in power reduction and removal of detrimental tramp elements (sulfur, oxygen).
- Methodology developed to predict segregation during ESR melting as a function of process parameters (i.e., slag temperature, melt rate, fill factor, etc.). Important for alloy element retention and tramp elements control.





Status and Summary

Progress and Accomplishments: Process Optimization

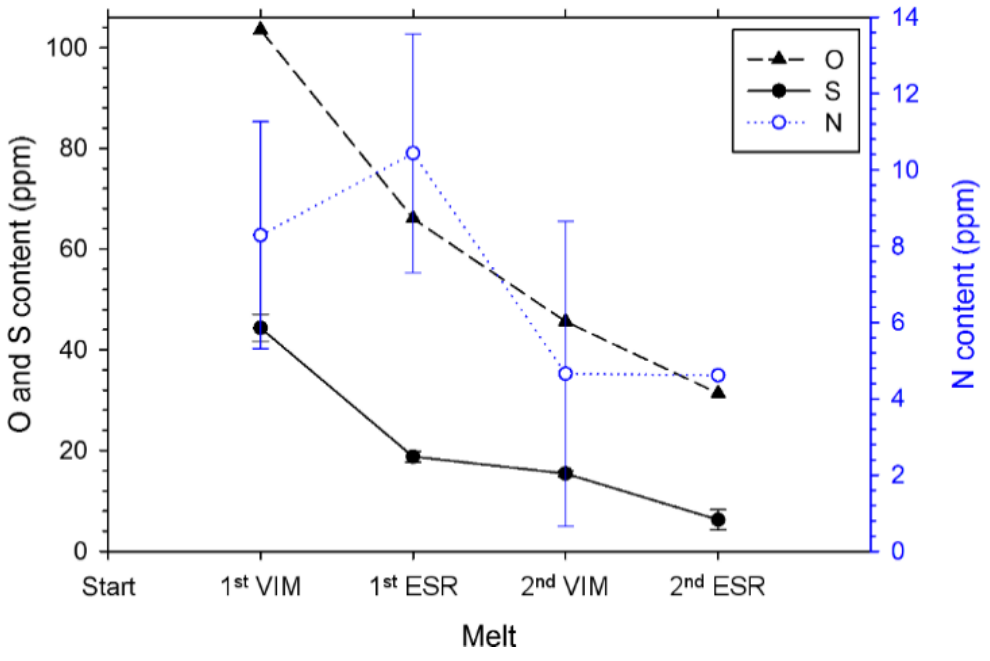
By applying models and careful melt control:

- Tramp element concentrations in ESR ingots can be reduced to desired levels.
- Tight element concentration ranges in advanced experimental alloys can be controlled.
- Yield for advanced & commercial alloys (e.g., CPJ7, JMP, XMAT 347, AFAs, etc.) can be improved.
- Reduction in power required to melt at a constant melt rate for master alloy production can be achieved.

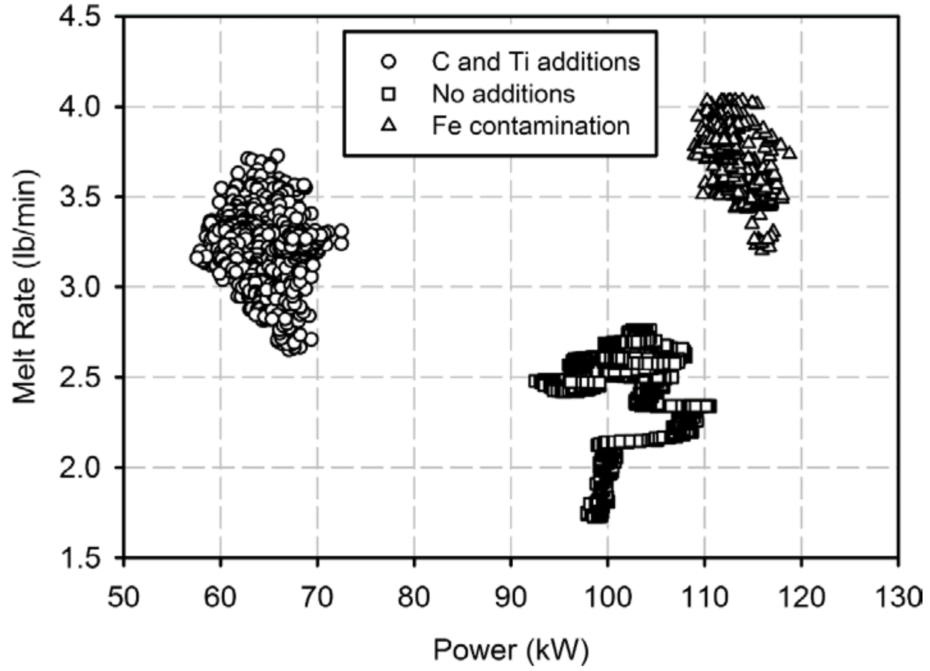
Removal of Unwanted Tramp Elements

Progress and Accomplishments To Date

- Successfully developed techniques and protocol for tramp element reduction/elimination via ESR.
- Demonstrated the benefits of minor additions of targeted elements (e.g., C, Ti) to improve the remelting of alloys.
  - The melt range of the alloy and volume of the mushy zone were extended.
  - Increased efficiency resulting in a >40% reduction in the power required to melt at an approximately constant melt rate.



Concentration of O, S and N following each step of melting.

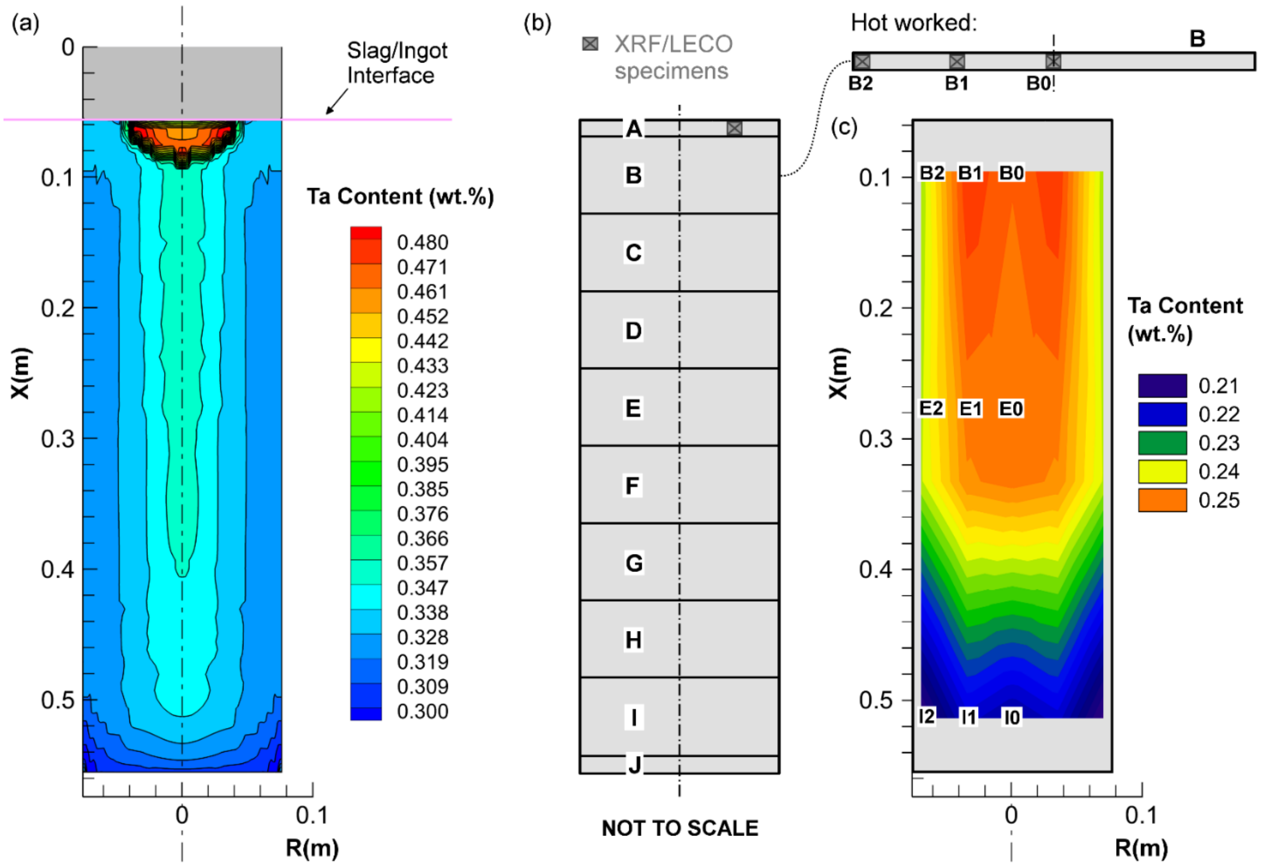


Melt rate as a function of power with minor additions.

Retention and Control of Key Elements

CFD and CALPHAD were combined with experiments to retain Ta in Advanced 9Cr Steel

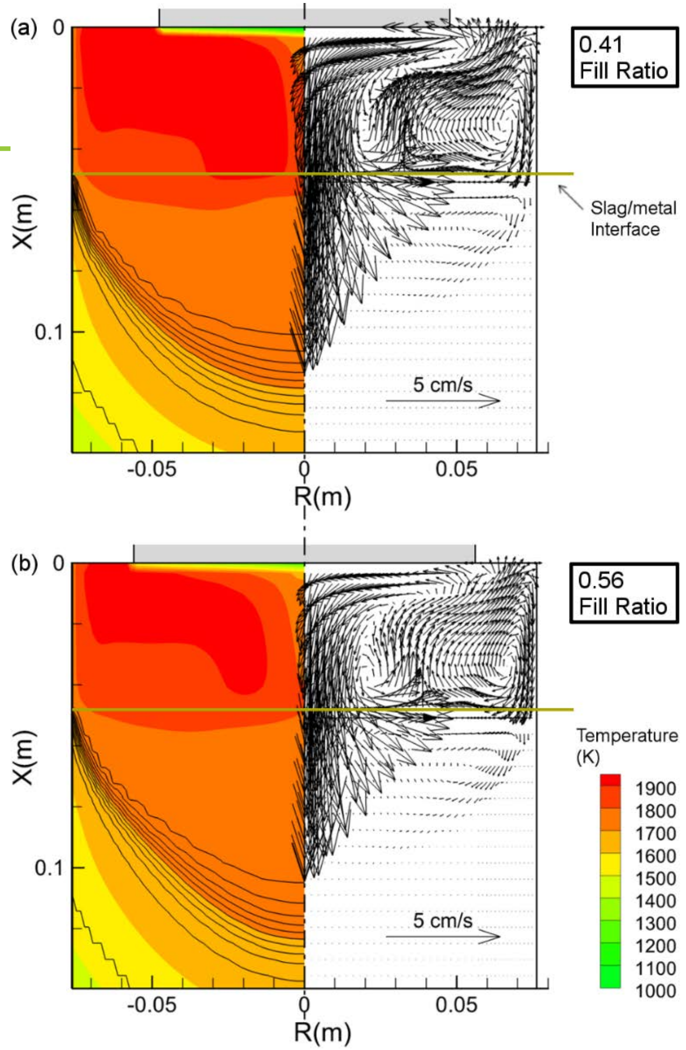
- Initial melt trials using our best methods resulted in unacceptable high loss of Ta.
- MeltFlow-ESR was used to predict the Ta concentration in the ingot.
  - A concentration profile was obtained for Ta in CPJ7.
  - Metallurgical investigations revealed Ta oxides were forming in VIM.
  - Inclusion trajectories were mapped for a range of densities and sizes.
- Based on these findings, the ESR process was adjusted so that the correct Ta concentration could be preserved for CPJ7, an advanced 9Cr steel.



Impact of Fill Ratio

Progress and Accomplishments To Date

- **Impact of electrode/crucible area ratio (fill ratio):**
    - Data input from the experiments with geometries (electrodes and crucibles)
- **The liquid velocity fields show two flow cells:**
    - One underneath the electrode with counterclockwise flow
    - One closer to the crucible wall with clockwise flow
- **Higher velocities are predicted underneath the electrode and at the slag/liquid interface**
- **Overall higher temperatures and liquid velocities during remelting of the smaller diameter electrode**
    - Higher current density beneath the center of the electrode as the fill ratio decreases
- **Average molten slag temperature in steady state:**
    - 1637°C for 0.41 fill ratio
    - 1598°C for 0.56 fill ratio



Temperature gradient and velocity field in the slag region and melt pool at steady state during ESR of 316 stainless steel with (a) 0.41 and (b) 0.56 fill ratios.

6in diam.
ESR Ingot

Simulate & Manufacture of Large-Scale Ingots

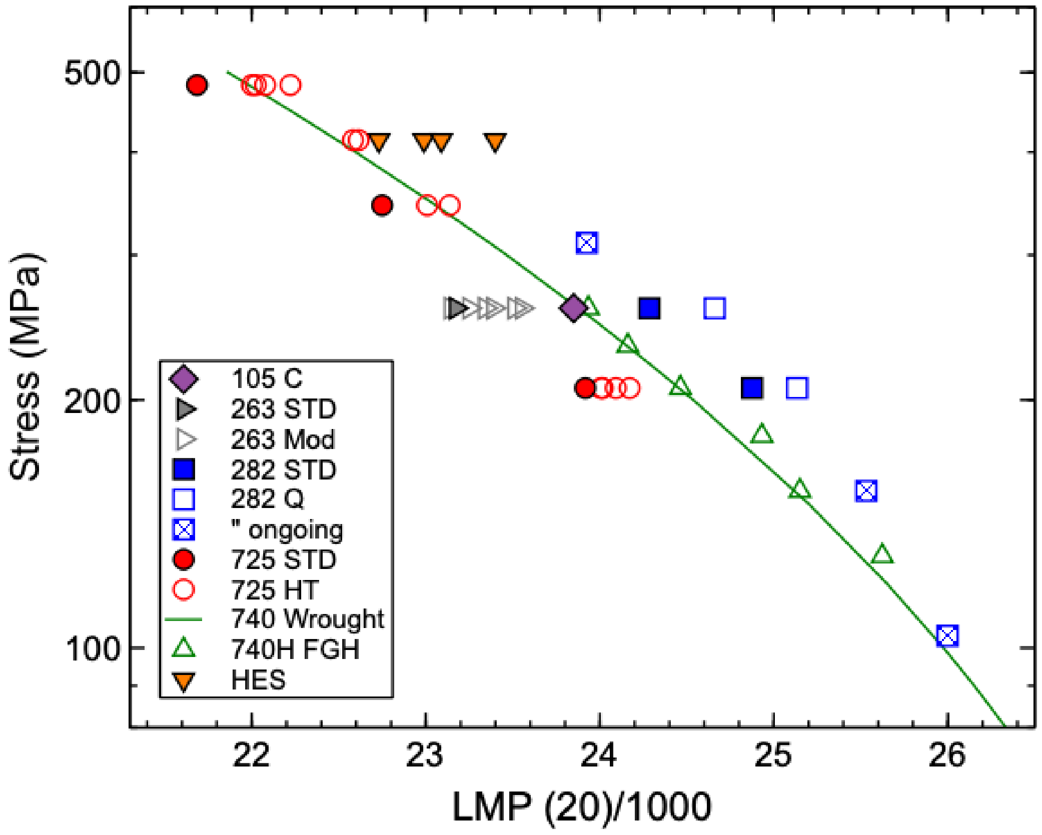
Ni-Alloy Development

Current Effort

Apply models to Ni-superalloy melting & scale-up:

- Emphasis on NETL developed Ni-superalloys – large range of chemistries & microstructures.
- Provides insights on processability of superalloys (pre-competitive).
- Increase the likelihood of transfer to U.S. industry by using commercial relevant technologies.

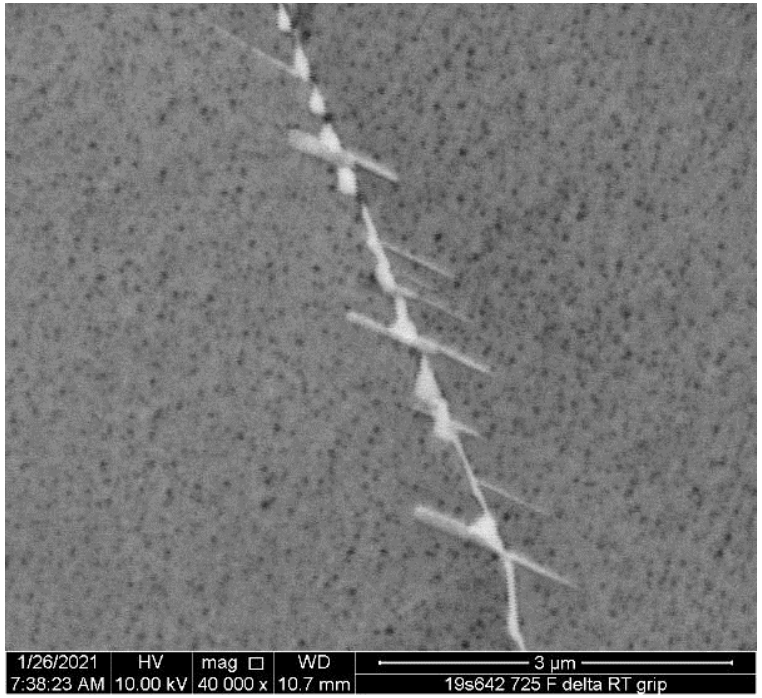
Creep of NETL Superalloys compared to wrought 740H



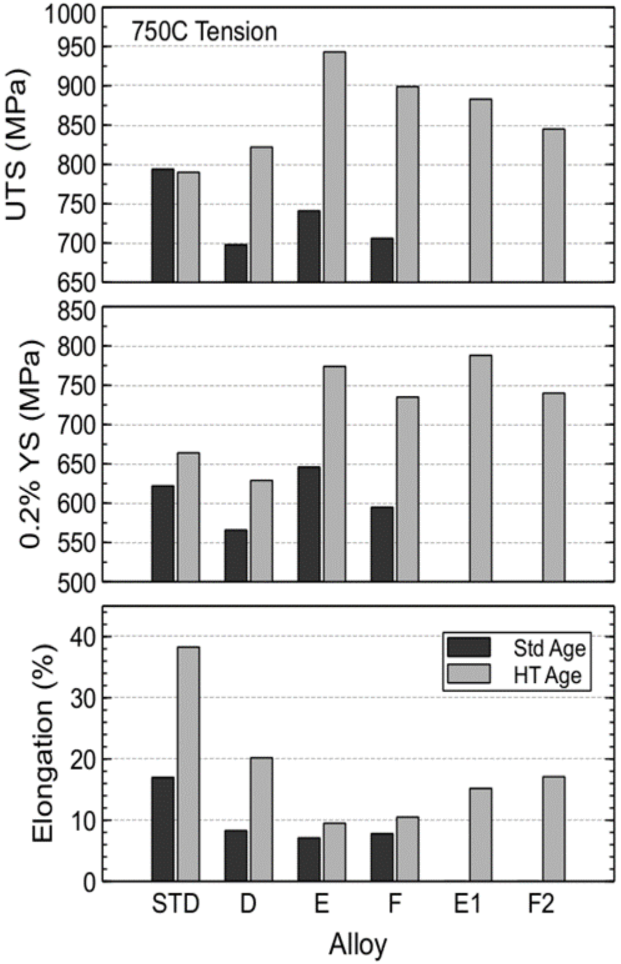
Grain Boundary Re-Design

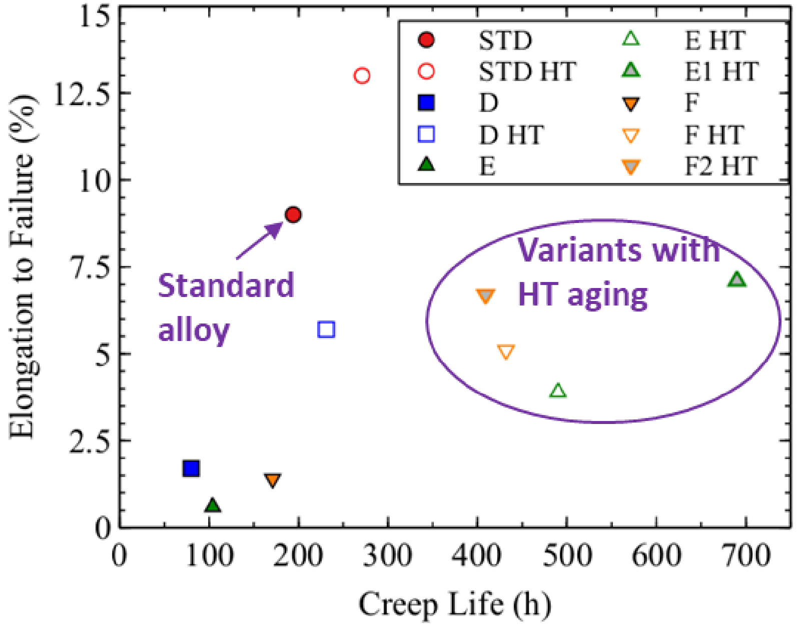
Improving a corrosion resistant alloy: IN725

Changes in the aging treatment and chemistry were designed to promote the complex formation of grain boundary phases and γ′/γ″ precipitates.



Microstructure of alloy F following HT aging in which precipitates are used to promote "grain boundary stitching"





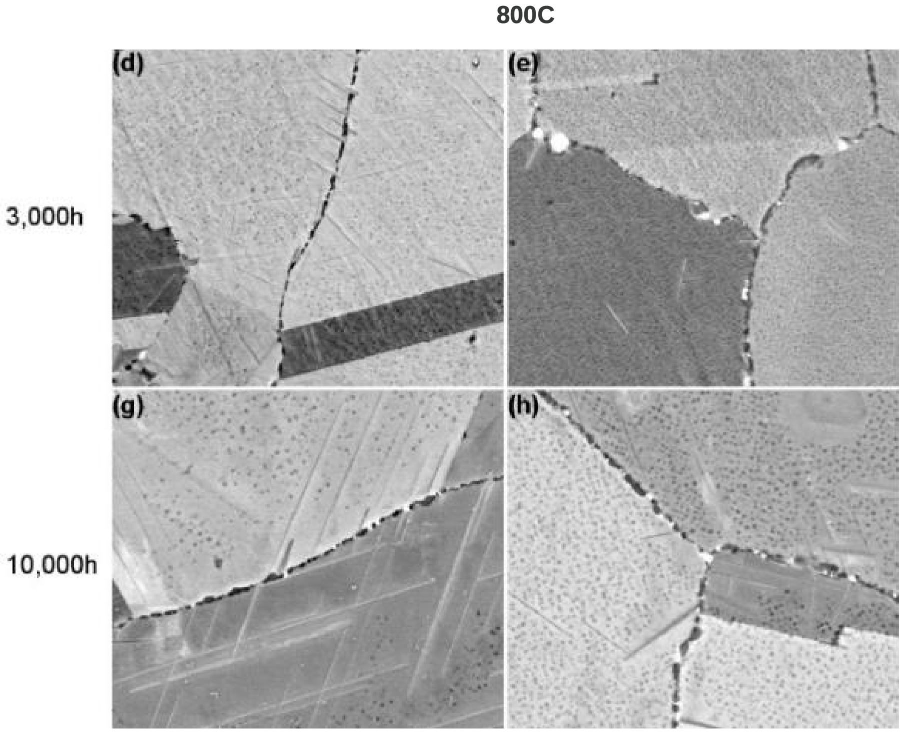
Outcome: Improvement in creep life of >250% with similar ductility from the standard alloy.

M. Detrois, P.D. Jablonski, J.A. Hawk, NiCrMoNb age hardenable alloy for creep-resistant high temperature applications, and methods of making, 63/125,888, 2020 (Provisional Filed: 15 Dec. 2020).

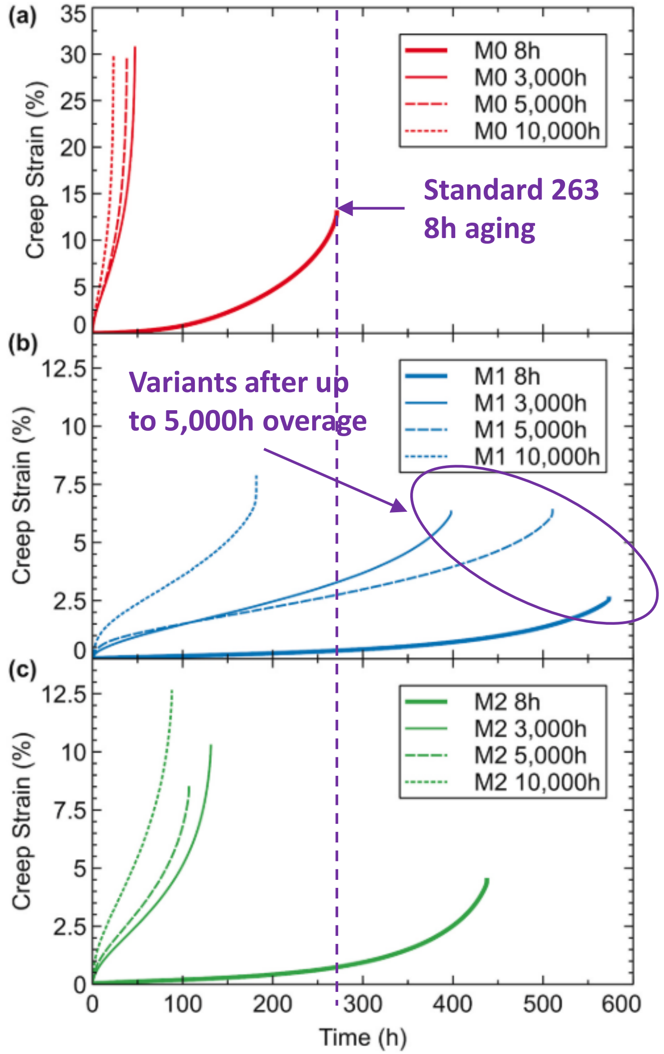
Enhancing Phase stability: Alloy 263

Impacting the phase stability at service temperature of Alloy 263

- Variants of commercial alloy Nimonic 263 were designed to possess higher fraction of γ' precipitates.
- Aging studies evaluated phase stability of the alloys around the service temperature.
- The standard alloy (M0) showed rapid η phase precipitation from the grain boundaries (detrimental to mechanical properties).
- NETL modified alloys (M1, M2) demonstrated much greater phase stability in both cast and cast/wrought forms.



SEM images show the standard alloy M0 on the left and NETL modified M1 on the right.



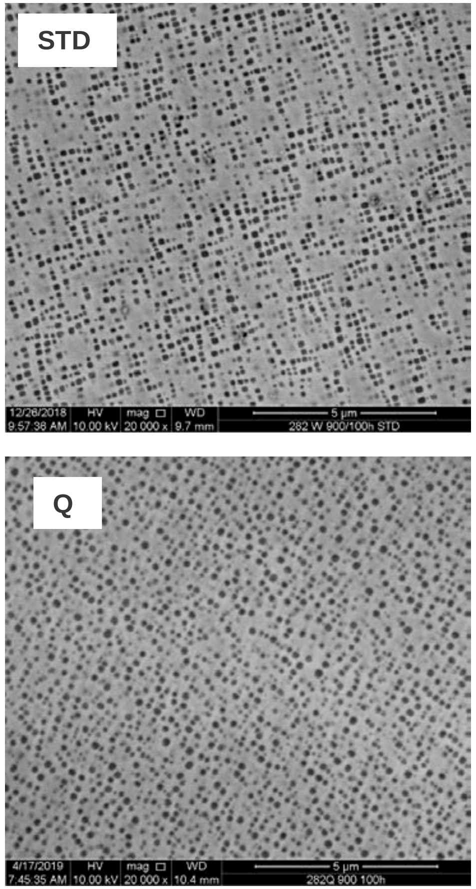
Outcome: **A stronger, more stable version of alloy 263.**

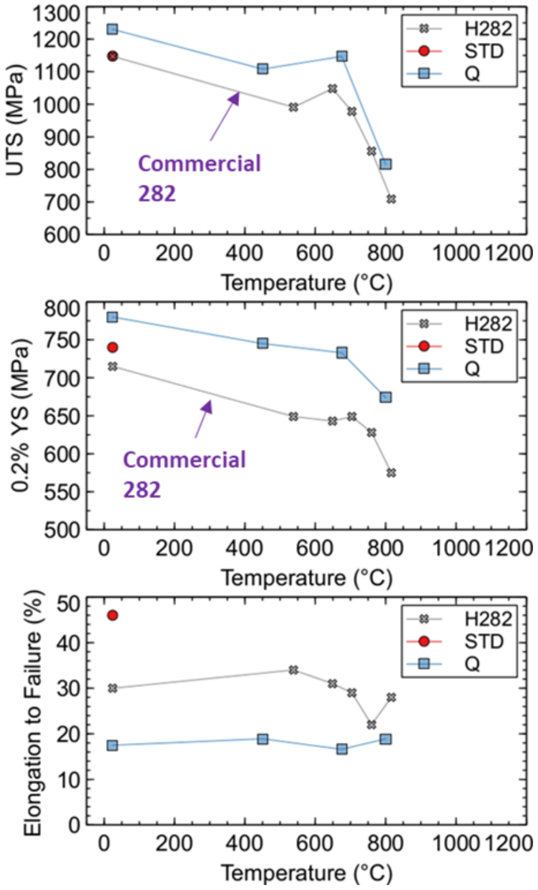
Increasing Strength

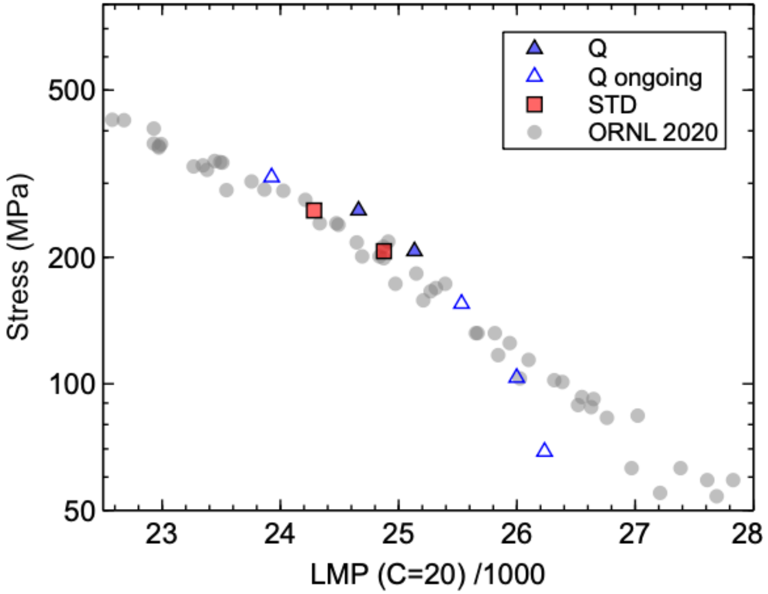
Enhancing γ′ content

- Modifications to alloy 282 were pursued by altering alloy chemistry to strengthen the alloy at ≥ 800°C by increasing γ′ precipitate volume fraction.
- The gamma prime fraction/solvus at 900°C was designed to equal that of the commercial alloy at 800°C.
- NETL variant 282 Q has thus far outperformed the commercial alloy by ~130% in creep (measured as creep life).

900°C / 100h





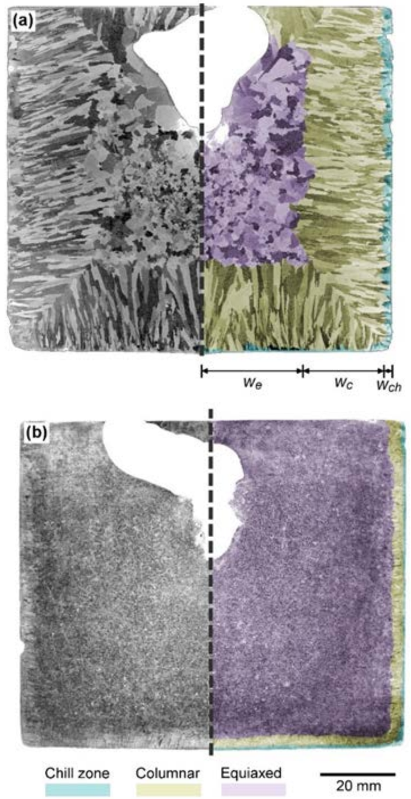


Outcome: Higher Temperature Version of H282

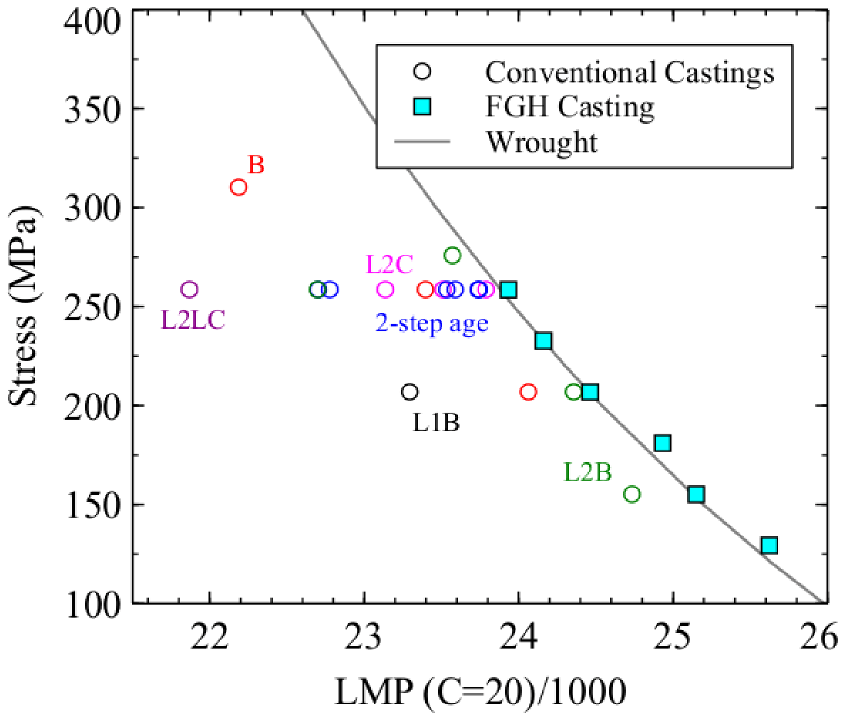
Cast version of 740H

Alloy (and supply chain) options for thick wall castings

Conventional casting
Non-Uniform Microstructure



NETL-modified casting
Uniform Microstructure



Larson-Miller plot for the different castings, conventional and FGH (compared to data from wrought processed Inconel 740)

- Modify the casting process for Inconel 740H to improve creep life.
- Conventional castings (open circles) showed poor and inconsistent creep lives.
- The NETL-process **(FGH)** produced a fine-grain casting with homogeneous precipitation of grain boundary phases to obtain a cast product matching the wrought alloy on the LMP plot.

Outcome: **Cast version of 740H that performs on par with wrought 740H in creep life.**

Current effort: **cast version of 617**

Summary

Progress and Accomplishments To Date

ESR Melting Outcomes:

- Improved melting efficiencies.
- Successful removal of tramp elements such as oxygen and sulfur.
- Tight control of critical elements such as Ta in CPJ 7.
- Ingot yield improvements.

Advanced Ni Alloy Outcomes:

- At least 250% improvement in creep life in IN725-class alloy (provisional patent filed).
- Showed conventional 263 becomes phase unstable with exposure at 800C. Significant improvements can be made by adjusting the chemistry.
- Improvements in creep life (130%) in a 282-class alloy.
- Produced the first cast 740H material with creep properties comparable to wrought 740H (patent application filed).

NETL Alloys Available for Evaluation