Nickel-Base Superalloy Development (Advanced Alloy Development-FWP Task 5)

NATIONAL ENERGY TECHNOLOGY LABORATORY

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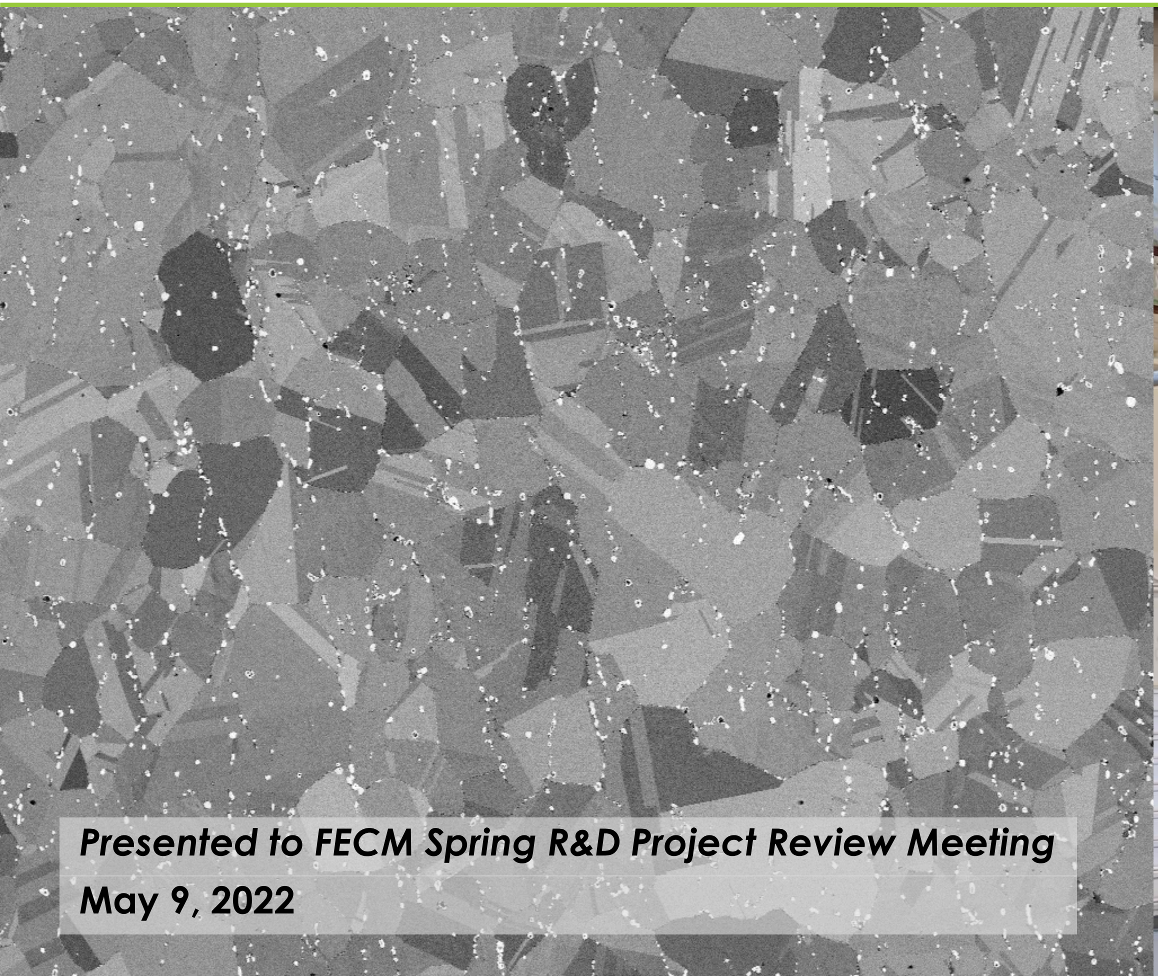

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U.S. DEPARTMENT OF ENERGY

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Introduction

Scope

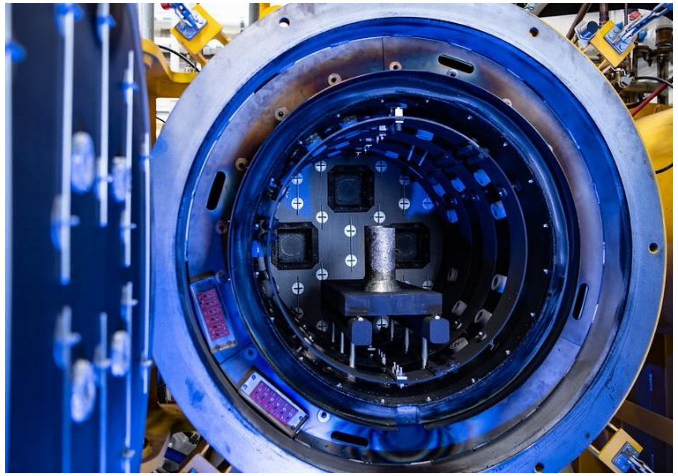
This task drives to design, produce and characterize cost-effective alloys and high-performance materials suitable for extreme environments that are found in next generation energy systems. The aim of this task is two-fold:

- To optimize industrial melting, such as electroslag remelting (ESR), practices used to produce heat-resistant alloys for power systems, thereby, contributing to the cost reduction of advanced alloys for energy systems. **(Outside the scope of this presentation.)**
- To maximize properties of Ni-base superalloys through alloy design and thermomechanical processing (TMP) control, thereby providing alloy solutions that will enable and improve advanced energy cycles. This includes the development of cast Ni-based superalloys from commercial wrought alloy formulations which will expand the supply chain for advanced heat resistant materials.

Introduction

Melt Processing Laboratory

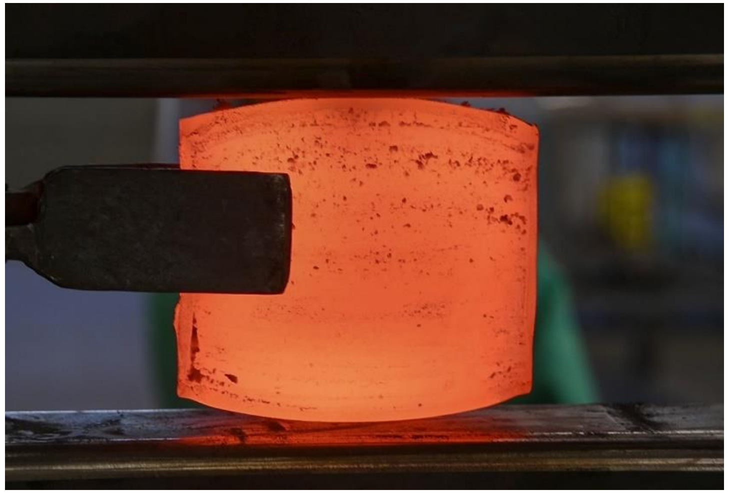
- **3 VIM furnaces for research scale ingots:**
    - 13 (1) and 22 kg (2) crucible capacities.
    - Typical ingots for alloy development at NETL are 8 or 16 kg.
- **Vacuum heat treatment furnaces (2) with forced Ar gas fan cooling for homogenization heat treatment.**
- **Computational homogenization heat treatment design.**
- **Chemistry analysis using XRF (major elements) and combustion (C, N, O and S).**



Introduction

Metals Fabrication Laboratory

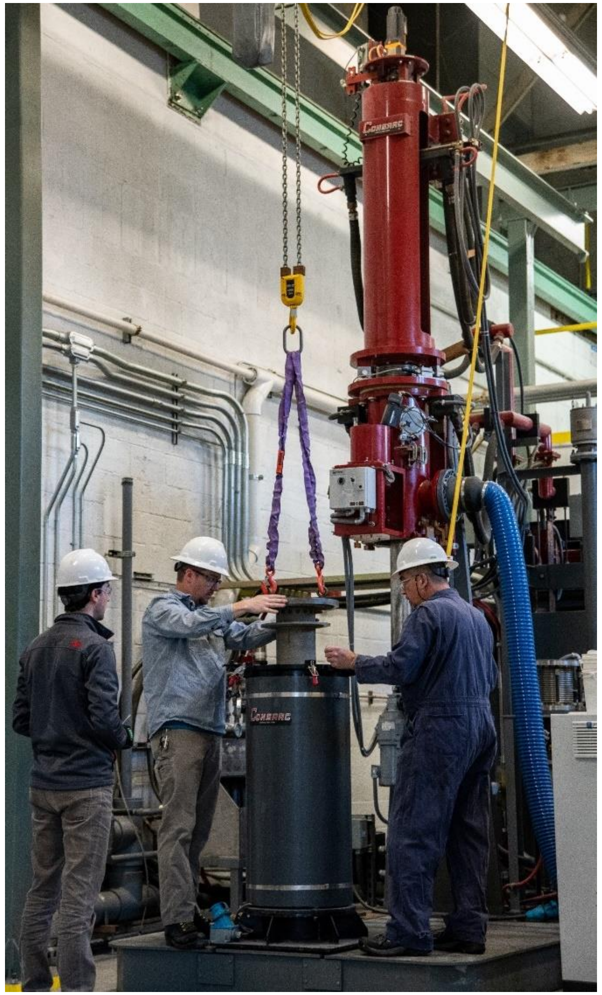
- Following homogenization, the surface of the ingots are prepared on a lathe.
- Hot working consists of steps of forging followed by hot rolling with reheating between each step.
- The cylindrical ingots are transformed into plates for characterization and mechanical testing.
- The cast structure is converted to an equiaxed microstructure during hot working.



Introduction

Scale-Up Capabilities

- **Scale-up capabilities:**
  - 136 kg VIM furnace (retired).
  - New 227 kg VIM furnace.
- **200 kg combination VAR/ESR research scale furnace.**
- **The large-scale VIM furnace is used to produce electrodes for VAR/ESR experiments.**



Effect of Key Elements and Aging in IN725 Variants

Introduction

- Inconel 725 possesses great resistance to elevated temperature corrosion, very good yield stress and tensile strength at temperatures typically <600°C.
- The goal is to improve the elevated temperature mechanical properties (specifically creep) of alloy 725. Approach using compositional design:
  - Variation in the Ti/Al ratio to favor the precipitation of γ′ over that of γ″.
  - Additions of Nb/Ta to promote the formation of fine and stable precipitates (such as δ) along the grain boundaries.
  - Custom high temperature aging (HTA): 800°C for 20h – cooling 1°C/min to 750°C – 750°C for 8h.

Composition of the various heats from XRF analysis for the main elements, combustion analysis for C and calculated from the melt addition for B (wt. %) and calculated Ti/Al ratio.

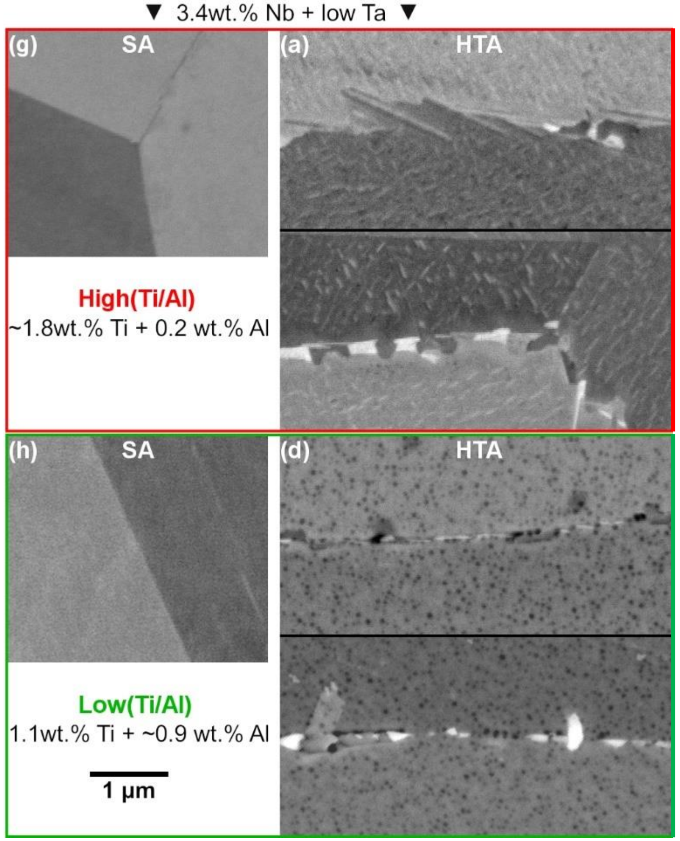
Alloy	Cr	Al	Ti	Nb	Ta	Fe	C	Mn	Mo	Si	B	Ni	Ti/Al
STD	21	0.24	1.82	3.4	0.2	3.9	0.039	0.04	7.1	0.018	0.003	Bal.	7.6
E1	21	0.20	1.87	3.4	**3.6**	3.8	0.049	0.05	7.0	<0.010	0.003	Bal.	9.4
F2	21	0.19	1.88	**4.6**	0.4	3.9	0.049	0.05	7.1	<0.010	0.003	Bal.	9.9
D	21	0.85	1.10	3.4	0.4	3.9	0.044	0.06	7.1	0.033	0.003	Bal.	1.3
E	21	0.89	1.15	3.4	**3.5**	3.9	0.038	0.04	7.0	0.021	0.003	Bal.	1.3
F	21	0.84	1.13	**4.5**	0.4	3.9	0.040	0.04	7.1	<0.010	0.003	Bal.	1.3

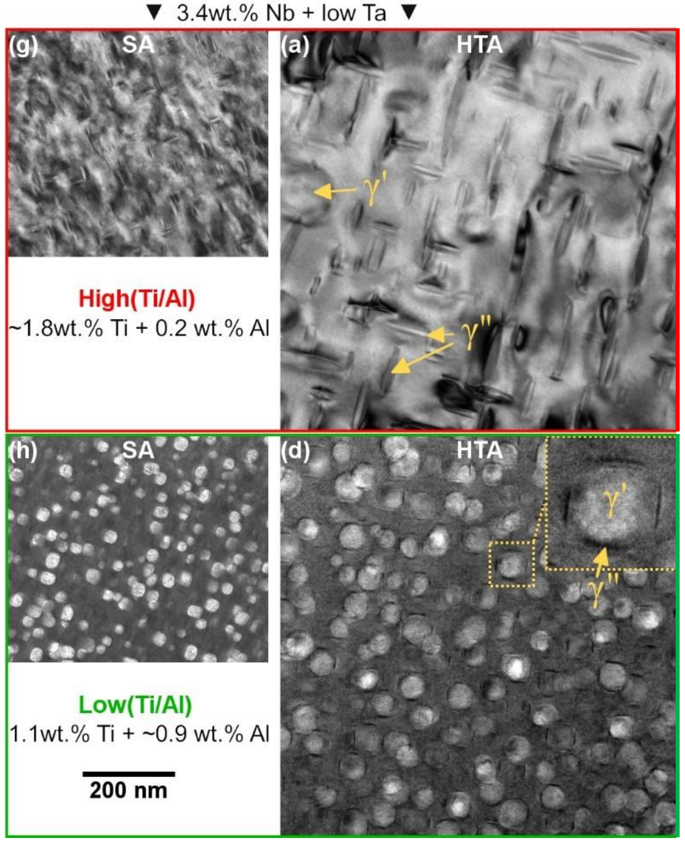
M. Detrois, S. Antonov, K.A. Rozman, J.A. Hawk, P.D. Jablonski, Improved Creep and Tensile Properties of a Corrosion Resistant Ni-Based Superalloy using High Temperature Aging and Nb/Ta Additions, Metall. Mater. Trans. A (2022). https://doi.org/10.1007/s11661-022-06690-6

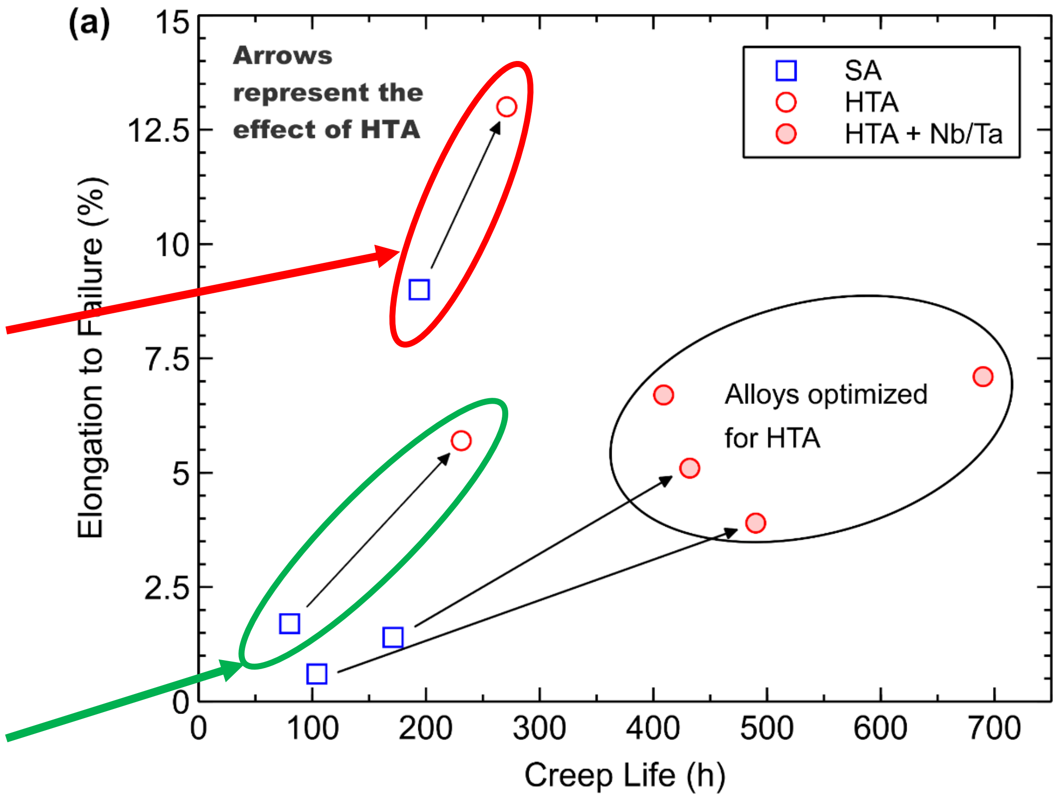
Effect of Key Elements and Aging in IN725 Variants

Effect of High Temperature Aging

The HTA promoted the formation and growth of γ″ precipitates as well as grain boundary phases, such as δ precipitates, among others. This resulted in significant increases in time and elongation to failure.







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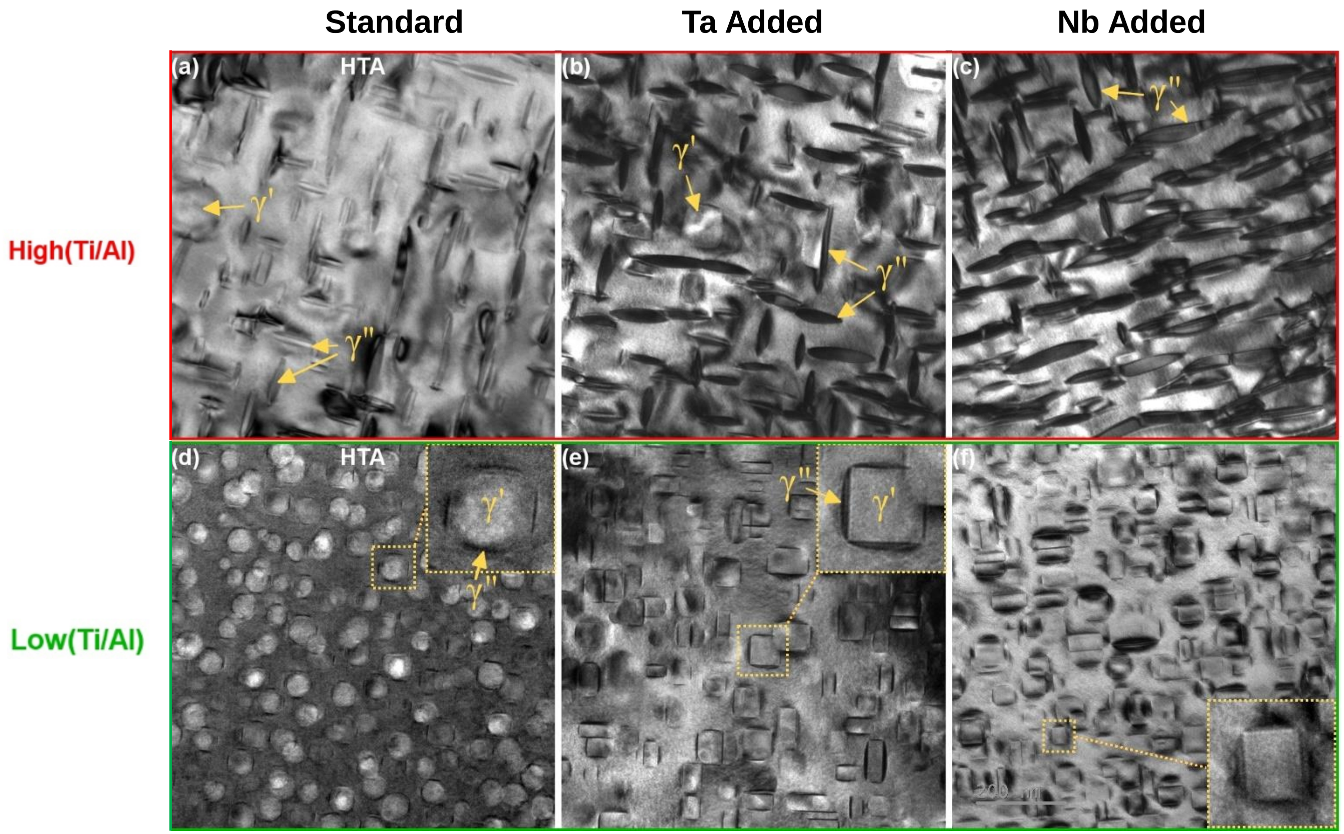
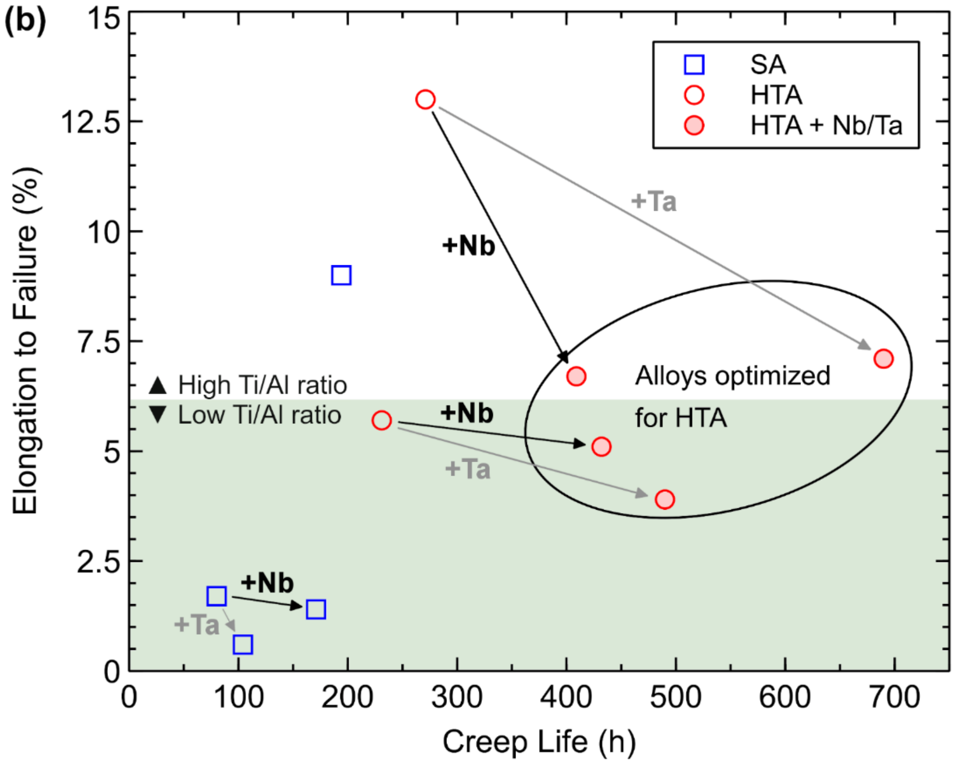
Effect of Key Elements and Aging in IN725 Variants

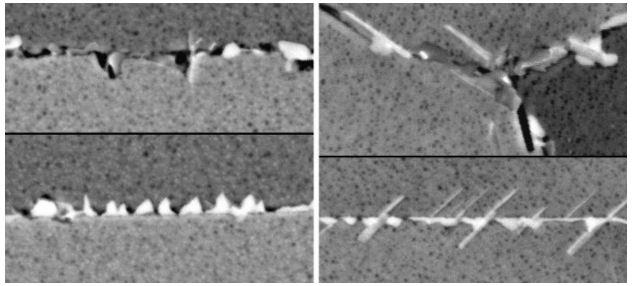
Effect of Ta/Nb Additions and Ti/Al Ratio

The creep life of IN725 was improved by up to 256% for testing at 700°C and 483 MPa and by up to 73% for testing at 790°C and 207 MPa by using additions of Nb or Ta coupled with a higher temperature aging heat treatment.





GB phases in alloys with added Ta and Nb following the HTA aging:



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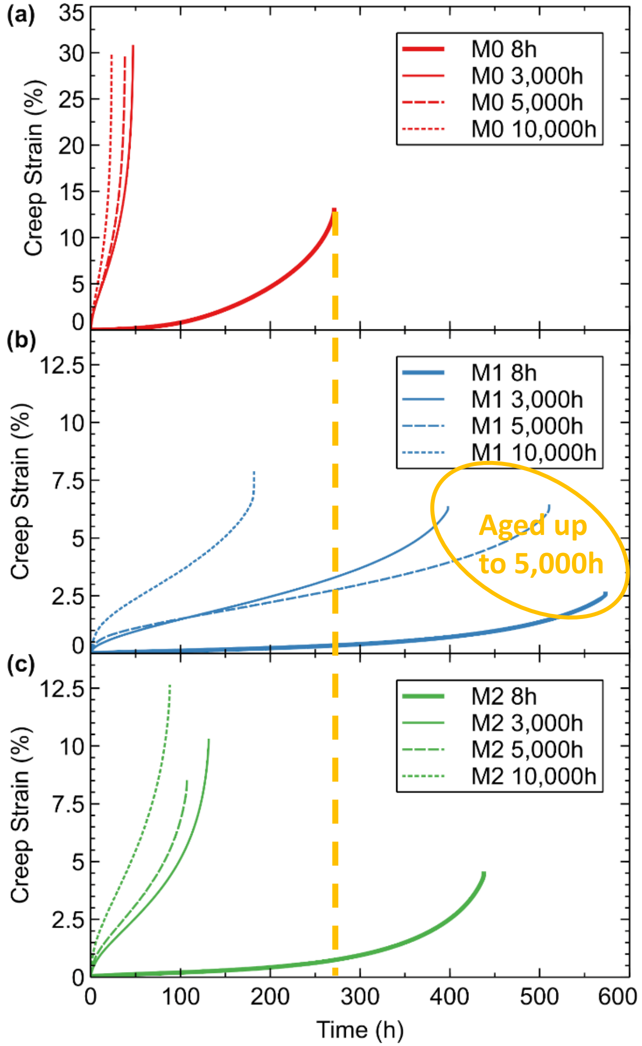
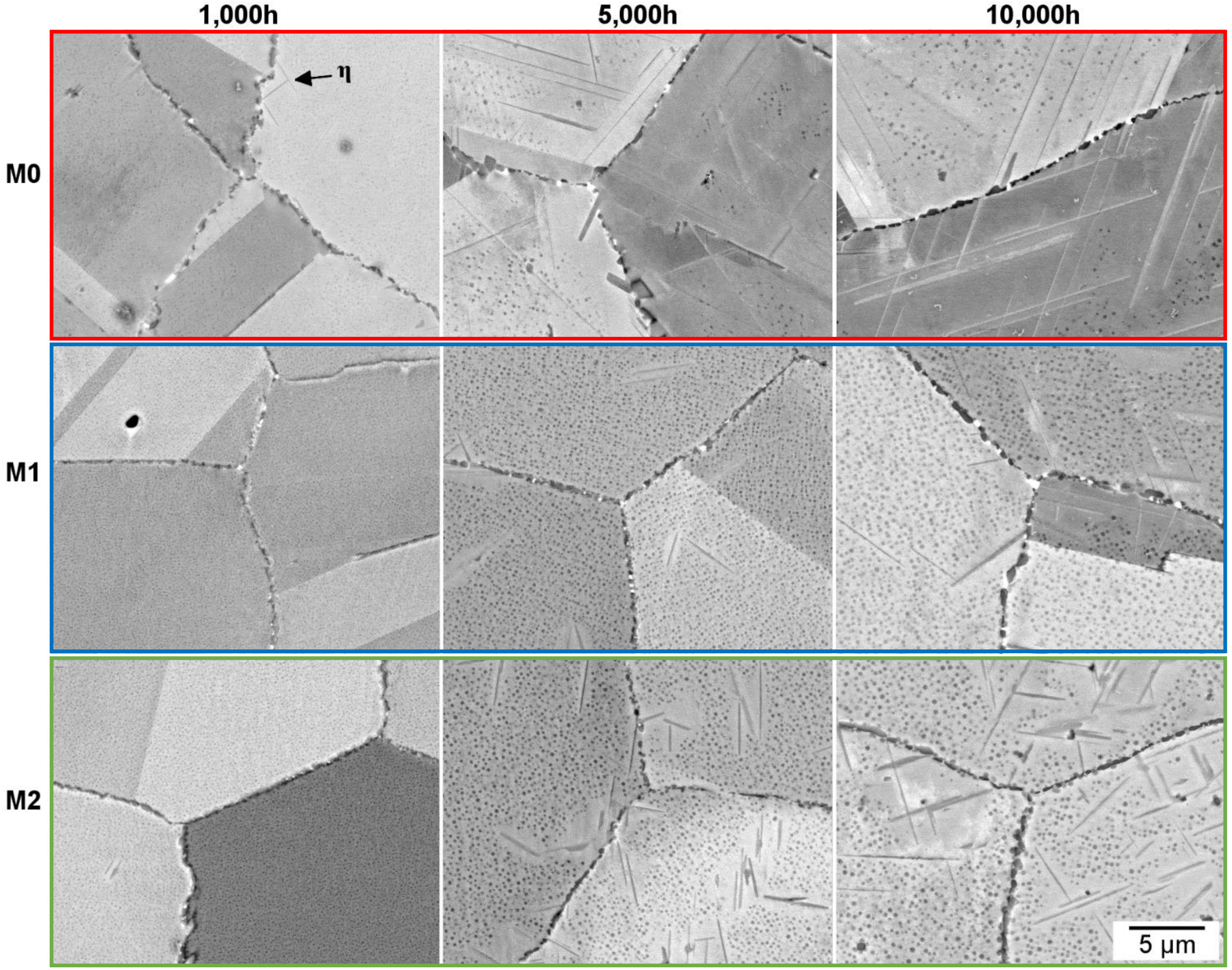
Influence of η Phase in Alloy 263

Over-aged Creep Testing

- With reduced formation and stability of the η phase, and increased γ′ fraction, alloy M1 outperformed the standard alloy in creep regardless of the exposure time at 800°C prior to testing.
- Exposures for 3,000 h and beyond resulted in creep lives below 50 h for the standard alloy, which is at least 3.6 times less than the life of M1 after exposure at 800°C for 10,000 h.
- Decreasing Co negatively impacted phase stability.



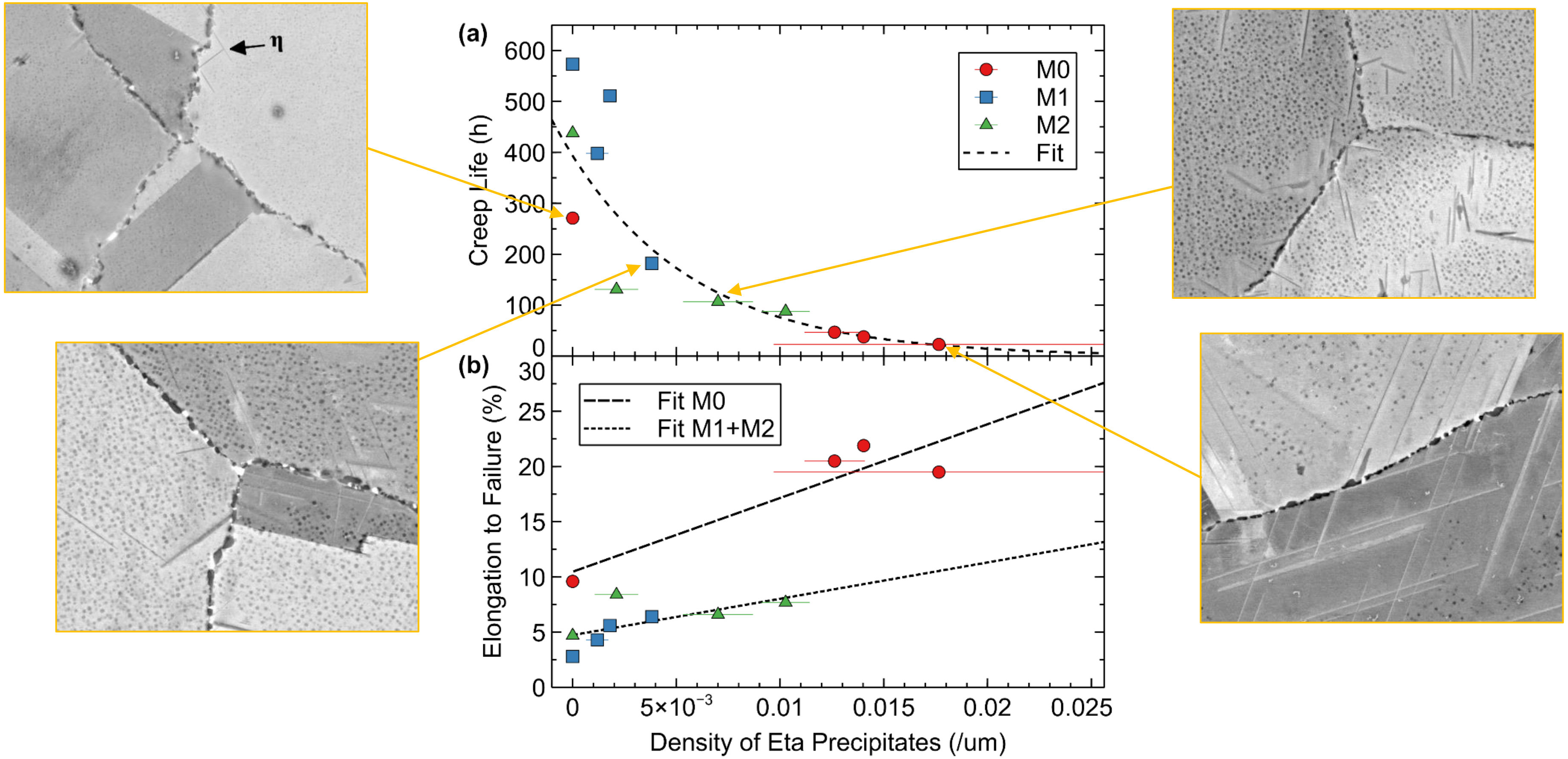


M. Detrois, P.D. Jablonski, J.A. Hawk, The effect of η phase precipitates on the creep behavior of alloy 263 and variants, Mater. Sci. Eng. A 799 (2021) 140337. https://doi.org/10.1016/j.msea.2020.140337

Influence of η Phase in Alloy 263

Influence of the η Phase

- The minimum creep rate increased exponentially with the density of η precipitates.
- The relationship was independent of the chemistry changes, and therefore, the γ′ precipitates (not reported).
- Consequently, the creep life decreased exponentially with the density of η precipitates while the elongation to failure increased.



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C Content in Nimonic 105

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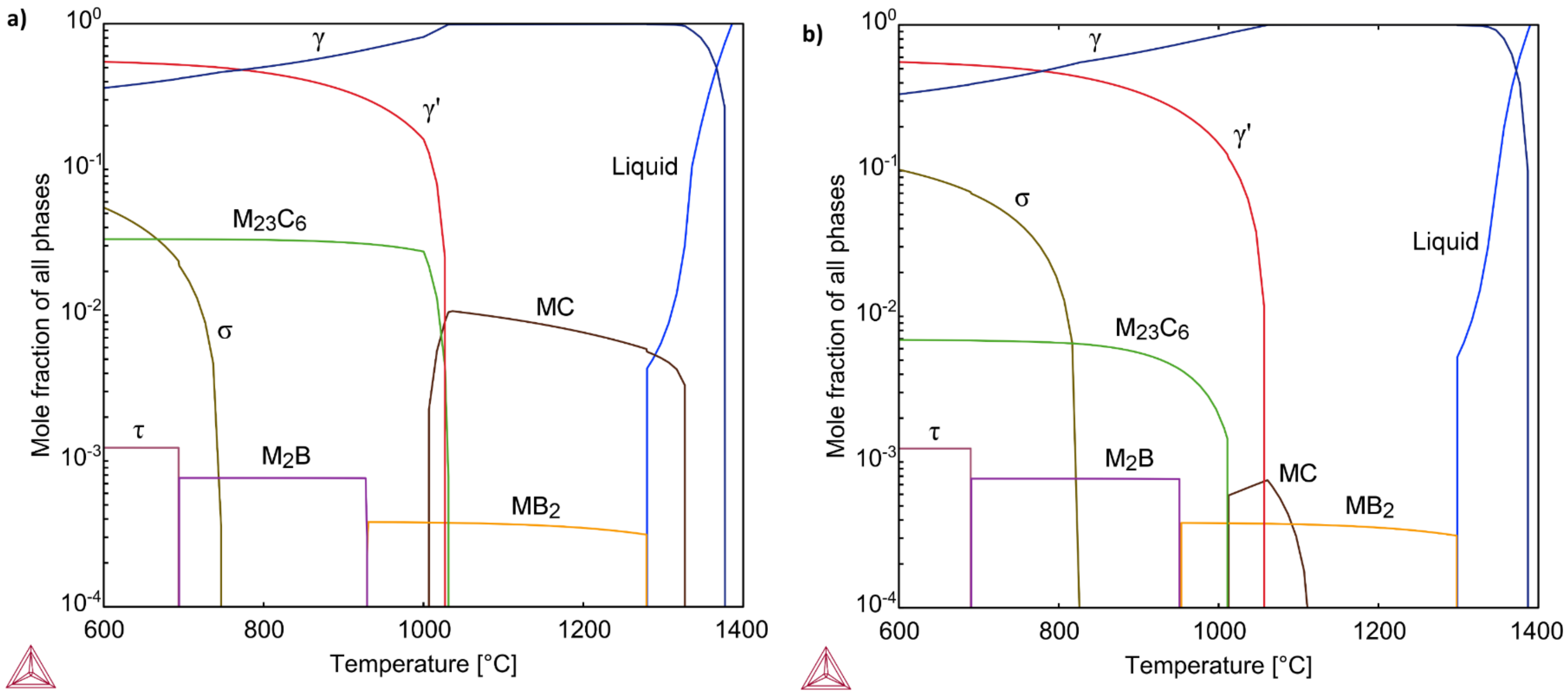
Introduction

- Nimonic 105 is a wrought Ni-base superalloy developed for service up to 950°C.
- The specified C content is high in Nimonic 105 with a maximum of 0.17 wt.%.
- **The influence of the C concentration was explored with regards to fabricability, mechanical properties in tension and creep, and phase stability of Nimonic 105.**



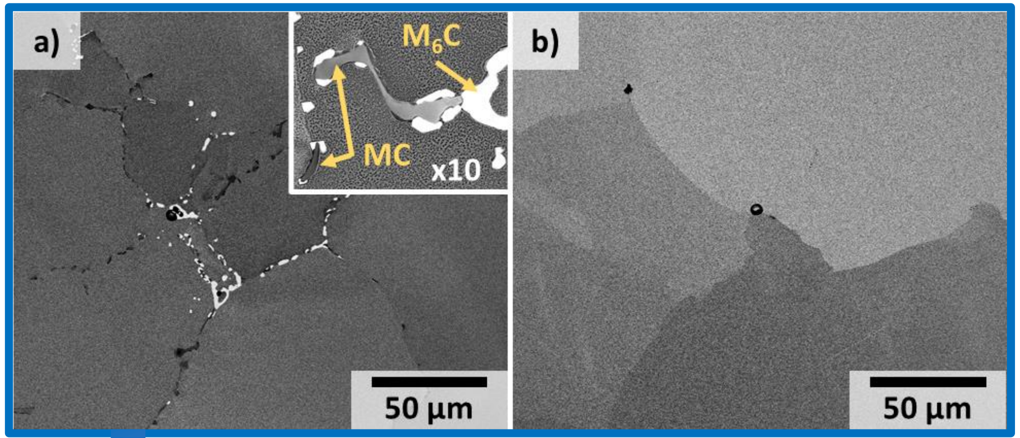
Compositions of the alloys in wt. % measured from XRF for the main elements, combustion analysis for C and addition to the melt for B.

Wt.%	Ni	Mn	Si	Cr	Co	Mo	Ti	Al	C	B	N	O	S
N105	Bal.	0.5	0.36	14.8	20.8	5.0	1.05	4.44	**0.150**	0.005	4 ± 0	19 ± 2	3 ± 0
N105LC	Bal.	0.5	0.36	14.8	20.8	5.0	1.09	4.49	**0.031**	0.005	4 ± 1	1 ± 1	3 ± 0

S. Antonov, K.A. Rozman, P.D. Jablonski, M. Detrois, Implications of carbon content for the processing, stability, and mechanical properties of cast and wrought Ni-based superalloys, (Upcoming).

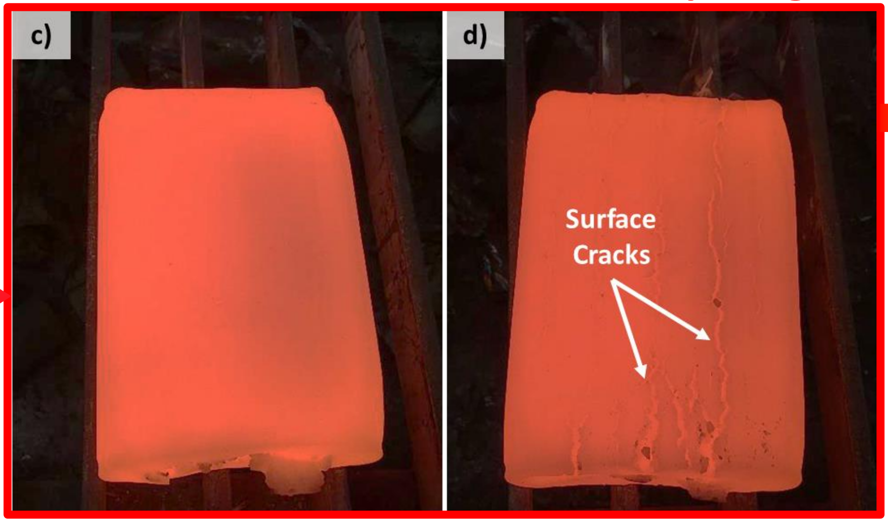
C Content in Nimonic 105

Processing – Microstructure Relationship



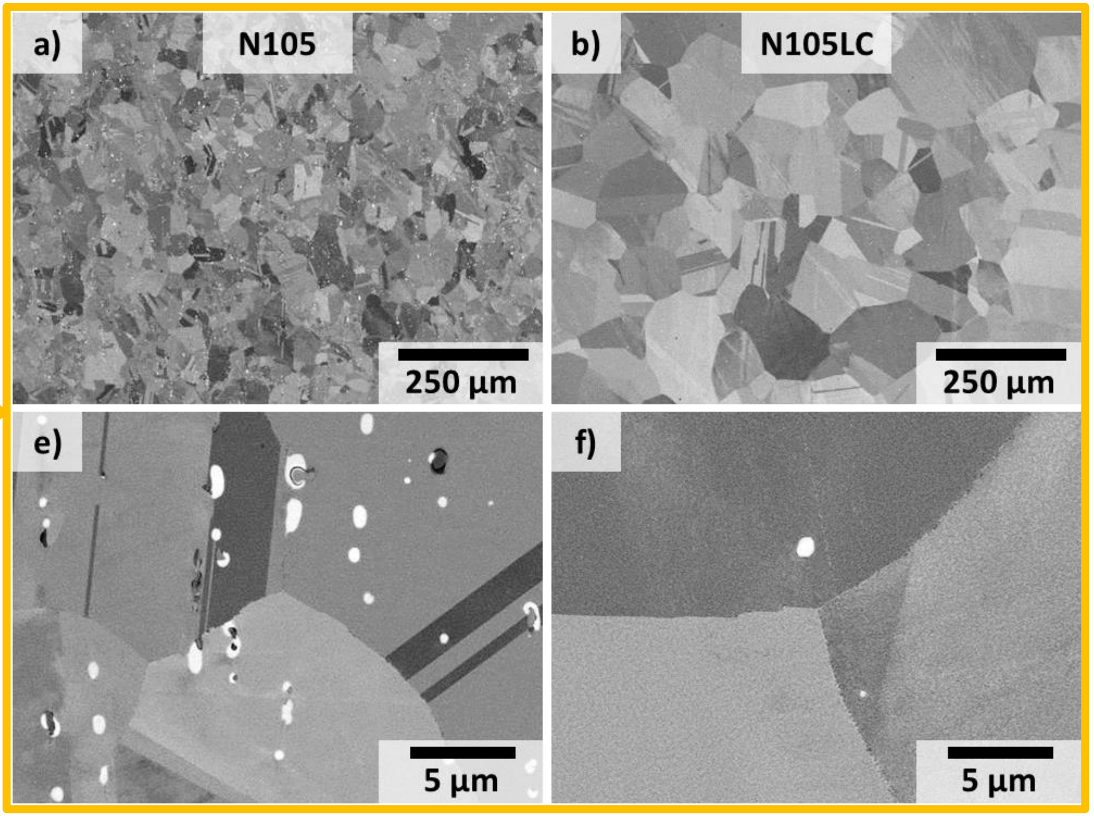
Homogenized

Step Forged



The finer grain size in N105 improved the fabricability in addition to the carbides likely promoting localized recrystallization.

Post Processing



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C Content in Nimonic 105

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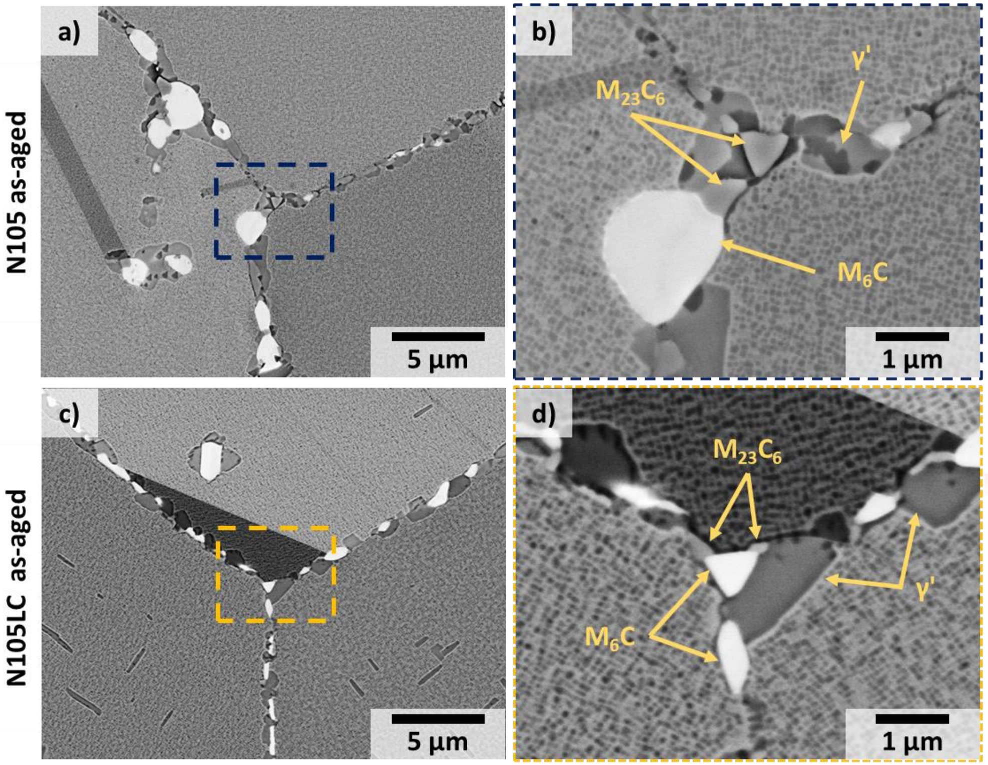
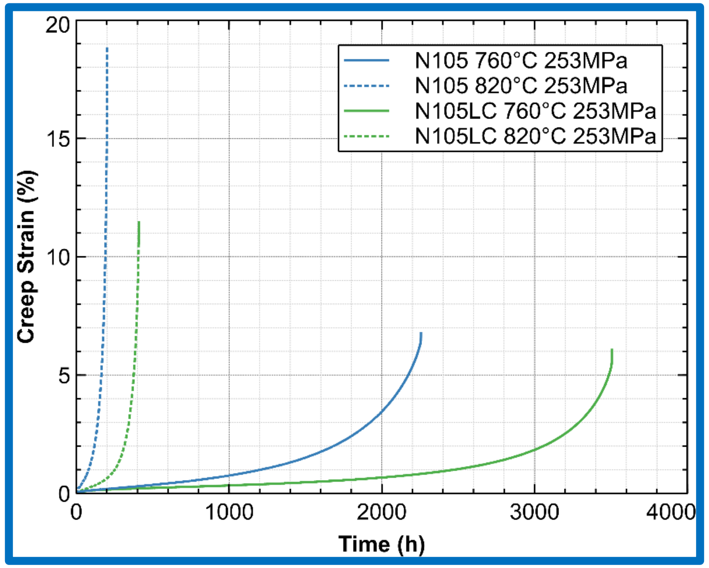
Microstructure - Property Relationship

► **The creep life of the low-C version was 55 to 102% greater** than N105 with the increase related to the grain size.

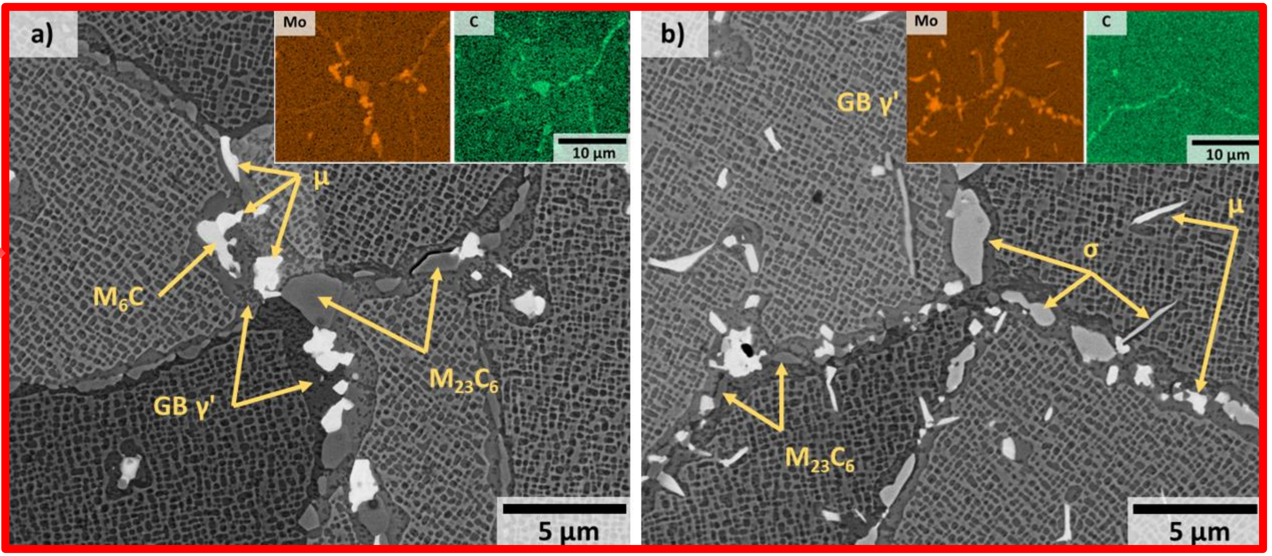




Creep Testing

Phase Stability 800°C / 5,000 h

▼ N105 was more stable with $M_{23}C_6$ carbides decorating the GBs and limited µ phase formation. **With less C available in N105LC, σ phase formed along the GBs while the significant µ phase formation occurred from the M_6C carbides.**



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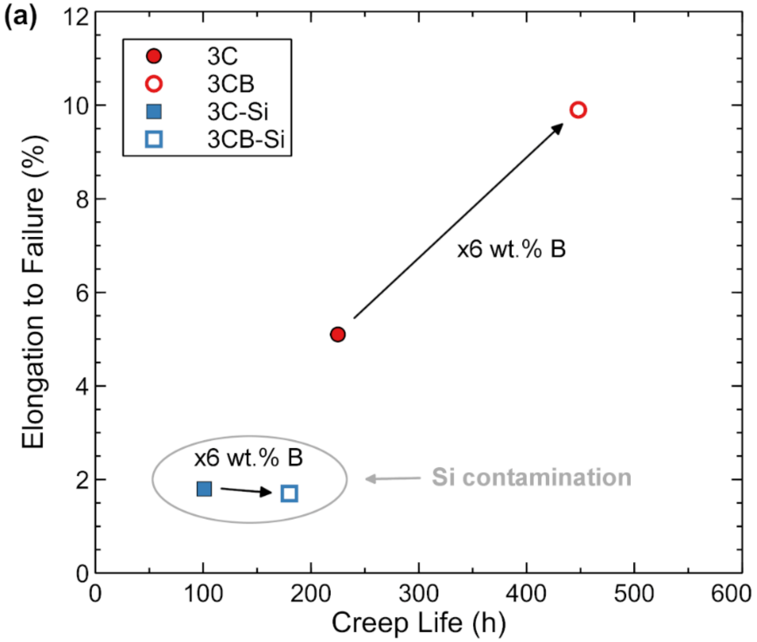
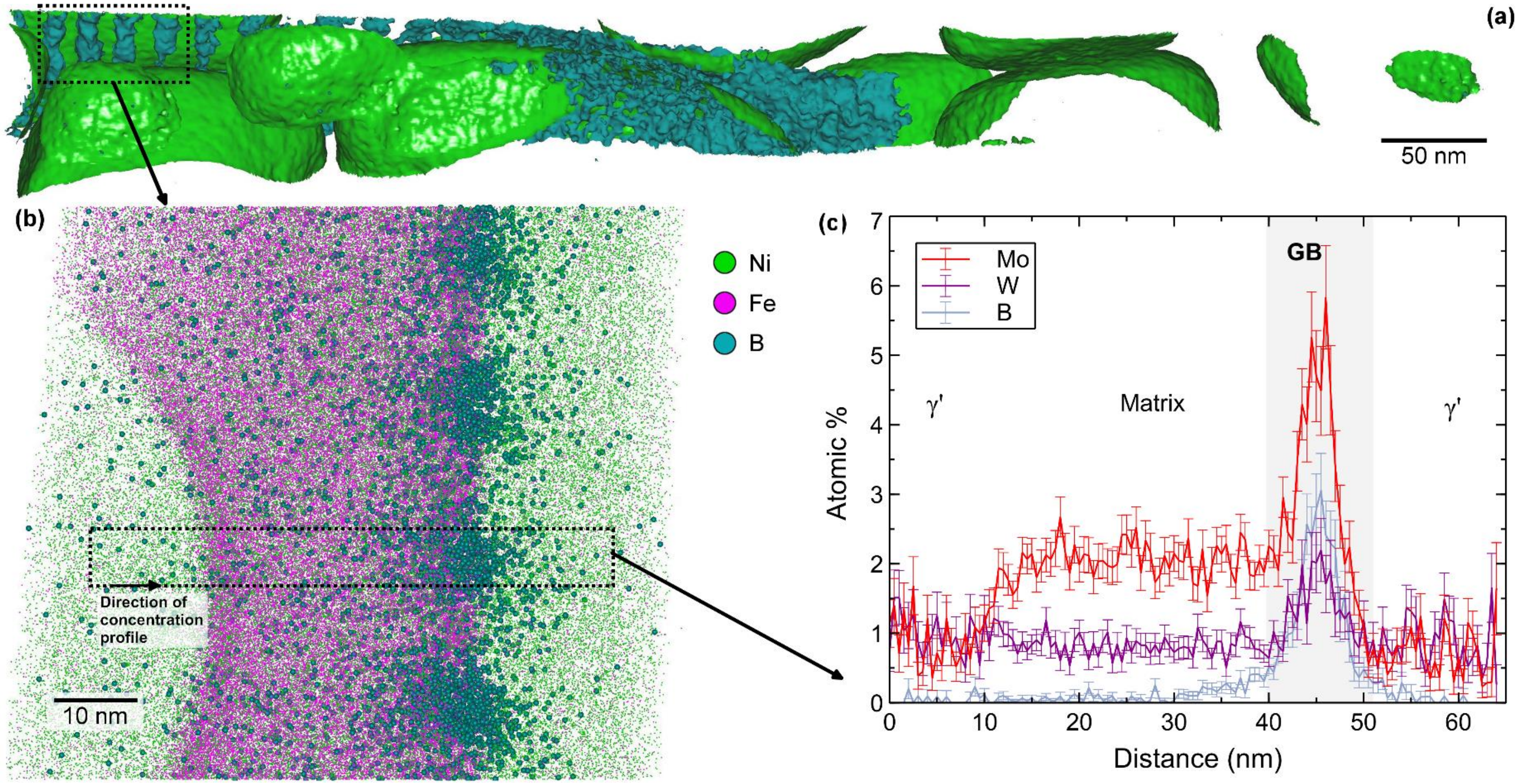
Influence of Trace Elements

B and Si at Grain Boundaries

The creep life and elongation to failure ~doubled in a high-purity Ni-based alloy with B additions while only the creep life was improved in the low-purity alloy (Si, Cu contamination). DFT calculations have shown that Si segregation to GBs decreases GB cohesion, which offsets the positive effect of B segregation.





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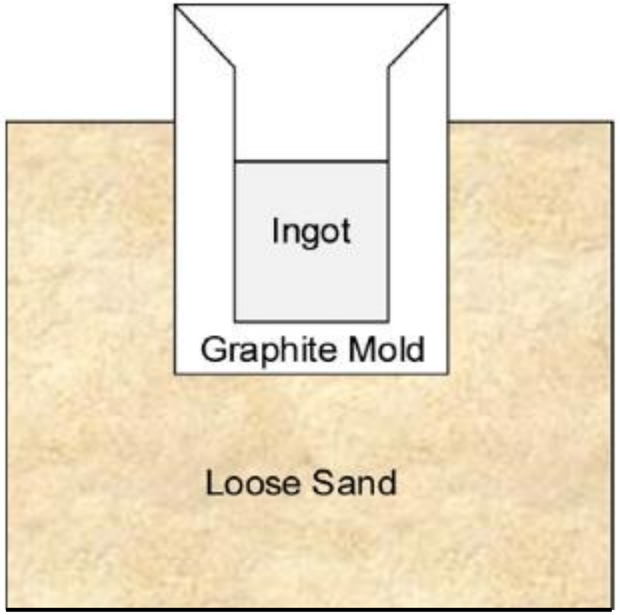
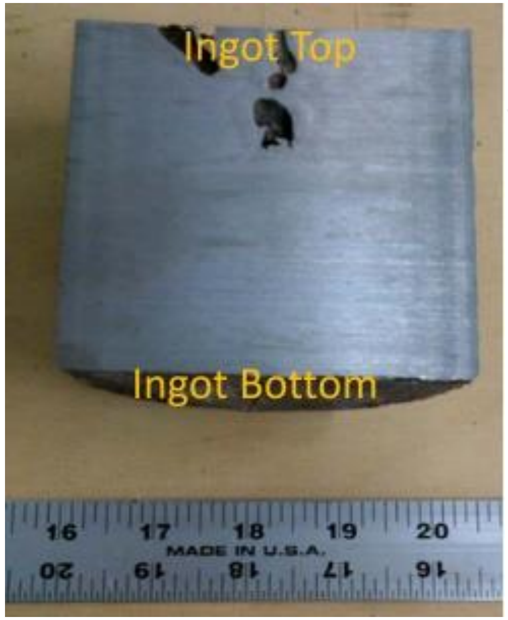
Ni-Base Superalloy Castings

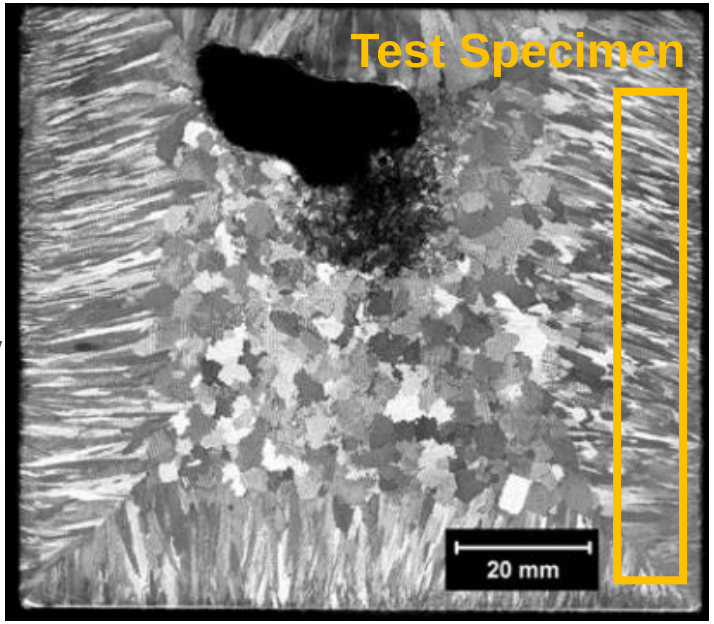
Overview



Small scale castings (7 kg) utilizing VIM melting and pouring in a graphite mold in loose sand to simulate cooling of thick wall castings.



Billet cross section macro-etched:



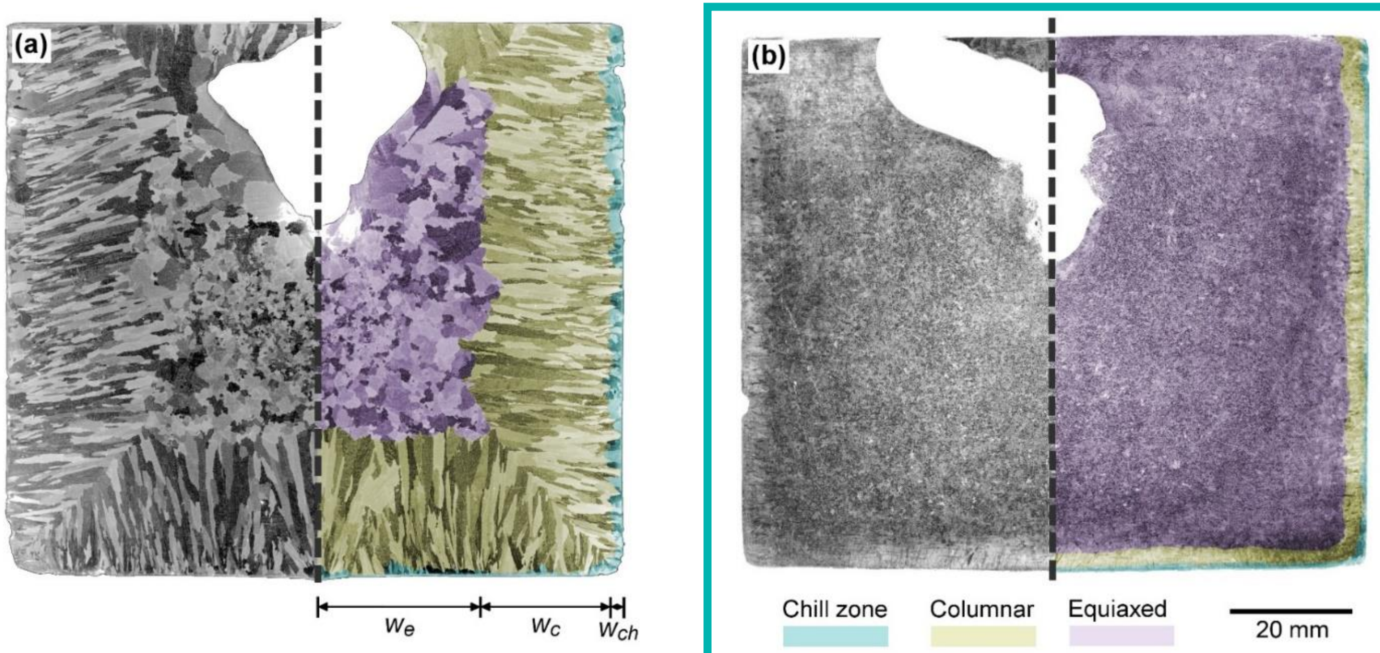
Typical casting grain structures are inhomogeneous with a chill grain zone, columnar zone and equiaxed zone in the center.

Testing is performed in both equiaxed and columnar regions.

M. Detrois, K.A. Rozman, P.D. Jablonski, J.A. Hawk, An Alternative Casting Technique to Improve the Creep Resistance of Cast INCONEL alloy 740H, Metall. Mater. Trans. A 51 (2020) 3819-3831. https://doi.org/10.1007/s11661-020-05822-0

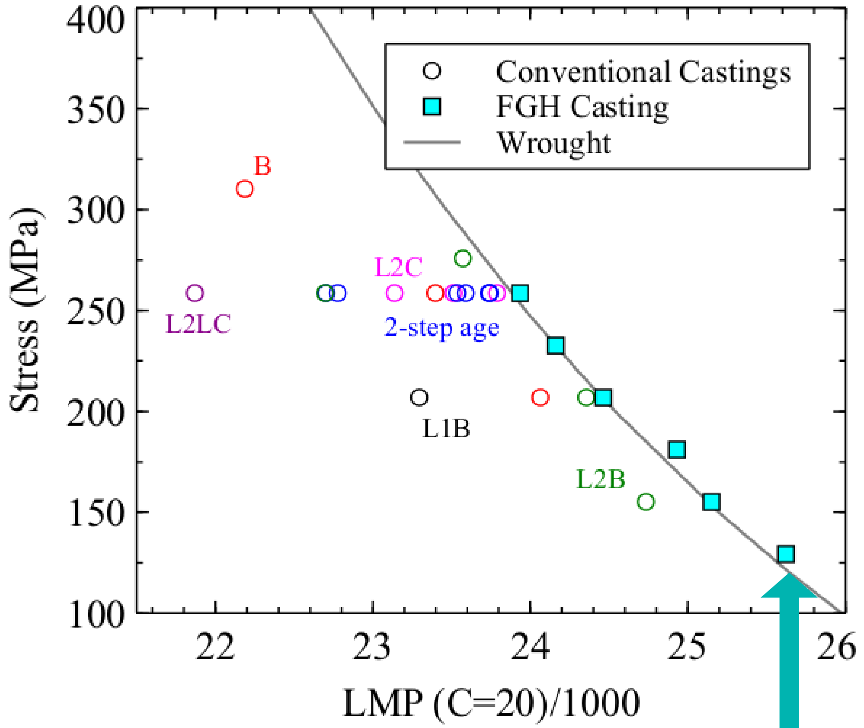
Ni-Base Superalloy Castings

740H Casting



Non uniform grain structure and grain boundary precipitates.

Uniform, refined grain structure and repartition of grain boundary precipitates (carbides) in the FGH casting.



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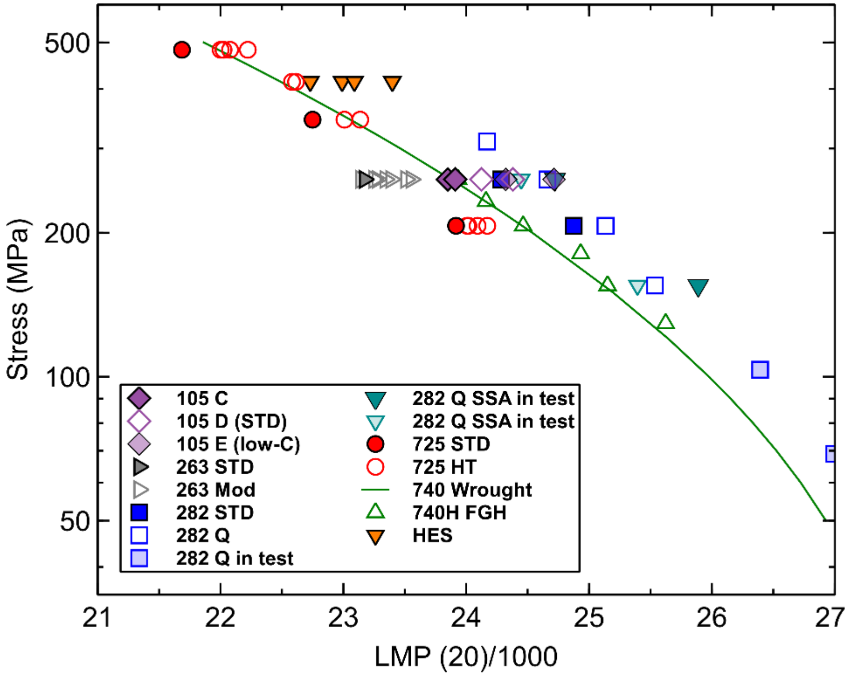
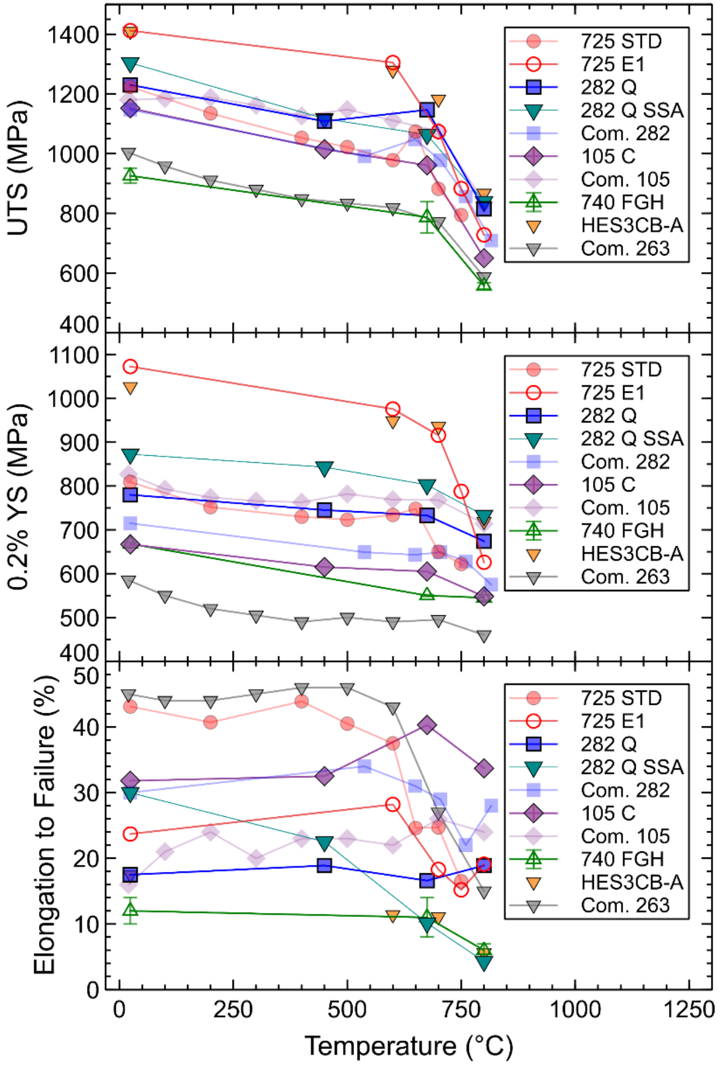
Conclusion/Overview

- Examples were selected within the research performed under Task 5 to showcase some of the findings related to the Ni-Base superalloy development work.
- Several other alloys were investigated to expand the supply chain for creep resistant materials in energy systems.
- The knowledge obtained from Task 5 will be leveraged to advance future studies, particularly in Task 23 which will aim at higher operating temperatures that can be found, for example, in hydrogen systems.





Acknowledgments

J.A. Hawk, K.A. Rozman, J.D. Poplawsky (CNMS at ORNL for Atom Probe Tomography), M.C. Gao, E.R. Argetsinger, J.A. Mendenhall. This work was performed in support of the US Department of Energy's Fossil Energy Crosscutting Technology Research Program.

Questions?

- **M. Detrois, S. Antonov, K.A. Rozman, J.A. Hawk, P.D. Jablonski, Improved Creep and Tensile Properties of a Corrosion Resistant Ni-Based Superalloy using High Temperature Aging and Nb/Ta Additions, Metall. Mater. Trans. A (2022). https://doi.org/10.1007/s11661-022-06690-6**
- **M. Detrois, P.D. Jablonski, J.A. Hawk, The effect of η phase precipitates on the creep behavior of alloy 263 and variants, Mater. Sci. Eng. A 799 (2021) 140337. https://doi.org/10.1016/j.msea.2020.140337**
- **M. Detrois, P.D. Jablonski, J.A. Hawk, Precipitate Phase Stability and Mechanical Properties of Alloy 263 and Variants in Wrought or Cast Form, Proceedings of the 14th International Symposium on Superalloys (Superalloys 2020), Seven Springs, PA, pp. 171-181, 2020. https://doi.org/10.1007/978-3-030-51834-9_17**
- **M. Detrois, Z. Pei, T. Liu, J.D. Poplawsky, M.C. Gao, P.D. Jablonski, J.A. Hawk, The detrimental effect of elemental contaminants when using B additions to improve the creep properties of a Ni-based superalloy, Scr. Mater. 201 (2021) 113971. https://doi.org/10.1016/j.scriptamat.2021.113971**
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