Enhancing the Creep Performance of a Corrosion Resistant Ni-Based Superalloy for Elevated Temperature Applications

Martin Detrois
Research Scientist
Research & Innovation Center

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Authors and Contact Information

Martin Detrois[1,2], Paul D. Jablonski[1], Jeffrey A. Hawk[1]

[1] National Energy Technology Laboratory, 1450 Queen Ave. SW, Albany, OR 97321, USA

[2] NETL Support Contractor, 1450 Queen Ave. SW, Albany, OR 97321, USA

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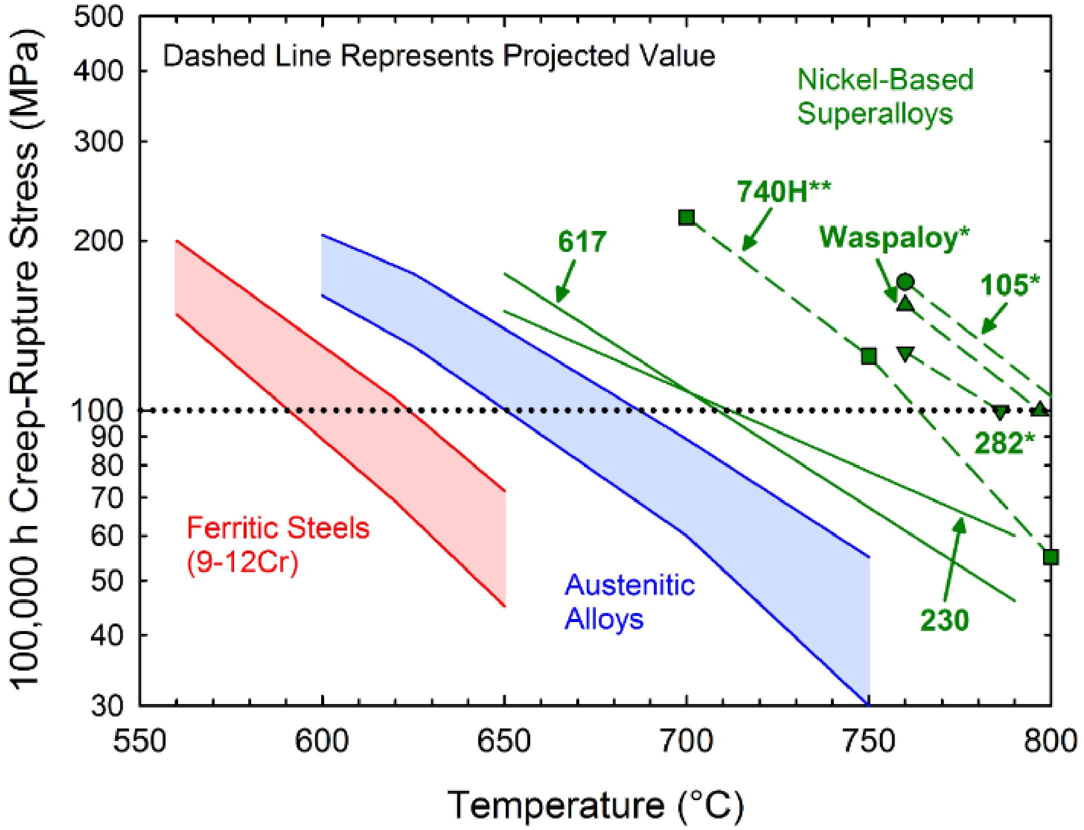
Introduction

Alloys for Next Generation Power Generation

- Operating temperatures for next generation power generation are targeted above 700°C (760°C in the US).
- Potential to raise the efficiency from ~35 to >45% (example of AUSC power plant).
- Reduction in levels of emissions.
- **Structural materials alternatives are needed to replace components in systems such as boilers and steam turbines typically made from austenitic stainless steels or ferritic martensitic steels.**
- Materials that can resist harsh environments (oxidation, corrosion) and long-term creep degradation.



M.C. Hardy, M. Detrois, E.T. McDevitt, C. Argyrakis, V. Saraf, P.D. Jablonski, J.A. Hawk, R.C. Buckingham, H.S. Kitaguchi, S. Tin, Metall. Mater. Trans. A. 51 (2020) 2626–2650. doi:10.1007/s11661-020-05773-6.

Introduction

Inconel 725

- Inconel 725 possesses great resistance to elevated temperature corrosion.
- Very good yield stress and tensile strength at temperatures typically <600°C.
- Typical microstructure consists of face-centered cubic (FCC) matrix with precipitate strengthening consisting of:
  - γ′ – $Ni_3(Al,Ti)$ – ordered FCC $L1_2$ crystal structure
  - γ′′ – $Ni_3(Nb,Al,Ti)$ – ordered tetragonal $D0_{22}$ crystal structure
- Inconel 725 finds use in marine applications where resistance to pitting corrosion or stress corrosion cracking is essential.
- Used for components such as hangers, landing nipples, mandrels, fasteners, and shafts, among others.

Table 1. Composition range for INCONEL 725 (wt.%).

Element	Cr	Al	Ti	Nb	Fe*	C	Mn	Mo	Si	P	S	Ni
Minimum	19.0	-	1.0	2.75	Bal.	-	-	7.0	-	-	-	55.0
Maximum	22.5	0.35	1.7	4.0	Bal.	0.03	0.35	9.5	0.20	0.015	0.010	59.0

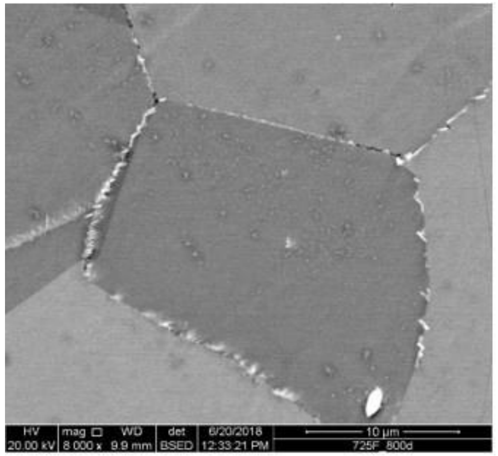
https://www.specialmetals.com/assets/smc/documents/alloys/inconel/inconel-alloy-725.pdf

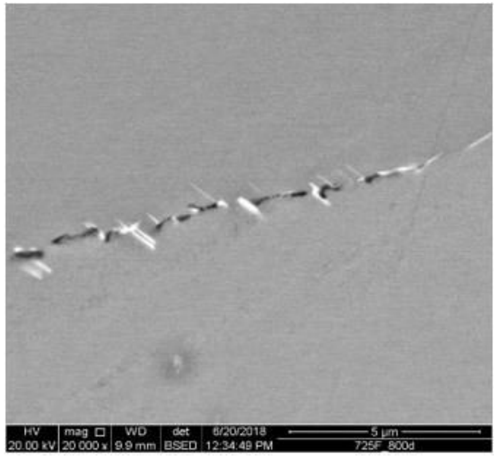
Alloy Design

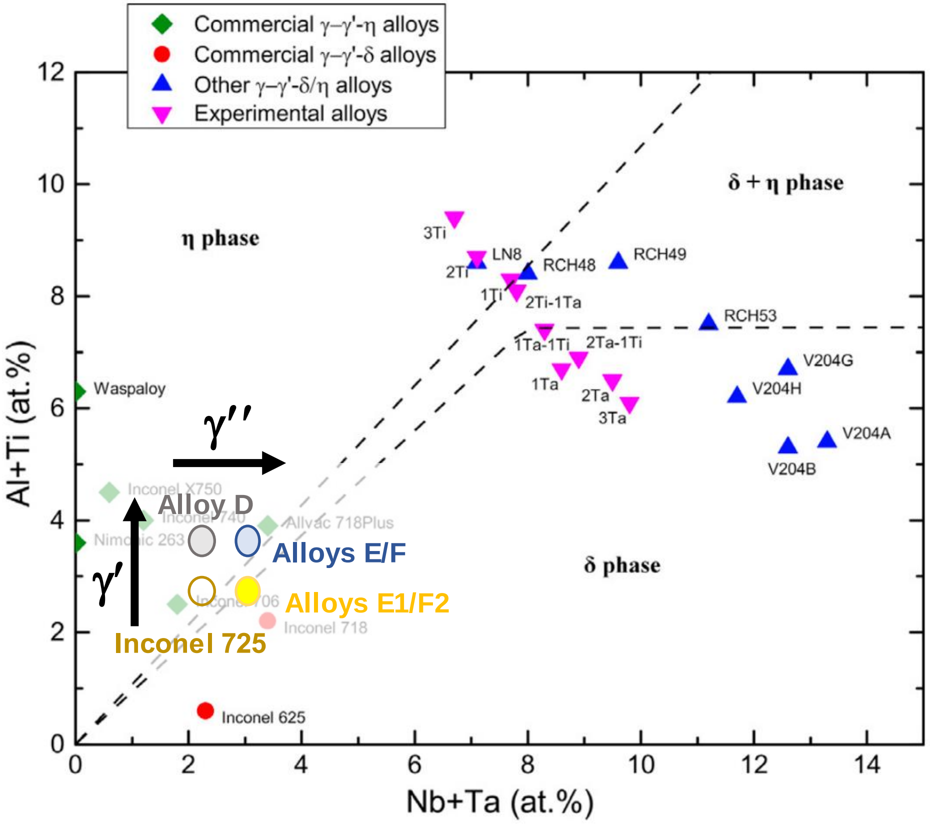
Design

- Improve the elevated temperature mechanical properties (specifically creep) of alloy 725.
- Combine corrosion resistance and creep performance.
- 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.
- Employ an aging heat treatment that would favor the precipitation of those phases.

► Microstructure of alloy F following exposure at 800°C for 20h.







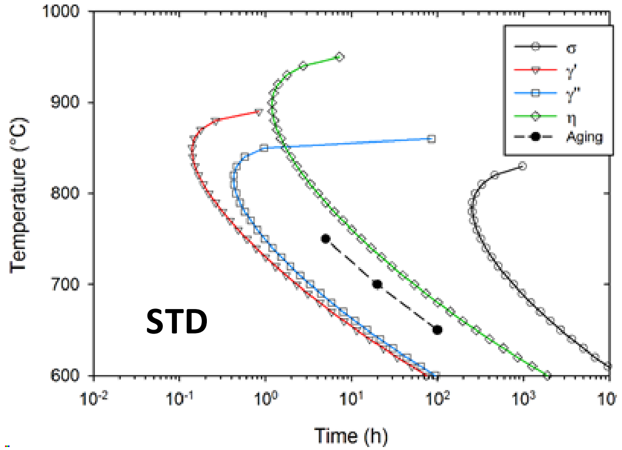
▲ Adapted from: S. Antonov, M. Detrois, R.C. Helmink, S. Tin, J. Alloys Compd. 626 (2015) 76–86.

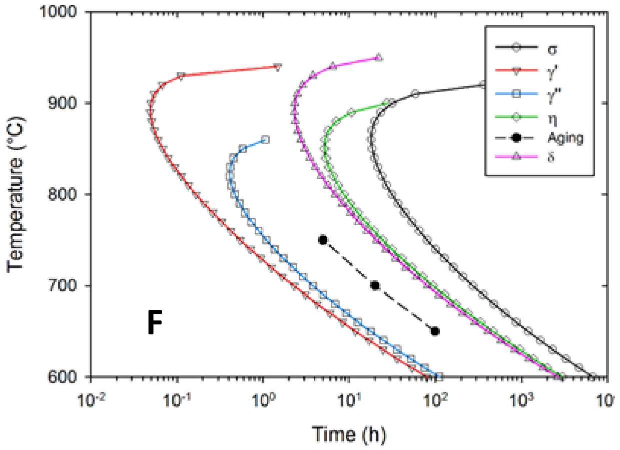
Alloy Design

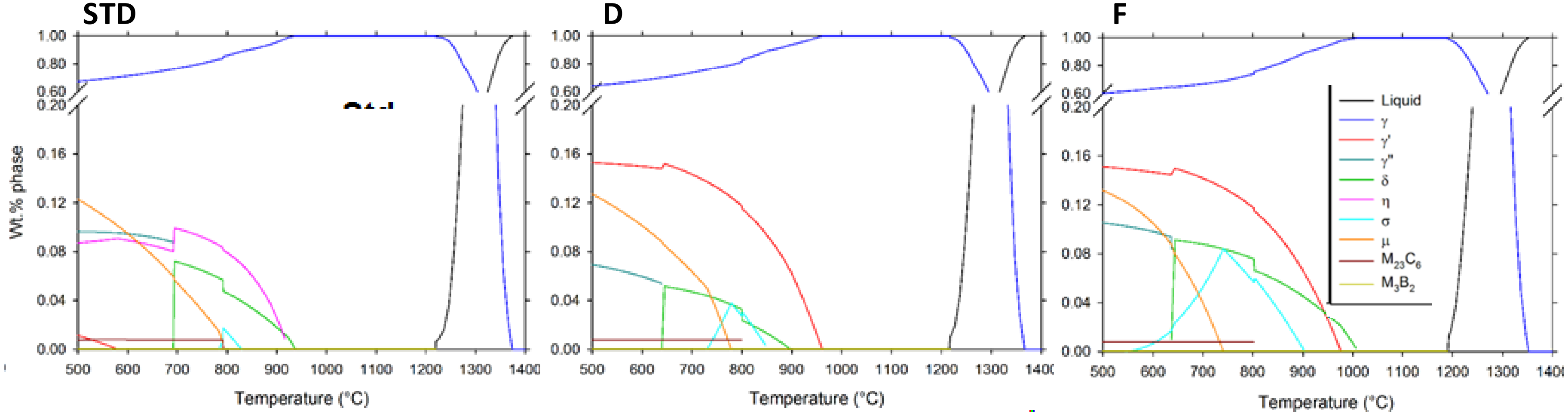
Computational Work

- From the standard alloy formulation (STD), decreasing the Ti/Al ratio (alloys D and F) increased γ′ stability.
- From D to F, increasing the Nb concentration increased the stability of the δ phase (or γ′′).
- Similarly, Ta was investigated and Ta + Nb = 3 at.% was chosen.
- A ”high-temperature” (HT) aging heat treatment was chosen to precipitate GB phases first and then γ′/γ′′ precipitates using JMatPro.

▼ JMatPro simulations.







▲ Thermo-Calc simulations.

Alloy Design

Alloy Formulations

- Six alloy compositions were investigated, Table 2. The standard alloy (STD) corresponds to alloy 725.
- Alloys E1 and F2 have a similar Ti/Al ratio than STD (high-Ti/Al).
- Alloys D, E and F have a low-Ti/Al ratio.
- Alloys E1 and E have greater Ta concentrations compared to STD and D, respectively.
- Alloys F2 and F have greater Nb concentrations compared to STD and D, respectively.

Table 2. 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

Manufacture and Characterization

Processing

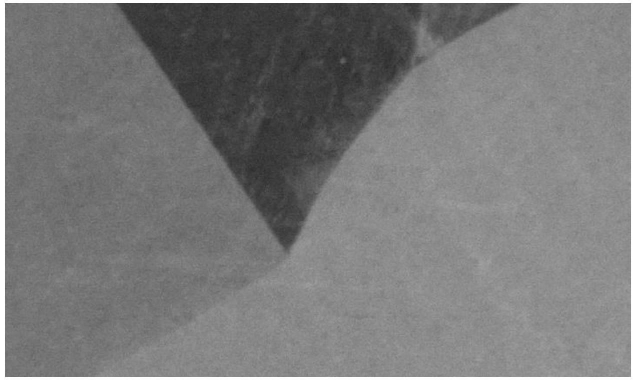
- The alloys were vacuum induction melted (VIM) using industry-grade stock material to obtain 8 kg ingots.
- Chemistry slices were cut from the ingots to perform XRF and combustion analysis.
- The ingots were homogenized following a computationally optimized homogenization heat treatment.
- The ingots were hot worked using steps of forging and hot rolling to obtain 10 mm thick plates that were solutioned.
- Two aging heat treatments were investigated:
  - Standard for alloy 725 (SHT): 730°C for 8h – cooling 1°C/min to 620°C – 620°C for 8h
  - Custom high temperature (HT): 800°C for 20h – cooling 1°C/min to 750°C – 750°C for 8h
- ASTM testing was performed following E139 standard for creep and E8 for tension.
- Microstructural observation was performed using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) was used for preliminary phase identification.

Manufacture and Characterization

Microstructural Characterization

- Complex precipitation along the GBs.
- "Clean" boundaries in the standard alloy as resolved in the SEM.
- Nb promoted δ phase formation.

▼ Alloy STD with SHT aging.

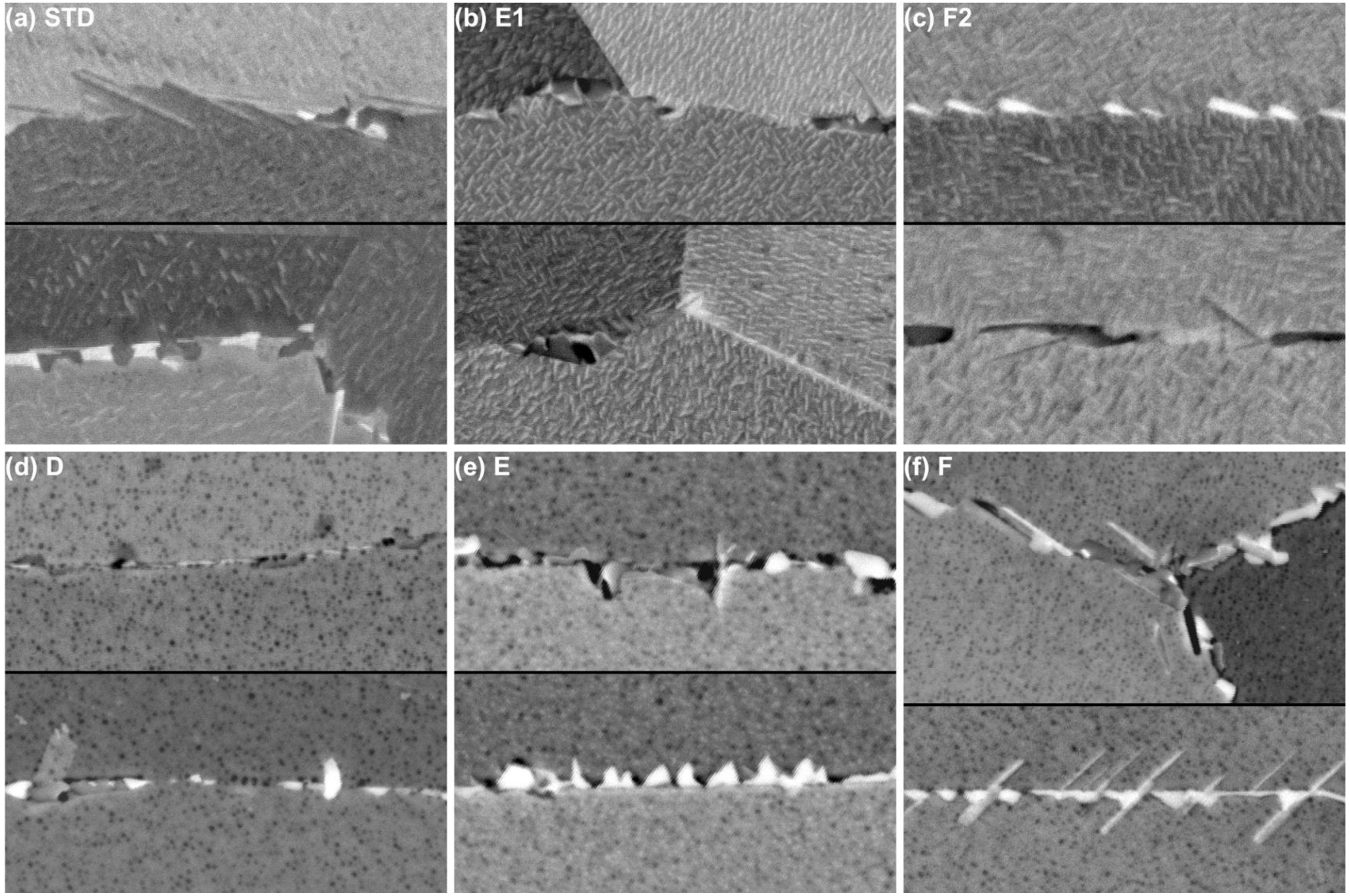


► Alloys with HT aging.

3.4wt.% Nb + low Ta | 3.4wt.% Nb + ~3.5wt.% Ta | ~4.5wt.% Nb + low Ta

~1.8wt.% Ti + 0.2 wt.% Al
Ti/Al = 9

1.1wt.% Ti + ~0.9 wt.% Al
Ti/Al = 1.2

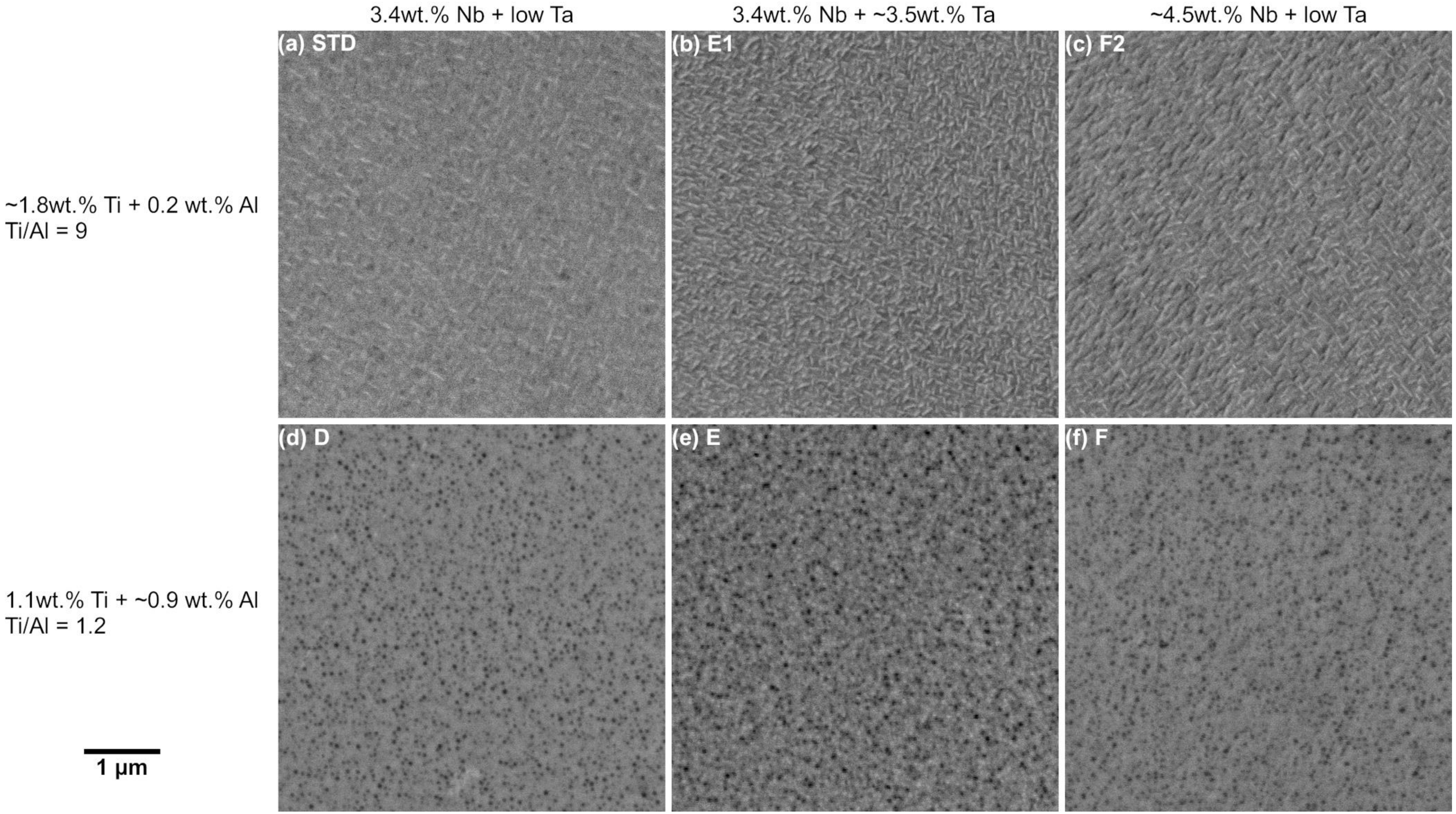


1 µm

Manufacture and Characterization

Microstructural Characterization

- Decreasing the Ti/Al ratio stabilized the γ′ precipitates over γ′′.
- Additions of Ta and Nb increased the density of precipitates in alloys E1, F2 and E.
- Co-precipitation of γ′ and γ′′ was observed in alloy E.
- Ta was found to stabilize γ′′.



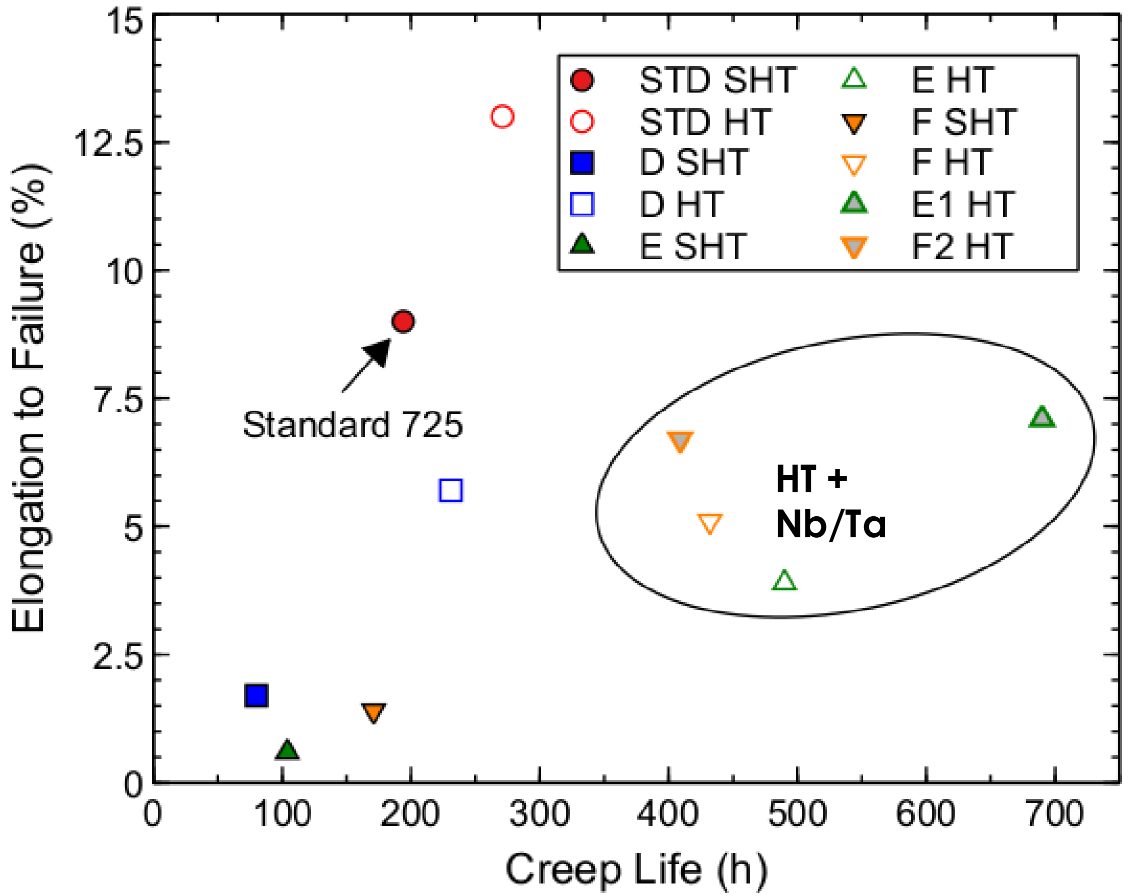
Mechanical Properties

Creep Properties

Testing at 700°C / 70 ksi

Comparing to Alloy STD in its standard aging heat treatment (SHT, or condition most representative of standard alloy 725/625 PLUS):

- The HT aging heat treatment resulted in an improvement of creep life by 40% and ductility by 53%.
- The HT aging treatment combined with Nb addition resulted in an improvement of creep life of >120% (although the Ti/Al ratio was changed).
- The HT aging treatment combined with Ta addition resulted in an improvement of creep life of >250% with similar ductility.



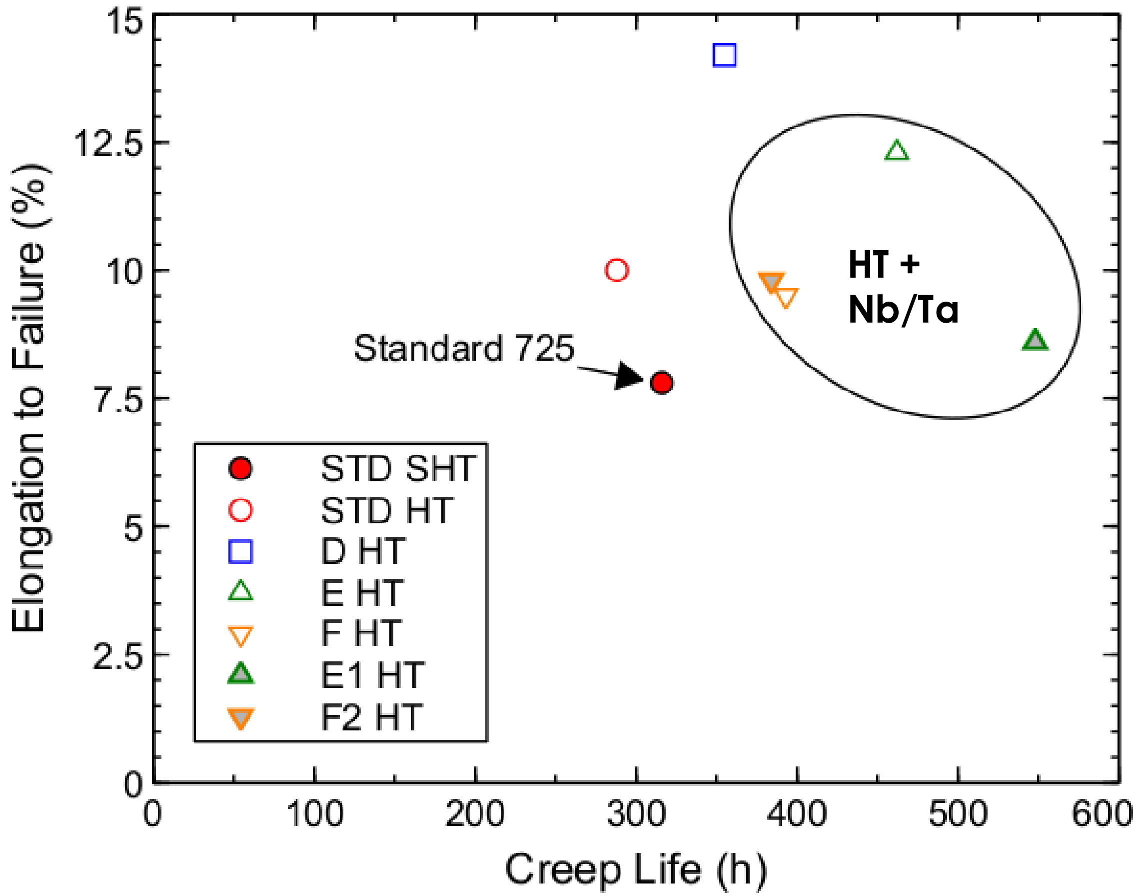
Mechanical Properties

Creep Properties

Testing at 790°C / 30 ksi

Comparison with Alloy STD (with SHT aging) showed that chemistry variations extended each alloy variant creep life.

- The creep life increased by 73% in alloy E1 with a slight increase in ductility.
- The creep life increased by 46% in alloy E with a 58% increase in creep ductility.

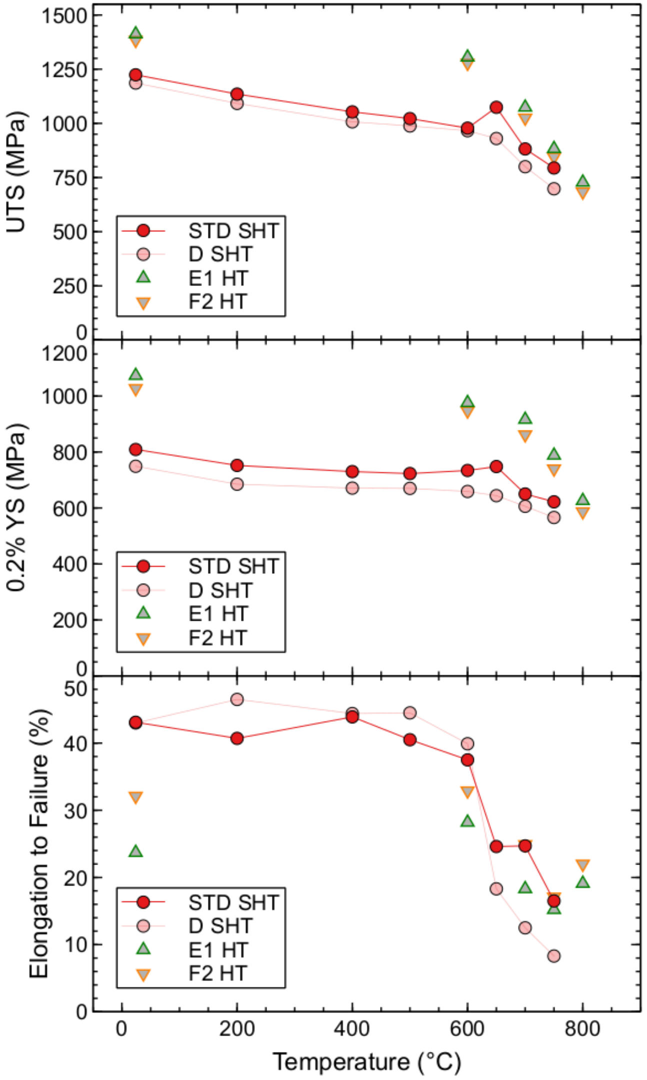


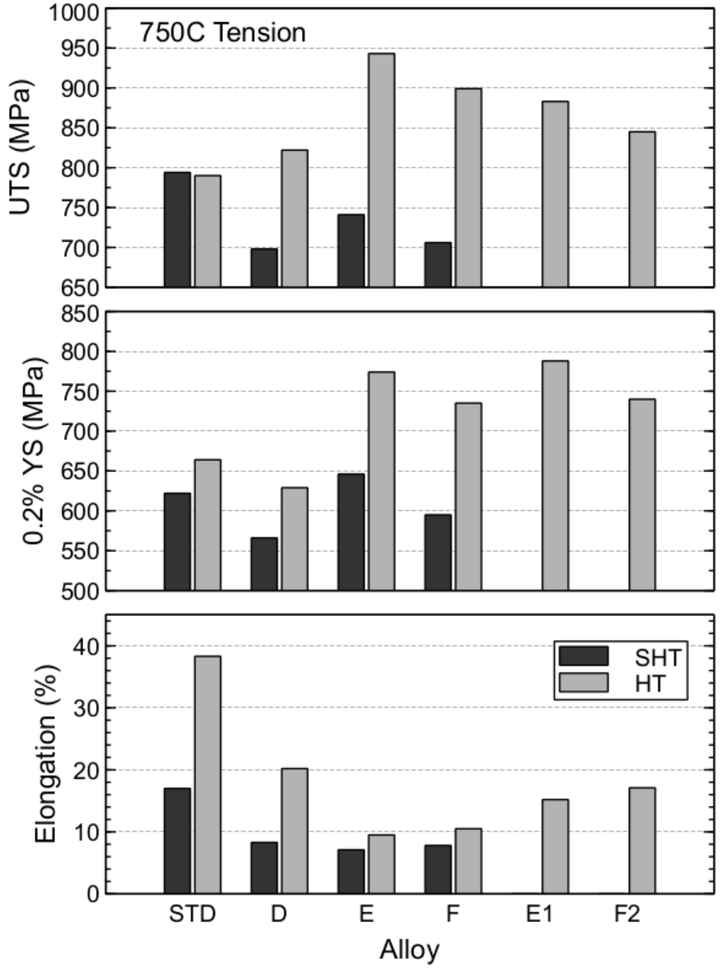
Overall, the improvements were less under the present testing conditions (790°C / 30 ksi) than at 700°C / 70 ksi.

Mechanical Properties

Tensile Properties

- The UTS and 0.2% YS were greatly improved in the alloys with Nb and Ta additions (E, F, E1 and F2) and with the HT aging heat treatment.
- The decrease in Ti/Al ratio was not beneficial to the properties of the alloy (comparing STD vs D).
    - Microstructure containing γ″ precipitates showed greater UTS and YS than the corresponding γ′ microstructure.
- Nb and Ta additions were not beneficial unless the HT aging heat treatment was employed.

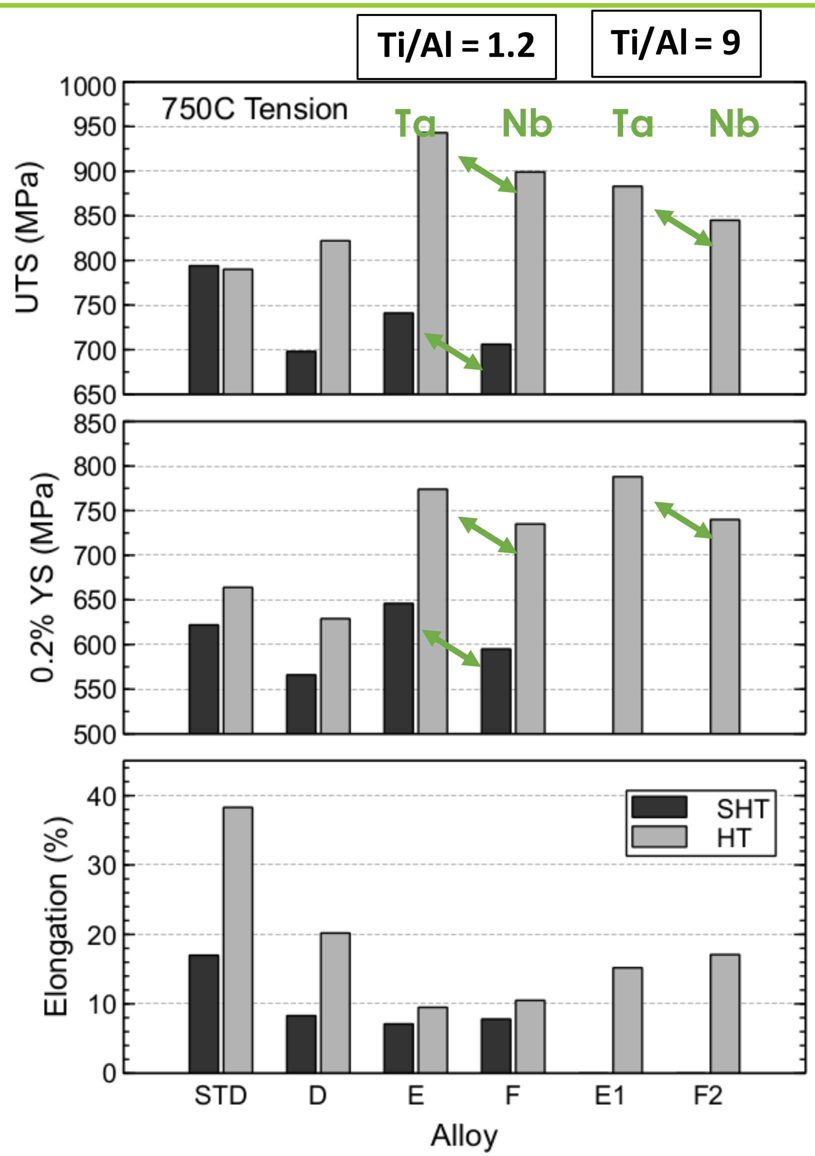


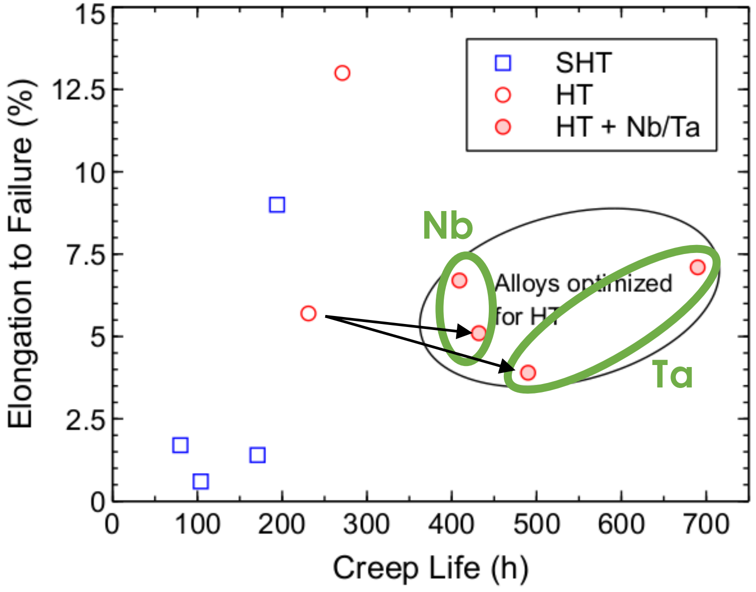


Discussion

Nb/Ta Additions

- Greater Ta content in the Nb + Ta = 3 at.% balance resulted in UTS and YS **5.8 ± 1.5 %** above that obtained with greater Nb.
- Additions of Nb and/or Ta significantly extended the creep life.
- The creep life of alloy D was more than doubled with additions of Nb or Ta (black arrows).
- Increased γ′′precipitation using Ta improved the mechanical performance of the alloy.





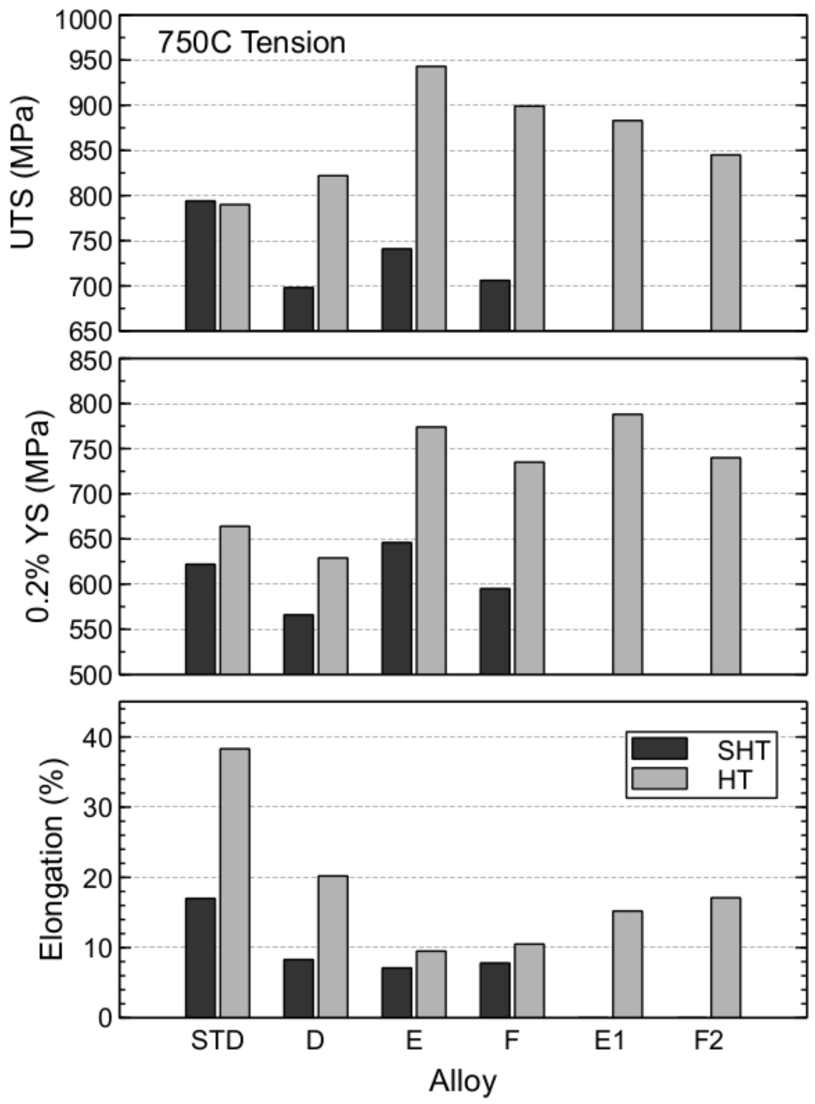
▲ Elongation to failure vs creep life following testing at 700°C / 70ksi.

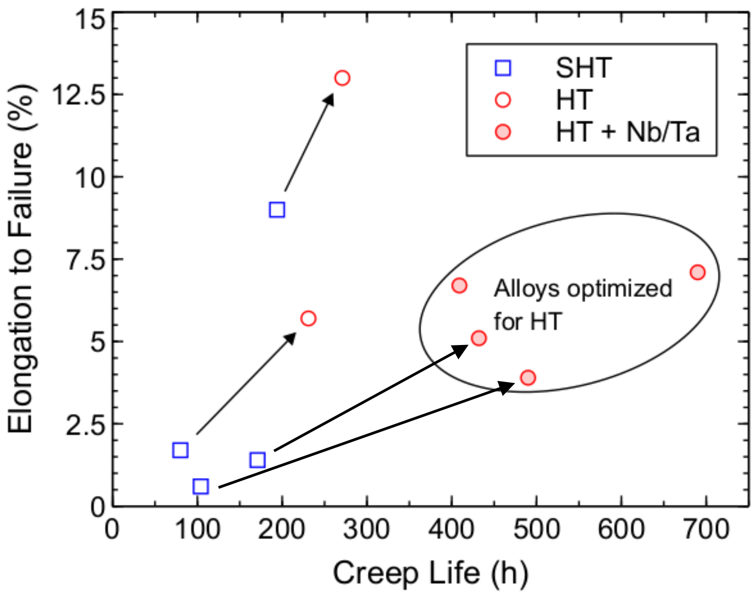
◄ Tensile test results at 750°C for the alloy with SHT or HT aging heat treatment.

Discussion

HT Aging Heat Treatment

- The HT aging heat treatment improved the UTS, YS and creep life of the alloys.
- More significant improvements were obtained with the Nb and Ta additions.
- The combination of HT aging and Nb/Ta additions enabled complex precipitation of fine GB phases (δ and carbides) known to prevent crack propagation or act as "grain boundary stitching".
- Thus, not only the creep life was improved but also the creep ductility.





▲ Elongation to failure vs creep life following testing at 700°C / 70ksi.

◄ Tensile test results at 750°C for the alloy with SHT or HT aging heat treatment.

Discussion Phase Stability – **Alloy STD**

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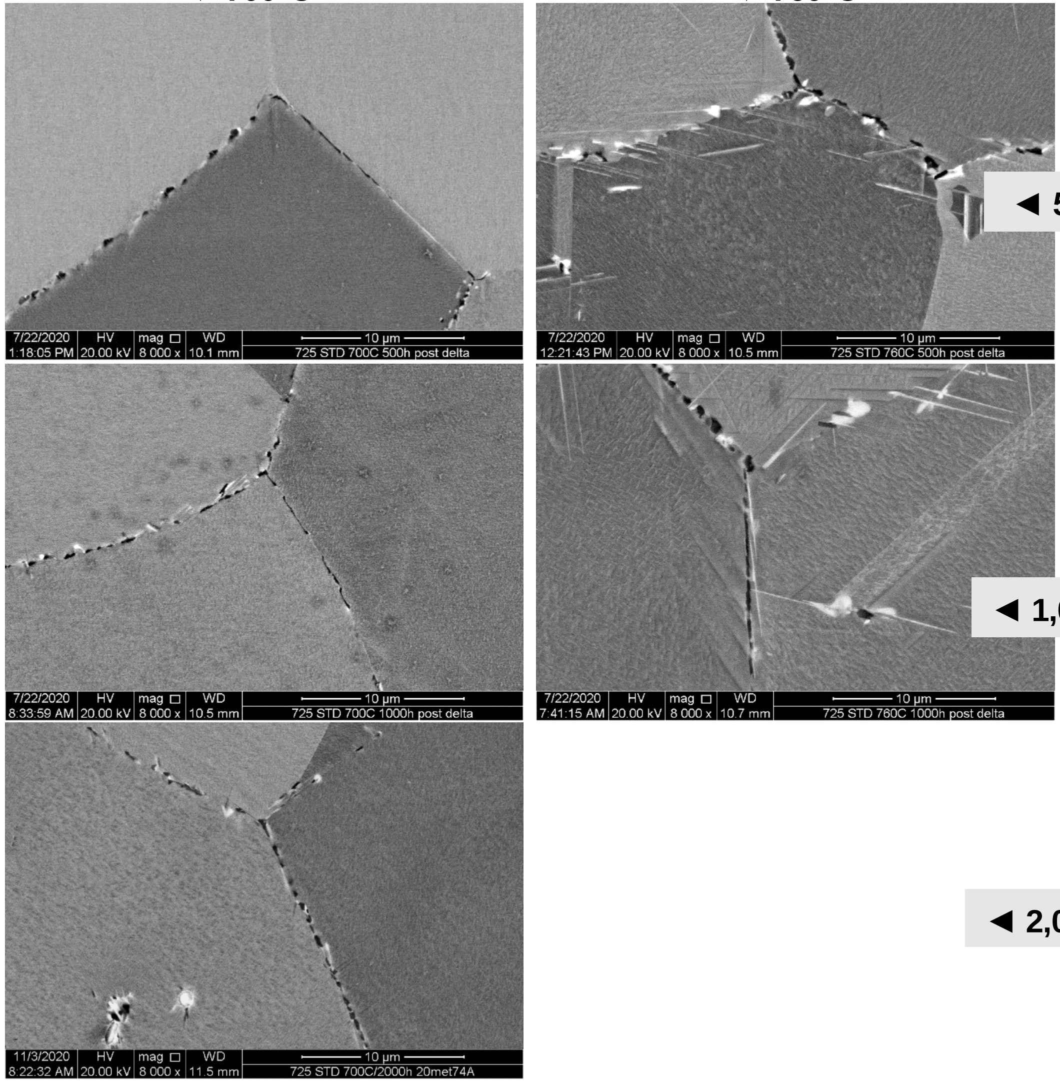
▼ 700°C ▼ 760°C

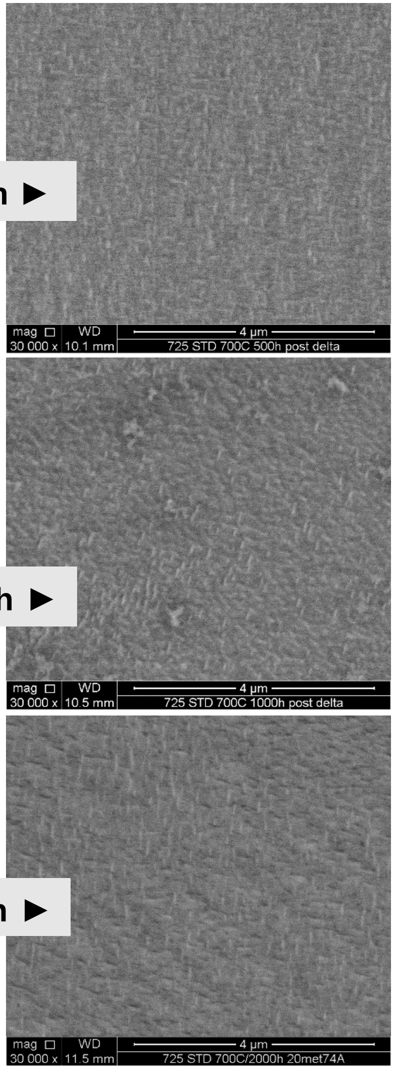


◄ 500h ►

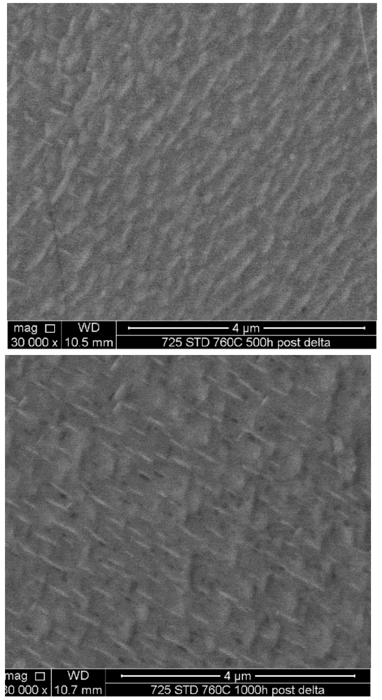
◄ 1,000h ►

◄ 2,000h ►

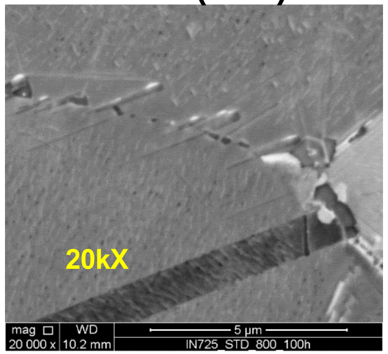
▼ 700°C



▼ 760°C



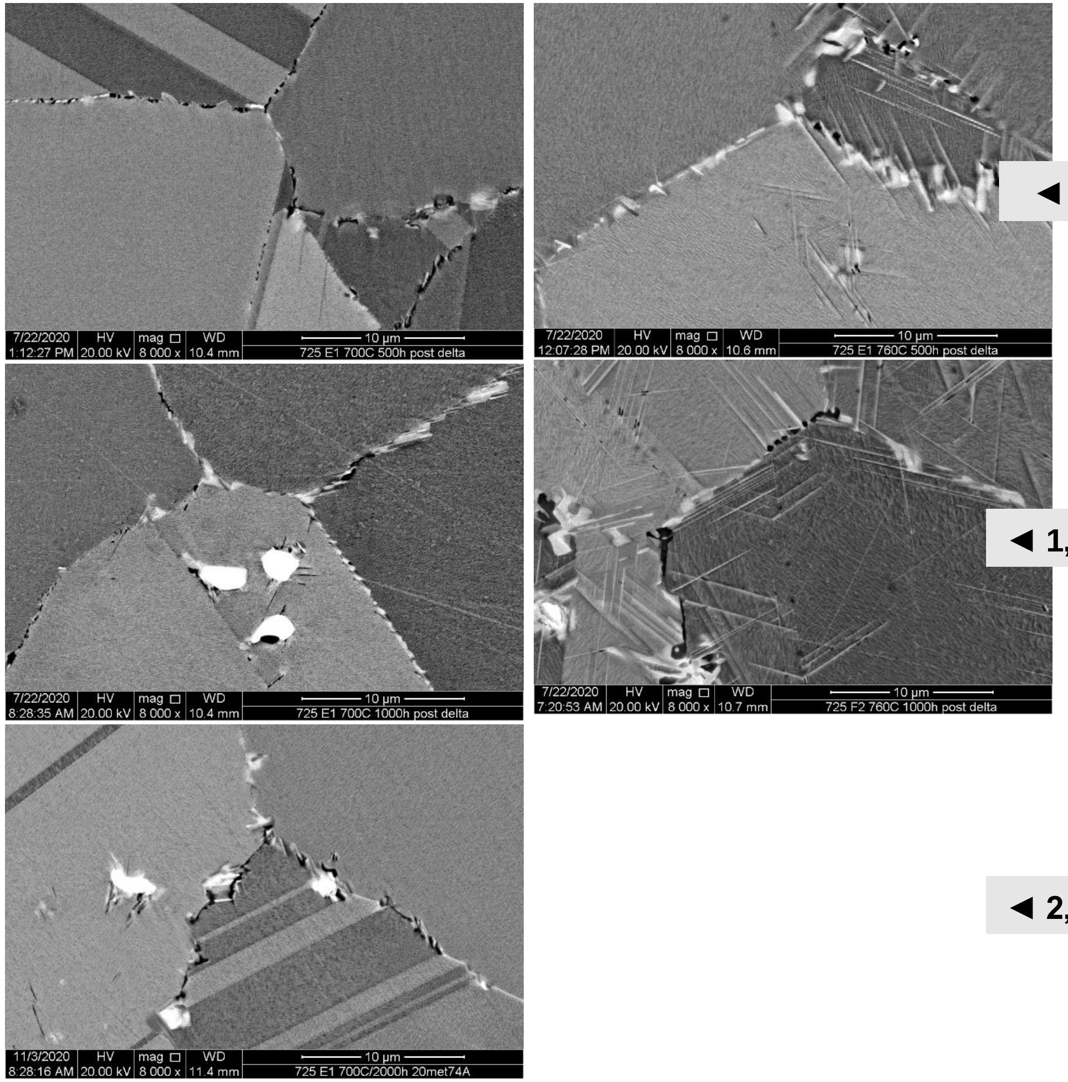
▼ 800°C (100h)



Alloy	Ti/Al ratio	Ta	Nb
STD	7.6	0.04	3.47
E1	9.4	2.40	3.47
F2	9.9	0.25	4.60

Discussion Phase Stability – **Alloy E1**

▼ 700°C ▼ 760°C

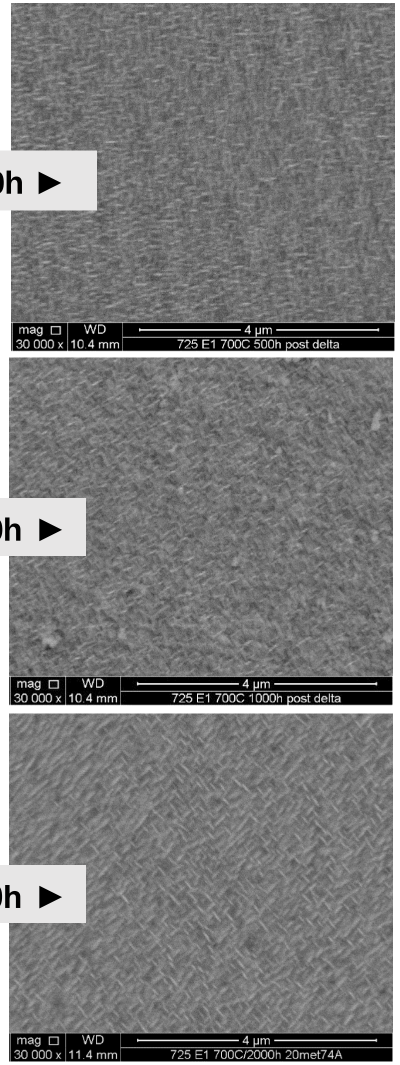


◄ 500h ►

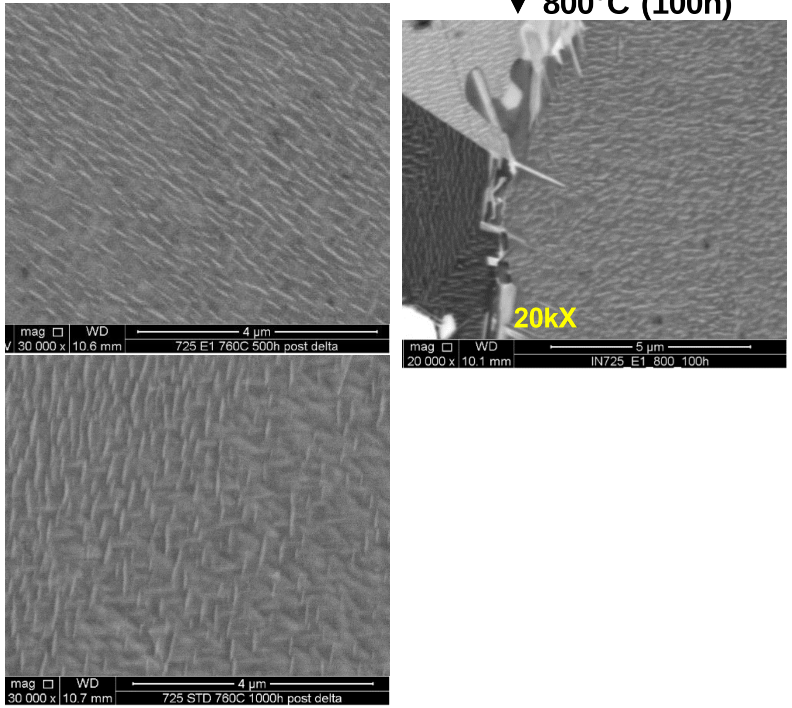
◄ 1,000h ►

◄ 2,000h ►

▼ 700°C



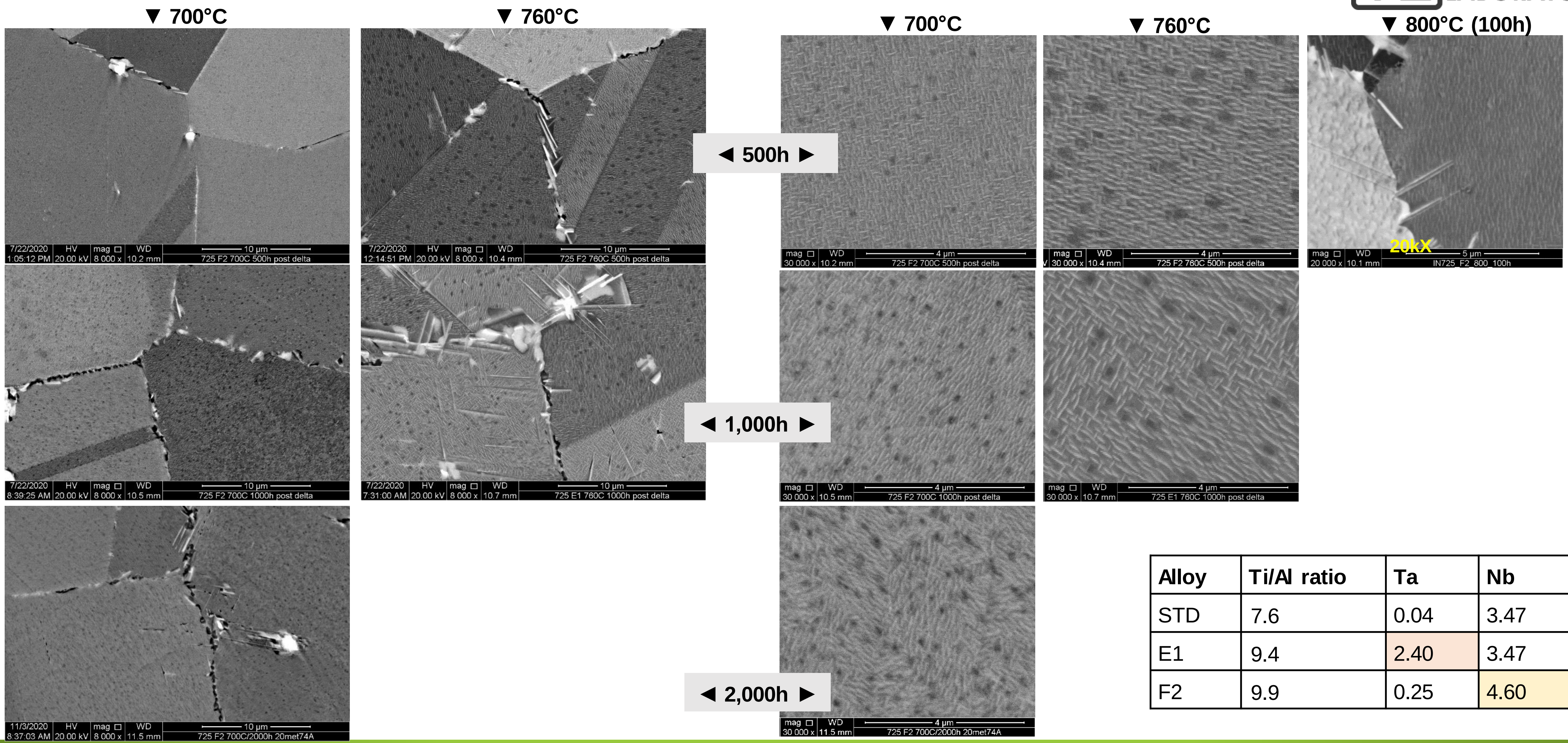
▼ 760°C ▼ 800°C (100h)



Alloy	Ti/Al ratio	Ta	Nb
STD	7.6	0.04	3.47
E1	9.4	2.40	3.47
F2	9.9	0.25	4.60

Discussion

Phase Stability – **Alloy F2**



Alloy	Ti/Al ratio	Ta	Nb
STD	7.6	0.04	3.47
E1	9.4	2.40	3.47
F2	9.9	0.25	4.60

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Conclusions

- **Alloys were designed from the base 725 composition using additions of Nb and/or Ta and a high-temperature "HT" aging heat treatment to enable the use of this corrosion resistant Ni-based superalloy in applications that also require resistance to creep deformation at elevated temperatures.**
- **Changes to the Ti/Al ratio resulted in preferred precipitation of γ′ (low-Ti/Al) or γ″ (high-Ti/Al).**
- **Additions of Ta stabilized the γ″ precipitates.**
- **The combination of fine γ′ and/or γ″ precipitates densely distributed in the matrix and complex repartition of fine precipitates along the grain boundaries improved the mechanical performance of the alloy.**
- **Comparing to Alloy STD in its standard aging heat treatment (SHT, or condition most representative of standard alloy 725/625 PLUS):**
  - The HT aging treatment combined with Nb addition resulted in an improvement of creep life of >120% (although the Ti/Al ratio was changed).
  - The HT aging treatment combined with Ta addition resulted in an improvement of creep life of >250% with similar ductility.
- **A phase stability study suggests a potential operating temperature between 700°C and 760°C (and <760°C) to prevent the growth of the GB precipitates and long-term creep degradation.**

Acknowledgments

This work was performed in support of the US Department of Energy's Fossil Energy Crosscutting Technology Research Program. The authors would like to thank E.R. Argetsinger and J.A. Mendenhall for assistance in melting, C.D. Powell for mechanical testing, R.E. Chinn and C. McKaig for chemistry analysis, M.B. Fortner for metallographic preparation and T.A. Godell for sample machining.

Questions?

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