



Integrated Process Improvement using Laser Processing and Friction Stir Processing for Nickel Alloys used in Fossil Energy Power Plant Applications

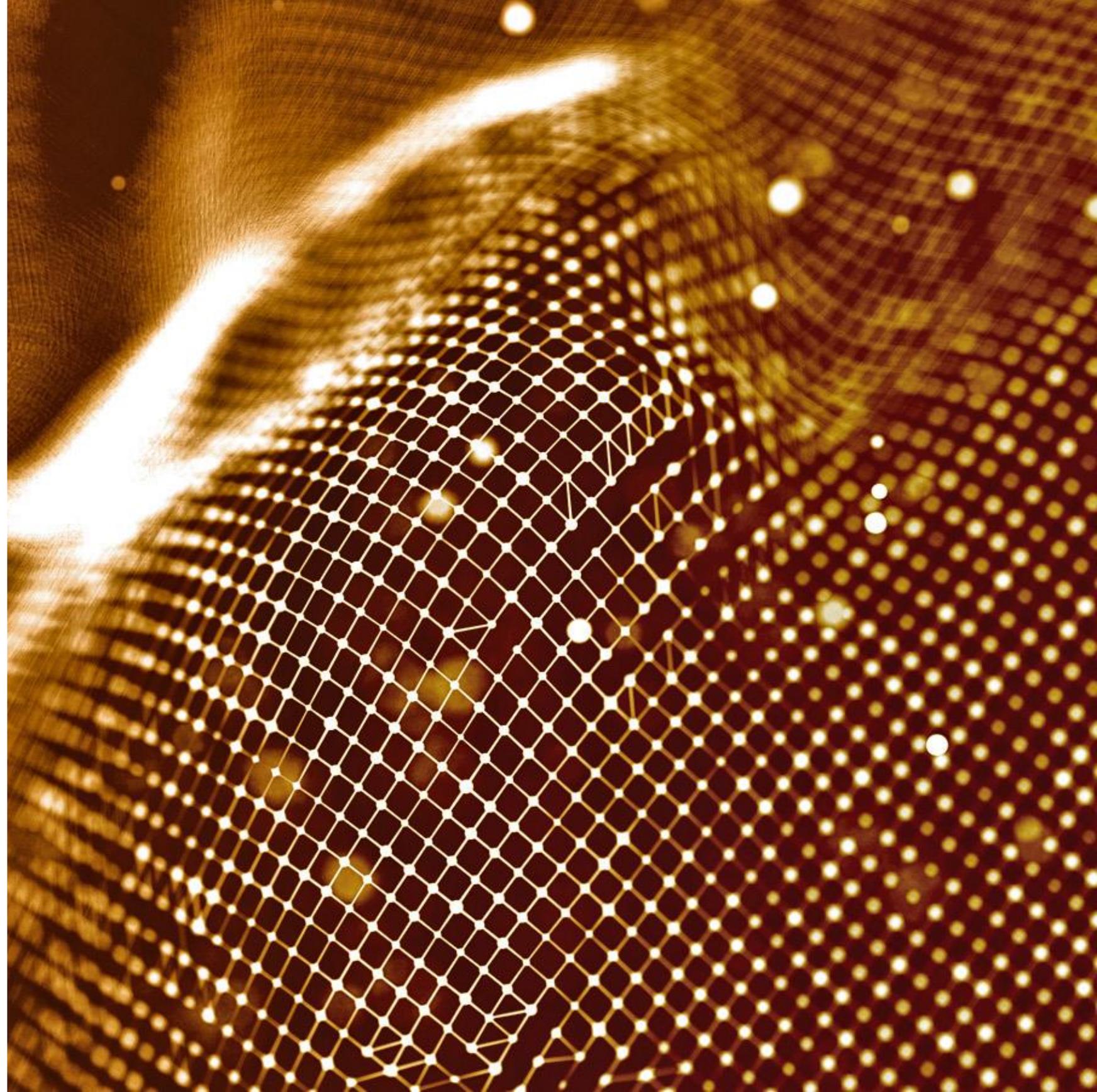
April 19, 2023

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Project Manager and Principal Investigator



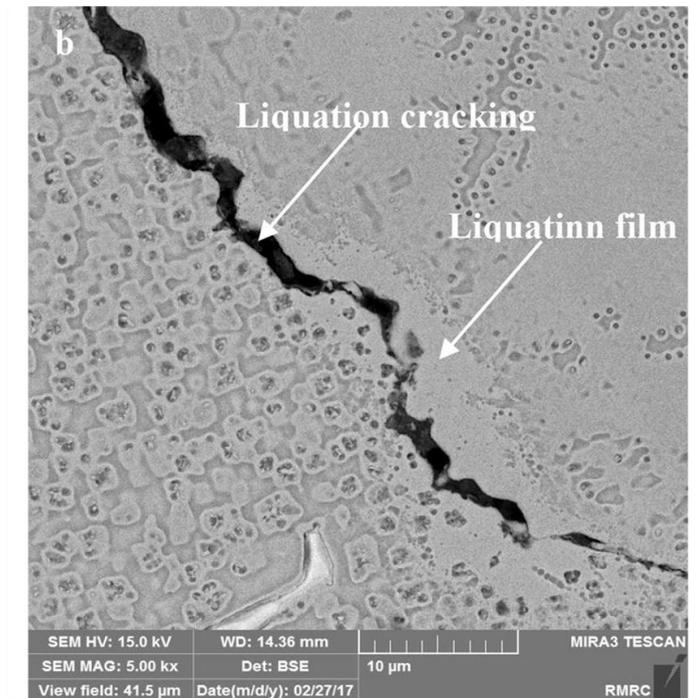
PNNL is operated by Battelle for the U.S. Department of Energy

PNNL-SA-184204



Background

- Nickel-based superalloys are difficult to join with other processes
 - Fusion based processes often lead to liquation cracking
 - Fusion processes require high level of skill and / or high level of process control
- Limited joining processes are used
 - Diffusion bonding
 - Plasma arc welding
 - GTAW Welding
- FSW/P of high melting point alloys is costly, however nickel-based superalloys represent opportunity space for FSW/P
 - Existing processes are costly and time consuming
 - Nickel-based super alloy products are high value

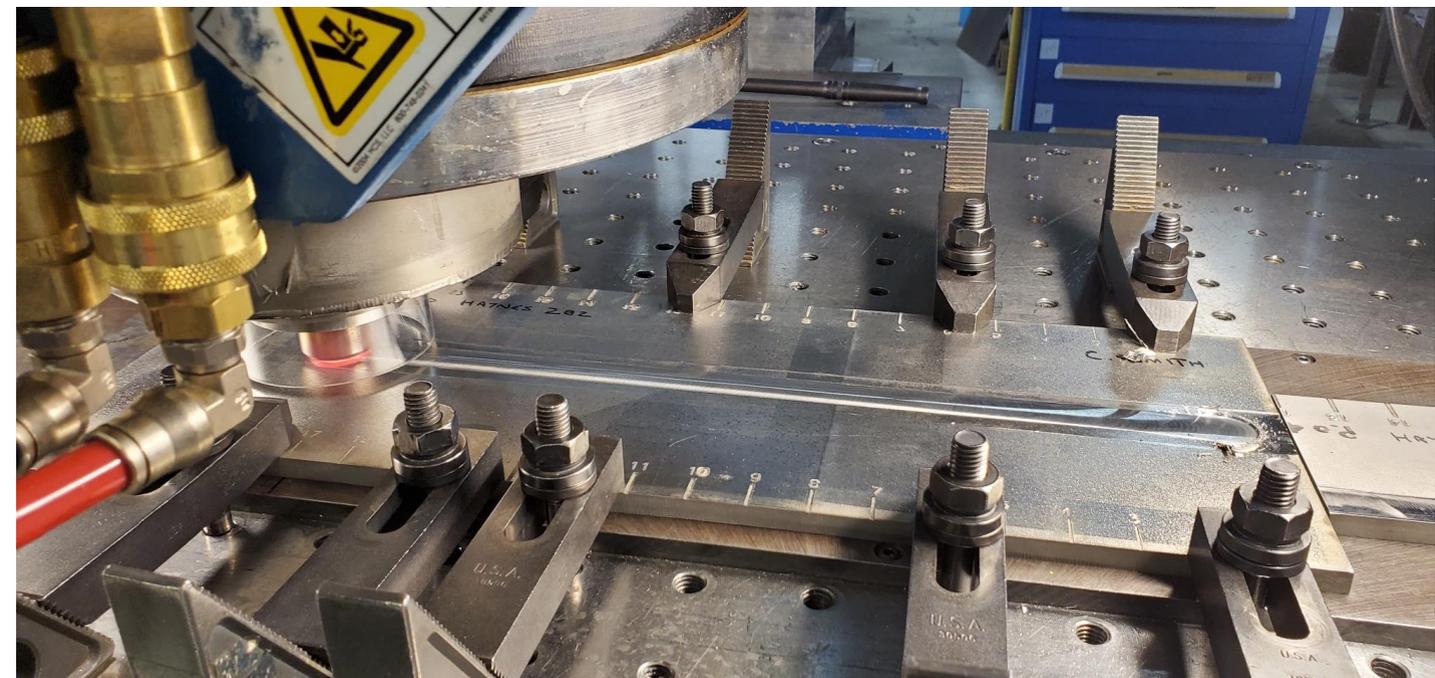


Liquation Cracking of IN-939 Nickel Alloy*

* Kazempour-Liasi et al, Effects of Filler Metals on Heat-Affected Zone Cracking in IN-939 Superalloy Gas-Tungsten-Arc Welds, Journal of Materials Engineering and Performance, 29, 1068-1079 (2020)

Background

- Limited investigation into FSW/P of nickel-based superalloys
 - Order of magnitude or more investigations into FSW/P of steel alloys and other high melting point materials
 - Order of magnitude or more investigations into of FSW/P of aluminum and other lower melting point materials versus that of other high melting point materials
- Literature review suggested limited success in FSW/P of nickel-based super alloys
 - Tool wear and failure noted as a common issue
 - Tensile testing results indicated potential to achieve failure outside FSW/P stirzone is possible
 - Lack of commonality in FSW/P efforts



FSP of Haynes 282 at PNNL

Approach: Focus on Feasibility

- FSW/P of wrought Haynes 282
 - Tensile testing
 - Microstructural characterization
 - ✓ Stirzone
 - ✓ Surrounding area
 - Characterize elevated temperature capability
 - ✓ Effect of high service temperatures on microstructure
 - ✓ Creep
- FSP of Haynes 282 castings: Healing of defects
- FSW/P of Inconel 617
- FSW of dissimilar joint: Haynes 282 to Haynes 233
- Fabricate prototypic part from Haynes 282
 - Fabricate tubing from two C-channels

Nominal Composition Weight %

Nickel:	57 Balance
Chromium:	20
Cobalt:	10
Molybdenum:	8.5
Titanium:	2.1
Aluminum:	1.5
Iron:	1.5 max.
Manganese:	0.3 max.
Silicon:	0.15 max.
Carbon:	0.06
Boron:	0.005

Haynes 282 Composition

FSW of Haynes 282

- Material Condition
 - 5 mm (0.20") and 9.5 mm (0.375")
 - Solution annealed condition
- Weld Parameters w/ argon shielding
 - Travel Speed: 0.42 mm/s (1.0 IPM)
 - Temperature Control: 800 to 850°C (1472 to 1562°F)
 - Axial Force: 62 kN (14000 lbs) → Based on visual quality
 - 50 – 100 RPM (output variable from temperature control)
 - MegaStir PCBN Q60 FSW Tools w/ 4 and 6 mm pin length
- Characterization before and after standard 2-step heat tx
 - Full solution anneal
 - Step 1: 1010°C(1850°F) for 2 hours then air cooled
 - Step 2: 788°C(1450°F) for 8 hours then air cooled



FSW/P Setup

Example
FS Tool

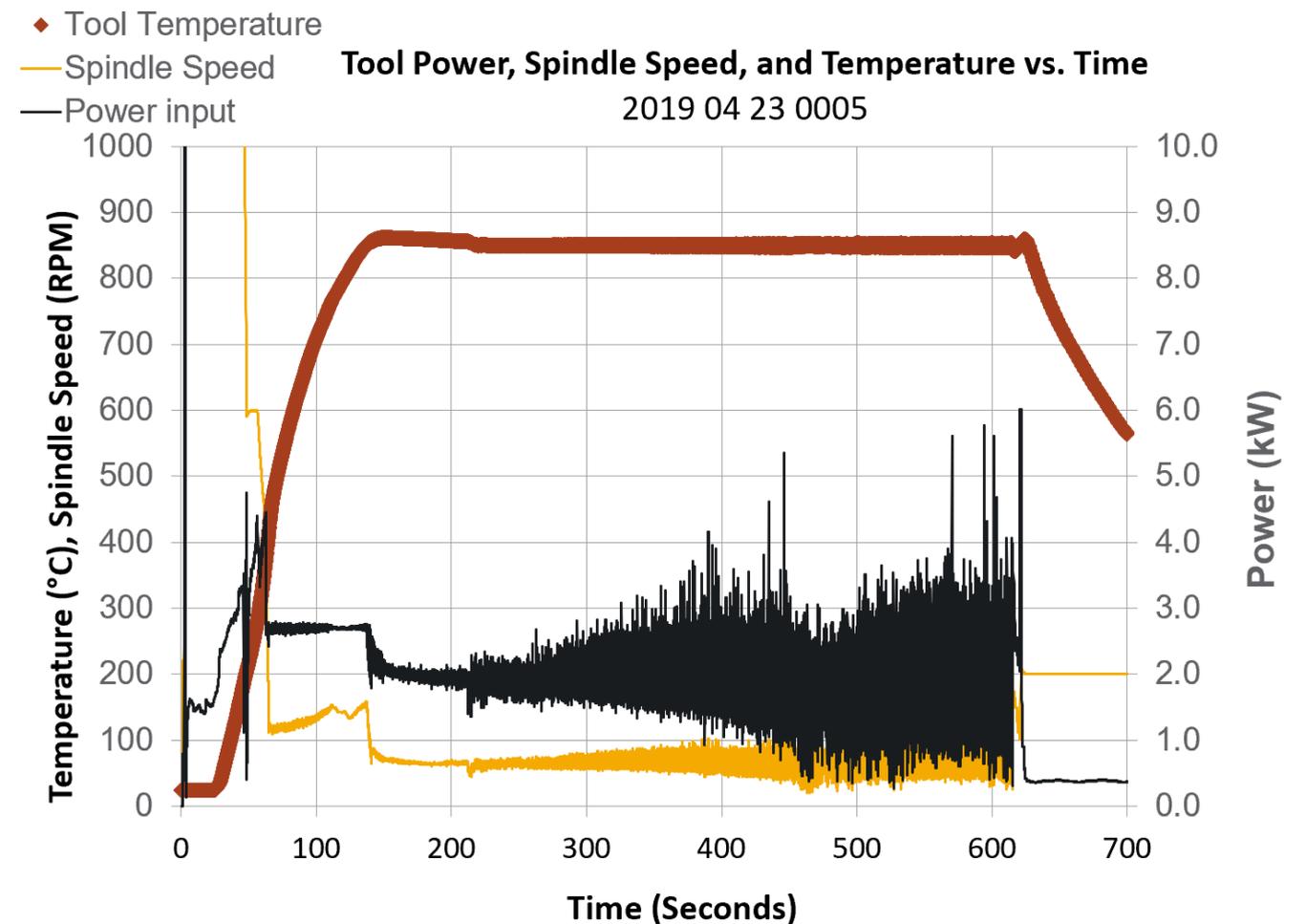


Example FSP of Haynes 282



FSW/P of Haynes 282

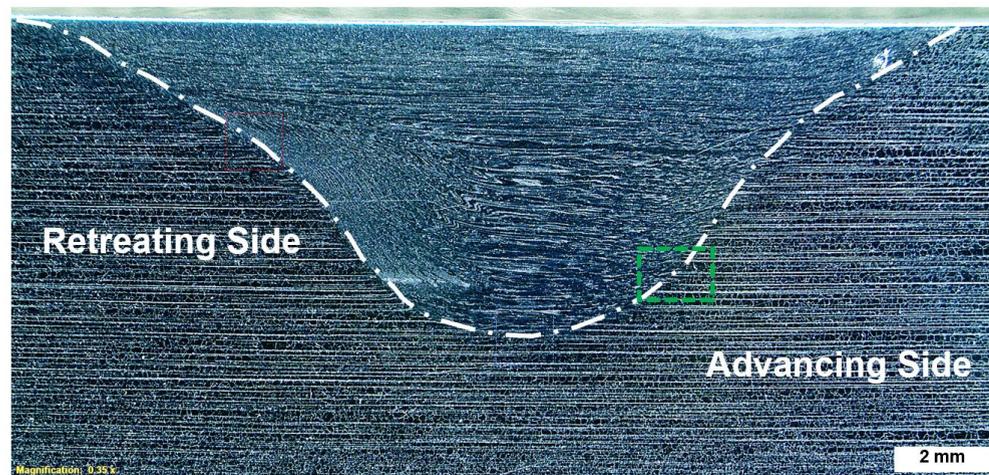
- Temperature control algorithm for successful FSW/P of steel alloys resulted in oscillatory behavior
 - Controller tuning required
 - Defaulted to manual temperature control for initial efforts
- Oscillation led to premature failure of FS tools
 - Pin failure
 - Shoulder cracking



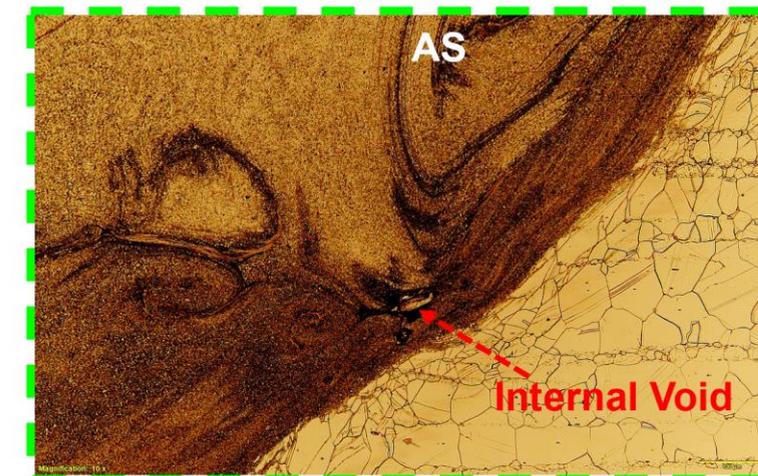
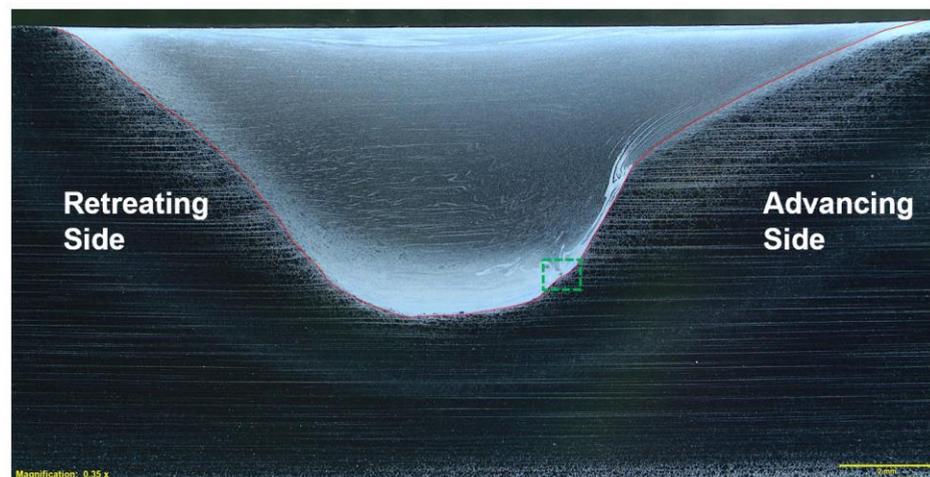
Power Input, Spindle Speed and Temperature during 850°C FSP

FSW/P of Haynes 282 Optical Microscopy

- Acceptable welds created at 850°C, with small internal void present at 800°C
- Further efforts focused on FS temperature range from 825 to 850°C



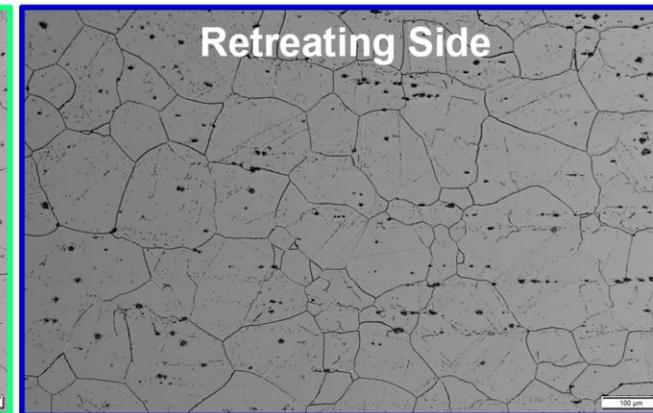
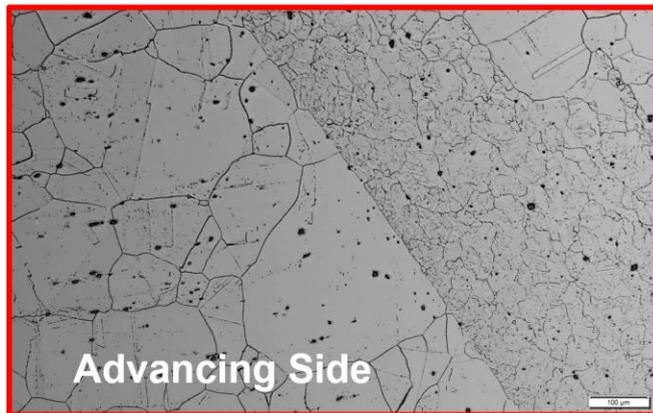
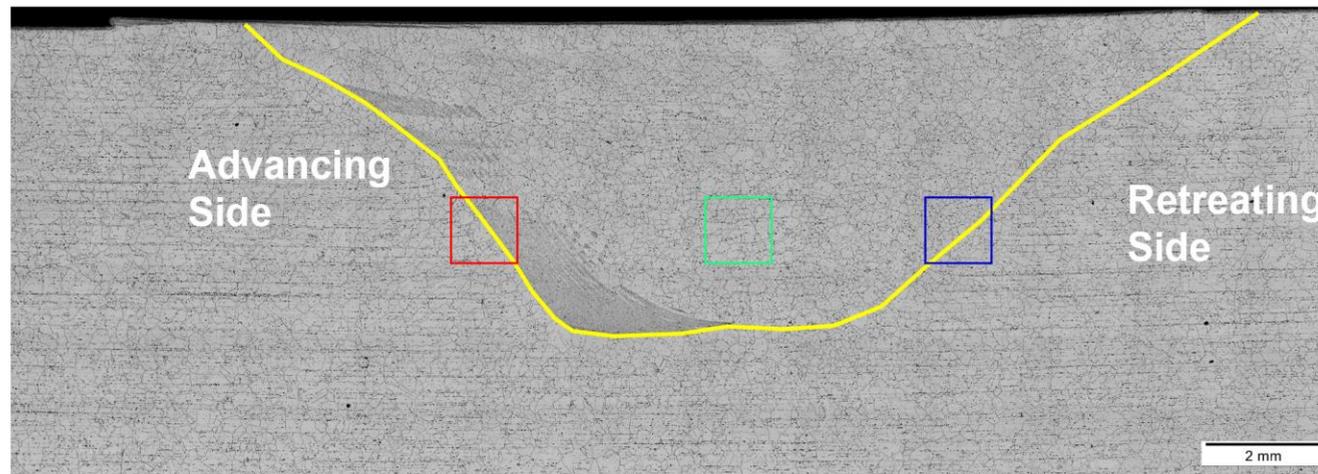
Haynes
282 FSP
at 850°C



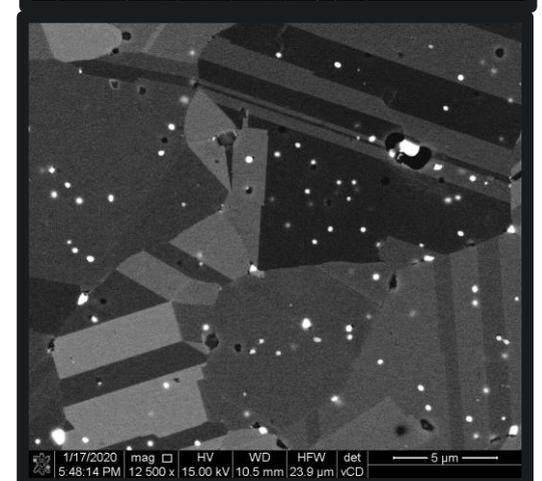
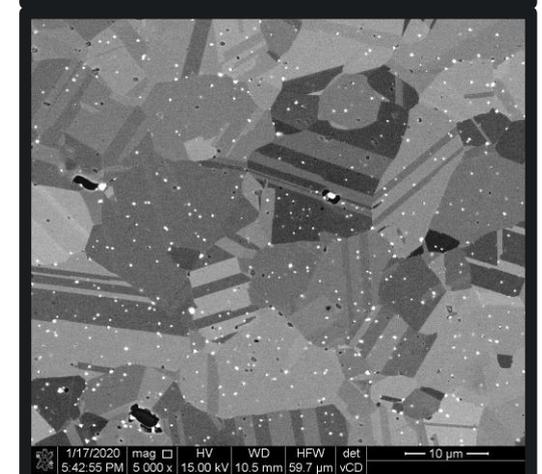
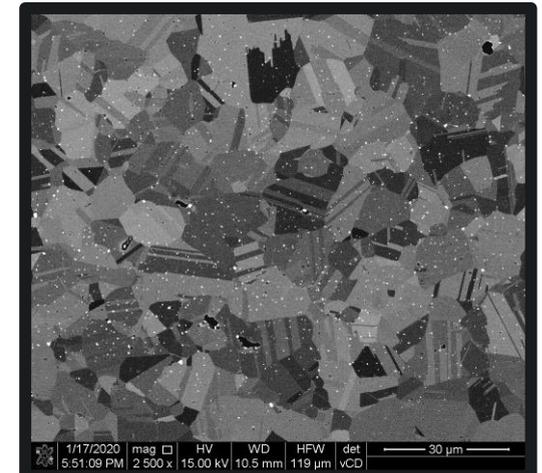
Haynes
282 FSP at
800°C

FSW/P Microstructural Characterization

- Microstructure after heat treatment
 - Consistent from base metal to FSW/P stirzone in all but small areas



FSW/P +
two-step
heat
treatment
micro-
structure



Increasing magnification

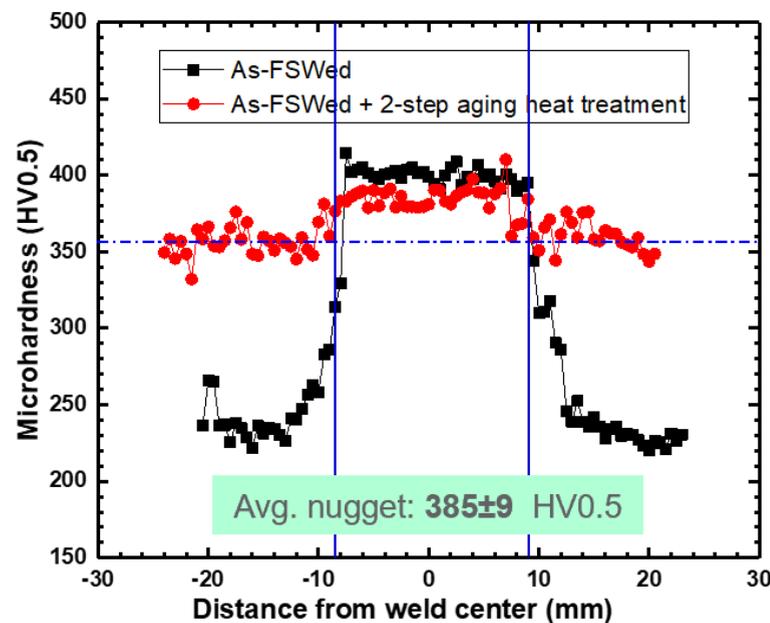


Microstructure after FSP + Heat Treatment in multiple locations

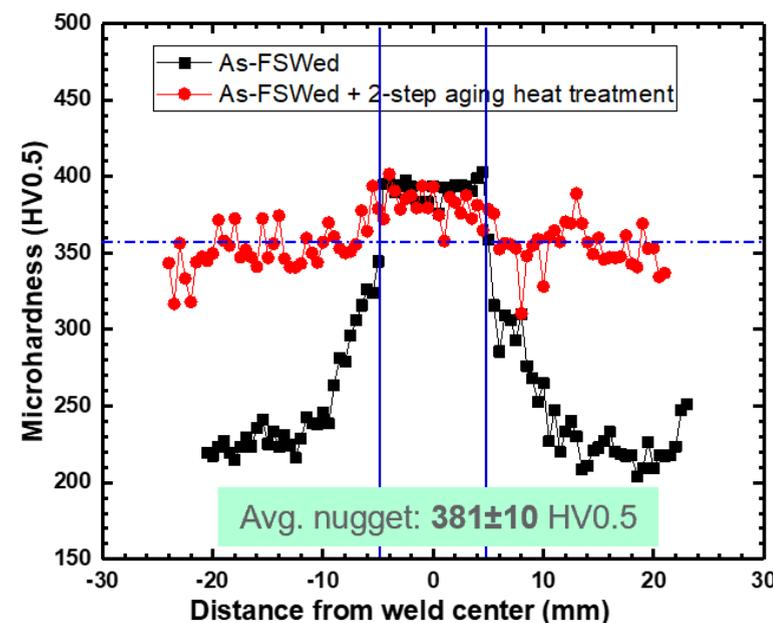
Microhardness Characterization

- Microhardness Testing
 - Before heat treatment and after heat treatment
- Results
 - FSP nugget harder than base material after FSP
 - After heat treatment microhardness consist from base material to nugget

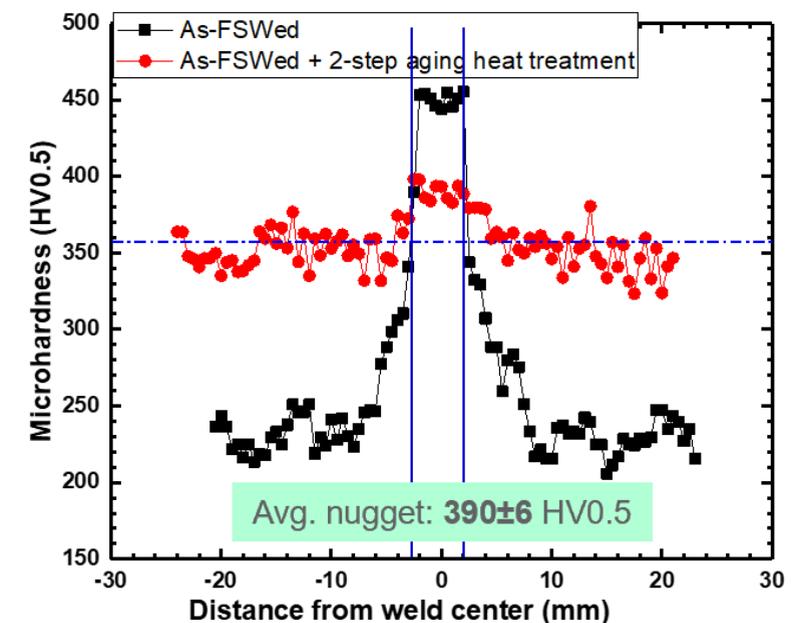
0.5 mm from top surface



3.0 mm from top surface



5.5 mm from top surface



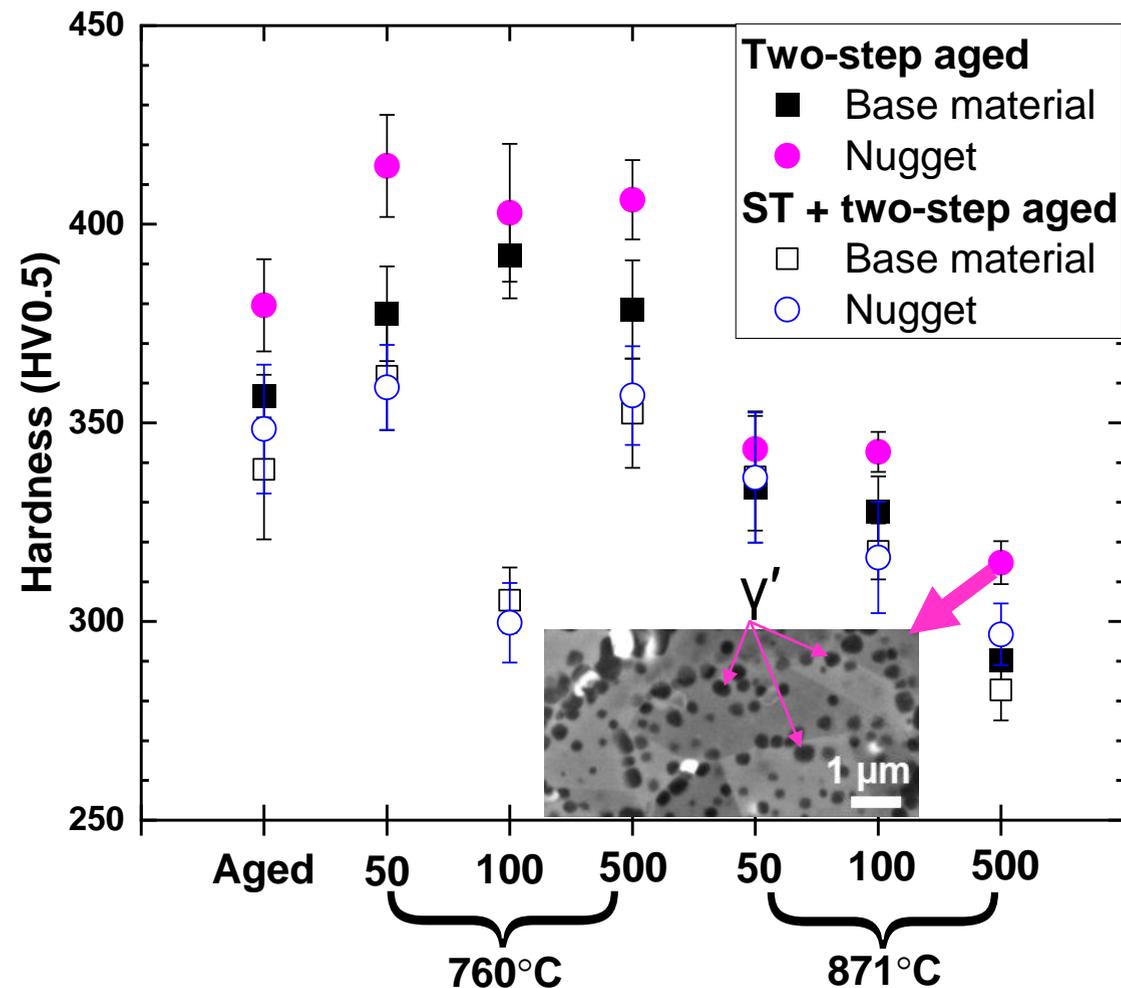
Thermal Stability Characterization

- 2 Heat treatment methods

- Method 1: Two-step aged
- Method 2: Solutionized + two-step aged



- Two post heat treatment thermal exposure conditions
 - Condition 1: 760°C (1400°F) for 50 hours, 100 hours, and 500 hours
 - Condition 2: 871°C (1600°F) for 50 hours, 100 hours, and 500 hours



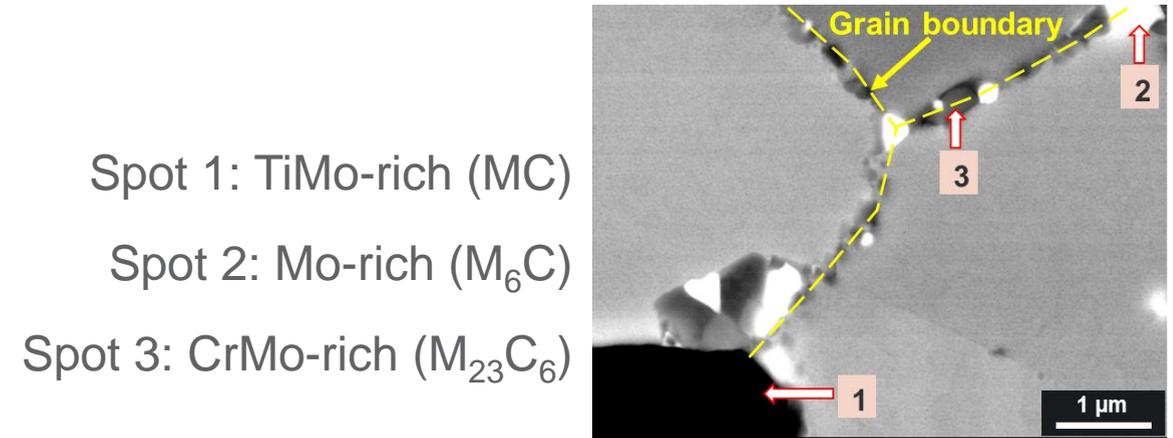
- In comparison with post-heat treated conditions both the methods exhibited similar observations.
- Upon 760°C exposure, hardness increase was observed.
- Upon 871°C exposure, reduction in hardness was noted due mainly to coarsening of the γ' precipitates ($\sim 200 \pm 150$ nm).
- All these observations are similar for both base metal and the processed region.

Hardening at 760°C and softening at 871°C

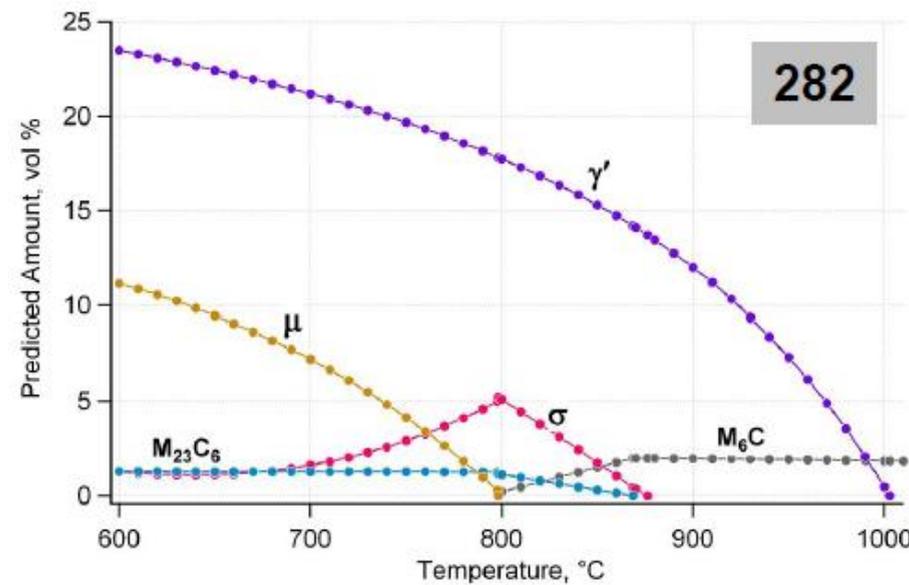
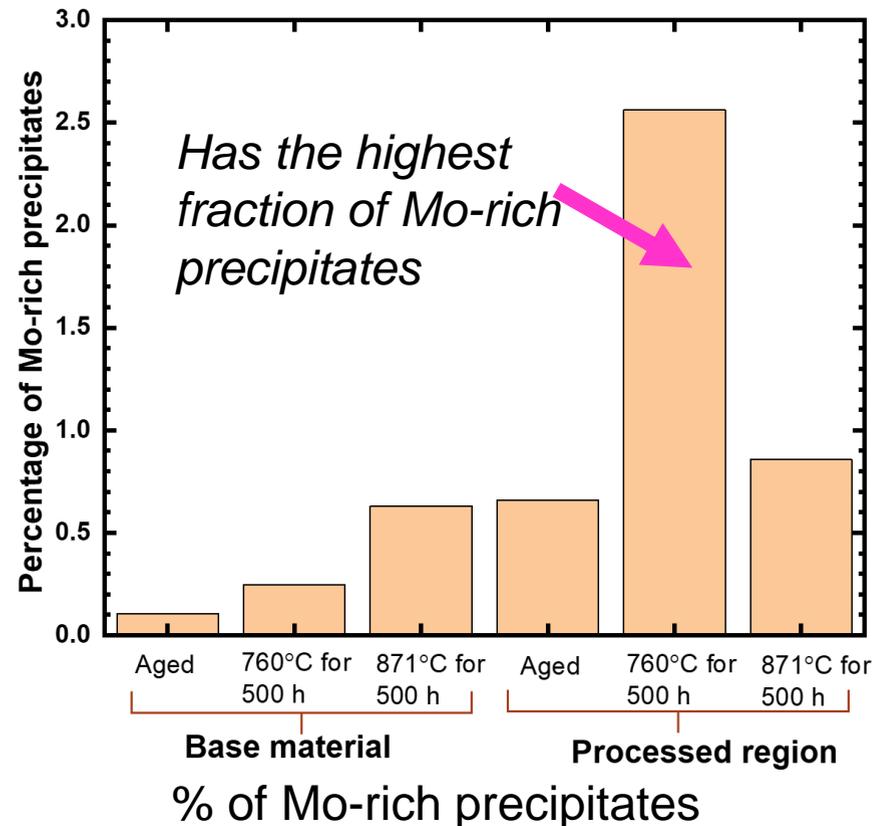
Thermal Stability Microstructural Analysis

- Precipitate analysis

- Mo-rich phases should be prevalent in 871°C but not in 760°C condition.
- M_6C occurs from 780°C to 1080°C
- Therefore, further analysis is underway in terms of Mo supersaturation in processed region

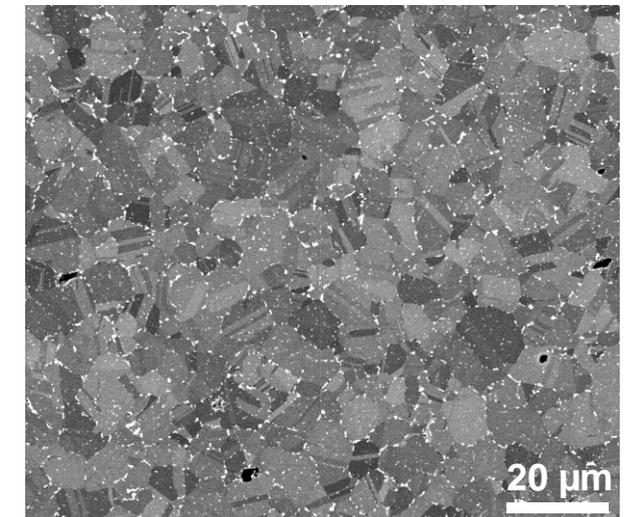


FSP region for two-step aged + thermal exposure at 760°C for 500 hours)



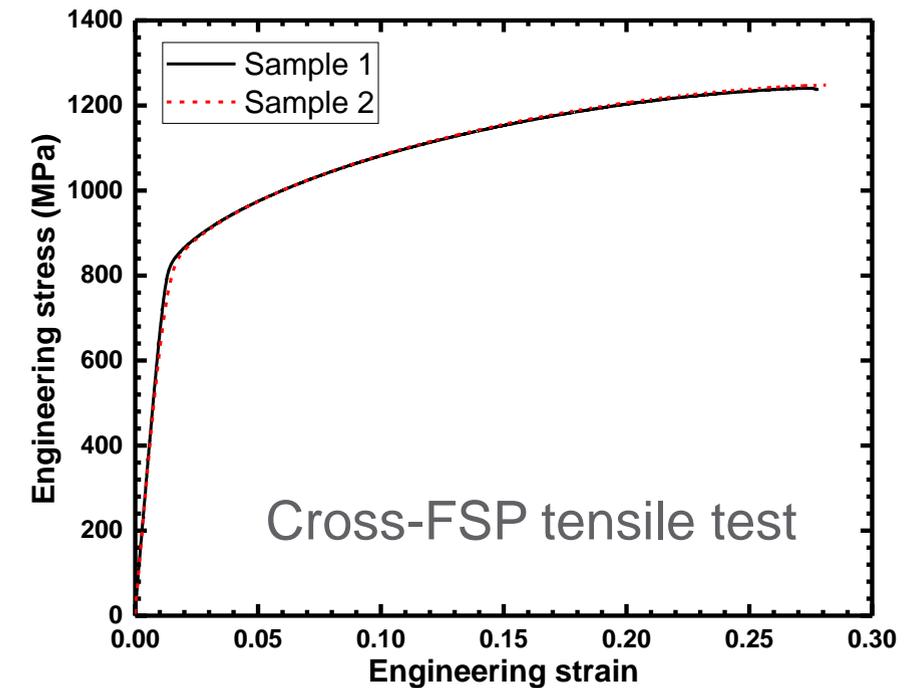
Phase diagram*

*Ni-based alloys for advanced ultrasupercritical steam boilers



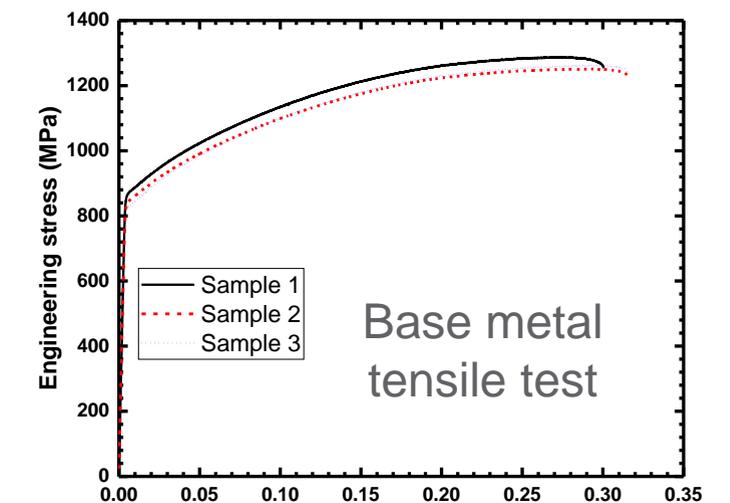
Tensile Testing

- 2 Heat treatment methods
 - Method 1: Two-step aged
 - Method 2: Solutionized + two-step aged
- Processed region tensile properties similar to the base metal resulting in a joint efficiency of 100%.
- All the cross-weld FSP samples failed in the base metal.



Tensile properties summary

Conditions	YS (MPa)	UTS (MPa)	TE (%)	Failure location
Base metal	850±18	1266±19	30±1	N/A
FSP method 1	824±9	1244±5	N/A	Base metal
FSP method 2	823	1211±1	N/A	Base metal
Fusion welding: Literature observations*				
Base metal	734±1	1119±2	43.2±0.3	N/A
Fusion weld	634±8	984±7	N/A	Fusion/base interface
Joint Efficiency	86.3%	88.0%		

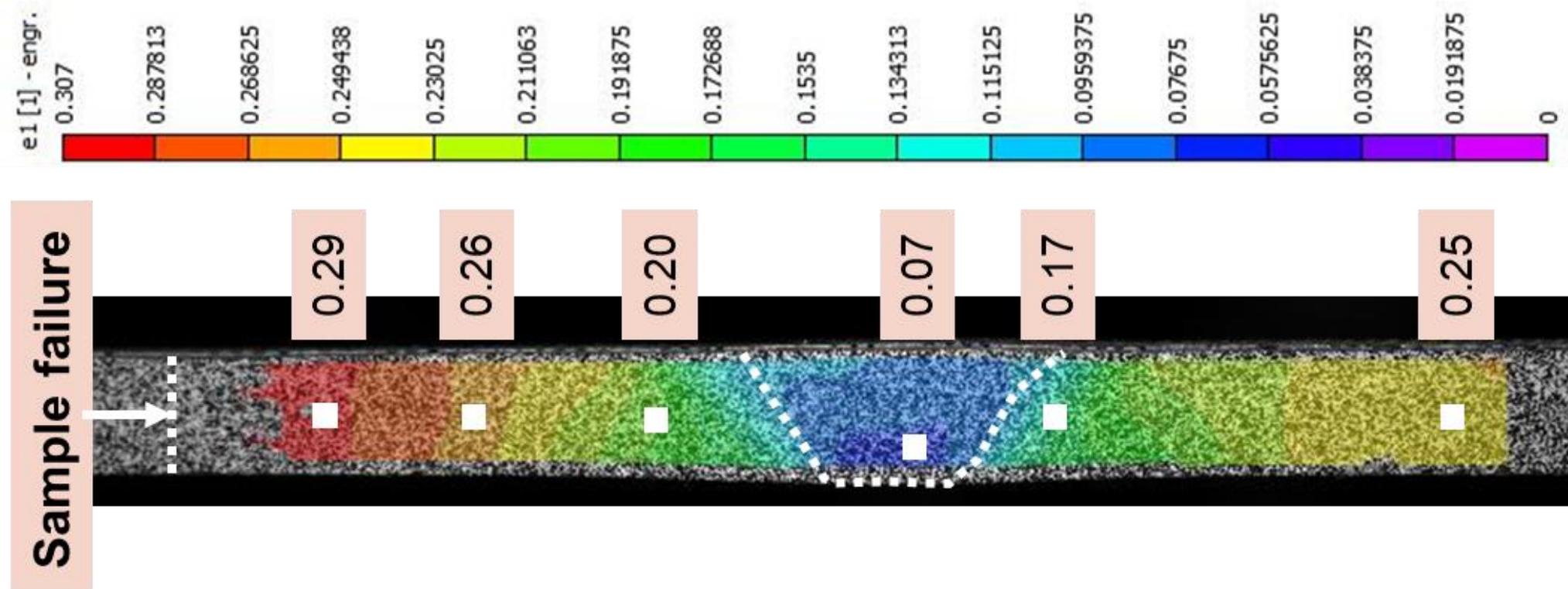


*A. Brittan, J. Mahaffey, M. Anderson, The performance of Haynes 282 and its weld in supercritical CO₂, Materials Science and Engineering: A 759 (2019) 770-777

Tensile Testing

- The lowest local strain was observed in the processed region
- The highest local strain was noted in the base metal

Tensile properties summary



Digital Image Correlation (DIC) analysis



FSW/P Characterization

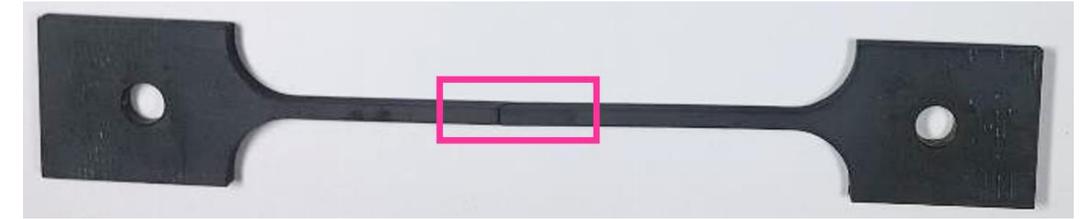
- Elevated Temp Creep Testing

- At 760°C
- 310 MPa (45 ksi)
- 190 MPa (27.5 ksi)
- Cross weld and all FSP

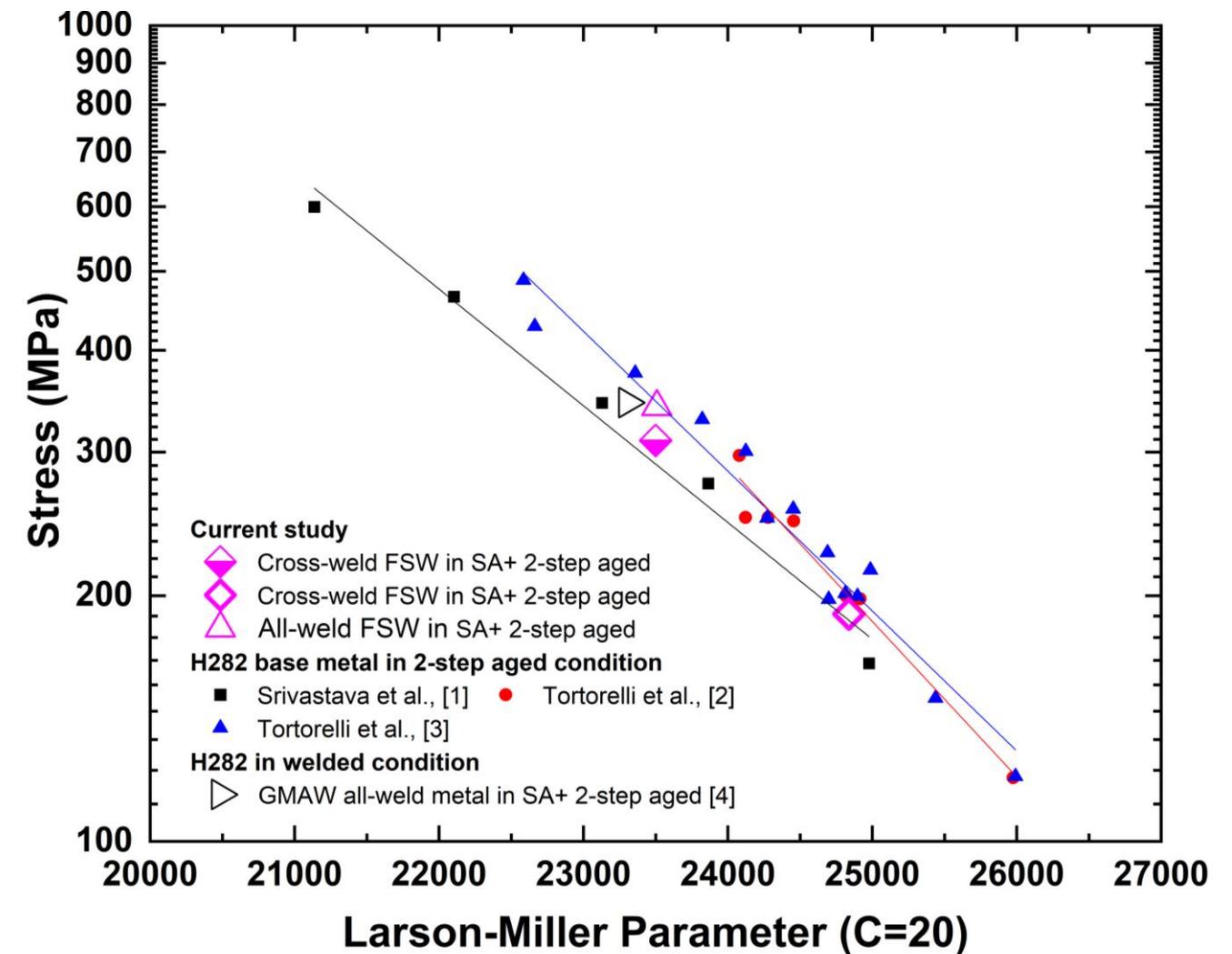
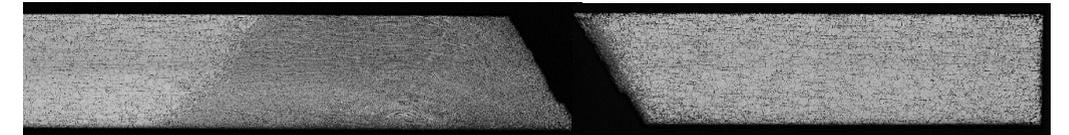
PNNL ATS 2330-MM Creep Testing Machine



Creep Testing Coupon Material Area



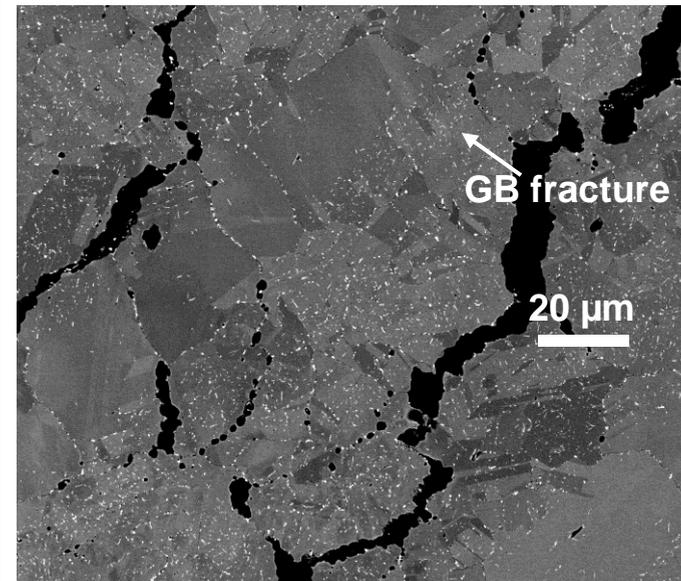
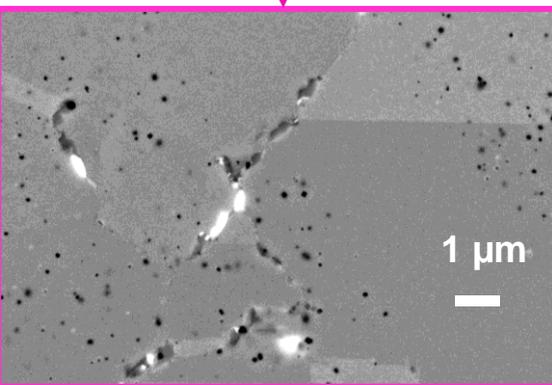
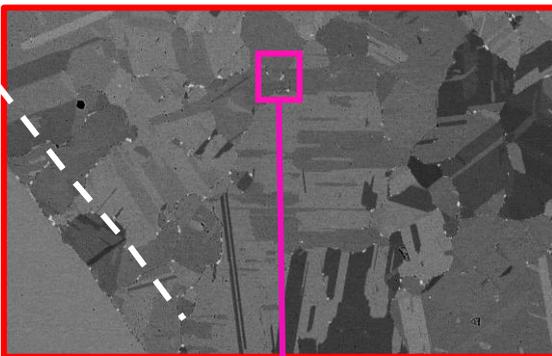
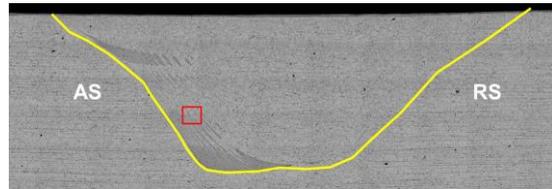
Creep Test Coupon and Failure Location



Creep – Microstructural observations

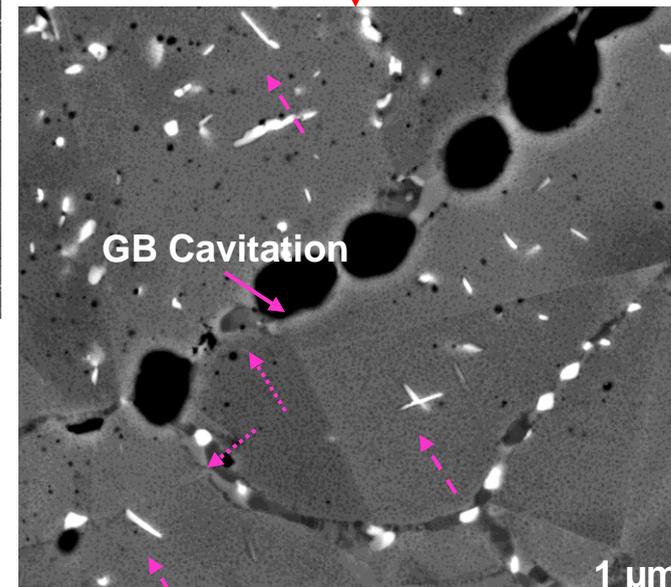
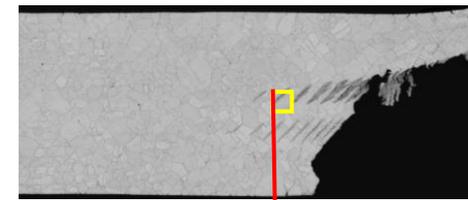
- Grain boundary fracture and sample failure were primarily in the banded region
 - As FSP (before creep testing), mostly, MC and $M_{23}C_6$ precipitates in the banded region.
 - Differences appear after creep testing

Before creep – banded region

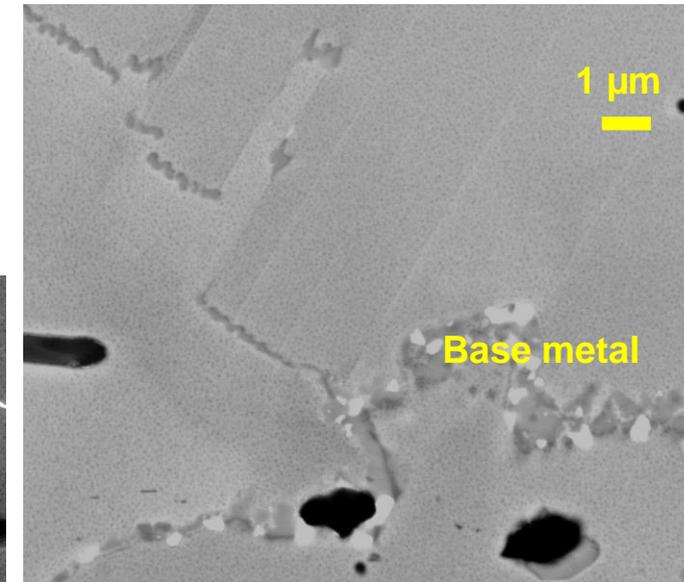


Presence of Mo-rich phases along grain boundary (GB) and grain interior.

After creep



Platelet-like phase could be μ phase $((Ni,Co)_7Mo_6)$ or σ $(FeCr/FeCrMo.CrCo)$, marked by dashed arrows.

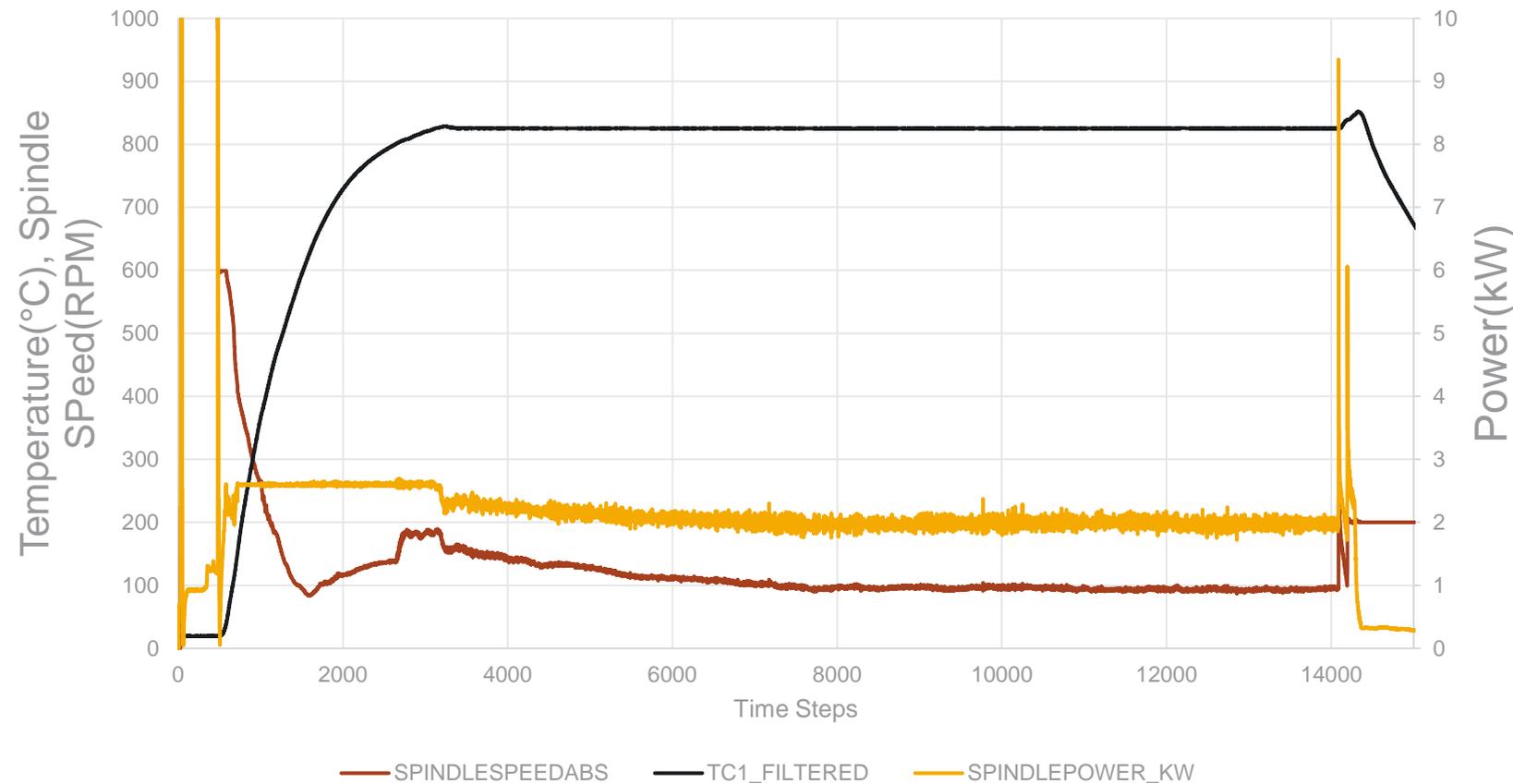


$M_{23}C_6$ and M_6C are marked with dotted arrows.

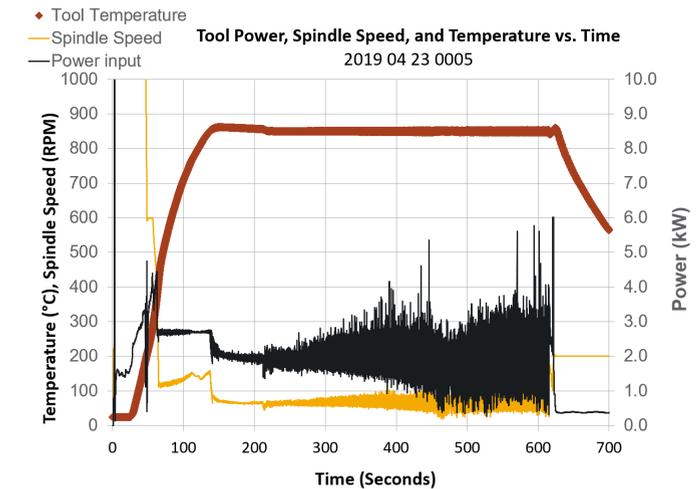
FSW/P of Haynes 282 Temperature Control

- Most recent temperature control results
 - Spindle speed and power stabilized

Tool Power, Spindle Speed, and Temperature vs. Weld Length



Power Input, Spindle Speed and Temperature with Temperature Control Active



FSP Data from early temperature control trials



Temperature Controlled FSP

Prototypic Haynes 282 Part Fabrication

- Rectangular tube from two friction stir welded C-Channels
 - 250 mm x 300 mm rectangular tube
 - 300 mm length
 - 6.35 mm wall thickness



Fixture



FSW in Progress

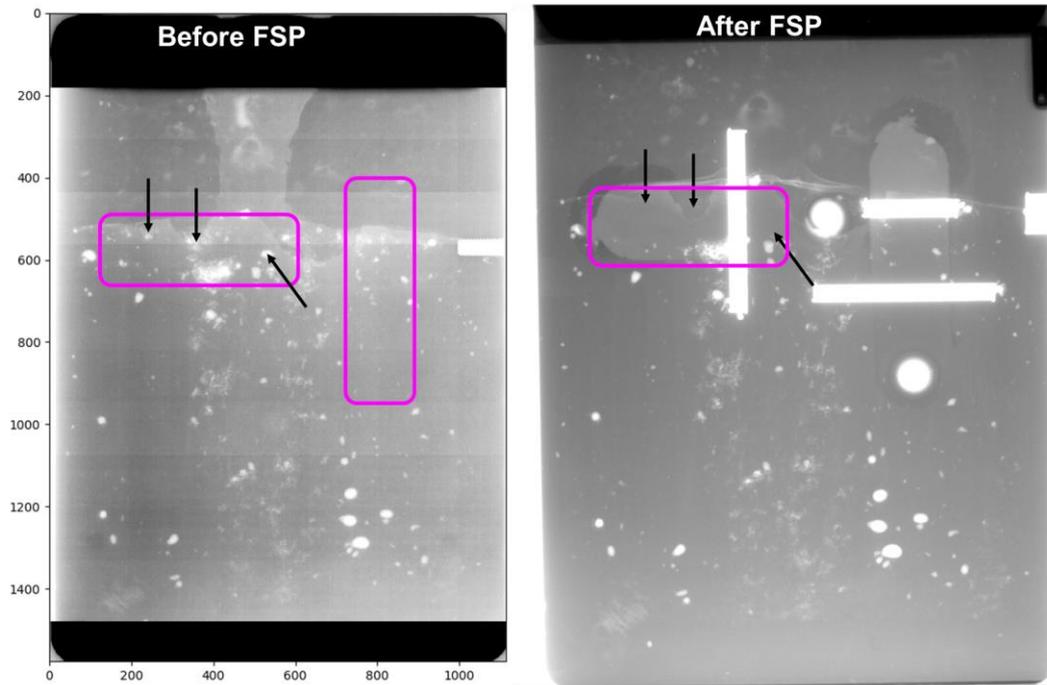


Example Welds

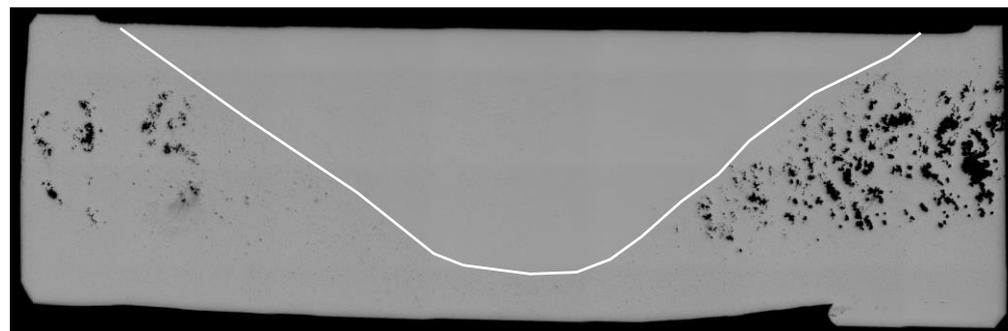


Other Efforts

- FSP of castings
 - Can FSP heal porosity or cold shuts?



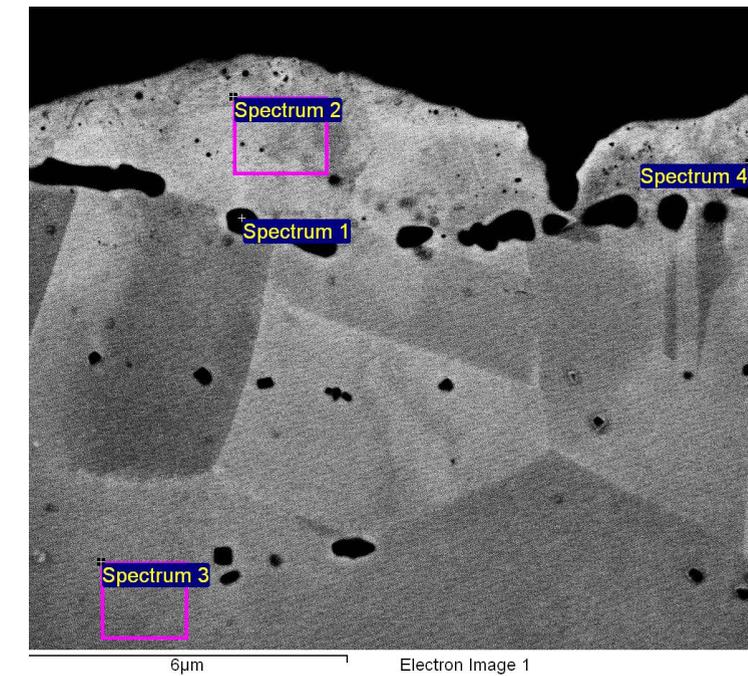
FSP Stirzone in region of porosity



FSP
of LA
plate



- Laser Ablation
 - Microscopy of and FSP of LA surfaces



Cross-section of LA surface

Chemistry in and around LA affected material

Spectrum	O	Al (↓)	Ti (↓)	Cr (↓)	Fe (↑)	Co	Ni	Mo (↑)	Total
Matrix (Spectrum 3)	0.26	1.13	1.27	15.70	0.76	10.25	62.33	8.29	100.00
Laser processed region (Spectrum 2)	0.36	0.33	0.58	10.80	2.12	11.06	63.54	11.20	100.00
Laser processed region (Spectrum 4)	0.37	0.22	0.60	10.73	2.86	10.60	63.92	10.70	100.00
Dark particles at the boundary (Spectrum 1)	43.00	38.25	0.81	4.22	0.00	1.48	6.52	5.72	100.00
Dark particles in the matrix	0.62	0.77	16.76	14.47	0.63	7.79	43.96	15.00	100.00

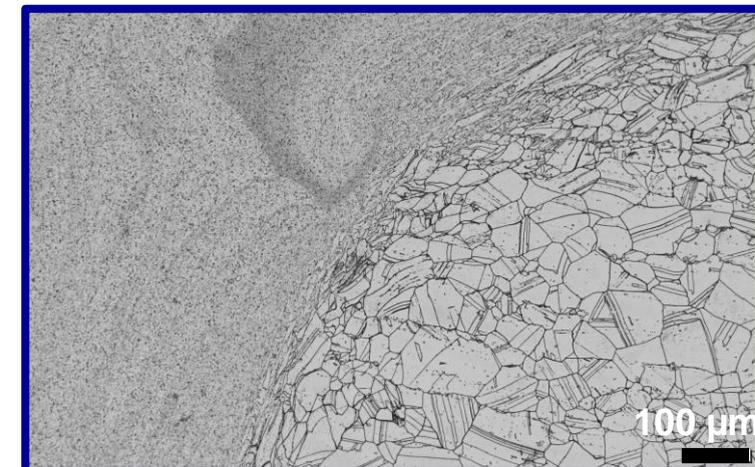
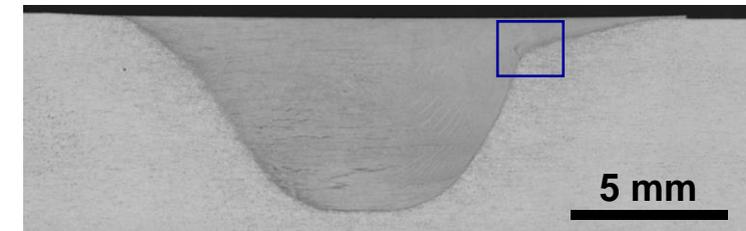
Other Efforts

- Dissimilar material FSW
 - Can FSW be used to locally customize material selection?



FSW Haynes 282 to Haynes 233

- FSW/P Inconel 617
 - Feasibility of FSW/P to join or process other nickel super alloys
 - Requires higher processing temperatures to avoid internal indications



FSP Inconel 617 Cross Section

Summary from Efforts to Date

- Nickel-based superalloys can be successfully friction stir welded and processed
- Efforts in this project suggest that low rotation speeds and processing temperatures are important to successful FSW/P of nickel-based superalloys
- FSW/P of solution annealed Haynes 282 significantly increases hardness in the stirzone. However, the standard two-step heat treatment yields similar hardness from the base material through the FSW/P regions
- Results indicate that FSW/P of Haynes 282 plus post solution anneal + FSW/P two-step heat treatment (standard fabrication process) yields FSW/P mechanical properties (creep and tensile) can meet base material properties.
- FSW/P can heal defects in cast material
- Inconel 617 similarly processable by FSW/P, but appears to require higher processing temperatures

Final Efforts: Effect of FSP on Diffusion Bonding

- Fine grain microstructure of FSP offers the potential opportunity to improve the diffusion bonding process
 - Reduce cycle time
 - Reduce surface preparation (polishing) requirements
- Status
 - Created FSP for effort
 - Samples cut into 1" x 1" coupons
 - Created test matrix with varying surface roughness and diffusion bonding times
 - Siemens will perform polishing and diffusion bonding
 - PNNL will perform post diffusion bonding characterization



FSP Haynes 282 Plate Cut Plan for
Diffusion Bonding Trials

Presentations and Publications

- Presentation at 2022 International Symposium on Friction Stir Welding
 - Focus on process development
- Presentation at TMS 2022
 - Focus on thermal stability analysis of FSP
- Journal Publications
 - M. Komarasamy, C. Smith, J. Darsell, W. Choi, G. Grant, and S. Jana, “Microstructure and mechanical properties of friction stir welded Haynes 282,” *Materials Characterization*, 182 (2021) 111558
- Planned journal publications
 - Elevated temperature mechanical properties
 - Thermal stability of FSP

Acknowledgements

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- Thank you to
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Thank you