



# Powder Metallurgy Hot Isostatic Pressing of 316H Stainless Steel

June 2023

*Changing the World's Energy Future*

Tate Patterson



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**Tate Patterson**

**June 2023**

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Idaho Falls, Idaho 83415**

**<http://www.inl.gov>**

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U.S. Department of Energy  
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# **Powder Metallurgy – Hot Isostatic Pressing of 316H Stainless Steel**

## **ASME Code Case Development in Support of Microreactors**

**Joint ART Materials/AMMT Program Review**

**DOE Headquarters, Germantown, MD**

**June 5-8, 2023**

**Tate Patterson**

**Idaho National Laboratory**



# Fiscal Year 2023 Work Package

- **AT-23IN0804094 – Structural Materials**

## Team

- **Tate Patterson (INL)**
- **Ryann Bass (now at US NRC)**
- **Richard Wright (Structural Alloys, LLC)**
- **Sam Sham (INL)**

# Background

- Powder metallurgy hot isostatic pressing (PM-HIP) is a manufacturing technique to produce metallic components by consolidating metal powder
  - Minimizes additional fabrication steps
  - Eliminates directional grains and solidification structures



MTC Powder Solutions



UK - Nuclear Advanced Manufacturing Research Center (NAMRC) System

# PM-HIP Adoption for Microreactors

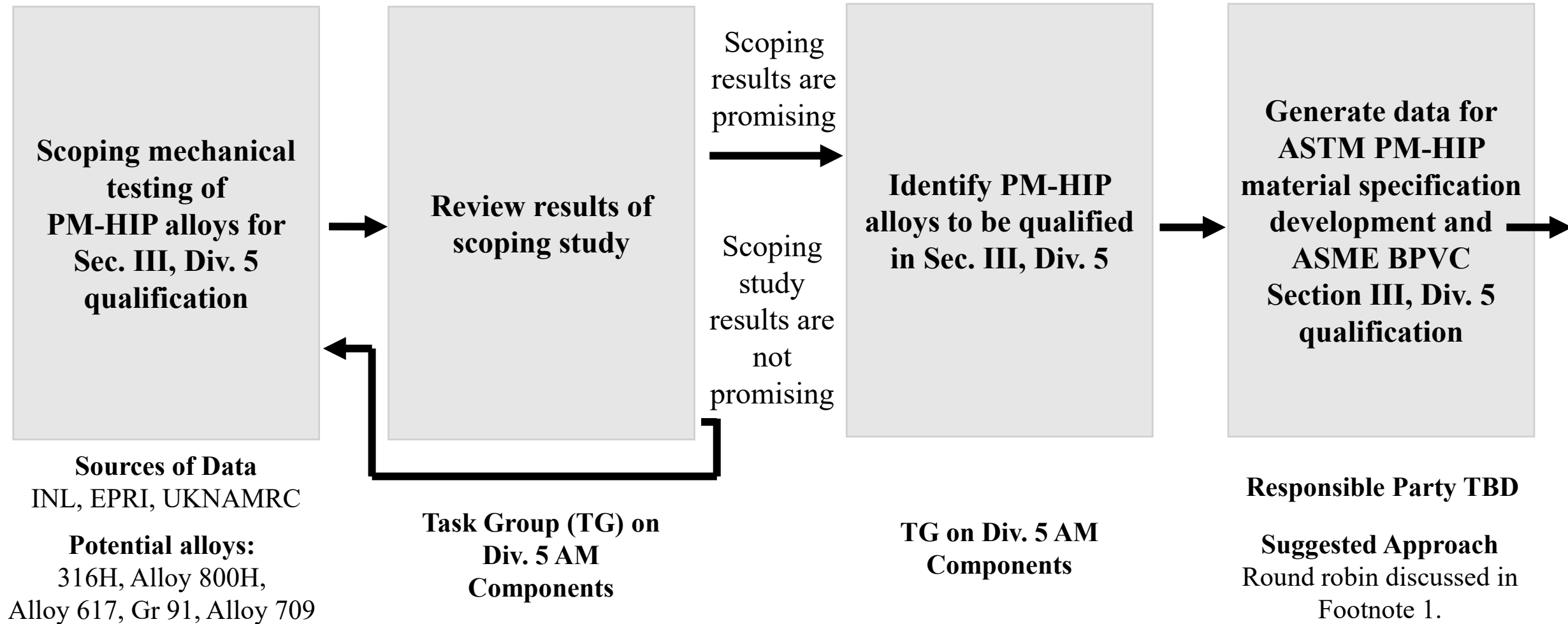
**PM-HIP can benefit microreactor construction to optimize designs, reduce construction time, reduce waste, and improve component/material availability**

- **Goals**

- Demonstrate high temperature mechanical properties of PM-HIP materials compared to wrought materials
- First address PM-HIP 316H stainless steel to support multiple advanced reactors
- Develop specifications and acceptance criteria for PM-HIP components

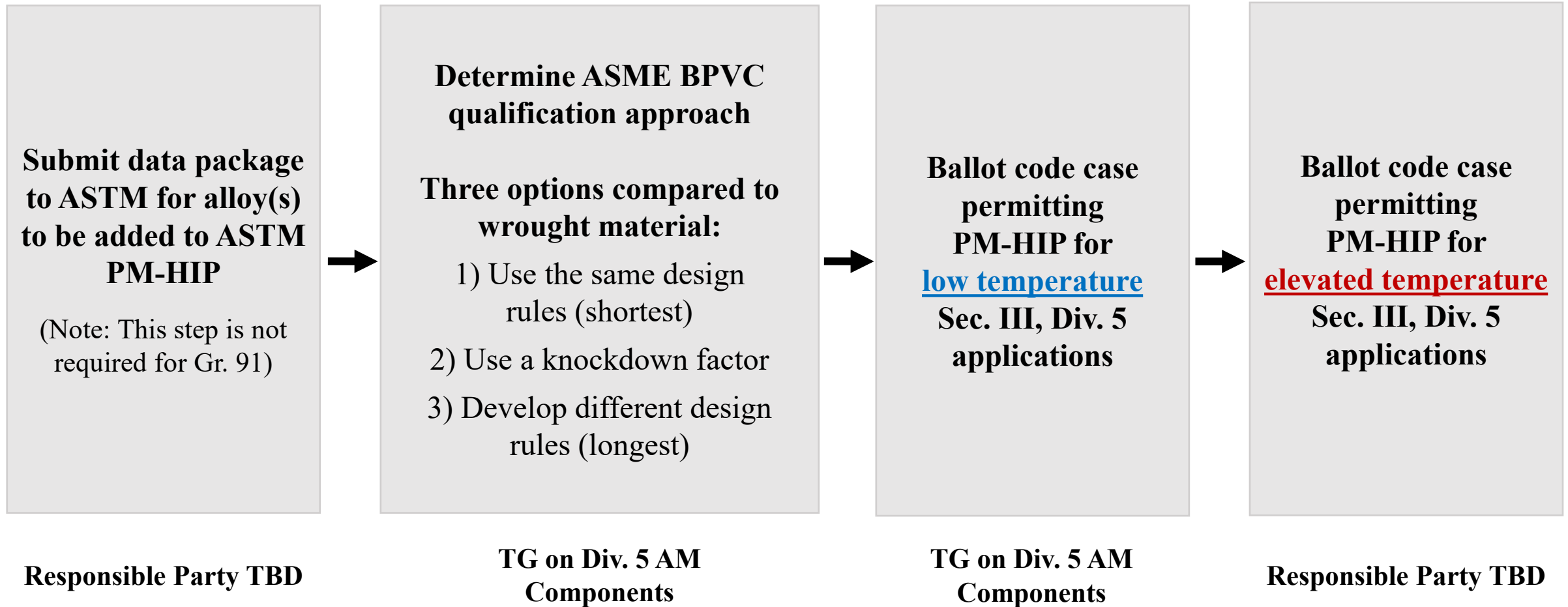


# PM-HIP Div. 5 Code Cases Roadmap



**Footnote:** “A Systematic Study of the Material Performance of Hot Isostatically Pressed Type 316L Stainless Steel Powder for the Civil Nuclear Sector” by Kyffin, Gandy, and Burdett.

# PM-HIP Div. 5 Code Cases Roadmap



# Major Milestones

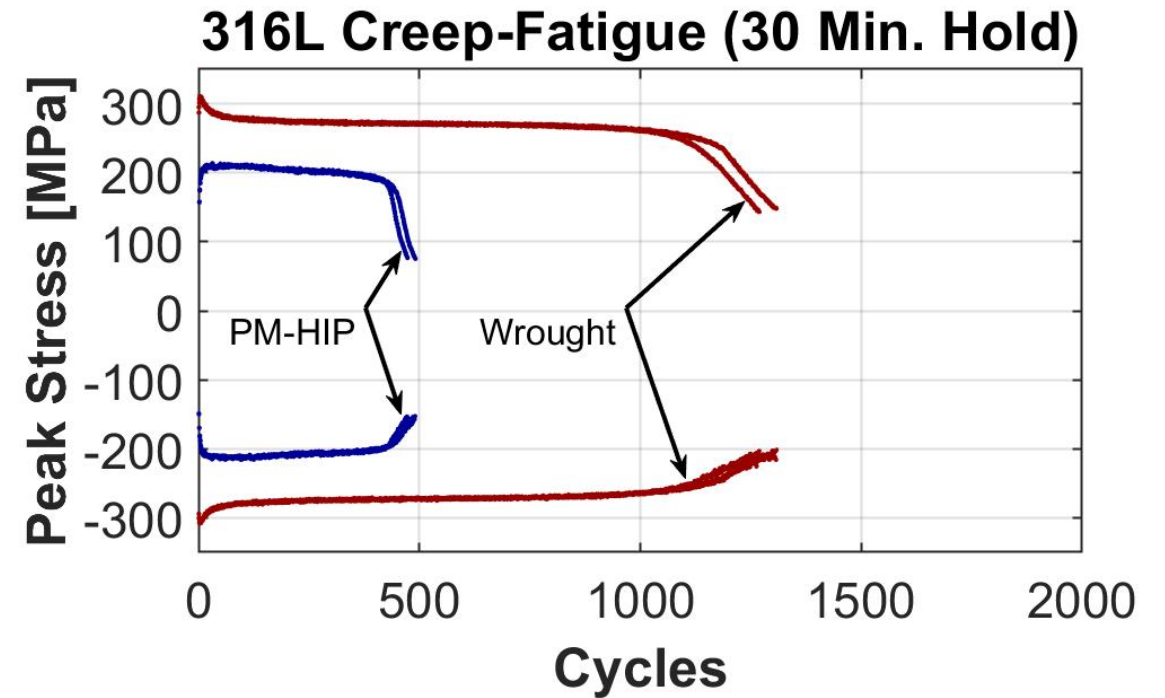
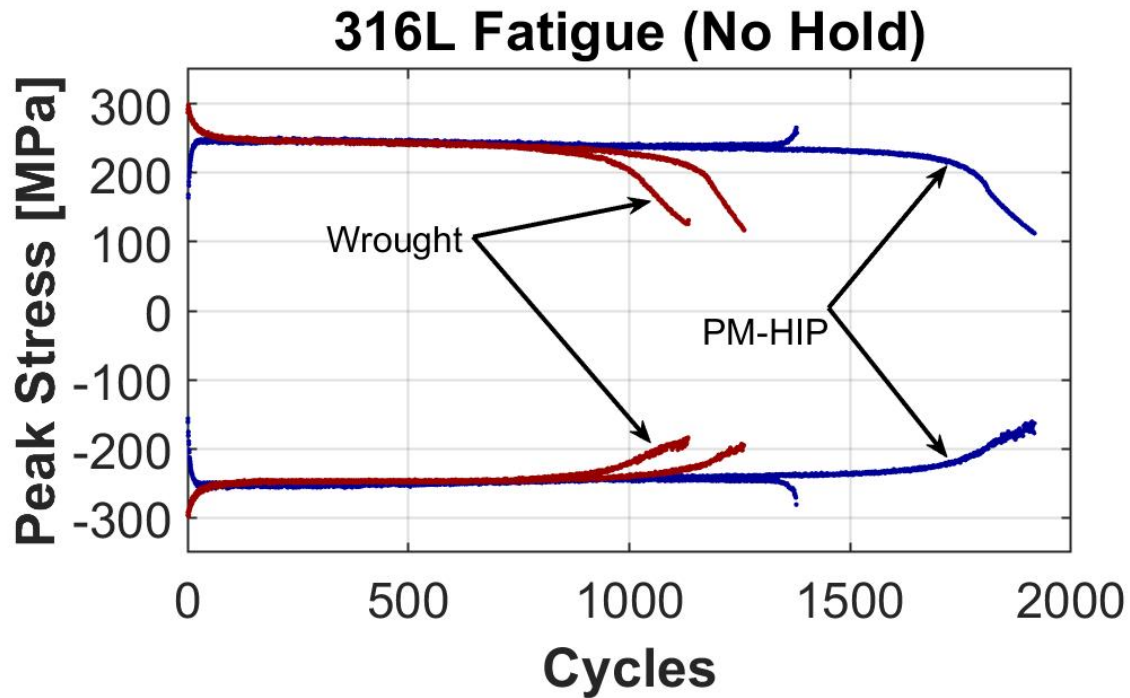
- **Complete development of PM-HIP 316H code cases for the American Society of Mechanical Engineers (ASME) Section III, Division 5, Class A applications by 2025**
  - **Low temperature PM-HIP 316H code case (up to 371°C)**
  - **High temperature PM-HIP 316H code case ( $371^{\circ}\text{C} < T < 816^{\circ}\text{C}$ )**



# Prior Results – 316L

- Fatigue and creep-fatigue at 650°C

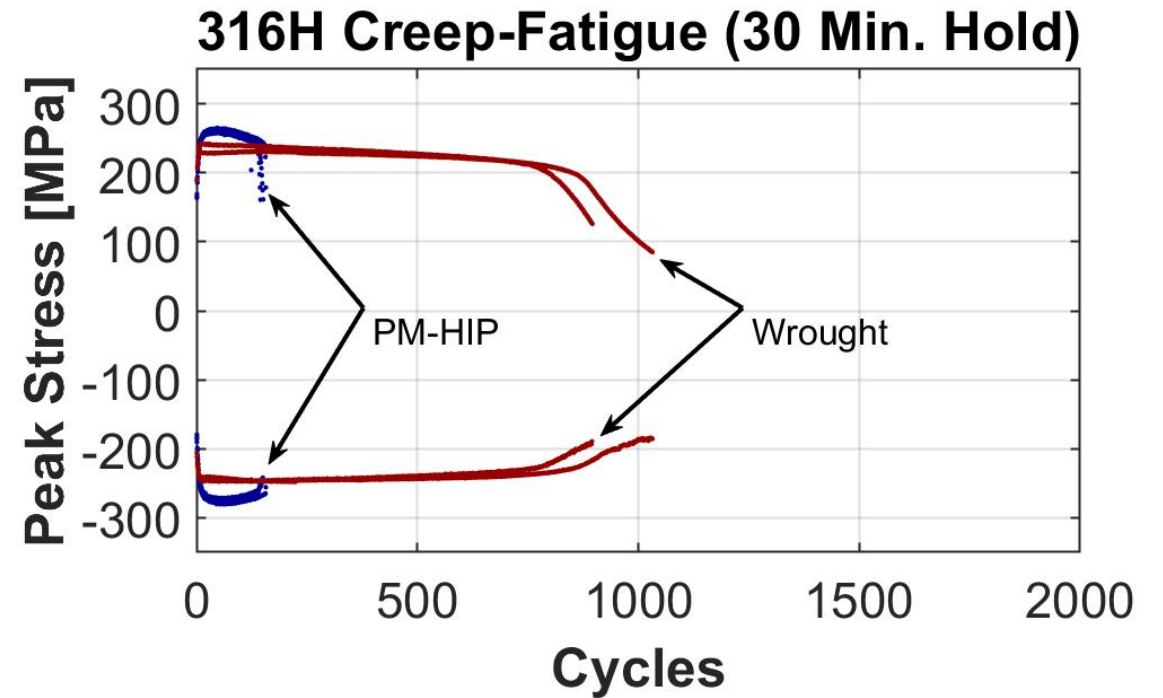
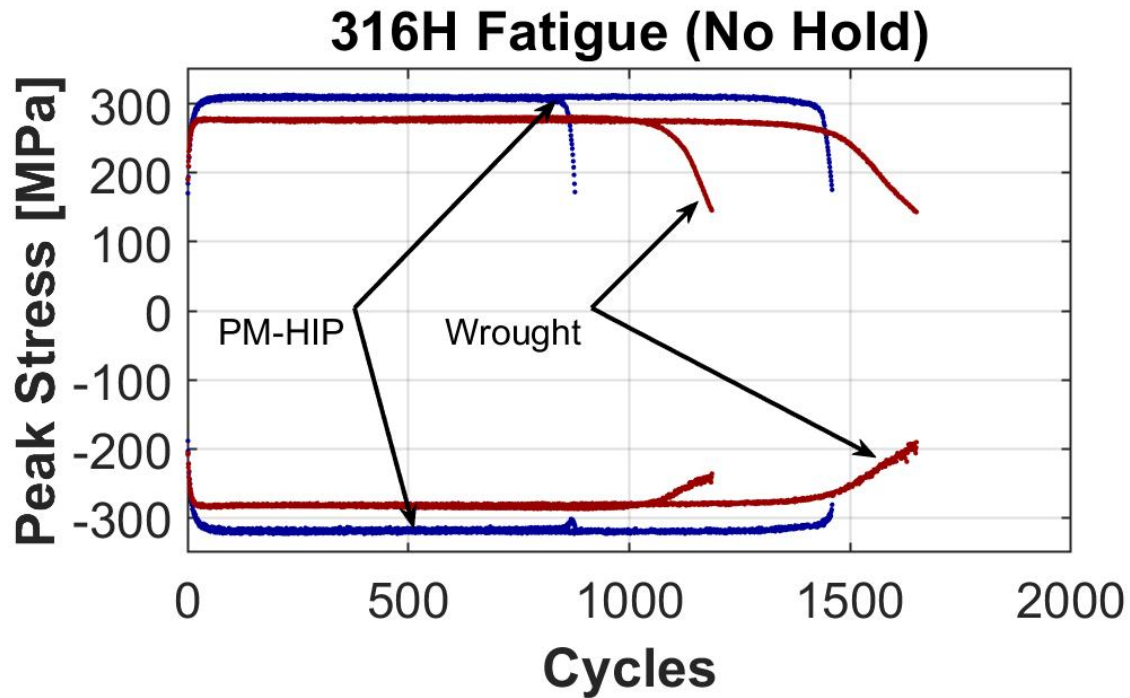
$$650^{\circ}\text{C}, \Delta\varepsilon = 1\%, R = -1, \dot{\varepsilon} = 0.001 \text{ s}^{-1}$$



# Prior Results – 316H

- Fatigue and creep-fatigue at 650°C

$$650^{\circ}\text{C}, \Delta\varepsilon = 1\%, R = -1, \dot{\varepsilon} = 0.001 \text{ s}^{-1}$$





# Properties and Process Optimization

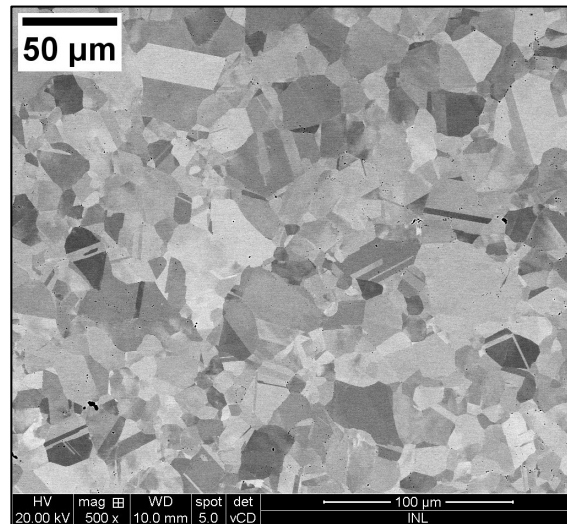
*Why are fatigue and creep-fatigue results showing inferior performance compared to wrought 316 stainless steel?*

- **Need to better understand how the composition and microstructure influence PM-HIP mechanical properties**
  - **Overall oxygen concentration**
    - Prior data has shown that >130 ppm oxygen content in PM-HIP SS may be deleterious to elevated temperature properties
  - **Oxide size and distribution**
  - **Grain size and grain size distribution**

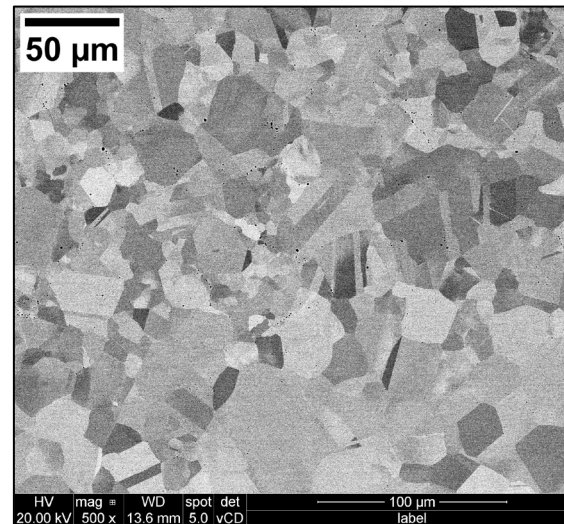
# Current Material Investigation

Consolidated Product Chemical Compositions (wt%)											Hardness (HV <sub>0.3</sub> )
	Ni	Cr	Mo	C	Si	Mn	S	P	O	N	
<b>316H – MTC Heat 1</b>	12.0	16.2	2.53	0.05	0.17	0.21	0.01	0.003	0.0190	0.141	<b>224</b>
<b>316H – UK-NAMRC</b>	11.8	17.3	2.53	0.04	0.17	0.18	<0.003	<0.005	0.015	0.069	<b>194</b>
<b>316L – UK-NAMRC</b>	11.9	17.7	2.44	0.015	0.83	1.88	0.008	0.008	0.0117	0.06	<b>173</b>

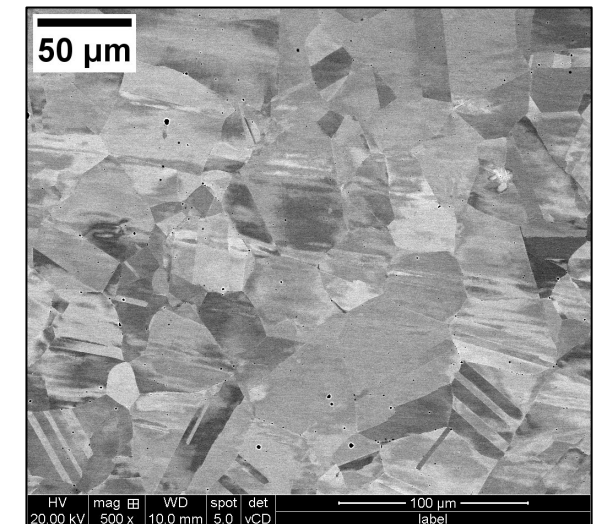
**316H – MTC Heat 1**



**316H – UK-NAMRC**



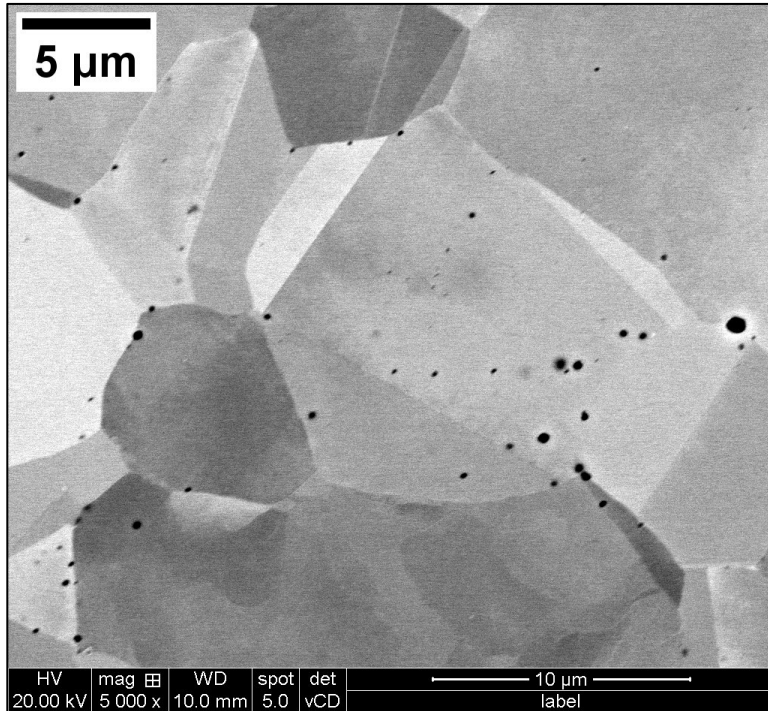
**316L – UK-NAMRC**





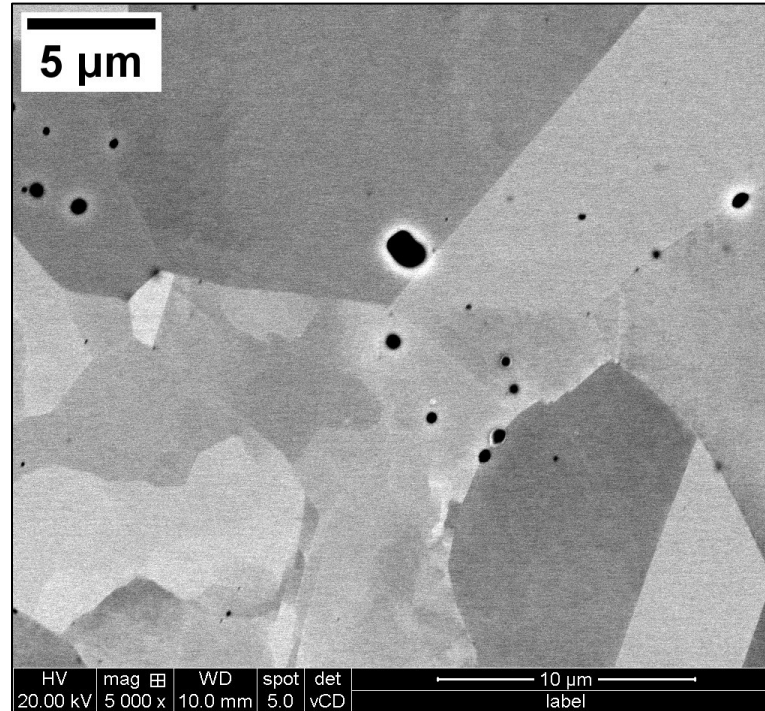
# Results – Oxide Analysis

316H – MTC Heat 1



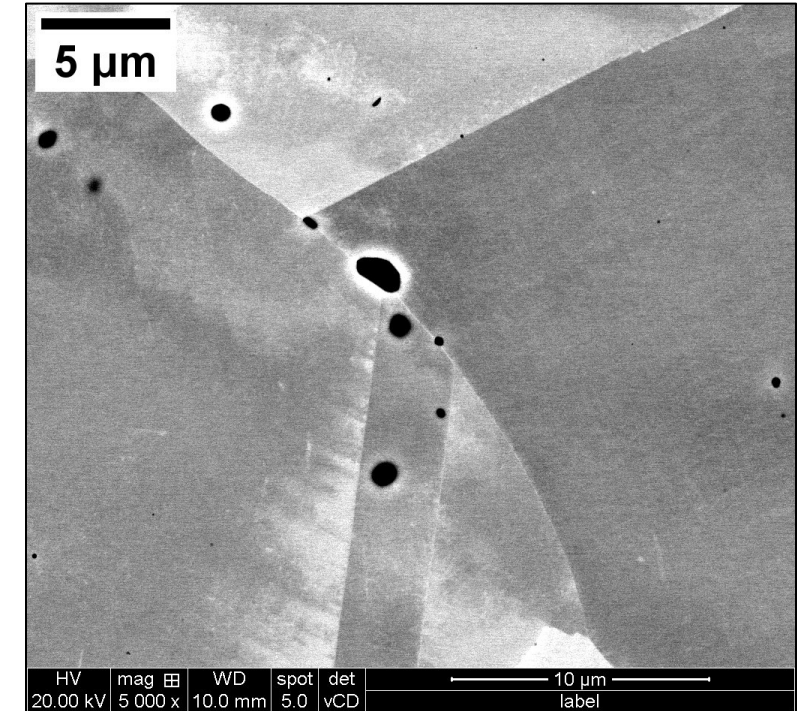
**Oxide Area Fraction = 0.10%**

316H – UK-NAMRC



**Oxide Area Fraction = 0.18%**

316L – UK-NAMRC

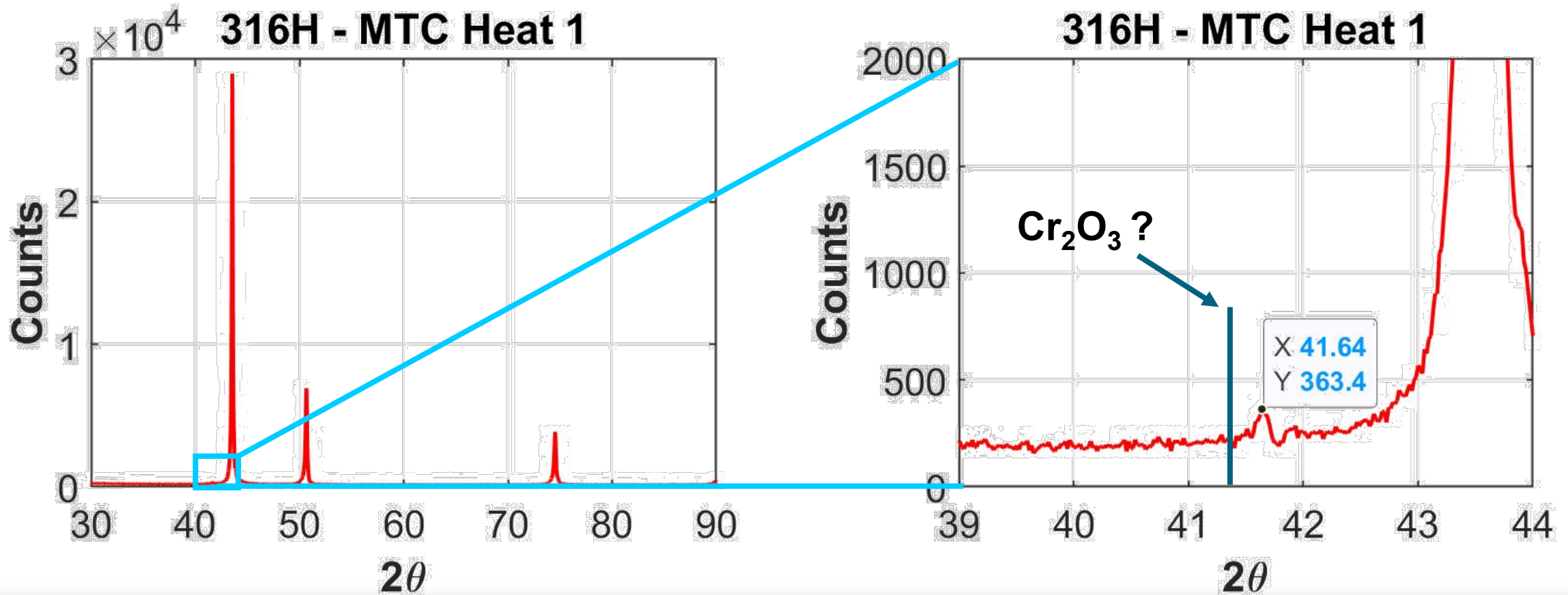


**Oxide Area Fraction = 0.18%**

\*Fraction averages are from five random images at an original magnification of 2500X captured at a 10 μm working distance

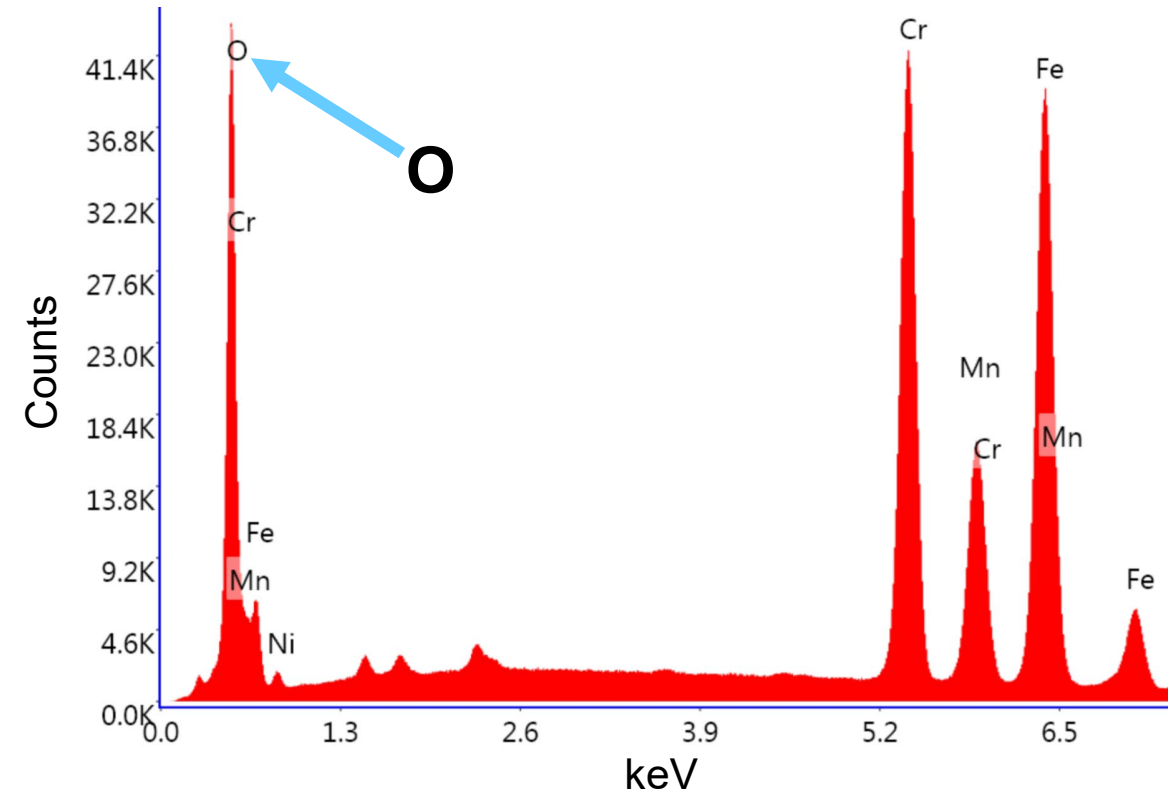
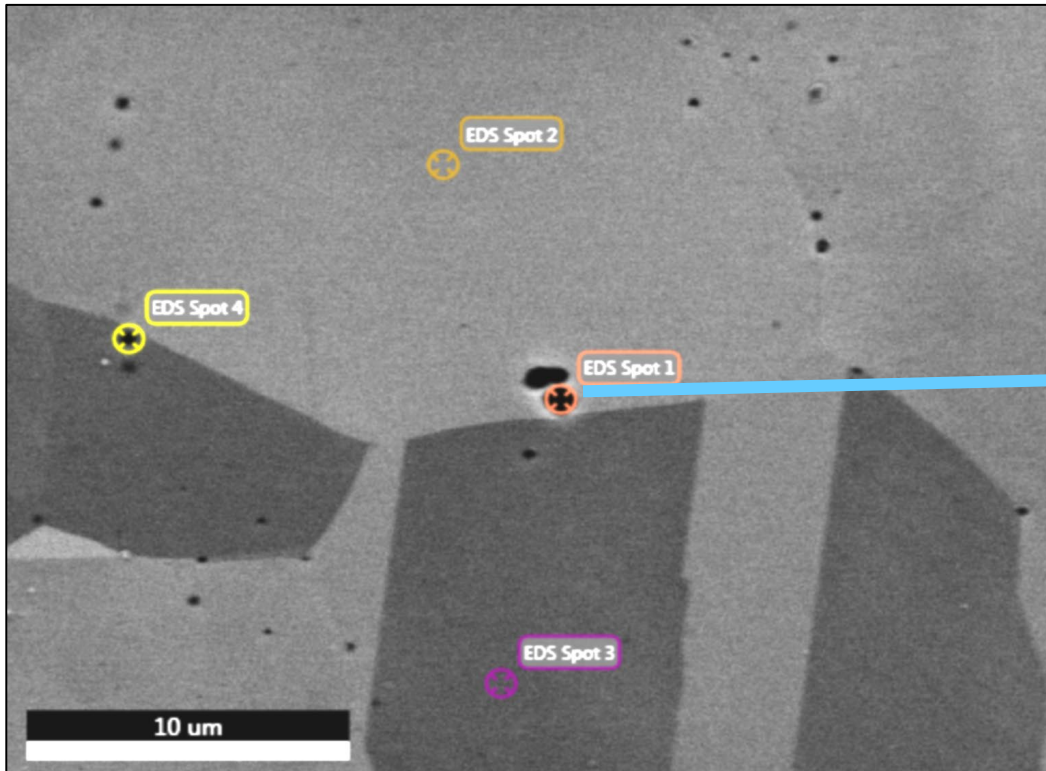
# Results – Oxide Analysis

- X-ray Diffraction Data



# Results – Oxide Analysis

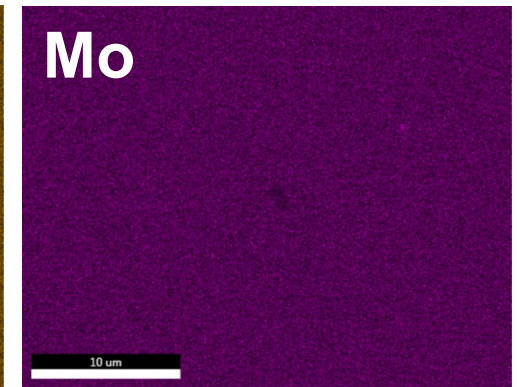
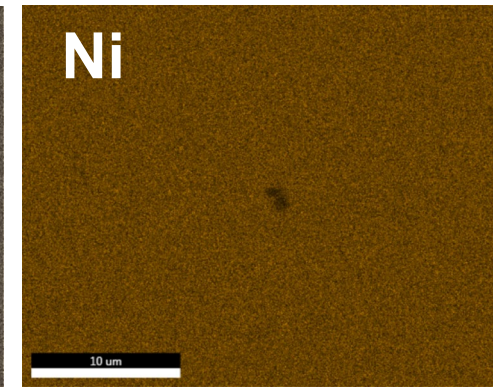
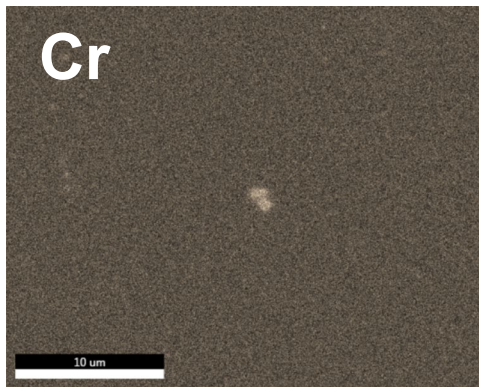
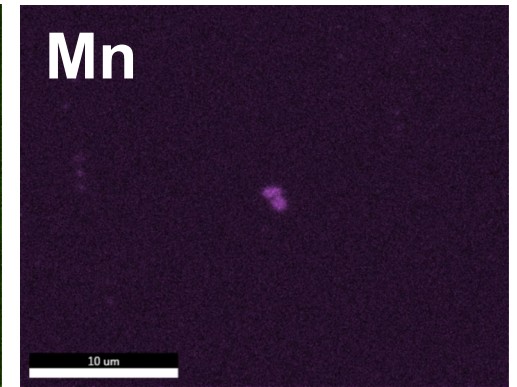
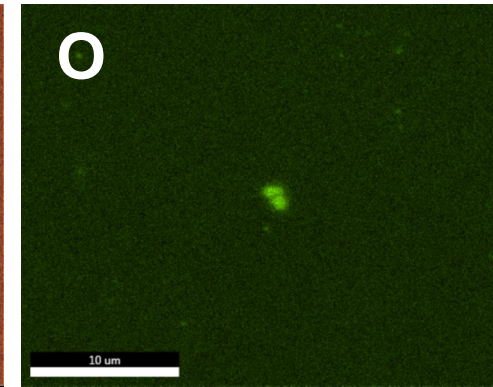
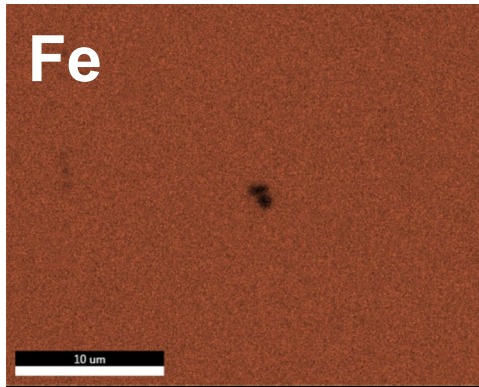
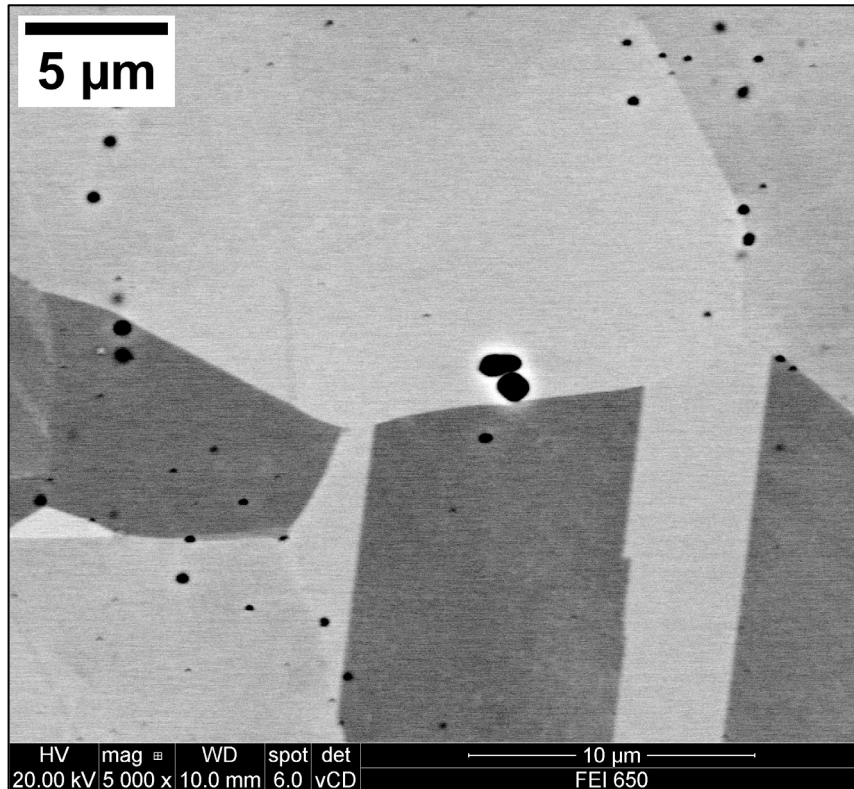
- Are the precipitates oxides?





# Results – Oxide Analysis

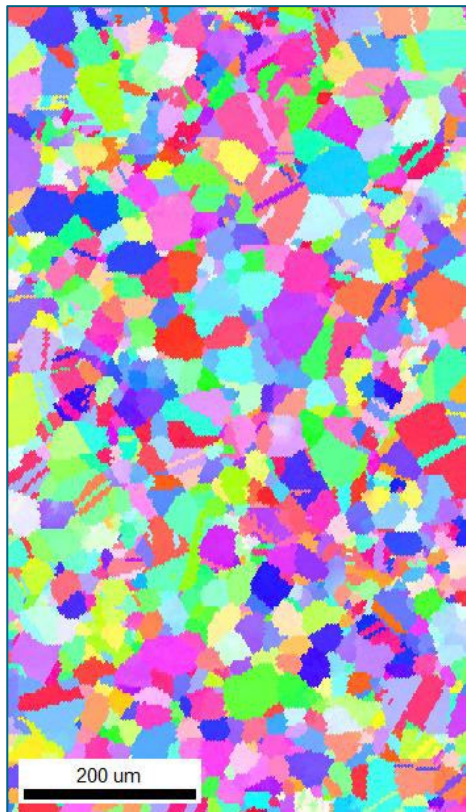
- EDS Analysis (Qualitative) – UKNAMRC 316H





# Results – Grain Size

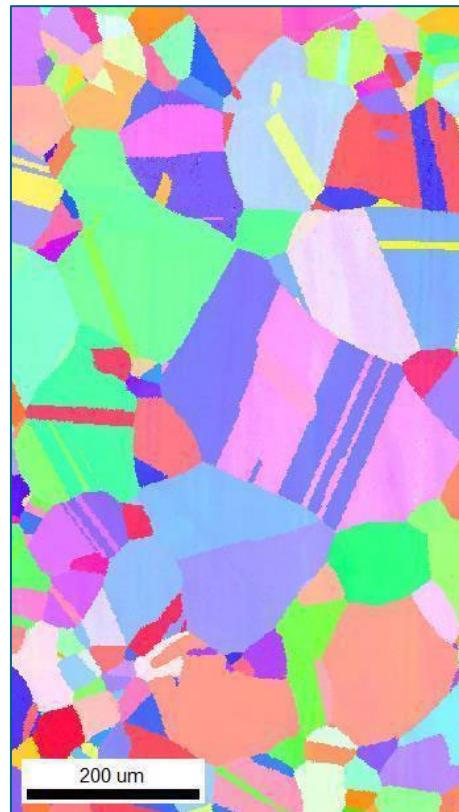
316H MTC Heat 1    316H UK-NAMRC    316L UK-NAMRC



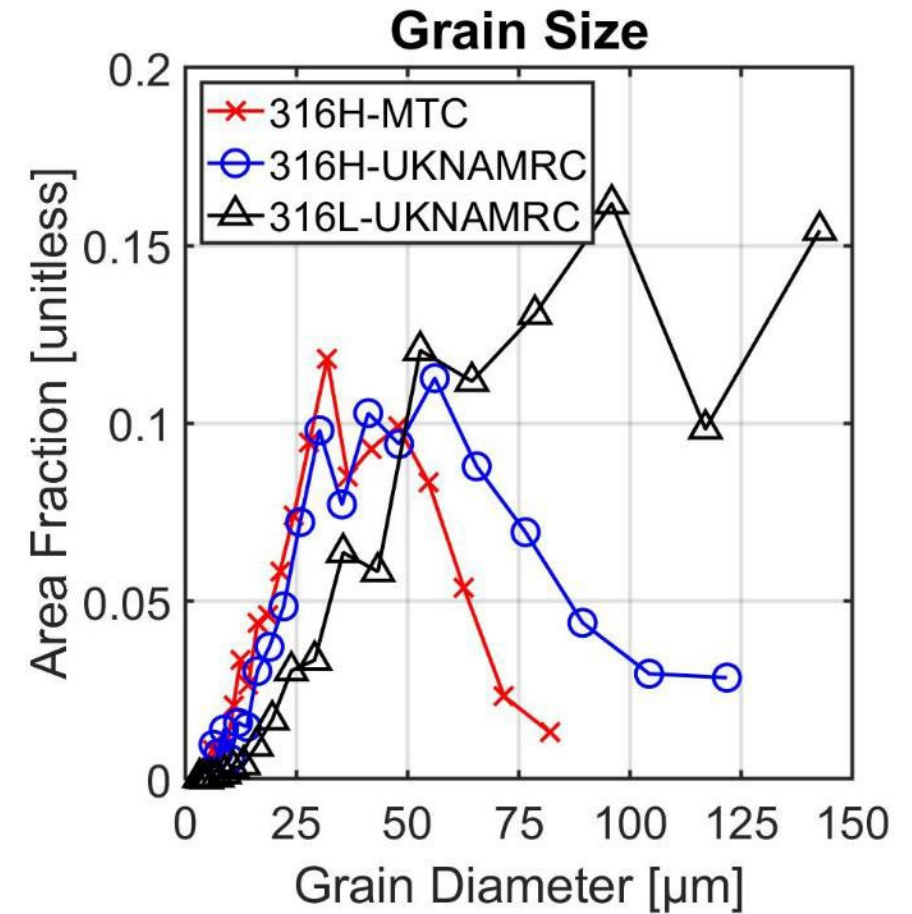
$d_{avg} = 35 \mu\text{m}$



$d_{avg} = 47 \mu\text{m}$



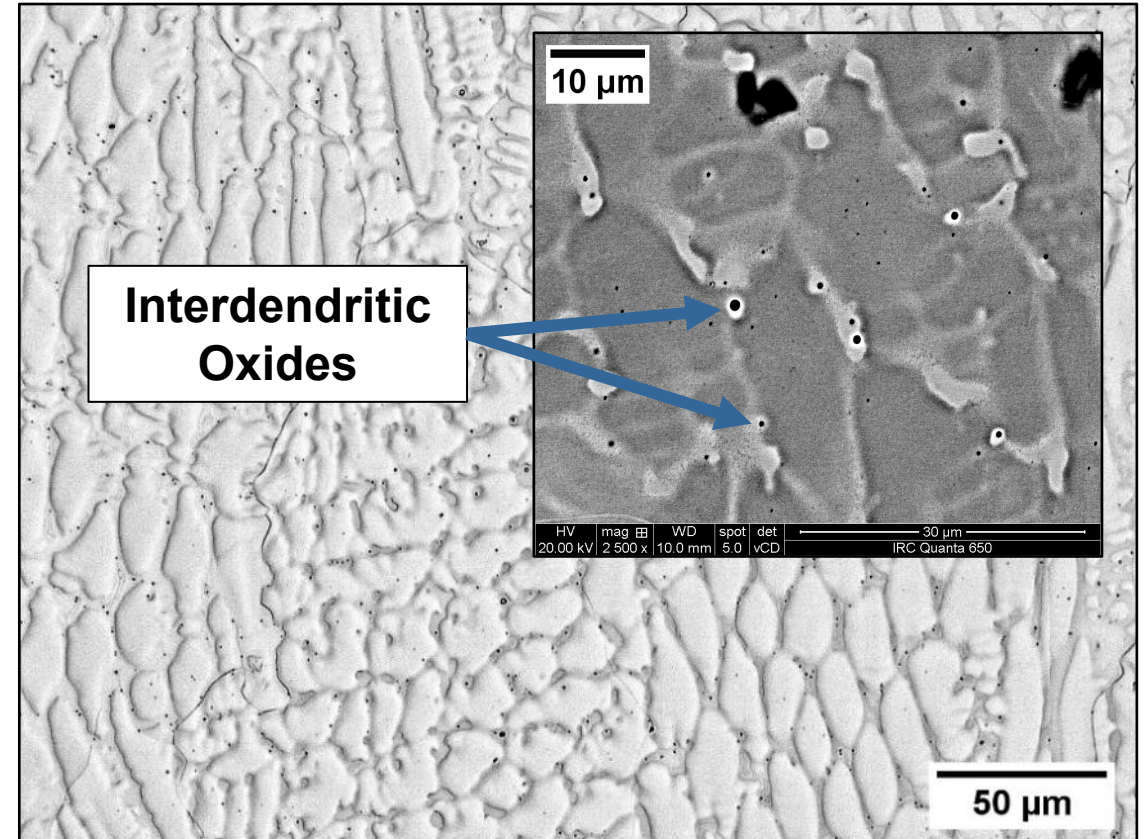
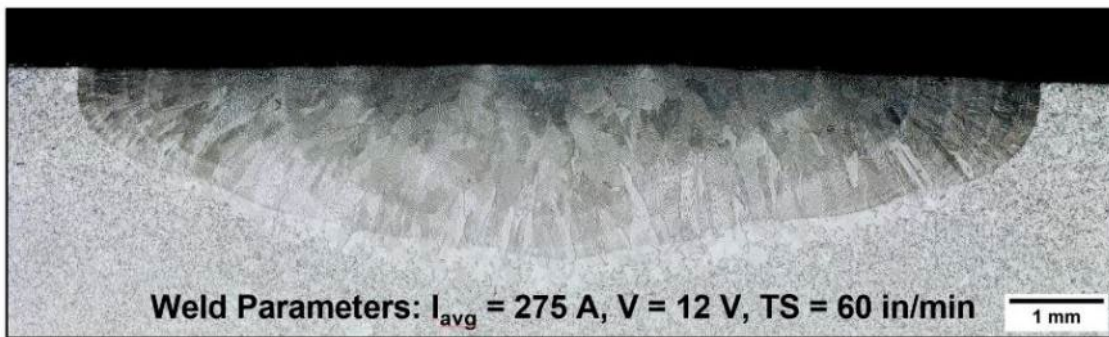
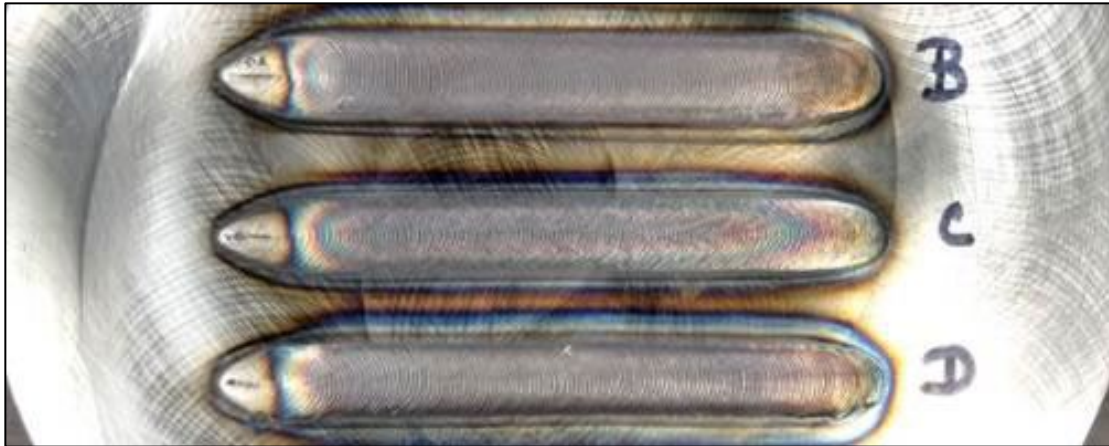
$d_{avg} = 80 \mu\text{m}$





# Results – Initial Weldability

- Bead-on-plate welds on 316H – MTC Heat 1





# 316H Procurement Status

- MTC Heat 2 was procured with an oxygen concentration in the powder of 120 ppm
- One-third of the powder was hot isostatically pressed and underwent a heat treatment identical to MTC Heat 1
- Another third is being heat treated at different conditions to try to influence the oxide size/distribution



# Conclusions

- Initial evaluation of 316L and 316H stainless steel showed poor creep-fatigue performance compared to wrought materials
- It is currently unknown as the exact mechanism resulting in the reduced creep-fatigue properties
- Evidence of oxide size and distribution may be attributing to the reduced properties regardless of the overall oxygen concentration
- Oxides remained in the gas tungsten arc weld metal

# Future Work

- Conduct elevated temperature mechanical testing on the MTC Heat 2 alloy and UK-NAMRC 316 alloys
- Determine if a lower overall oxygen content improves the creep-fatigue properties
- Compare oxide size and distribution at lower oxygen content

Powder Chemical Composition (wt%)											D50 Powder Size (um)
	Ni	Cr	Mo	C	Si	Mn	S	P	O	N	
316H – MTC Heat 2	12.0	17.0	2.53	0.05	0.20	0.21	0.003	0.004	0.0120	0.1010	52
316H – UK-NAMRC	11.9	17.1	2.52	0.05	0.17	0.18	0.002	0.004	0.0064	0.0755	100
316L – UK-NAMRC	11.9	17.7	2.44	0.015	0.83	1.88	0.008	0.008	0.0117	0.06	-

# **FY23 Milestone**

## **M3AT-23IN0804091, 9/15/2023**

- **Complete an initial evaluation of the elevated-temperature cyclic properties of optimized Alloy 316H fabricated by powder metallurgy hot isostatic pressing**

## **M4AT-23IN0804093, 9/29/2023**

- **Provide status of procurement of several heats of PM-HIP 316H stainless steel with optimized processing**



# Thank you

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**ENERGY**

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