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An Initial Evaluation of the Elevated-Temperature Cyclic Properties of Optimized 316H Stainless Steel Fabricated by Powder Metallurgy Hot Isostatic Pressing

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ABSTRACT

Powder metallurgy hot isostatic pressing (PM-HIP) is a mature advanced-manufacturing technology that can consolidate metallic powder under high temperature and pressure to generate near-net shape components. This process can lower production costs and reduce component lead times for nuclear-reactor construction and can be readily deployed in the near term for microreactor components because of their smaller size. The purpose of this work is to continue the evaluation of elevated-temperature cyclic-material properties for PM-HIP 316H stainless steel. Prior scoping results have shown that commercially procured PM-HIP stainless-steel materials have creep-fatigue performance that are reduced from wrought 316 stainless steels. To better understand how PM-HIP-processed material properties compare to traditional manufacturing methods, additional billets of PM-HIP 316H stainless steel were procured and tested. This testing is necessary to understand how elevated-temperature properties are influenced by the PM-HIP process and what data are needed for incorporating PM-HIP 316H into the American Society of Mechanical Engineers Boiler Pressure Vessel Code, Section III, Division 5, for high-temperature reactor construction. Specifically, an additional PM-HIP 316H stainless steel billet with lower oxygen and nitrogen concentrations was fabricated by the Nuclear Advanced Manufacturing Research Centre of the United Kingdom and tested to understand if elevated-temperature cyclic properties can be improved. The creep-fatigue resistance of the material from this new billet did not show appreciable improvement compared to prior testing.

ACKNOWLEDGEMENTS

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ACRONYMS

ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
BPVC	Boiler and Pressure Vessel Code
CF	Creep-fatigue
DOE	Department of Energy
EDS	Energy dispersive spectroscopy
FEI	Field Electron and Ion Company
FY	Fiscal year
HIP	Hot isostatic pressing
INL	Idaho National Laboratory
LCF	Low cycle fatigue
MRP	Microreactor Program
MTR	Material test record
MTS	Material Test Systems
NRC	Nuclear Regulatory Commission
PM-HIP	Powder metallurgy-hot isostatic pressing
SEM	Scanning electron microscope
UK-NAMRC	United Kingdom-Nuclear Advanced Manufacturing Research Centre
US	United States

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An Initial Evaluation of the Elevated-Temperature Cyclic Properties of Optimized 316H Stainless Steel Fabricated by Powder Metallurgy Hot Isostatic Pressing

1. INTRODUCTION

Powder metallurgy hot isostatic pressing (PM-HIP) is a mature advanced-manufacturing (AM) process. It is a near-net-shape process that starts with powder that satisfies the same chemistry specification as its wrought-product counterpart. The powder is confined within a can prefabricated to the net shape of the desired component. Using a fluid, the canned powder is consolidated under elevated temperature and constant, isostatic (all around) pressure. The PM-HIP manufacturing process can be beneficial by reducing additional fabrication steps, improving component availability, reducing additional welding, and creating uniform microstructures.

The code endorsed by the Nuclear Regulatory Commission (NRC) for nuclear-reactor construction is the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code (BPVC). Section III, Division 5 [1], for high-temperature reactor construction. Currently, Section III, Division 5, does not include PM-HIP as a qualified manufacturing process, but the ASME Section III, Division 5, Task Group on AM Components has deemed PM-HIP as a mature technique for incorporation into Section III, Division 5. One of the metallic alloys qualified for use in ASME Section III, Division 5, for high-temperature metallic pressure-boundary components is 316 stainless steel.

Prior work has shown that PM-HIP 316 stainless steels of various grades generally have room-temperature strengths comparable to the wrought product. However, Rupp and Wright [2] showed that elevated-temperature creep-fatigue properties of 316L stainless steel may be reduced and expressed the need to further identify the mechanisms responsible for low cycles to failure. Further investigation of PM-HIP 316H stainless steel also showed reduced cycles to failure than the wrought product [3]. The peak stress versus cycles to failure from this work are shown in Figure 1 for low-cycle-fatigue (LCF) conditions (left) and creep-fatigue (CF) conditions (right). Because of the low cycles to failure for the PM-HIP 316H versus wrought 316H, more work was planned to understand whether improvements were possible and what mechanisms are responsible for reduced elevated-temperature CF properties.

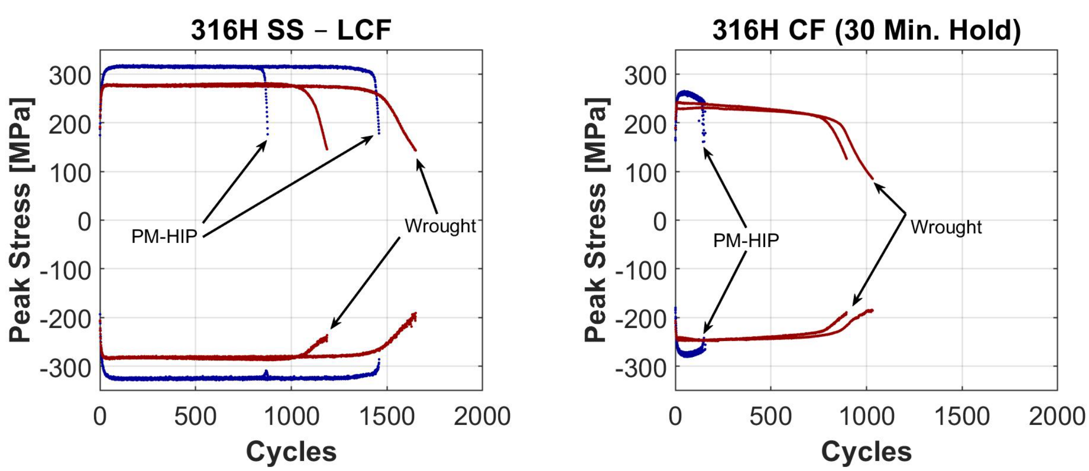

Figure 1. Peak stress versus cycles to failure for 316H stainless steel tested at 650°C with a fully reversed, total strain of 1%. Data extracted from Rupp [3].

This report contains the additional work to better understand PM-HIP 316H stainless steel elevated-temperature cyclic performance and the composition and microstructural mechanisms resulting in reduced PM-HIP creep-fatigue properties. Specifically, the additional billet of PM-HIP 316H analyzed in this investigation contained a 50-ppm lower oxygen content for the consolidated billet. The analysis of a lower oxygen content is to understand whether improved creep-fatigue performance is possible based on prior reports indicating that controlling the oxygen content below a threshold value will improve mechanical properties [4]. Ultimately, the goal of this work is to aid code-qualification efforts and understand potential composition requirements and/or mechanical-property limitations. This report contains high-temperature LCF and CF properties and microstructure analysis for the second billet of PM-HIP 316H.

2. EXPERIMENTAL METHODS

The following subsections describe the material compositions, material test record (MTR) data, elevated-temperature cyclic test results, and microstructure-examination procedures. The purpose of these data and reported microstructure and mechanical test results are to identify possible mechanisms for mechanical-property behavior and understand fatigue and creep-fatigue performance. These will be compared with the PM-HIP 316H stainless steel previously evaluated and produced by MTC Powder Solutions [3].

2.1. Materials

The 316H material evaluated in this report was hot isostatic pressed by the United Kingdom-Nuclear Advanced Manufacturing Research Centre (UK-NAMRC) and had a 50-ppm lower oxygen concentration than the MTC PM-HIP 316H [3]. However, both 316H powders were produced by Sandvik. The compositions for the 316H powders are shown in Table 1. The chemical composition of the UK-NAMRC PM-HIP 316H after consolidation and heat treatment is shown in Table 2. Table 2 also contains the composition of the first billet of the MTC PM-HIP 316H, the composition requirements for ASME Section II Part A [5] for 316H stainless steel plate (S31609), additional requirements for high-temperature use of 316 stainless steel as defined in ASME BPVC Section III Division 5 [1], and the 316 composition reported in American Society of Testing of Materials (ASTM) A988/A988M-17 “Standard Specification for Hot Isostatically-Pressed Stainless Steel Flanges, Fittings, Valves, and Parts for High Temperature Service” [6].

Table 1. Powder compositions for the UK-NAMRC PM-HIP 316H stainless steel and the MTC 316H stainless steel. All measurements, including oxygen, are from the full powder fraction (500 μm).

	316H UK-NAMRC	316H MTC Billet 1
C	0.050	0.055
Ni	11.9	11.8
Cr	17.1	16.3
Mo	2.52	2.51
Ti	<0.01	0.01
Al	0.01	0.01
Si	0.17	0.18
Mn	0.18	0.22
S	0.002	0.01
P	0.004	0.003
N	0.076	0.140
O	0.0093	0.0167

Table 2. Consolidated billet chemical compositions in weight percent for the UK-NAMRC PM-HIP 316H stainless steel and the MTC 316H stainless steel compared with composition requirements.

	316H UK-NAMRC	316H MTC Billet 1	SA 240 S31609 (316H)	ASME III Div. 5 (>595°C)	ASTM A988 S31600
C	0.04	0.04	0.04–0.1	0.04–0.1	0.08
Ni	11.8	12.0	10.0–14.0	10.0–14.0	10.0–14.0
Cr	17.3	16.4	16.0–18.0	16.0–18.0	16.0–18.0
Mo	2.53	2.48	2.00–3.00	2.00–3.00	2.00–3.00
Ti	<0.01	0.005	-	0.04 *	-
Al	<0.01	0.007	-	0.03 *	-
Si	0.17	0.17	1.00	1.00	1.00
Mn	0.18	0.21	2.00	2.00	2.00
S	<0.003	0.003	0.030	0.030	0.030
P	<0.005	0.002	0.045	0.045	0.045
N	0.069	0.147	-	≥0.05 *	≤0.10
O	0.015	0.02	-	-	-
** Additional requirements for Section III, Division 5 Class A and Class SM construction.*					

After HIP, the billets were subjected to solutionizing heat treatments. The UK-NAMRC 316H was heat treated at 1050°C for 4 hours, followed by water quenching, and the MTC billet was solutionized at 1050°C for approximately 2 hours.

2.2. Microstructure Characterization

Sample preparation involved conventional metallographic procedures such as sectioning and hot mounting in a thermosetting polymer. Grinding followed successive steps from 400- to 800-grit silicon carbide abrasive paper. Electrochemical etching was performed using 10% oxalic acid at 3 V. The etch time was approximately 20 seconds. Scanning electron microscopy (SEM) was performed using an Field Electron and Ion Company (FEI) Quanta FEG 650 scanning electron microscope. The accelerating voltage was 20kV with a 6.0 spot size, and the working distance was 10 mm. Energy dispersive spectroscopy (EDS) was performed using an Octane Elect Plus C5 detector and EDAX TEAM software, version 4.6. SEM analyses were performed with the specimens in the as-polished (unetched) condition.

2.3. Mechanical Testing

Table 3 contains the mechanical properties reported in the MTRs for the PM-HIP 316H billet produced by the UK-NAMRC and the PM-HIP 316H billet produced by MTC Powder Solutions. These MTC billet results were previously reported by Rupp in fiscal year (FY) 2021 [3]. The mechanical properties and hardness all met the ASTM/ASME specification requirements. The grain sizes reported in the MTRs were ASTM 8.8 and ASTM 7 for the UK-NAMRC and MTC billet, respectively. These reported MTR measurements were based on the linear-intercept method in ASTM E112-21 “Standard Test Methods for Determining Average Grain Size” [7]. ASTM and ASME material specifications for 316H wrought product [5,8] are required to have an average grain size of ASTM 7 or coarser (smaller ASTM No.), and the UK-NAMRC material would not meet this grain size restriction due to smaller grains.

Table 3. Ambient temperature mechanical properties reported in the material test report for the UK-NAMRC 316H stainless steel and the FY 2021 tested MTC 316H Billet 1 stainless steel [3].

	316H UK-NAMRC	316H Billet 1
Hardness (HV)	187	182
Yield Strength (MPa)	341	370
Tensile Strength (MPa)	623	671
Elongation (%)	47	69
Reduction in Area (%)	73	50
Grain Size (ASTM No.)	8.8	7

2.3.1. Elevated-Temperature Cyclic Testing

Elevated-temperature cyclic testing was based on E606/E606M-21 "Standard Test Method for Strain-Controlled Fatigue Testing" [9] and ASTM E2714-13 "Standard Method for Creep Fatigue Testing" [10], but conducted in accordance with INL's internal procedure PLN-3346 "Creep Fatigue Testing" [11]. Tests were conducted using Material Test Systems (MTS) servo-hydraulic load frames with MTS 653, three-zone furnaces. Strain was controlled using a direct-contact extensometer, and temperature was controlled and monitored using two direct-contact R-type thermocouples, spot welded onto the shoulder of the gauge section.

The reported LCF tests were performed at 650°C using full reversed strain of ±0.5% and a strain rate of 0.001/s. A schematic representation of the LCF and CF procedures are shown in Figure 2. The only difference between the LCF and CF test conditions is that the CF tests incorporated a 30-minute hold at a peak tensile strain of 0.5%. The criterion for determining cycles to failure was based on a 25% drop in the ratio of peak tensile stress to peak compressive stress for each cycle. The equation and schematic representation of this is shown in Figure 3.

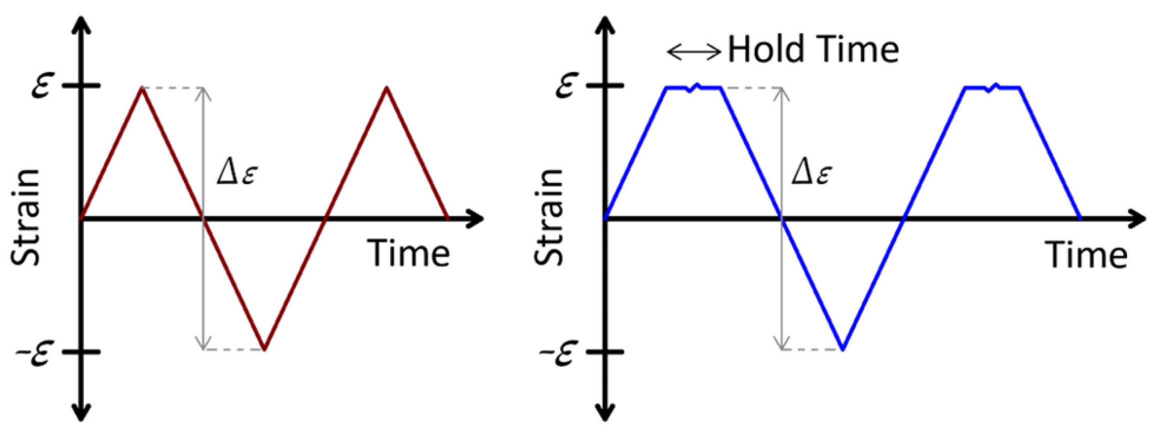

Figure 2. Schematics showing strain-controlled LCF (left) and CF (right) testing procedures.

$$r = \left| \frac{\sigma_{tensile}}{\sigma_{compression}} \right|$$

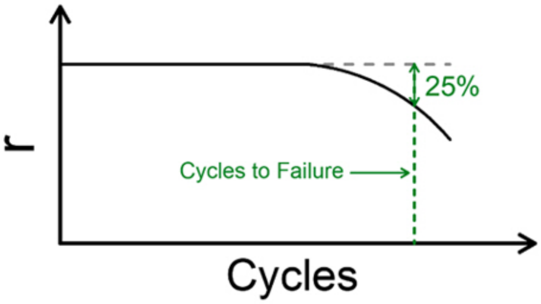

Figure 3. Evaluation criteria for cycles to failure based on a 25% stress reduction (N25).

3. RESULTS

The following results include the peak stress data for elevated-temperature cyclic testing, the cycles to failure (N_{25}) based on a 25% drop in the peak tensile stress to peak compressive stress ratio, and the microstructure analysis before and after testing. The wrought-alloy comparison is from 316H stainless steel reported by Rupp [3].

3.1. Mechanical Testing

3.1.1. Elevated-Temperature Cyclic Testing

The left plot in Figure 4 shows the maximum/minimum stress versus cycles plot for two LCF tests for the UK-NAMRC PM-HIP 316H stainless steel with a consolidated-billet oxygen concentration of 170 ppm. The max/min stress was consistent between the wrought and PM-HIP products. Similarly, the max/min stress was consistent in the creep-fatigue plot shown on the right in Figure 4. However, the cycles to failure were reduced by nearly 1000. The ratio for the peak tensile and peak compressive stress to compute the 25% reduction is shown in Figure 5 for both the LCF and CF tests. This is the criterion that was used to determine the cycles to failure. These results are shown in Figure 6. The LCF cyclic life for the UK-NAMRC 316H PM-HIP material was nearly comparable to the wrought product with the usual data scatter on cycles to failure. However, the CF cyclic performance was significantly reduced compared to the wrought product even accounting for data scatter.

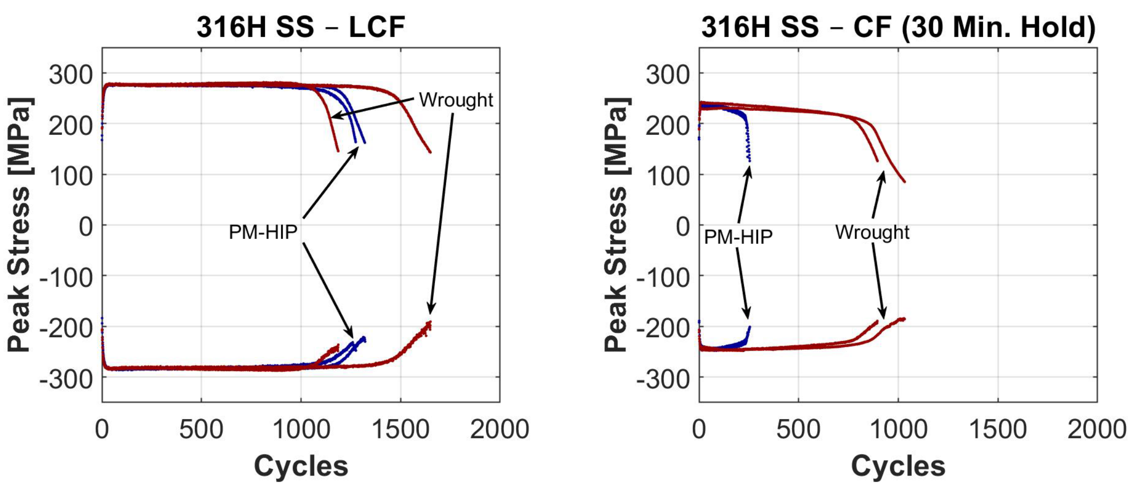

Figure 4. LCF and CF peak stress versus cycles for the UK-NAMRC PM-HIP 316H stainless steel tested at 650°C and ±0.5% strain compared to a wrought 316H stainless steel.

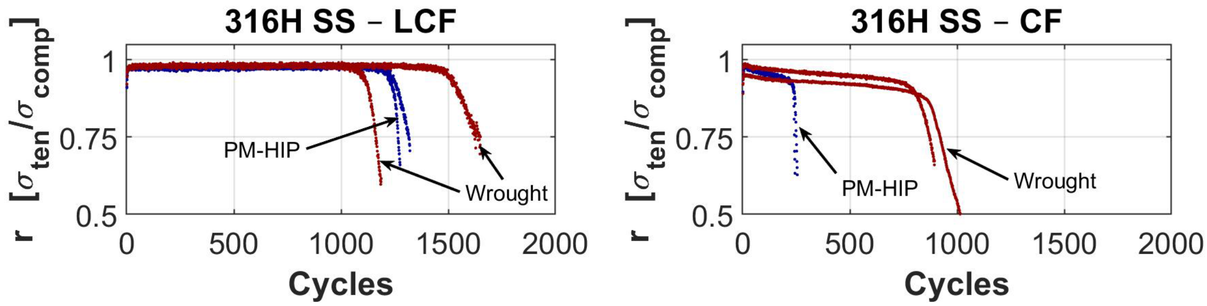

Figure 5. Ratios of maximum tension and maximum compression stress for LCF (left) and CF results (right) for the UK-NAMRC 316H results shown in Figure 4 and with comparison to a wrought 316H stainless steel.

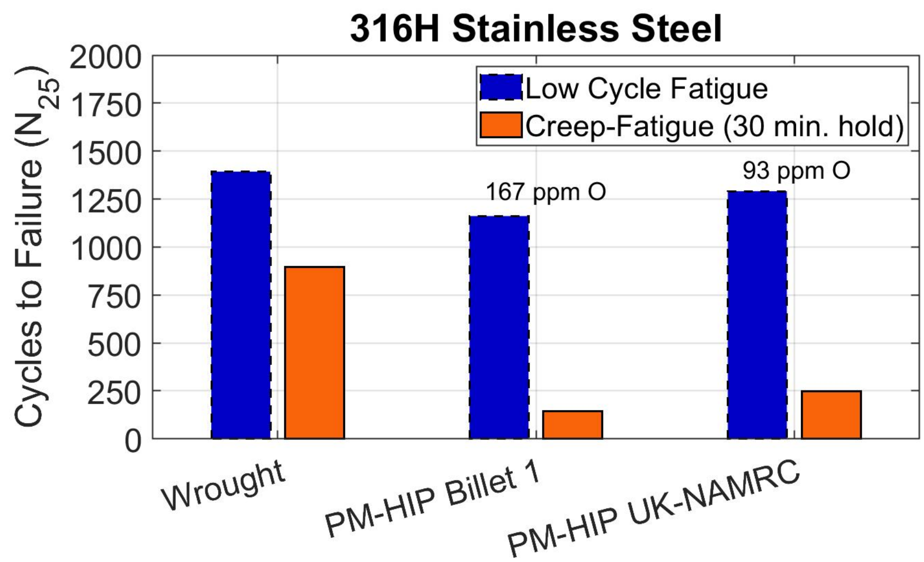

Figure 6. Comparison of cycles to failure between wrought 316H and the PM-HIP 316H materials. The reported oxygen contents are from the powder measurements.

3.2. Microstructure Analysis

3.2.1. Microstructure Analysis Prior to Testing

Figure 7 shows backscattered electron images for the UK-NAMRC 316H and MTC Billet 316H, and Figure 8 shows higher-magnification images. The higher-magnification backscattered images show precipitates through high compositional contrast. The precipitates are believed to be oxides as a result of the powder-production process. Although Cooper et al. [12] reported similar observations to be porosity, EDS indicates these locations are depleted of iron and nickel, while enriched in chromium, manganese, oxygen. Therefore, it is highly likely that the precipitates are chromium/manganese-rich oxides, and these EDS results are shown in Figure 9.

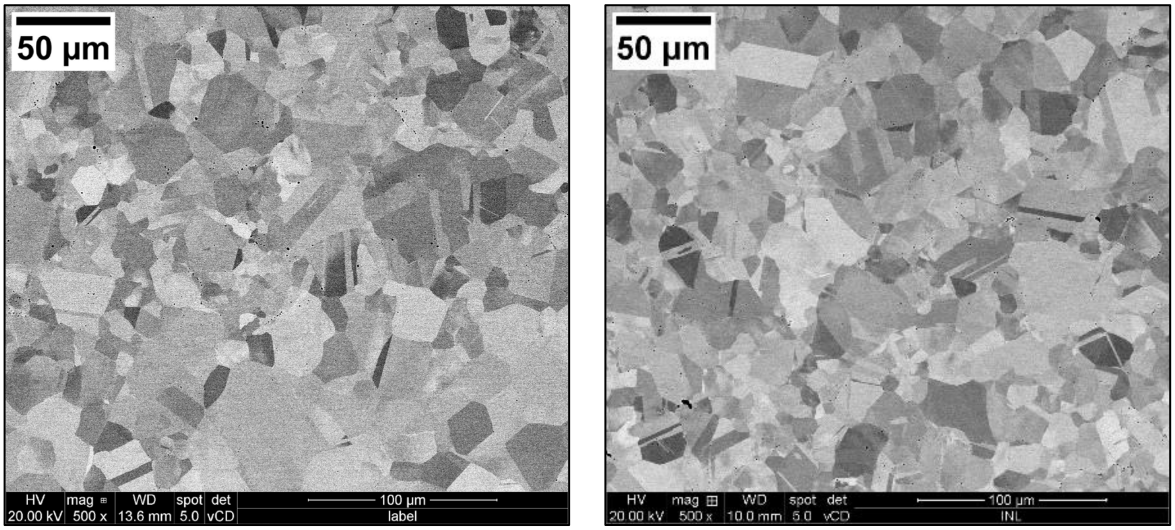

Figure 7. Backscattered electron images of the UK-NAMRC PM-HIP 316H stainless steel (left) and the MTC PM HIP 316H stainless steel (right) in the as-received condition.

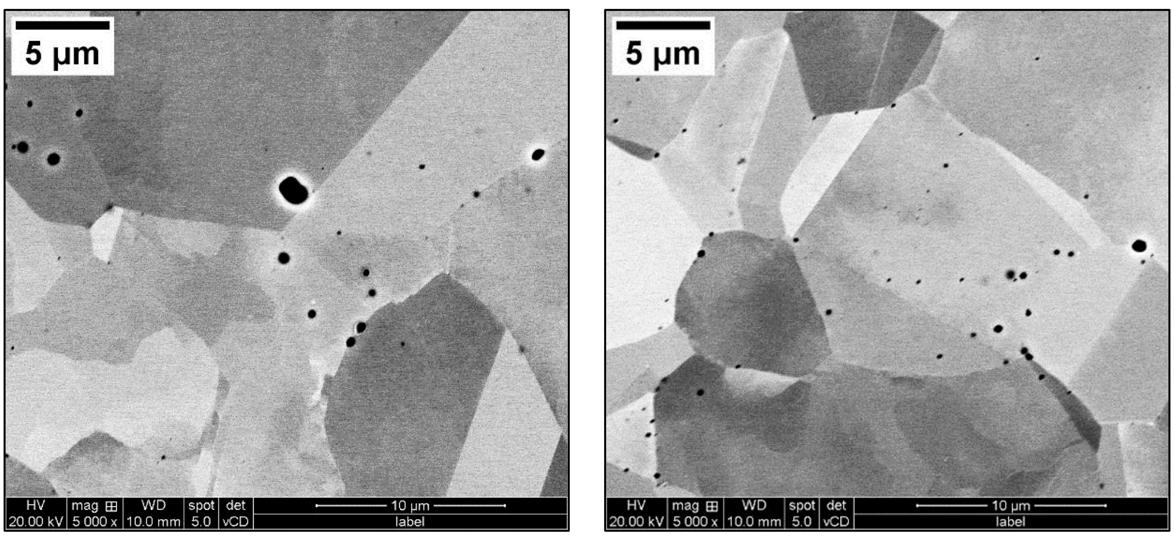

Figure 8. Backscattered electron images showing the precipitates at higher magnification.

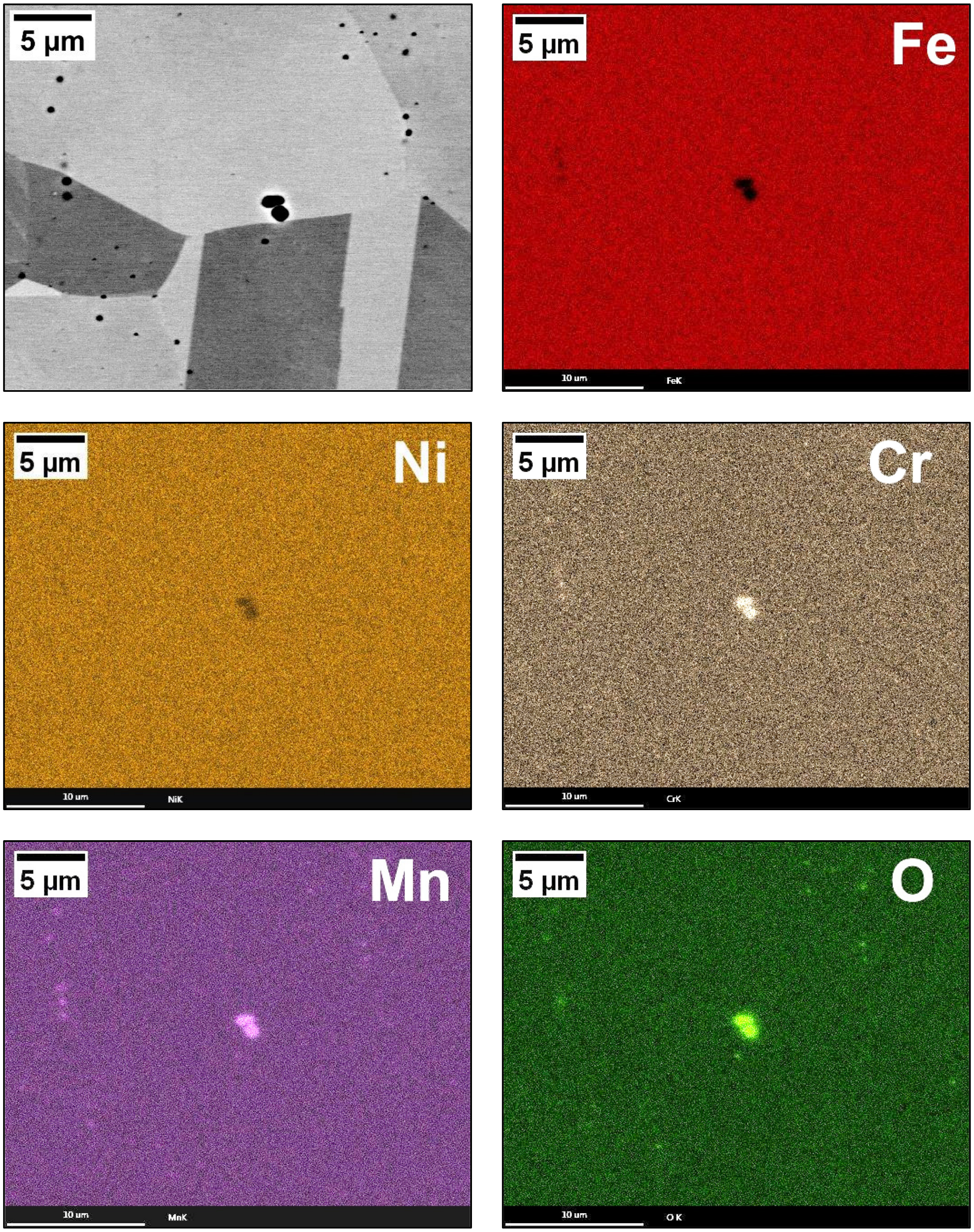

Figure 9. EDS scans of the UK-NAMRC PM-HIP 316H showing relative concentrations of elements. The upper-left backscatter electron image shows the microstructure of the EDS scanned area.

3.2.2. Microstructure Analysis after Testing

Figure 10 shows the microstructures from one of the completed UK-NAMRC PM-HIP 316H LCF tests. The crack propagation is mostly transgranular, or through the grain interiors, with numerous cracks nucleating from the sample surface. Contrary to the observed LCF crack propagation, the CF sample showed only intergranular cracking. As with the LCF test, the CF test also nucleated multiple cracks along the sample surface. Figure 11 and Figure 12 show the CF intergranular cracks in addition to grain-boundary void nucleation and grain-boundary facet cracks in front of and around the crack tip. It is hypothesized that oxide particles decorate the grain boundaries and may exacerbate creep and void nucleation during the high-temperature hold time. Therefore, the large oxides are likely a main factor regarding reduced cycles to failure during creep-fatigue testing.

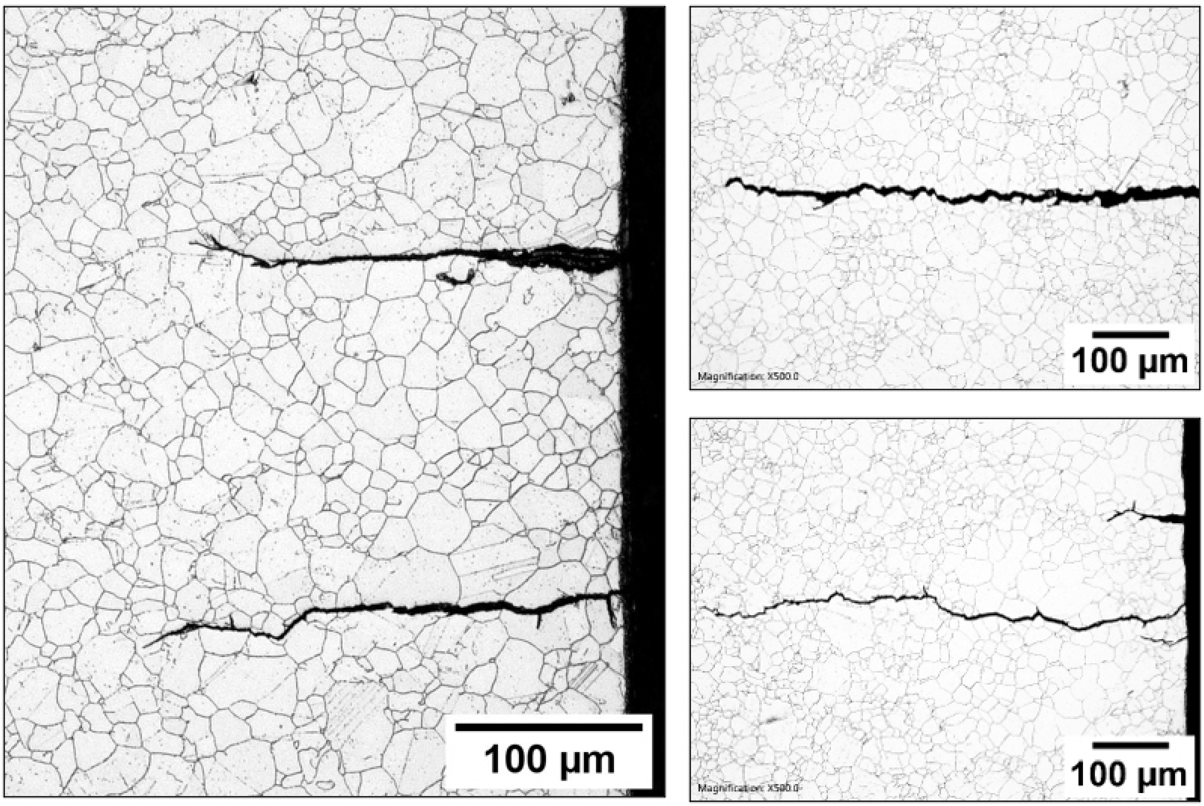

Figure 10. Optical micrographs from of the UK-NAMRC 316H stainless steel after LCF testing. Cracking appears to propagate entirely through the grain interiors (transgranular).

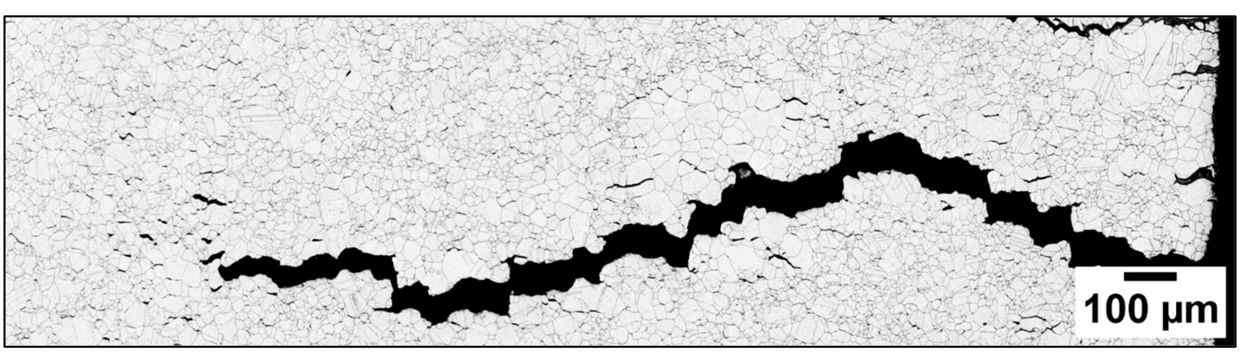

Figure 11. Optical micrograph of the UK-NAMRC 316H stainless steel from a CF test. The intergranular voids are present around and ahead of the main crack tip.

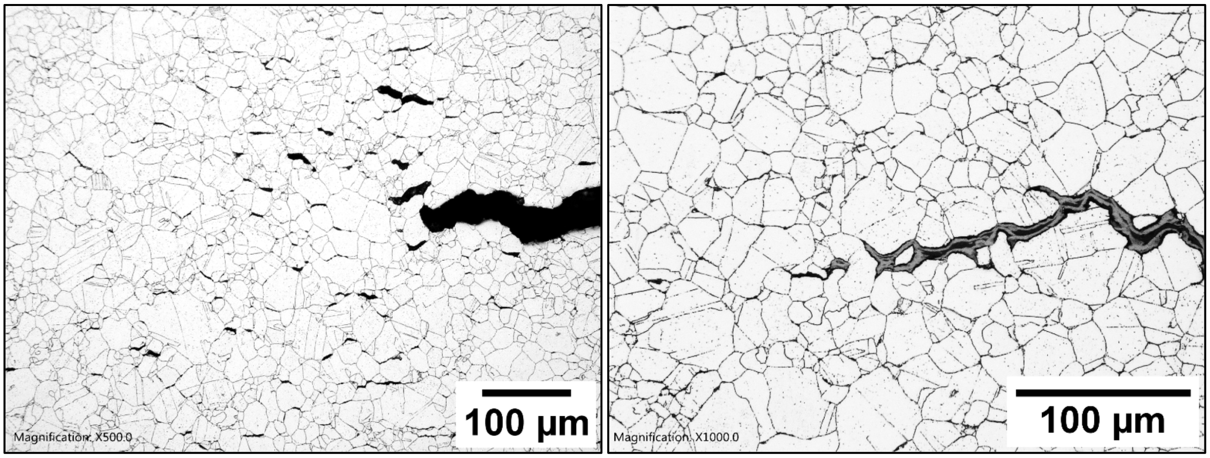

Figure 12. Optical micrographs of the UK-NAMRC 316H stainless steel after CF testing. The left photomicrograph shows the voids ahead of the main crack, and the right photomicrograph shows another smaller crack propagating along grain boundaries (intergranular).

4. SUMMARY

A reduction of 50 ppm of oxygen in the consolidated PM-HIP 316H stainless steel has shown negligible improvement in the CF cyclic life. The lack of improvement appears consistent with the microstructural observations of numerous micron-size precipitates within the structure. The current belief is that these precipitates are oxides and are introduced during the powder-production process. The fracture behavior between the LCF and CF testing revealed the difference between failure mechanisms of the two tests. It is possible that these oxides are weakening the grain boundaries and degrading creep resistance during elevated-temperature (650°C), strain-controlled CF testing. At the time of this writing, there is no requirement for oxygen concentrations for PM-HIP 316 stainless steel components. If reductions in oxygen content prove to be successful in improving elevated-temperature cyclic properties, it will be necessary to limit the maximum oxygen content in the powders or consolidated billets, such as the requirements for nitrogen in ASTM 988-17/A988M-17 [6].

5. FUTURE WORK

Further testing will evaluate the microstructure and elevated-temperature cyclic performance of three additional billets of PM-HIP 316H stainless steel. All three billets will be from the same heat of powder, but hot isostatic pressed at different conditions. The two billets procured have consolidated-product oxygen concentrations of 149 and 156 ppm. These results will indicate whether further reduction in oxygen can improve elevated-temperature cyclic properties and/or if CF cycles to failure can be improved under different processing techniques for a single heat of powder.

6. REFERENCES

1. ASME. 2023. "BPVC Section III-Rules for Construction of Nuclear Facility Components-Division 5-High Temperature Reactors." American Society of Mechanical Engineers. United States.

2. Rupp, R., and R. Wright. 2020. "The Elevated-Temperature Cyclic Properties of Alloy 316L Manufactured Using Powder Metallurgy Hot Isostatic Pressing." INL/EXT-20-59327, Idaho National Laboratory, Idaho Falls, ID. https://doi.org/10.2172/1975808.

3. Rupp, R. 2021. "The Elevated-Temperature Cyclic Properties of Alloy 316H Fabricated by Powder Metallurgy Hot Isostatic Pressing." INL/EXT-21-62993 Rev. 1, Idaho National laboratory, Idaho Falls, ID. https://doi.org/10.2172/1975813.

4. Kyffin, W., D. Gandy, and B. Burdett. 2020. "A Study of the Reproducibility and Diversity of the Supply Chain in Manufacturing Hot Isostatically Pressed Type 316L Stainless Steel Components for the Civil Nuclear Sector." *Journal of Nuclear Engineering and Radiation Science* 6(2): 021116. https://doi.org/10.1115/1.4044752.

5. ASME. 2021. "BPVC-Section II-Part A-Materials Specifications (Ferrous materials beginning to SA-450)." American Society of Mechanical Engineers. United States.

6. ASTM. 2023. "Standard Specification for Hot Isostatically-Pressed Alloy Steel Flanges, Fittings, Valves, and Parts for High Temperature Service." ASTM A988/A988M-17, ASTM International, West Conshohocken, PA. https://doi.org//10.1520/A0988_A0988M-17.

7. ASTM. 2021a. "Standard Test Methods for Determining Average Grain Size." ASTM E112-21, ASTM International, West Conshohocken, PA. https://doi.org/ 10.1520/E0112-13R21.

8. ASTM. 2022. "Standard Specification for Chromium and Chromium-Nickel Stainless Steel Plate, Sheet, and Strip for Pressure Vessels and for General Applications." A240/A240M-22a, ASTM International, West Conshohocken, PA. https://doi.org/10.1520/A0240_A0240M-22A.

9. ASTM. 2021b. "Standard Test Method for Strain-Controlled Fatigue Testing." E606/E606M-21, ASTM International, West Conshohocken, PA. https://doi.org/10.1520/E0606_E0606M-21.

10. ASTM. 2020. "Standard Test Method for Creep-Fatigue Testing." E2714-13 2020, ASTM International, West Conshohocken, PA. https://doi.org/10.1520/E2714-13R20.

11. INL. 2017. "Creep Fatigue Testing." INL/MIS-10-18953, PLN-3346, Revision 9, Idaho National Laboratory, Idaho Falls, ID. https://inldigitallibrary.inl.gov/sites/sti/sti/Sort_1624.pdf.

12. Cooper, A. J., N. I. Cooper, J. Dhers, and A. H. Sherry. 2016. "Effect of Oxygen Content Upon the Microstructural and Mechanical Properties of Type 316L Austenitic Stainless Steel Manufactured by Hot Isostatic Pressing." *Metallurgical and Materials Transactions A* 47: 4467–4475. https://doi.org/10.1007/s11661-016-3612-6.