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High Temperature Mechanical Properties of 316H Stainless Steel Fabricated by Powder Metallurgy Hot Isostatic Pressing

Microreactor Program

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ABSTRACT

Powder metallurgy (PM) hot isostatic pressing (HIP) is a manufacturing technique that consolidates metallic powders to produce near-net-shaped parts. Potential benefits of the PM-HIP process are to reduce the time required to fabricate large-scale parts and/or improve designs, specifically for high temperature reactors and/or microreactors. However, the PM-HIP process is not an approved manufacturing method in Section III, Division 5 of the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code (BPVC) for high temperature reactors. Therefore, it is not readily available for use by commercial reactor manufacturers. Introduction into ASME Sec. III. Div. 5 BPVC would require high temperature creep, creep-fatigue, and low cycle fatigue data to allow for developing design criteria. Prior work showed that PM-HIP 316 stainless steels have reduced creep-fatigue performance compared to a traditionally manufactured, wrought 316 stainless steel. Because of the large discrepancy between PM-HIP 316 and wrought 316 alloys, there is a need for additional high temperature, mechanical property data, which will allow for a better understanding of the mechanical behavior and development of a code case. The purpose of this work was to generate creep data to 1) understand the creep behavior of PM-HIP 316H relative to the previously generated creep-fatigue properties and 2) continue to acquire data for the ASME Sec. III, Div. 5 BPVC.

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ACRONYMS

ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
BPVC	Boiler and Pressure Vessel Code
HIP	hot isostatic pressing
INL	Idaho National Laboratory
MRP	Microreactor Program
PM	powder metallurgy
UTS	ultimate tensile strength
YS	yield strength

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1. INTRODUCTION

Powder metallurgy (PM) hot isostatic pressing (HIP) has been considered as an advanced manufacturing method capable of near-net-shape component fabrication. The process starts with metallic powders contained within a vessel called a can that approximates the shape of the final part. This powder-filled can is loaded into a HIP chamber where temperatures are elevated to as high as 0.7–0.9 times the melting temperature and isostatic pressure is applied on the order of 100 MPa. The high temperature and pressure coalesce the metallic particles to generate a fully dense part. After HIP, the can material is removed, either mechanically or chemically, from the interior billet composed of the original powder.

It is possible that the PM-HIP manufacturing process can reduce fabrication steps to improve manufacturing efficiency and help improve component design compared to traditional forging, rolling, and casting processes. Therefore, it is necessary to understand the material properties that can be achieved with PM-HIP processing, and data must be produced to understand limitations of the process for each alloy. Specifically, if PM-HIP is desired for advanced, high temperature nuclear reactor construction, data must be collected to develop the design rules and introduce the process and materials into the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code (BPVC) Section III "Rules for Construction of Nuclear Facility Components", Division 5 High Temperature Reactors [1], or the methods and data would need to be approved by the United States Nuclear Regulatory Commission.

Therefore, an effort was undertaken to demonstrate the feasibility of incorporating PM-HIP, specifically starting with 316 stainless steels, into ASME Sec. III, Div. 5. Specifically, the Sec. III, Div. 5 Task Group on Advanced Manufactured Components considered PM-HIP to be a relatively mature technique based on prior work. This work was from the incorporation of PM-HIP 316L stainless steel into ASME Sec. III "Rules for Construction of Nuclear Facility Components", Div. 1 – Subsection NB Class 1 Components [2] as Code Case N-834 [3]. The work behind Code Case N-834 concluded that the lowest possible oxygen content and nitrogen contents up to 0.1 weight percent (wt%) are beneficial for mechanical properties. Albeit, these mechanical properties were only low temperature mechanical testing using methods such as Charpy V-notch impact toughness. Therefore, it is irrelevant to the high temperature properties required of Sec. III, Div. 5.

To demonstrate high temperature, time-dependent properties of 316L PM-HIP material, Rupp and Wright [4] performed scoping creep-fatigue testing. Their results indicated that a large reduction in cycles to failure (approximately half) was occurring compared to a wrought 316L stainless steel. This work continued and expanded to include PM-HIP 316H stainless steel [5]. Similar to PM-HIP 316L, the PM-HIP 316H data showed a high reduction in cycles to failure compared to wrought 316H. An additional heat of powder was procured with lower oxygen content and used to create two billets of PM-HIP 316H, which were hot isostatic pressed at two different HIP conditions. Neither of the billets showed an improvement in the creep-fatigue performance [6].

The purpose of this work is to expand on the elevated temperature testing to better understand the issues associated with the low creep-fatigue performance. The PM-HIP 316H material was subjected to creep testing and elevated temperature tensile testing. Each billet was tested in the same condition as previous creep-fatigue tests, which was hot isostatically-pressed and solution-heat treated by a commercial vendor as reported in Reference [7]. This report contains additional high temperature mechanical properties and the corresponding microstructures from failed creep test specimens.

2. EXPERIMENTAL METHODS

2.1. Materials

The composition of the commercially produced powder and compositions of the consolidated billets produced via two different HIP parameters are shown in Table 1. For reference, Table 1 also shows the chemical composition required by ASME Sec. II, Part A [8] for 316H stainless steel plate (S31609), the chemical composition for 316 stainless steel as mandated by ASME BPVC Sec. III Div. 5 [1] for high temperature (>595°C) use, and the 316 composition reported in American Society of Testing and Materials (ASTM) A988/A988M, "Standard Specification for Hot Isostatically-Pressed Stainless Steel Flanges, Fittings, Valves, and Parts for High Temperature Service" [9].

Table 1. Measured powder and consolidated billet chemical compositions in weight percent.

	316H Powder	PM-HIP 316H Billet 2A	PM-HIP 316H Billet 2B	SA 240 S31609 (316H)	ASME III Div. 5 (>595°C)	ASTM A988 S31600
Fe	Bal.	Bal	Bal.	Bal.	Bal.	Bal.
C	0.05	0.043	0.043	0.04–0.1	0.04–0.1	0.08
Ni	12.0	12.06	12.01	10.0–14.0	10.0–14.0	10.0–14.0
Cr	17.0	17.13	17.05	16.0–18.0	16.0–18.0	16.0–18.0
Mo	2.53	2.54	2.53	2.00–3.00	2.00–3.00	2.00–3.00
Cu	—	<0.01	<0.01	—	—	—
Ti	<0.003	<0.003	<0.003	—	≤0.04 *	—
Al	<0.01	0.003	0.005	—	≤0.03 *	—
Si	0.20	0.18	0.19	1.00	1.00	1.00
Mn	0.21	0.19	0.19	2.00	2.00	2.00
S	0.003	0.003	0.003	0.030	0.030	0.030
P	0.004	0.004	0.004	0.045	0.045	0.045
B	—	0.0003	0.0003	—	—	—
N	0.101	0.090	0.088	—	≥0.05*	≤0.10
O	0.012	0.0156	0.0149	—	—	—

NOTE: * Div.5 supplementary requirement for use at temperatures above 1,100°F (595°C).

2.2. Mechanical Testing

Elevated temperature tensile tests and creep tests were performed on billets A and B to better understand the performance of these PM-HIP 316H stainless steels. The parameters for each test are defined in the following subsections.

2.2.1. Elevated Temperature Tensile Testing

Tensile testing was performed at 650°C using a 100 kN load frame and furnace heating under atmospheric conditions. The strain rate was performed at 0.002/s and controlled with an extensometer up to a 20% strain. After a 20% strain, the extensometer was removed for the remainder of the tensile test, and the strain was based on stroke control with a crosshead speed of 1.75 mm/min. The specimen geometry was identical to the creep specimens and is shown in Figure 1.

2.2.2. Creep Testing

Creep testing was performed using a statically loaded frame with a 20:1 lever arm. Tests were conducted at 650°C in air with an applied stress of 160 MPa. The specimens were machined, and tests were conducted in accordance with ASTM E21 "Standard Test Methods for Elevated Temperature Tension Tests of Metallic Materials" [10]. Figure 1 shows a fully machined cylindrical specimen geometry used for creep. The geometry used threaded grips, a nominal total length of 3.90 in [100 mm], and a nominal gauge section diameter of 0.250 in [6.35 mm].

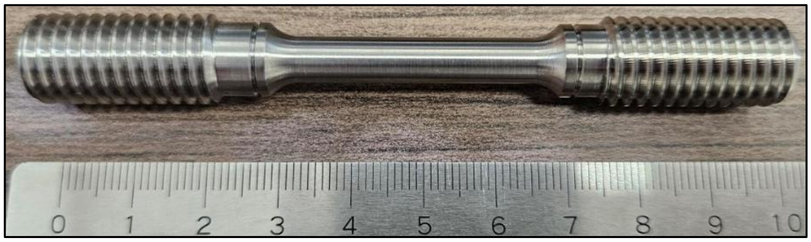

Figure 1. Machined specimen geometry used for creep testing and elevated temperature tensile testing.

2.3. Microstructure Characterization

For metallographic evaluation, specimens were abrasively sectioned and mounted in a thermosetting polymer. All specimens were mechanically ground with 400-, 600-, and 800-grit silicon carbide abrasive paper followed by polishing with 6 and 3 µm polycrystalline diamond suspension. Electrochemical etching was performed using a 10% oxalic acid solution and a constant voltage of 3 V for nearly 20 seconds.

3. RESULTS

3.1. Mechanical Test Results

3.1.1. Elevated Temperature Tensile Tests

Figure 2 shows plots of engineering stress versus strain for billet 2A and billet 2B from tensile tests at 650°C. For comparison, a plot of wrought 316H stainless steel using data extracted from Zhang et al. [11] is shown in Figure 2. Both billets A and B showed similar tensile properties for yield strength (YS), ultimate tensile strength (UTS), and percent elongation to failure. Further evaluation of the results and the measured reduction in area is shown in Figure 3.

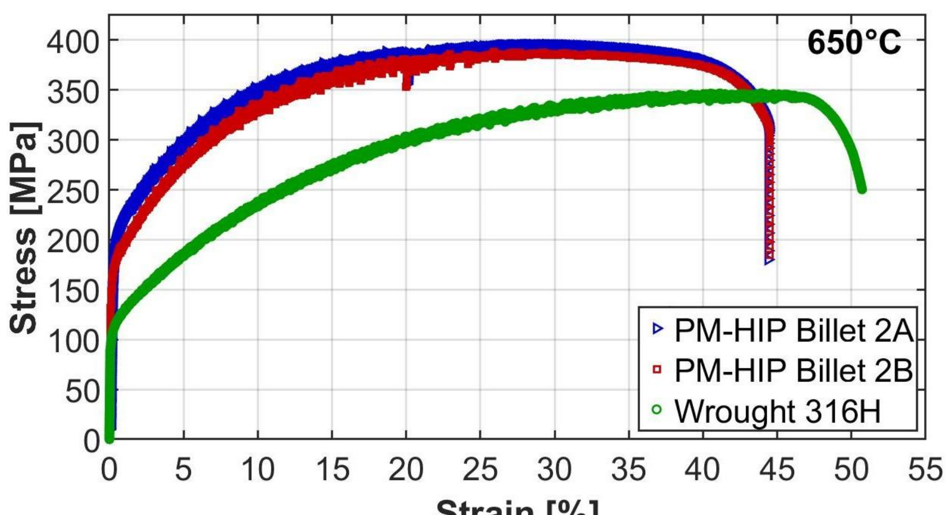

Figure 2. Stress versus strain curves for elevated temperature tests of billet 2A and billet 2B at 650°C compared with wrought 316H data from Zhang et al. [11].

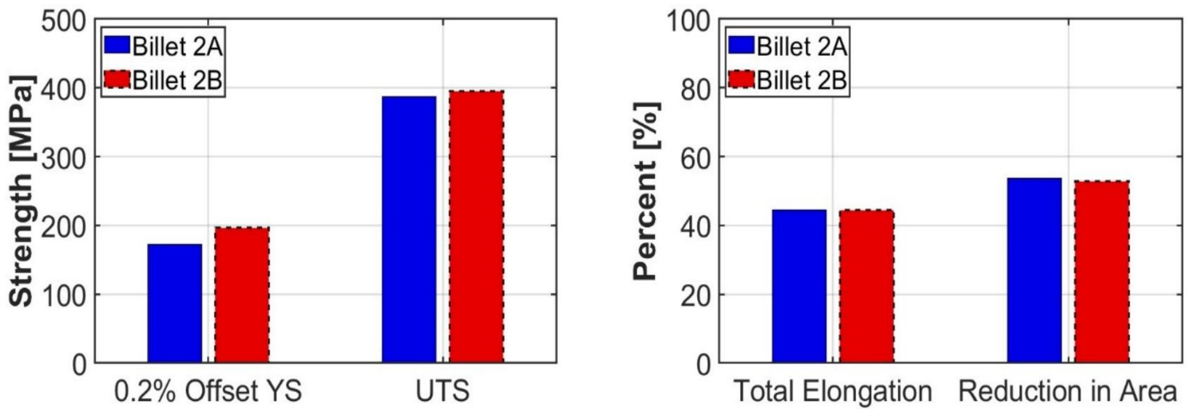

Figure 3. Yield strength, ultimate tensile strength, elongation, and reduction in area for the PM-HIP 316H stainless steels tested at 650°C.

Compared to the wrought material, the 316H PM-HIP elongation to failure was nearly 8% lower, but the YS and UTS were higher. This is likely attributed to the finer grain size, where PM-HIP billets A and B had ASTM grain size numbers of 8 and 7, respectively. Whereas the finest grain size allowed for 316H in plate is ASTM No. 7 based on ASTM A240 “Standard Specification for Chromium and Chromium-Nickel Stainless Steel Plate, Sheet, and Strip for Pressure Vessels and for General Applications” [12]. Therefore, it was expected that the wrought material would have a lower strength, solely based on a larger grain size. Even though there was variation in the low cycle fatigue and creep-fatigue performance of these two 316H billets [6], [7], the elevated temperature tensile tests showed similar results with negligible variation when comparing strength or ductility.

3.1.2. Creep Tests

Figure 4 shows results for four creep tests performed at 650°C and 160 MPa. Two tests were with billet 2A, and two tests were with billet 2B. Billet 2A, with the finer grain size, showed better creep strain and time to failure than billet 2B. Although billet 2A showed high variability between both tests, the average creep strain to failure was approximately 8% higher with a longer lifetime of nearly 250 hours. Figure 5 shows a plot of time-to-failure and total creep strain at failure for billet 2A and 2B in comparison to historical, unpublished data for wrought 316H.

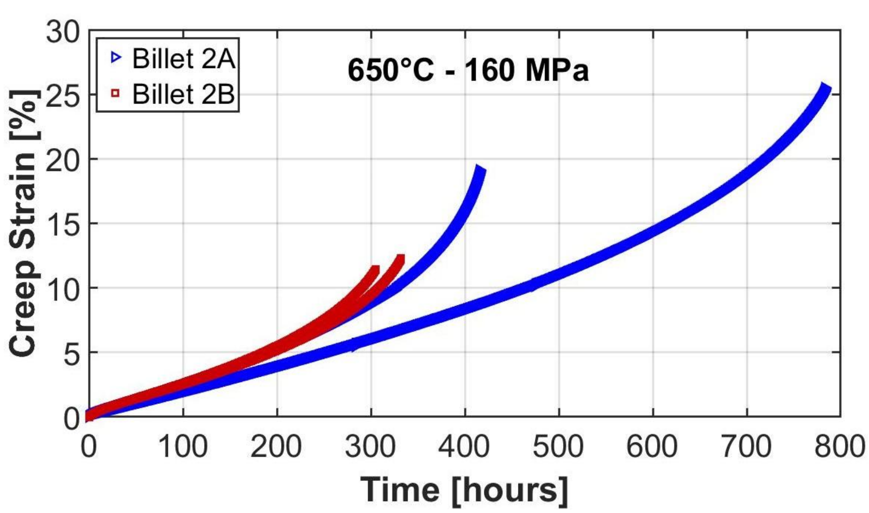

Figure 4. Strain versus time for PM-HIP 316H billets 2A and billets 2B creep tested at 650°C and a stress of 160 MPa.

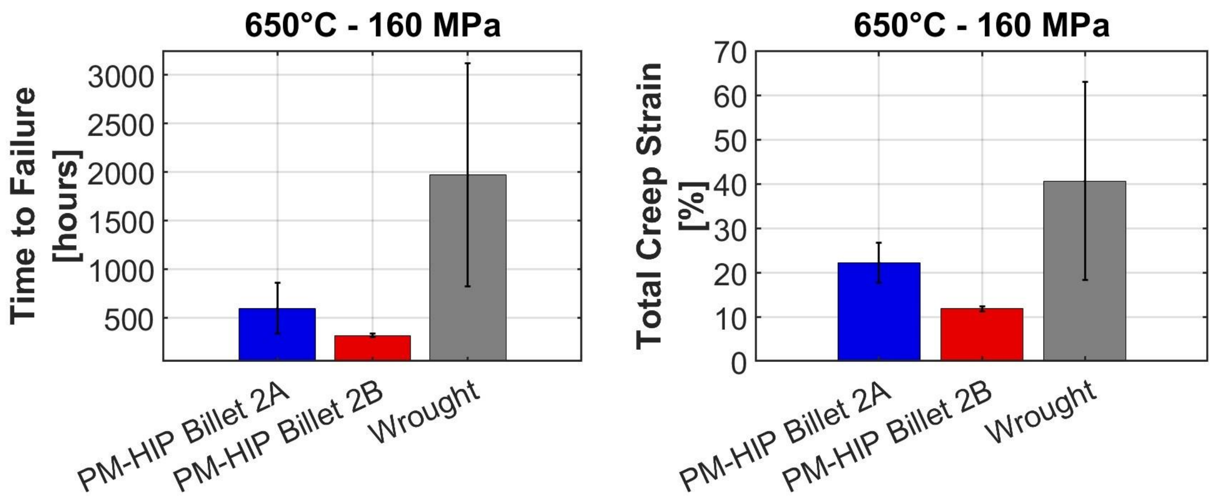

Figure 5. Time to failure (left) and total creep strain (right) for PM-HIP 316H billets 2A and 2B compared to wrought 316H stainless steel from unpublished data.

Although a much greater sample size, wrought 316H has an average time to failure of nearly four times that of the PM-HIP materials. Similarly, the creep strain is reduced by a factor of two for billet 2A and nearly a factor of four for billet 2B. The differences in the creep results were consistent with results obtained from creep-fatigue testing, where billet 2A showed better performance (higher cycles to failure) for low cycle fatigue and creep-fatigue tests at 650°C [6]. This data PM-HIP stainless steel creep data is in contrast to data originally published by Östlund and Berglund [13] who reported PM-HIP 316L creep performance as nearly equivalent to wrought 316L.

3.2. Microstructure Analysis

3.2.1. Creep test microstructures

Figure 6 shows photos of a failed billet 2A creep specimen. Each PM-HIP specimen failed with minimal reduction of area and initiated numerous cracks throughout the gauge section. Optical micrographs at two different magnifications of an as-tested creep specimen from billet 2A are shown in Figure 7. Similarly, optical micrographs at the same magnifications for billet 2B are shown in Figure 8.

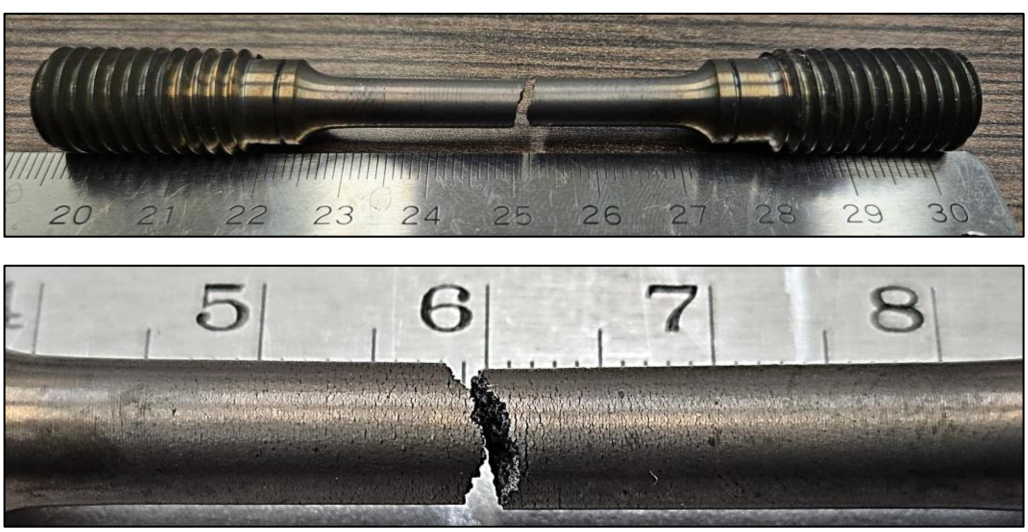

Figure 6. Photos of a failed billet 2A creep specimen.

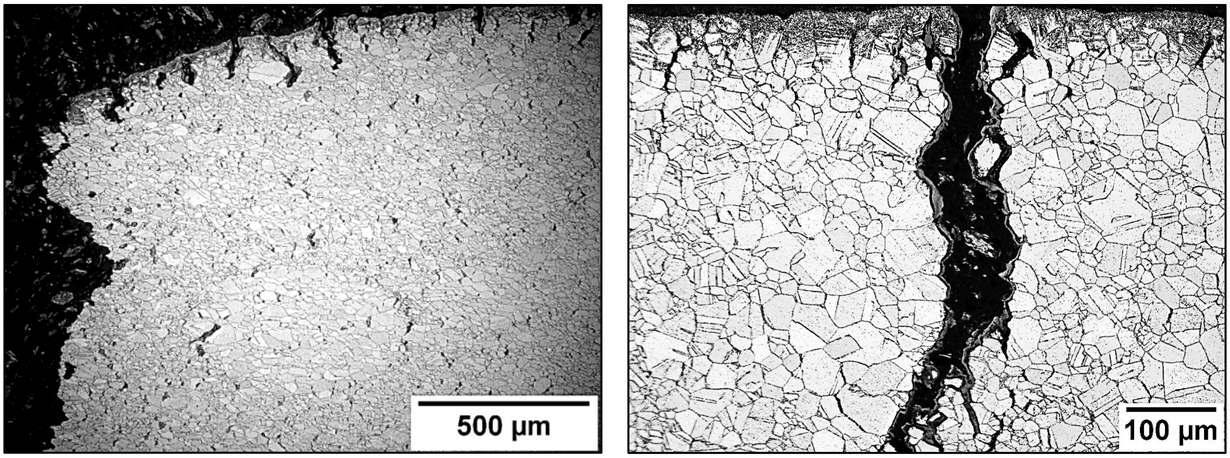

Figure 7. Optical micrographs of billet 2A creep tested at 650°C and 160 MPa.

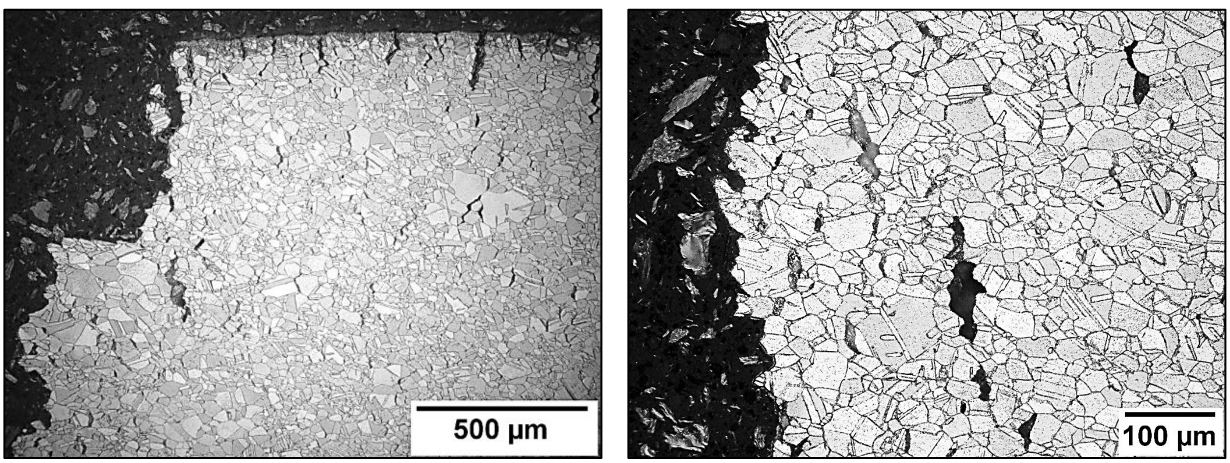

Figure 8. Optical micrographs of billet 2B creep tested at 650°C and 160 MPa.

Figure 7 and Figure 8 depict the cracks nucleated and propagated along the grain boundaries. The importance of the intergranular crack morphology is that it is the cracking mechanism seen in the creep-fatigue test specimens [6], [7]. Due to the similarities, it can be inferred that the cracking mechanism and reduced cycles-to-failure associated with the creep-fatigue test is mostly caused by intergranular creep damage. Because these PM-HIP materials contain oxides/nitride particles along the grain boundaries, which are likely prior particle boundaries, it is probable that the reduced creep and creep-fatigue performance is attributed to these oxides/nitrides and their interaction with the prior particle/grain boundaries.

4. SUMMARY

This work showed that the 650°C creep properties for 316H PM-HIP contained trends that were consistent with the results obtained from creep-fatigue tests. Billet 2A, which had a finer grain size (ASTM No. 8), showed longer creep life and higher creep ductility compared to billet 2B (ASTM No. 7). The only difference between these two conditions was the HIP temperature and time, as the powders were the same. Also, the PM-HIP 316H creep tests showed substantially reduced creep ductility and creep rupture life than wrought 316H. The properties from these scoping tests indicate that PM-HIP 316H stainless steels are not comparable to wrought 316H and cannot use the same design criteria.

5. FUTURE WORK

If PM-HIP 316 alloys are intended to be used in structural applications for high temperature reactors, many more tests are needed to understand the mechanical property degradation compared to wrought counterparts, i.e., different temperatures, stresses, strains, and strain rates. It is also necessary to analyze other alloys produced via PM-HIP with compositions approved for high temperature use in ASME Sec. III, Div. 5 BPVC. Without collecting this data, PM-HIP will not be available as a BPVC allowable process for commercial, high temperature reactor developers within the United States.

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