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The Elevated-Temperature Cyclic Properties of Alloy 316L Manufactured Using Powder Metallurgy Hot Isostatic Pressing

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Changing the World's Energy Future

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SUMMARY

According to developers at the GAIN 2019 microreactors workshop, advanced manufacturing (AM) is needed to fabricate microreactor components. A methodology has not been established to qualify AM for elevated-temperature nuclear construction. A potential approach is to use the wrought design methodology by establishing the AM properties as equivalent to or better than wrought. This investigation aims to understand the relationship between powder metallurgy hot isostatic pressing (PM HIP) and material characteristics for Alloy 316L as they relate to elevated-temperature component design and construction. The elevated-temperature cyclic behavior of Alloy 316L manufactured by PM HIP differs from wrought. Of particular significance is the reduced number of cycles to failure for the PM HIP Alloy 316L compared to wrought. Additional work to identify the microstructural feature that control the elevated-temperature cyclic behavior is needed.

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ACRONYMS

AM	Advanced manufacturing
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
BPVC	Boiler and Pressure Vessel Code
BSE	Backscattered electrons
EBSD	Electron backscattered diffraction
IPF	Inverse pole figure
MTR	Materials test report
MTS	Materials Test Systems
PM HIP	Powder metallurgy hot isostatic pressing
SE	Secondary electrons
SEM	Scanning electron microscope
w.r.t.	With respect to

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The Elevated-Temperature Cyclic Properties of Alloy 316L Manufactured Using Powder Metallurgy Hot Isostatic Pressing

1. INTRODUCTION

Advanced manufacturing (AM) offers many potential benefits including the ability to fabricate geometrically complex components and obsolete replacement parts. Consequently, a wide variety of industries are taking advantage of AM including automotive and aerospace. The nuclear industry has expressed interest in utilizing AM to fabricate nuclear components [1]. At the GAIN 2019 microreactors workshop, developers communicated a need for AM to fabricate microreactor components. AM, in this report, refers to any fabrication technique that is currently not qualified in Section III, Division 5 of the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code (BPVC). This section of the code pertains to the construction of elevated-temperature nuclear components [2]. A methodology to qualify AM for elevated-temperature nuclear construction has not been established. Qualification requires establishing that the component will behave as excepted for the entirety of its design life [1]. Time-dependent properties including creep and creep-fatigue come into play for elevated-temperature applications further complicating qualification.

A performance-based qualification methodology approach is proposed. It is hypothesized that if specific mechanical properties and microstructural features of an AM material are equivalent to those of wrought, then the wrought design methodology can be applied to the AM material. This would significantly reduce the resources required for code qualification. It should be noted a performance-based qualification approach is in line with the expectations of the Nuclear Regulatory Commission. The alternative is the qualification of specific AM techniques [1, 3]. The objective of this investigation is to develop an understanding of the role of AM on material characteristics as they relate to elevated-temperature component design and construction. This investigation focuses on powder metallurgy hot isostatic pressing (PM HIP) of Alloy 316L.

PM HIP was selected for this investigation as it is more mature than other AM techniques and is more readily deployable in the near term. Its maturity is supported by the following observations. First, PM HIP has been qualified in the ASME BPVC for the construction of low-temperature nuclear components. ASME BPVC Case N-834 permits the construction of Alloy 316L components fabricated by PM HIP for Section III, Division 1, Subsection NB applications. This code case specifies that the wrought design methodology shall be used if the PM HIP is in conformance with the American Society for Testing and Materials (ASTM) standard A988 and a list of fabrication and post-fabrication requirements for the component are met [4, 5]. Second, Alloy 316L PM HIP creep properties have been demonstrated to be equivalent to wrought [6].

2. EXPERIMENTAL PROCEDURE

2.1 Material

This investigation necessitated the procurement of Alloy 316L fabricated by both PM HIP and conventional processing. This enabled both fabrication methods to be tested using the same methodology so that their properties could be directly compared. The Alloy 316L fabricated by conventional processing is referred to as wrought material in this report.

2.1.1 PM HIP

An Alloy 316L PM HIP bar, 36241-13, was purchased off the shelf from commercial vendor Sandvik Powder Solutions, AB, Surahammar, Sweden. Sandvik has since been acquired by Metal Technology Co., Ltd. The materials test report (MTR) states conformance to ASTM standard A988 and ASME BPVC Case N-834. The bar had a diameter of 190 mm and length of 590 mm. Specimens were machined so that the longitudinal axis was parallel to the length of the PM HIP bar.

The chemistry and room-temperature tensile properties of the PM HIP bar reported in the MTR are provided in Table 1 and Table 2 below. The properties reported in the MTR were determined from bar 36241-12. This bar came from the same powder batch and was heat treated with the procured bar. It should be noted that ASTM standard A988 specifies the chemical composition and minimum room-temperature tensile properties. The reported chemistry and room-temperature tensile properties meet these requirements. The PM HIP grains per the MTR were determined to have an average ASTM grain size number of six. The comparison technique in ASTM standard E112 was used to determine average grain size [7].

Table 1. Chemical composition in weight percent of PM HIP, heat 36241, and wrought, heat A26M, Alloy 316L bar.

	C	Si	Mn	P	S	Cr	Ni	Mo	Al	B
PM HIP	0.012	0.65	0.58	0.011	0.009	17.44	11.50	2.22	< 0.002	0.0004
Wrought	0.015	0.23	1.34	0.035	0.0230	16.56	10.53	2.003	NR	NR
	Co	**Cu**	**Nb**	**Ta**	**Ti**	**V**	**Fe**	**N**	**O**	**Ta+Co**
PM HIP	0.021	0.033	< 0.005	0.005	0.006	0.020	67.40	0.049	0.020	0.026
Wrought	0.29	0.46	NR	NR	NR	NR	NR	0.034	NR	NR

NR = not reported

Table 2. Room-temperature tensile properties of PM HIP, heat 36241, and wrought, heat A26M, Alloy 316L bar.

	Yield stress	**Ultimate tensile strength**	**Elongation**	**Reduction in area**
	MPa	MPa	%	%
PM HIP	294	575	59.0	72
Wrought	519.4	660.7	40.74	69.42

2.1.2 Wrought

Wrought Alloy 316L was procured off the shelf from commercial vendor Best Stainless and Alloys. The product form was 25.4 mm diameter round bar that was annealed with a cold draw finish. The MTR indicates the material was produced by North American Stainless, heat A26M. The MTR states compliance with a number of standards including ASME SA479-17 [8]. The chemistry and room temperature tensile properties as reported in the MTR are provided in Table 1 and Table 2 above. The grain size, according to the MTR, is an ASTM grain size number of four to six. This wrought material was selected because of its similar carbon content and grain size to the PM HIP bar.

2.2 Microstructure characterization

The as-received PM HIP microstructure was characterized using both the Archimedes' principle and microscopy. The experimental procedure for both techniques will now be discussed.

2.2.1 Archimedes' principle

Density was determined using Archimedes' principle. Both an oil impregnated and non-oil impregnated specimen were used. Measurements were in accordance with ASTM standard B962 for the oil impregnated specimen and ASTM standard B311 for the non-oil impregnated specimen [9, 10].

2.2.2 Microscopy

Imaging with both secondary electrons (SE) and backscattered electrons (BSE) as well as electron backscattered diffraction (EBSD) were used to characterize microstructure with the scanning electron microscope (SEM). Adequate specimen preparation for SEM characterization was achieved using standard grinding and polishing techniques. The SEM was operated at a 20 keV accelerating voltage and spot size of 6. EBSD data was collected using a 2 μm step size and 4 × 4 binning. EBSD data was processed using MTEX, a free MATLAB toolbox for the analysis of crystallographic texture. A misorientation between neighboring pixels larger than 5° defined a grain boundary. Grains were required to be comprised of a minimum of 5 pixels.

2.3 Elevated-temperature cyclic testing

Fully reversed, strain-controlled fatigue testing with a symmetric triangular wave form was performed. These tests were at 600 and 650°C for a total strain of 1%. All tests had a strain rate of 10^{-3} s^{-1}. Creep-fatigue testing was also performed. These tests were equivalent to the fatigue tests with the addition of a 30-minute hold at the peak tensile strain. These tests were in accordance to PLN-3346 and generally followed the specifications in ASTM standard E606 for fatigue and ASTM standard E2714 for creep-fatigue testing [11, 12, 13].

Testing was performed in air in a three-zone resistance furnace equipped to a servo-hydraulic test frame. Materials Test Systems (MTS) manufactured both the furnace and test frame. Two R-type thermocouples spot welded to the specimen shoulders were used to monitor specimen temperature during testing. A direct-mounted extensometer with a 12 mm gauge length and symmetric total travel range of 5 mm from Epsilon Technology recorded strain. Button-head specimens with a 7.49 mm diameter were used. Before testing, essential specimen dimensions were measured with a 50× optical comparator.

Upon test completion, the data were analyzed. The number of cycles to failure, N_f, was identified. Its determination depended on the location of the primary crack. The primary crack located within the extensometer gauge length is optimal. For this report, this location is referred to as IX. For an IX primary crack, N_f is defined as a 25% drop in maximum peak stress from N_o, the number of cycles to crack initiation. N_o is identified as the cycle where the maximum peak stress deviates from linearity. Schematics defining N_f and N_o are available in Figure 1 of ASTM standard E2714 [13]. Sometimes, the primary crack falls outside the extensometer gauge length. This crack location is referred to as OX. During a test with an OX primary crack, the extensometer is unable to record crack initiation and propagation. Once the crack initiates, the specimen is strained each cycle an amount greater than desired. As a result, a test with an OX crack is not considered valid. The test, however, still provides useful information. The number of machine cycles to failure is reported as N_f. The final possible primary crack location is at the boundary of the extensometer gauge length. This primary crack location is referred to as AX. The analysis of these samples depends on whether the extensometer is able to capture crack initiation and propagation. If it does, the analysis is treated in the same manner as a test with and IX primary crack. Otherwise, the test analysis is the same as one with an OX primary crack.

3. RESULTS

3.1 As-received PM HIP microstructure

The density of the PM HIP bar was measured to be 99.25% of the density typical of wrought and is therefore in accordance with ASTM standard A988 [5]. Pores were observed using a SEM with the SE and BSE modes at magnifications of 300× and greater. Figure 1 below shows porosity in the as-received microstructure at a magnification of 2,000×. It should be noted the density and microstructure requirements specified in ASTM standard A988 are still met since the Archimedes' principle confirmed that the density exceeded 99% of the density typical of wrought [5]. However, ASME BPVC Case N-834 requirement e.1 is not met which requires the microstructure to be free of porosity at magnifications up to and including 2,000× [4]. The as-received PM HIP microstructure is fairly homogenous with a reasonably uniform grain size and an extremely random texture. This is evident from the inverse pole figure (IPF) map and IPFs in Figure 2 and Figure 3 below.

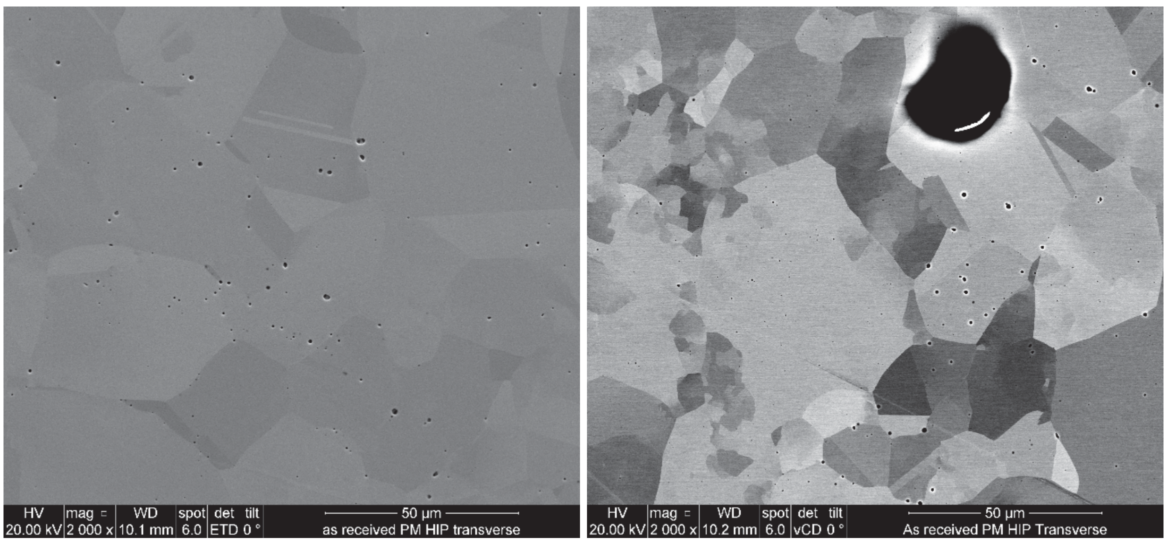

Figure 1. SE image (left) and BSE image (right) of the as-received PM HIP at a magnification of 2,000×. Porosity is present in the as-received PM HIP. There is a wide distribution in porosity size.

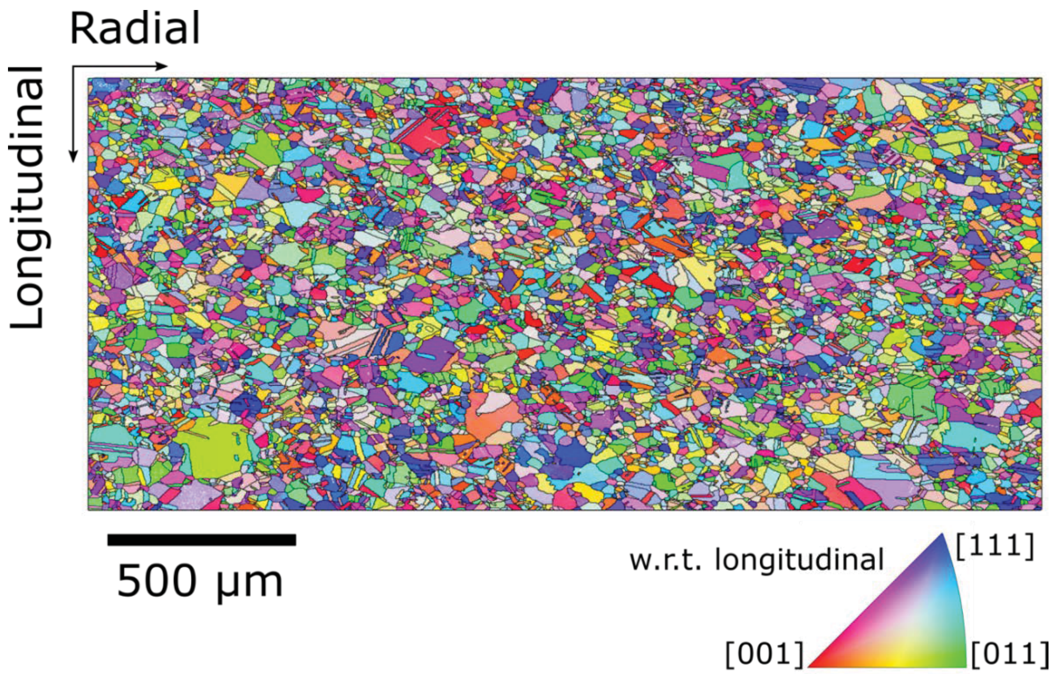

Figure 2. IPF map of EBSD data from the as-received PM HIP bar. The map is with respect to (w.r.t.) the longitudinal direction of the PM HIP bar.

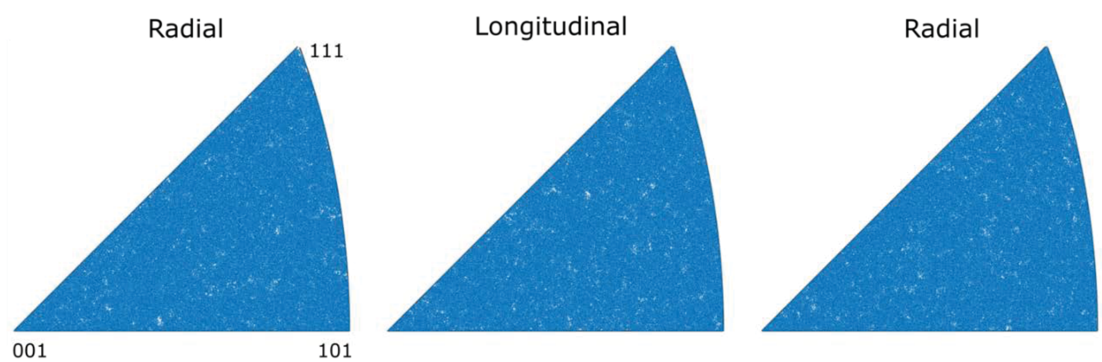

Figure 3. IPFs of EBSD data from the as-received PM HIP bar.

3.2 Elevated-temperature cyclic properties

The elevated-temperature cyclic behavior of the PM HIP material is different than that of the wrought. This is evident from Table 3 and Figure 4 below. Table 3 reports N_o, N_f, and various stresses at cycle 10 and midlife. Figure 4 shows the peak stress as a function of cycle. The steady state peak stress of the wrought material is greater than the PM HIP material for the conditions tested. This indicates the wrought material is stronger than the PM HIP material. The number of fatigue cycles to failure of the PM HIP material is greater than the wrought material. The opposite is observed for creep-fatigue. The PM HIP material initially cyclically hardens prior to reaching its steady state behavior. The wrought material, on the other hand, initially cyclically softens prior to reaching its steady state behavior. It is postulated that the hardening and softening behavior can be attributed to the PM HIP material starting from the solution annealed condition while the finish for the wrought material was cold drawn. The duplicate tests run for each material and test condition emphasize the reproducibility of these results. This indicates that the differences in elevated-temperature cyclic behavior between the PM HIP and wrought materials are significant.

Table 3. Fatigue and creep-fatigue tests results at 600°C and 650°C for PM HIP and wrought Alloy 316L material.

Specimen	Hold time	N_o	N_f	Primary crack location	Cycle 10				Cycle Midlife			
					σ_{max}	σ_{min}	$\sigma_{hold\ start}$	$\sigma_{hold\ end}$	σ_{max}	σ_{min}	$\sigma_{hold\ start}$	$\sigma_{hold\ end}$
	minutes	cycle	cycle		MPa	MPa	MPa	MPa	MPa	MPa	MPa	MPa
600°C												
316L-HIP-35	0	1,859	2,048	AX	235	-242	-	-	264	-270	-	-
316L-HIP-36	0	1,822	2,002	IX	236	-244	-	-	270	-276	-	-
316L-W-07	0	1,226	1,375	IX	330	-324	-	-	295	-296	-	-
316L-W-08	0	1,062	1,112	IX	325	-326	-	-	298	-302	-	-
316L-HIP-41	30	506	580	IX	223	-228	167	165	237	-244	210	181
316L-HIP-42	30	445	705	IX	220	-225	216	172	232	-241	205	161
316L-W-03	30	1,371	1,464	IX	307	-310	306	249	271	-275	261	216
316L-W-05	30	1,189	1,358	IX	308	-313	307	264	267	-277	267	218
650°C												
316L-HIP-37	0	1,588	1,811	IX	231	-236	-	-	239	-242	-	-
316L-HIP-38	0	-	1,379	OX	231	-238	-	-	244	-249	-	-
316L-W-04	0	1,087	1,235	IX	305	-304	-	-	270	-273	-	-
316L-W-06	0	1,057	1,196	IX	300	-300	-	-	270	-272	-	-
316L-HIP-39	30	417	459	IX	206	-208	149	91	202	-206	198	103
316L-HIP-40	30	381	442	IX	205	-206	144	104	205	-210	204	118
316L-W-01	30	1,047	1,198	IX	277	-280	277	204	241	-247	223	157
316L-W-02	30	901	1,050	IX	275	-273	274	191	244	-248	236	145

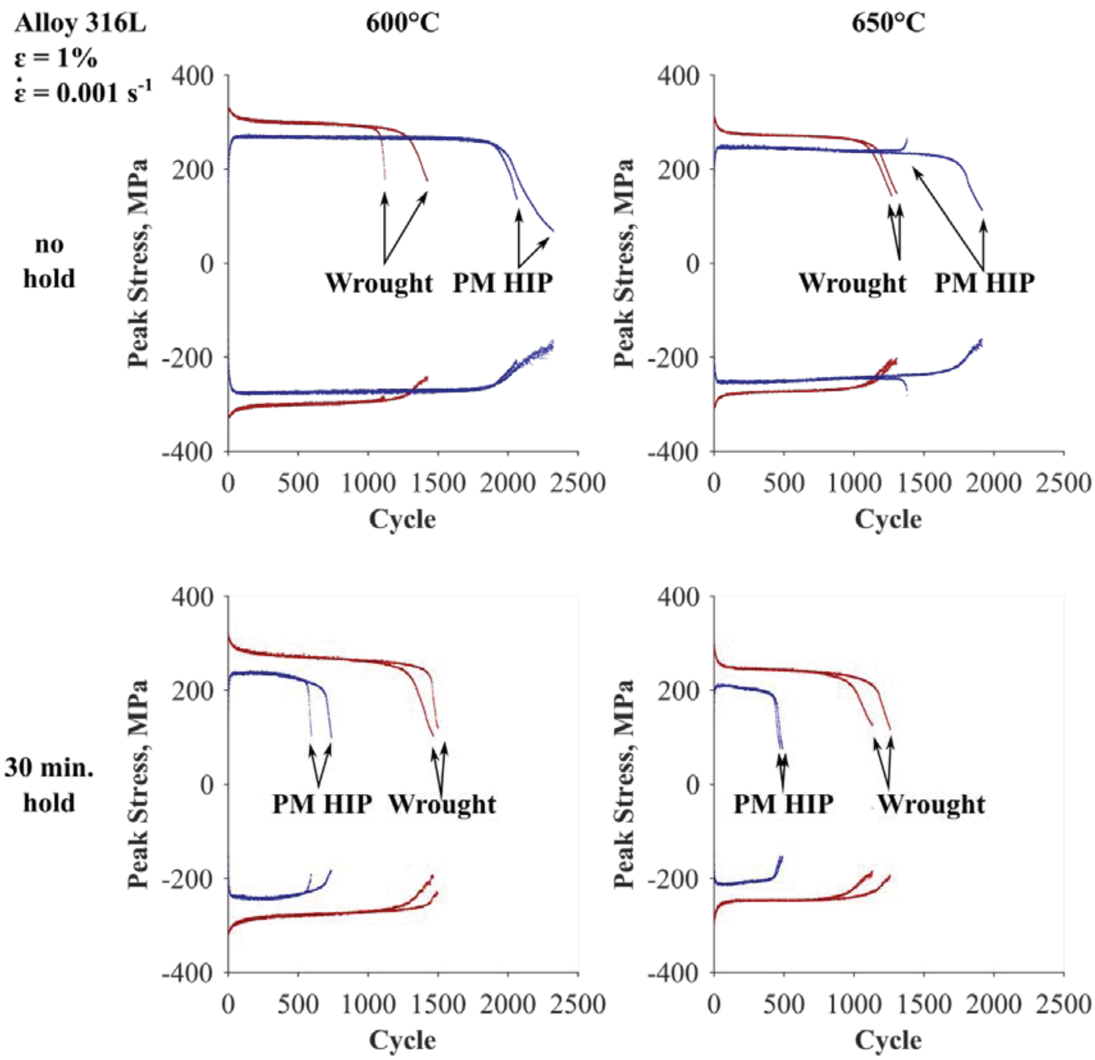

Figure 4. Peak stress versus cycle of the PM HIP and wrought Alloy 316L material from 1% total strain fully-reversed fatigue and creep-fatigue tests at 600°C and 650°C.

4. DISCUSSION

A potential route for AM code qualification is to establish the AM properties as equivalent to wrought [3]. Code qualification may be simplified as the wrought design methodology would be used and less data would likely need to be generated. This route would necessitate establishing mechanical properties and microstructural features that must be met to ensure equivalency.

The elevated-temperature cyclic properties of the Alloy 316L manufactured by PM HIP in this investigation were different than wrought. The creep-fatigue properties of the Alloy 316L manufactured by PM HIP are significantly reduced compared to wrought. The room-temperature tensile properties of the Alloy 316L manufactured by PM HIP exceeded the minimums specified in ASTM standard A988 [5]. Therefore, room-temperature tensile properties alone are not sufficient to establish equivalent or improved properties compared to wrought. The microstructural features that caused these differences need to be identified so that they can possibly be adjusted in the future to achieve equivalency with wrought. Some potential sources of variation are the porosity, grain size, chemistry, oxides, inclusions, and texture.

Future work will involve looking at each of these potential sources of variation in detail. For example, X-ray computed tomography will be used to characterize the porosity of the as-received microstructure. This will enable the distribution in pore size to be quantified. The crack path of the tested PM HIP specimens will be characterized in order to identify the role of porosity on fatigue and creep-fatigue behavior.

5. CONCLUSIONS

The elevated-temperature cyclic behavior of Alloy 316L manufactured by PM HIP differed from wrought. The number of cycles to failure for the Alloy 316L manufactured by PM HIP were significantly reduced compared to wrought for creep-fatigue. The room-temperature tensile properties of the Alloy 316L PM HIP bar exceeded the minimums specified in ASTM standard A988 [5]. This demonstrates that room-temperature tensile properties alone do not ensure equivalent or improved properties compared to wrought. Easily identifiable microstructural features and/ or mechanical properties that control the elevated-temperature cyclic behavior need to be identified.

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