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The Elevated-Temperature Cyclic Properties of Alloy 316H Fabricated by Powder Metallurgy Hot Isostatic Pressing

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SUMMARY

Powder metallurgy hot isostatic pressing (PM HIP), an advanced manufacturing technique, is viewed by the Department of Energy as a "transformational technology" for heavy section manufacturing of nuclear components. Although PM HIP is a mature technology, it has not been qualified in Section III, Division 5 of the American Society for Mechanical Engineers Boiler and Pressure Vessel Code for elevated-temperature nuclear construction. This work seeks to demonstrate a PM HIP qualification method for elevated-temperature nuclear construction that is based on allowable material characteristics. Specifically, the elevated-temperature cyclic properties of PM HIP 316H are investigated. The creep-fatigue properties of PM HIP 316H are significantly degraded compared to those of wrought products. Current PM HIP standards, however, do not ensure fabrication of elevated-temperature nuclear components with creep-fatigue properties equivalent or superior to components fabricated from traditional wrought product. A research and development program is proposed for qualifying PM HIP 316H for elevated-temperature nuclear construction.

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ACRONYMS

AM	advanced manufacturing
AMTs	Advanced Manufacturing Technologies
ART	Advanced Reactor Technologies
ASME	American Society of Mechanical Engineers
ASTM	American Society for Testing and Materials
ATLAS	Advanced Technology for Large-Scale
BPVC	Boiler and Pressure Vessel Code
BSE	back-scattered electron
DOE	Department of Energy
EBSD	electron backscattered diffraction
EDS	energy dispersive X-ray spectroscopy
INL	Idaho National Laboratory
IPF	inverse-pole figure
MARVEL	Microreactor Applications Research Validation and EvaLuation
MRP	Microreactor Program
MTS	materials test systems
NE	Office of Nuclear Energy
NRC	Nuclear Regulatory Commission
PM HIP	powder metallurgy hot isostatic pressing
SEM	scanning electron microscope
w.r.t.	With respect to

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The Elevated-Temperature Cyclic Properties of Alloy 316H Fabricated by Powder Metallurgy Hot Isostatic Pressing

1. INTRODUCTION

Powder metallurgy (PM) hot isostatic pressing (HIP) is an advanced manufacturing (AM) technique that enables the fabrication of near-net-shape components. PM HIP consolidates powder by high isostatic pressure at elevated temperature under vacuum. The powder is typically produced by gas atomization and encapsulated in an evacuated steel can prior to densification. The can is fabricated so that it is the shape of the desired component but slightly larger (Gandy, 2013).

PM HIP has been identified as a "transformational technology" for heavy section manufacturing in the Department of Energy's Advanced Manufacturing Methods for Nuclear Energy Roadmap (Gandy, 2013). As a result of the small size of their components, microreactors are ideally suited to take advantage of this near net-shape AM process to support reduction in cost and schedule of microreactor construction. PM HIP also offers a myriad of other advantages for microreactor construction. These include the improved ability to inspect PM HIP components compared to those fabricated using conventional processes. This is a consequence of PM HIP producing a microstructure that is uniform and homogenous. This microstructure is also better for welding. Furthermore, PM HIP enables fabrication of complex shapes and the elimination of welds (Gandy, 2013; Burdett & Hookham, 2009; Cooper, Cooper, Dhers, & Sherry, 2016; Sulley, Wallace, Warner, & Jones, 2020). There has been recent advancement in the technology development of the PM HIP fabrication process for the pressure vessel of the small modular light water reactor. A large-scale facility that can be used to HIP a full-size pressure vessel of a small modular light water reactor is being planned through a public-private partnership, the Advanced Technology for Large-Scale (ATLAS) PM HIP Project. Such development for PM HIP can be leveraged for microreactor applications.

Nuclear reactors and nuclear reactor designs are regulated and licensed by the United States Nuclear Regulatory Commission (NRC). Nuclear components that require regulatory oversight must be qualified to ensure the component will behave as expected for the entirety of its design life. Time-dependent properties, including creep and creep-fatigue, come into play for elevated-temperature applications, further complicating qualification. One approach for qualifying a material is through the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code (BPVC). This route is specifically mentioned in the NRC's "Draft Action Plan for Advanced Manufacturing Technologies (AMTs)," which encompasses PM HIP (United States Nucler Regulatory Commission, 2019).

Elevated-temperature nuclear construction falls under Section III, Division 5 of the ASME BPVC. Currently, components fabricated by AM, including PM HIP, are not qualified in Section III, Division 5. The ASME BPVC Task Group on Division 5 AM Components is engaged in identifying the appropriate methods for qualifying AM components for elevated-temperature nuclear construction. The Task Group has identified PM HIP as one of two AM techniques closest to being ready for Section III, Division 5 qualification. This is because PM HIP is a mature technology that has already been incorporated into several sections of the ASME BPVC. This includes a code case for low-temperature nuclear construction. Code Case N-834 permits the construction of PM HIP 316L components for Section III, Division 1, Subsection NB applications. In this code case, the wrought-design methodology and allowables are used. "Wrought" refers to material fabricated by conventional methods. PM HIP creep properties have also been observed to be equivalent to wrought for Alloy 316L and V-modified 9% Cr-1% Mo steel (Gr 91) (Gandy, 2013; Östlund & Berglund, 2017). Time-dependent elevated-temperature data from PM HIP materials necessary for Division 5 qualification are not available, or at least not publicly.

This work seeks to demonstrate a PM HIP qualification method for elevated-temperature nuclear construction that is based on allowable material characteristics. This contrasts with a qualification approach that is dependent solely on the testing of witness specimens. Qualifying based on allowable material characteristics is a cross-cutting approach that can be applied to similar classes of materials such as solid-solution strengthened alloys. The fastest qualification route is to utilize the wrought design methodology and allowables in Section III, Division 5 of the code. This reduces the resources necessary for qualification. This route, however, requires the PM HIP material to have specific mechanical properties and microstructural features that are equivalent or superior to wrought.

In this study, the elevated-temperature cyclic properties of PM HIP 316H are investigated. Alloy 316H was selected as it is one of six materials fabricated by conventional processing qualified in ASME Section III, Division 5 for elevated-temperature nuclear construction. It is the reference construction material for gas-cooled reactors, liquid-metal reactors and molten-salt reactors. Hence PM HIP 316H is relevant for applications to microreactors based on these reactor concepts. For example, 316H is a construction material for the DOE Microreactor Applications Research Validation and EvaLuation (MARVEL) microreactor based on liquid-metal reactor design. Elevated-temperature cyclic properties are investigated because the creep-fatigue properties of PM HIP 316L were degraded compared to Wrought 316L (Rupp & Wright, 2020).

2. EXPERIMENTAL PROCEDURE

2.1 PM HIP Material

An Alloy 316H bar, fabricated by PM HIP, was procured from MTC Powder Solutions AB. This bar was fabricated specifically for this project. A list of targets for both the powder and bar were provided to the vendor. The vendor agreed to fabricate the bar on a best-effort basis. There is currently not an American Society for Testing and Materials (ASTM) standard for PM HIP 316H, but it would belong in ASTM A988, "Standard Specification for Hot Isostatically-Pressed Stainless Steel Flanges, Fittings, Valves, and Parts for High Temperature Service" (ASTM A988/988M-17, ASTM, 2017). The vendor stated that applicable ASTM A988 requirements were met when fabricating the PM HIP 316H bar.

The bar, Heat S0114, was 927-mm long with a 190-mm diameter. The following information was reported in the Material Compliance Dossier. The bar was solution annealed at 1050°C for 136 minutes, then water quenched. The microporosity requirements specified in ASTM A988 8.1 were met. The chemistry and room-temperature mechanical properties of the bar are shown in Table 1 and Table 2. The average grain size of the bar in an ASTM grain size number 7. Grain size was determined by the ASTM E112, "Standard Test Methods for Determining Average Grain Size," comparison procedure (ASTM E112-13, 2013).

Table 1. Chemical composition of Heat S0114 in weight percent.

C	**Si**	**Mn**	**P**	**S**	**Cr**	**Ni**	**Mo**	**Al**	**B**
0.040	0.17	0.21	0.002	0.003	16.44	11.95	2.48	0.007	0.0003
Co	**Cu**	**Nb**	**Ta**	**Ti**	**V**	**Fe**	**N**	**O**	**Ta+Co**
0.011	0.012	<0.005	0.006	0.005	<0.005	68.47	0.147	0.0202	0.017

Table 2. Mechanical properties of Heat S0114.

Tensile Strength	**Yield Strength**	**Elongation**	**Reduction in area**	**Hardness**
MPa	MPa	%	%	HV30

671	370	50	69	180, 183, 183

2.2 Microstructural Characterization

A combination of optical and scanning electron microscopy (SEM) were used to characterize the as-received and post-cyclic-testing microstructure of the PM HIP material. The following sections describe the experimental procedure for both microscopy techniques.

2.2.1 Optical Microscopy

A Keyence VHX-6000 microscope was used for optical microscopy. Specimens were prepared using standard grinding and polishing techniques and electro-etched with a 10% oxalic acid solution. The specimens were etched for 30 seconds with a voltage of 2.2 V.

2.2.2 Scanning Electron Microscopy

An FEI Quanta FEG 650 microscope was used for SEM characterization. Specimens were prepared using standard grinding and polishing techniques. The SEM was operated at 20 kV with a spot size of 5.5 or 6.

Electron backscattered diffraction (EBSD) data was collected with the Hikari detector and TEAM software from EDAX. EBSD data was collected with a 2-µm step size and 4 × 4 binning and processed using a free MATLAB toolbox, MTEX. A misorientation of five degrees or greater defined a grain boundary. Grains required a minimum of five pixels. Energy dispersive spectroscopy (EDS) data was also collected with the TEAM software.

2.3 Elevated-Temperature Cyclic Testing

Axial strain-controlled, elevated-temperature cyclic testing was conducted in accordance with PLN-3346, "Creep Fatigue Testing" (PLN-3346, Revision 9, 2017). The test procedures were derived from ASTM E606, "Standard Test Method for Strain-Controlled Fatigue Testing," and ASTM E2714, "Standard Test Method for Creep-Fatigue Testing" (ASTM E606/E606M-19, 2019; ASTM E2714-13, 2013). The following paragraphs detail the testing setup and procedures.

Testing was conducted using Materials Test Systems (MTS) servo-hydraulic test frames. Strain was measured with a direct-mounted extensometer from Epsilon Technology with a 12-mm gauge length and 5-mm fully-reversed total travel range. Fatigue testing used a symmetric, triangular strain waveform that was fully reversed with a 1% total strain range. Creep-fatigue testing used the same strain waveform as fatigue testing with an additional 30-minute tensile hold at the peak tensile strain. The strain rate for all tests was 10^{-3} s^{-1}.

Testing was conducted in air with a three-zone resistance furnace procured from MTS. Temperature was measured with two R-type thermocouples spot-welded outside each side of the gauge section on the specimen shoulders. The temperature for all tests was 650°C.

Specimens were machined so that the longitudinal direction was parallel to the length of the PM HIP bar. The specimens were cylindrical with a button-head end connection and 7.49-mm minimum diameter. Prior to testing, the diameter of the reduced section is measured with a 50× optical comparator. Challenges with machining the specimens resulted in each specimen having non-conformance issues with respect to the engineering drawing. The most critical was non-concentricity. Consequently, any cyclic data collected from these specimens are considered trend. After testing, no buckling or other obvious bending was evident in the specimens associated with these machining errors.

A cyclic test was terminated when the peak tensile force was a predetermined percent lower compared to an early test cycle. Ideally, the test should be stopped before the specimen fractures into two.

The number of cycles to crack initiation, N_o, and the number of cycles to failure, N_f, were identified using the procedures prescribed by Totemeier and Tian (Totemeier & Tian, 2007). In this work, N_f is defined as a 25% drop in the ratio of the peak tensile to peak compressive stress from N_o.

3. RESULTS

3.1 As-Received PM HIP Microstructure

The as-received PM HIP microstructure was characterized using SEM and EBSD. A back-scattered-electron (BSE) image and inverse-pole figure (IPF) map of the as-received microstructure are shown in Figure 1 and Figure 2, respectively. The following observations were made about the as-received PM HIP 316H bar.

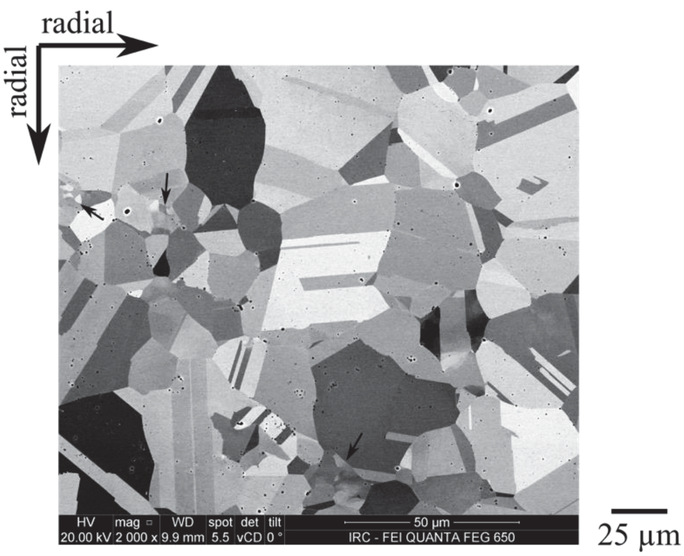

Figure 1. BSE image of the as-received PM HIP microstructure. The arrows highlight instances of substructure.

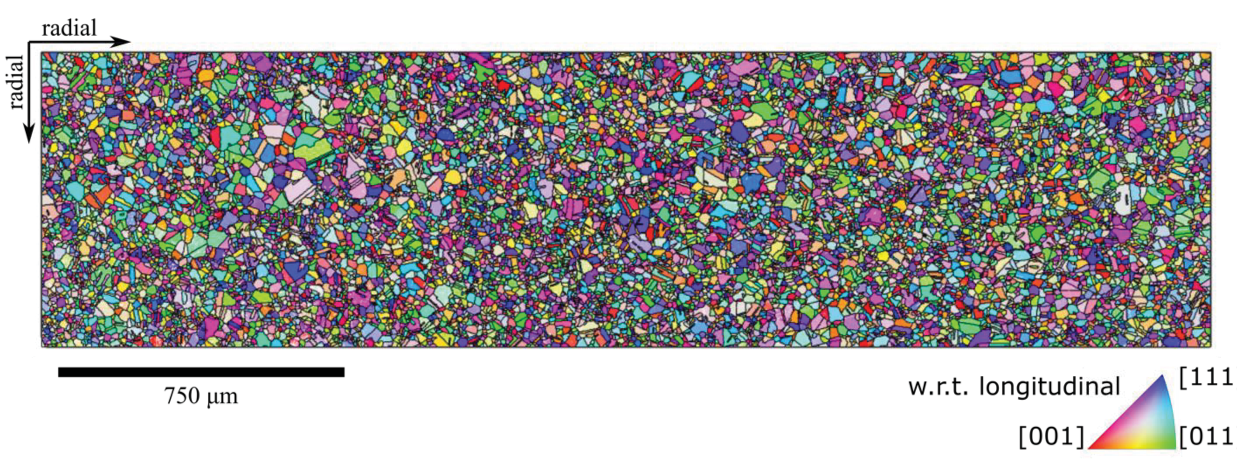

Figure 2. IPF map of EBSD data from the as-received PM HIP bar. The x- and y-direction of the map correspond to the radial direction of the bar. The map is with respect to (w.r.t) the longitudinal direction of the bar.

Porosity that is homogenously distributed throughout the microstructure at grain boundaries and within grains is visible in Figure 1. This porosity may be an artifact of particle pullout during sample preparation (Cooper, Cooper, Dhers, & Sherry, 2016). A technique, such as electropolishing, to investigate this was not conducted. In-house testing of the PM HIP bar verified conformance to the microporosity requirements specified in ASTM A988 8.1 (ASTM A988/988M-17, ASTM, 2017).

Substructure is present in the microstructure. Instances of substructure are highlighted with arrows in Figure 1. The cause for this substructure is unknown.

The crystallographic orientation of the grains, which is also known as texture, is random. This is shown by the IPF map in Figure 2 which is oriented with respect to the longitudinal direction. PM HIP is known for producing material with a random texture.

The microstructure contains a mixture of small and large grains. This is apparent in Figure 2. It is suspected that the grain size is bimodal. Analysis to confirm the grain size distribution was not performed.

3.2 Elevated-Temperature Cyclic Properties

The results from elevated-temperature cyclic testing are shown in Table 3 and Figure 3. Results from wrought product are also included for comparison. The following observations were made about the fatigue and creep-fatigue behavior.

The fatigue behavior of the PM HIP material is similar to wrought, with a comparable number of cycles to failure. The peak stress at the midlife cycle is greater for the PM HIP material than for wrought, even though they are similar at Cycle 10. The reason for the higher peak stress in the PM HIP material is unknown. It is suspected the difference results from dislocations being pinned in the PM HIP material. This may potentially be from oxides or the substructure observed in the as-received grain structure.

The creep-fatigue behavior of the PM HIP material is degraded compared to wrought. The number of cycles to failure for the PM HIP material is approximately 16% of the wrought product form. The peak stress in the PM HIP material is greater than wrought at both Cycle 10 and midlife. While cyclic softening is observed in both materials, the softening is more rapid in the PM HIP material. The reason for the difference in the peak stress and cyclic softening between the two materials is unknown. It should be noted that a total of four PM HIP creep-fatigue tests were conducted with very little variability observed between these tests. This demonstrates the reproducibility of the data. It also indicates that whatever mechanism is responsible for the observed properties is fairly homogeneous throughout the PM HIP bar.

Table 3. PM HIP 316H fatigue and creep-fatigue data. Cyclic testing was at 650°C, with a 1% fully-reversed strain range and a 10^{-3} s^{-1} strain rate. Creep-fatigue had a 30-minute tensile hold at the peak tensile strain. Wrought 316H data is included for comparison. Specimen IDs with HIP in the name are from the PM HIP 316H bar, while IDs with 76B in the name are from the wrought product.

				Cycle 10				Cycle Midlife			
Specimen	**Hold time**	N_o	N_f	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
316H-HIP-1	0	1,381	1,453	272	-281	-	-	310	-320	-	-
316H-HIP-5	0	845	872	266	-274	-	-	308	-315	-	-
76B6-1	0	1,450	1,635	263	-270	-	-	276	-281	-	-
76B6-2	0	1,060	1,165	267	-274	-	-	279	-285	-	-
316H-HIP-2	30	122	124*	250	-260	227	151	262	-278	261	152
316H-HIP-3	30	154	157*	248	-259	246	157	262	-279	258	153
316H-HIP-6	30	141	151*	244	-254	242	150	255	-272	254	149
316H-HIP-7	30	137	145*	246	-256	195	155	258	-272	257	150
76B-4	30	765	887	241	-245	239	153	232	-243	231	142
76B-5	30	774	945	229	-241	229	132	224	-244	224	128

* The last cycle of the test is reported because a 25% drop in stress was not captured. This was because either the specimen fractured in two or the test was terminated prematurely to decrease the likelihood of specimen fracture. The tests that were terminated prematurely were only a few cycles away from reaching the 25% stress drop.