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On the stress corrosion crack growth behaviour in high temperature water of 316L stainless steel made by laser powder bed fusion additive manufacturing

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ABSTRACT

This paper reports a comprehensive study of the stress corrosion crack growth behaviour of laser additively-manufactured (AM) 316L stainless steel in high temperature water. A wide range of parameters and their effects were evaluated, including microstructure, heat treatment, stress intensity factor, cold work, crack orientation, oxidizing vs. reducing conditions, and porosity. Stress-relieved material exhibits anisotropic microstructure and preferred crack path along material's build direction. With high-temperature annealing, the material re-crystallizes to equiaxed structure and behaves like wrought material. The retained unrecrystallized grains in an annealed AM part may not affect the cracking. Increased porosity may enhance the crack growth rate.

1. Introduction

In recent years, laser powder bed fusion (L-PBF) additive manufacturing (AM) has been widely explored in both academia and industry [1–5]. It uses a high-power laser to precisely melt and solidify alloy powder layer-by-layer and create a final geometry directly from its 3D computer model. This novel process offers higher degrees of design freedom and can significantly accelerate the deployment schedule of a new component from design to production. Given the enthusiasm of the aerospace industry for this technology, the nuclear industry is realizing its potentials to produce reactor internal components with improved performance, and reduced supply chain, cost and time to market [6]. The technology also provides a unique capability to rapidly design and fabricate parts and tools during the plant's refuel outage.

A wide range of materials have been studied and manufactured based on L-PBF, including austenitic stainless steel (SS) [7], precipitation hardened SS [8], Co-Cr alloy, Alloy 718 [9], and Titanium 6Al-4V alloy [10]. While extensive work has been conducted to additively fabricate austenitic SS parts, including complex components, most effort focused only on basic attributes such as porosity, residual stress, basic tensile properties, along with component yield and process monitoring [4,7,11–14]. Compared to tensile properties, the current understanding of the fracture and cracking behaviour of AM metals is limited [4,15–17], and little work has been done to define and evaluate the unique material requirements for nuclear applications.

Austenitic 316L SS is used extensively for components both inside and outside the reactor pressure vessel. While it is still challenging to achieve acceptable density and microstructure in other alloys by additive manufacturing, AM 316L SS has achieved significant success with near full density, reasonable tensile properties and fatigue crack growth behaviour, and good component yield [7,11,14,18,19]. However, for nuclear applications, it is well known that austenitic SS is susceptible to stress corrosion cracking (SCC) in high temperature water. SCC can be influenced by temperature, residual plastic strain in the alloy, irradiation damage, sensitization, water chemistry, and electrochemical potential [20–22]. L-PBF utilizes the high local heat density from the laser to fuse and solidify the metal powder. This process results in extremely fast heating and cooling rate and leads to high levels of residual stress and strain inside the material. In addition, higher porosity (compared to wrought material) is expected in AM parts due to incomplete fusion. Hot isostatic pressing (HIP) and heat treatment are normally performed following L-PBF to ensure good microstructure and low porosity. The microstructure of the AM products highly depends on the process parameters. The SCC susceptibility of AM 316L SS in high temperature water, one of the most critical material properties for nuclear service, had not been evaluated in either academia or industry.

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This paper summarized our extensive data and understanding of the SCC growth behaviour of AM 316L SS in high temperature water. Different heat treatments were performed to evaluate the effects of the unique microstructures (from unrecrystallized anisotropic grains to recrystallized equiaxed grains) on SCC behaviour. Additionally, the effects of stress intensity factor (K), oxidizing normal water chemistry (NWC) vs. reducing hydrogen water chemistry (HWC), cold work (CW), crack propagation orientation, retained unrecrystallized grains and porosity were also studied in detail. Giving the importance of SCC in nuclear applications, this study was designed to provide comprehensive SCC growth rate data for AM 316L SS in high temperature water environments, and discuss the cracking processes and mechanisms. The SCC response of AM 316L SS under different conditions also contributes to the development of nuclear specifications for AM materials. This study shows that, after proper high temperature annealing, AM 316L SS by L-PBF exhibits similar SCC growth behaviour as its wrought counterpart.

2. Experimental

2.1. Materials

Commercial 316L SS powder from Carpenter Powder Products (Bridgeville, PA, USA) was used to fabricate AM 316L SS samples for this research. The powder was sieved using 325 mesh screens. To ensure the reasonable flowability, powder size below 15 μm was limited to < 3 wt%. AM 316L SS samples were fabricated at Quad City Manufacturing Laboratory (Rock Island, IL, USA) using an EOS M270 metal additive manufacturing system. The process parameters of 195 W laser power, 1.2 m/s laser scan speed, and 20 μm powder thickness were selected to minimize porosity. Table 1 shows the chemical compositions of the as-received powder and the AM sample. The powder and the fabricated sample had a similar composition and met the ASTM A276 specification [23]. The AM samples had a slight increase in oxygen and carbon, and a decrease in nitrogen, which was probably due to reactions (e.g. oxidation, nitrogen removal) during laser melting.

Four heat treatment conditions (Table 2) were applied to AM 316L SS samples by Quad City Manufacturing Laboratory to assess the effects of microstructural variations: (1) as-built condition from L-PBF; (2) stress relief at 650 °C for 2 h in argon, (3) HIP in argon for 4 h at 1150 °C and 1000 bar, followed by solution annealing (SA) at 1066 °C for 1 h, and (4) heat treatment at 955 °C for 4 h in argon. Treatment 2 is called stress relief because it releases some of the residual stress from the as-built component using a low-temperature treatment. Most AM 316L SS parts by AM vendors are supplied using either Treatment 1 or Treatment 2 if a heat treatment condition is not specified by the buyer. Treatment 3 utilized HIP to densify the as-built part and recrystallize its microstructure. After the HIP process, a solution annealing at 1066 °C for 1 h followed by a water quench was conducted to remove carbides that might form during the slow cooling following HIP. Treatment 4 (955 °C for 4 h) was selected to produce a bimodal microstructure involving both recrystallized equiaxed grain (~30% recrystallized) and as-built characteristics, which was used in this study to understand the impact of the retained unrecrystallized grains on SCC. In an AM part, full recrystallization is not always achieved due to the strain variations in the part. Solution-annealed AM materials generally possess some unrecrystallized grains, as discussed later.

A higher porosity AM 316L SS from a different vendor was also evaluated to understand the effect of porosity on SCC. This material was recrystallized by solution-annealing, but HIP was not performed. Table 3 shows the difference in porosity between the stress-relieved condition (Treatment 1) and the HIP + SA condition (Treatment 2). The primary heat of AM 316L SS used in this study had reasonably low porosity with small defect size, so the HIP in Treatment 2 did not yield significant improvement. The high porosity heat from the other vendor showed higher porosity and larger pore size.

2.2. Stress corrosion crack growth tests

Compact tension (CT) specimens with 5% side grooves on each side were machined from the AM 316L SS blocks after all heat treatments. The specimen's dimensions followed ASTM E647 [24] with a thickness of 12.7 mm and a width of 25.4 mm. Fig. 1 shows specimen orientation relative to the powder bed of the EOS system. The directions were designated by the axes of the coordinate system shown in the figure. "X" and "Y" axes refer to the directions in the plane parallel to the powder bed, while "Z" axis refers to the material build direction. In a crack growth test, the notation "Z-X" means that the loading is in the "Z" direction and the crack advances in the "X" direction.

The validity and accuracy of the SCC growth rate measurement capabilities have been proven by a Round Robin organized by the Swedish Nuclear Power Inspectorate and Electric Power Research Institute in the 1990s [25] and adopted by many labs around the world [26–33]. As shown in Fig. 2, CT specimens were instrumented with platinum (Pt) current and potential leads for direct current potential drop (DCPD) measurements, which provides a resolution of ~1 μm in crack length or $\sim 10^{-9}$ mm/s in crack growth rate. The 2.5A DC current flowing through the specimen was supplied by an Agilent Technologies 6611C DC power supply, and reversed about once per second by a solid-state relay bridge to reduce the measurement errors associated with thermocouple effects. An Agilent Technologies multiplexer and digital voltmeter were used to measure the DCPD potentials, along with temperature, inlet and outlet conductivities, corrosion potentials, and other signals. DCPD active potential leads were positioned at the front face of the specimen to measure the voltage change during crack propagation. To compensate the voltage drift caused by the changes in metal resistivity, DCPD reference potential leads were attached on the back-face of the specimen to monitor the resistivity drift without any influence from the growing crack. Computer software controlled the current reversal, data acquisition, data averaging, and the crack length calculation, and maintained a constant K by automatically lowering the load as the crack advanced [20,21]. The specimen was electrically insulated from the loading rig by zirconia sleeves and washers. Extensive finite element modelling (FEM) and experimental validation using bench marking were used in the early development of DCPD algorithms at GE. FEM simulation determined the current distribution and the relationship of DCPD potential vs. crack length for both active and reference potential leads. The DCPD data throughout the test is divided by the initial DCPD reading. The actual crack length in the test is obtained based on the pre-determined relationship of DCPD ratio vs crack length by FEM simulation. This DCPD ratio vs. crack length relationship strongly depends on the locations of the probes, but not on the material type, temperature, or specimen size, as long as the specimen size and probe location remain in proportion. Extensive averaging of data was used to improve the measurement resolution. DCPD is very accurate if the crack is straight, e.g. during fatigue pre-cracking in air, but errors

Table 1
Measured chemical compositions of AM 316L powder and AM 316L part studied in this work by inductively coupled plasma optical emission spectrometry and instrumental gas analysis.

Material	Fe wt%	Cr wt%	Mn wt%	Si wt%	Ni wt%	Cu wt%	Mo wt%	V wt%	P wt%	Co wt%	W wt%	C ppm	S ppm	O ppm	N ppm
Powder	67.7	16.7	1.02	0.74	10.7	0.19	2.29	0.05	0.02	0.13	0.04	221	62	326	1163
AM Part	67.7	16.9	1.13	0.71	10.7	0.20	2.24	0.05	0.02	0.12	0.03	266	61	384	935

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