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Fatigue crack growth of additively and conventionally manufactured Hastelloy X in pressurized hydrogen gas

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ABSTRACT

This study examines the fatigue crack growth behavior of Hastelloy X, a common gas turbine burner alloy, in both wrought and powder bed fusion-laser beam (PBF-LB) conditions. Room temperature fatigue crack growth tests were performed in 4.6 MPa hydrogen gas, with emphasis on crack propagation in the Paris regime. Compact tension specimens were tested either with or without hydrogen pre-charging, performed at 343 °C in 34.5 MPa hydrogen gas for 35 days. When tested at room temperature in gaseous hydrogen, the wrought alloy exhibited an approximately three-fold higher crack growth rate. The PBF-LB material showed accelerated crack growth in hydrogen at stress intensity factor ranges below $\Delta K = 30 \text{ MPa } \sqrt{\text{m}}$, whereas at higher ΔK values, the crack growth rates in hydrogen converged with those measured in air. Interestingly, the fatigue crack growth rates in hydrogen were similar irrespective of hydrogen pre-charging despite the six-to seven-fold increase in bulk hydrogen content after pre-charging. Although the acceleration of fatigue crack growth in hydrogen was modest compared with that typically reported for high-strength steels, fractography revealed clear evidence of hydrogen-assisted damage in the wrought material, including textured cleavage facets. No comparable features were observed in the PBF-LB material. However, a larger number of secondary cracks were observed in regions corresponding to $\Delta K < 30 \text{ MPa } \sqrt{\text{m}}$. Overall, the results suggest that hydrogen embrittlement was more prominent in the wrought Hastelloy X than in the PBF-LB Hastelloy X under the investigated conditions.

1. Introduction

Additive manufacturing by means of powder bed fusion-laser beam (PBF-LB) builds components layer by layer via rapid melting and solidification of metal powder. This approach offers significant design freedom, enabling the production of complex geometries [1]. In gas turbines, this technology has been leveraged to fabricate monolithic burners with internal fuel-air mixing channels that are impractical to produce through conventional methods. As the energy sector shifts toward low-carbon-emission operation, gas turbines are increasingly adapted for hydrogen-rich fuels [2]. The enhanced combustion capability achieved via PBF-LB positions this technology as a key enabler for hydrogen integration.

Hastelloy X is widely used for gas turbine burners due to its excellent mechanical properties and oxidation resistance [3]. Hastelloy is a

trademark of Haynes International, Inc., Kokomo, IN. For service in hydrogen, however, its resistance to hydrogen embrittlement (HE), a phenomenon that reduces ductility and fracture resistance in many alloys, must be well understood [4–9]. Hastelloy X is widely recognized as a benchmark alloy for hydrogen-rich environments and is considered a promising candidate material for hydrogen burner applications, outperforming alternatives such as Inconel 625 [10–12]. Wrought Hastelloy X has demonstrated HE resistance under low-cycle fatigue regime at temperature between 538 and 871 °C. Furthermore, low-cycle fatigue tests at room temperature using 2% total strain range have shown no measurable reduction in fatigue life when tested in hydrogen [13,14]. However, increased HE susceptibility has been observed when fine, continuous carbide precipitates have formed along the grain boundaries during ageing. In such conditions, hydrogen traps associated with the carbides is expected to promote embrittlement. In contrast, the un-aged

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material tested at room temperature retains its characteristic HE resistance [15].

Unlike the extensively studied wrought Hastelloy X, the HE of PBF-LB Hastelloy X is poorly understood. Compared with the hot-rolled, solution-annealed (wrought) alloy, the PBF-LB microstructure exhibits columnar grains parallel to the build direction, and a higher density of crystallographic defects due to strains arising from in-situ thermal cycling during processing [16]. This microstructure results in anisotropic mechanical properties along the build direction versus horizontal directions [17]. Slow-strain rate tensile (SSRT) tests on both PBF-LB and wrought Hastelloy X, using hollow-specimens, showed no reduction in tensile ductility in hydrogen at pressure of 200 bar, either at room temperature or at 800 °C [18]. Although these results suggest promising resistance to HE, the performance of cyclically loaded engineering components cannot be reliably assessed based solely on monotonic material properties. Instead, a fracture mechanics framework is required to evaluate material performance under environmentally assisted crack growth condition. This need is further supported by the material assessment conducted by the National Aeronautics and Space Administration (NASA), which classified Hastelloy X for cautious use in certain applications and recommended detailed fracture mechanics and crack-growth analyses [19]. Such considerations are particularly important because hydrogen-embrittlement susceptibility can depend strongly on the applied loading mode [4,20,21].

In service, this becomes critical for gas turbine burners, whose life-time is primarily governed by crack initiation and growth driven by thermomechanical fatigue (TMF) [22]. Hydrogen has higher solubility in austenite at elevated temperatures (~700 °C), whereas embrittlement effects tend to be most severe near room temperature [11,23,24]. This combination creates a plausible service scenario. Hydrogen uptake occurs to a considerable extent at high temperature. Upon cooling, dissolved hydrogen is partially retained, which may influence the mechanical response at lower temperatures. Notably, tensile residual stresses arising from prior compressive loading at high temperature can promote crack advance during low temperature part of cycles governed by out-of-phase thermomechanical fatigue (OP-TMF). Given this combination of hydrogen availability and stress state at low temperature, fatigue crack growth testing performed in hydrogen at room temperature (which is close to practical minimum temperature of OP-TMF loading in hydrogen) can serve as a useful indicator of HE susceptibility.

Also, to date, no relevant data have been reported regarding the influence of hydrogen on the fatigue crack propagation rates of PBF-LB Hastelloy X in the Paris regime. Considering this regime is sensitive to environmental effects, it is therefore of critical importance to assess the performance in hydrogen. In order to improve understanding of the aforementioned issue, the present study focusses on fatigue crack growth behaviour of PBF-LB and wrought Hastelloy X in 4.6 MPa hydrogen gas and in air. A detailed fractography is performed and is correlated to hydrogen embrittlement mechanisms. Additionally, the relevance of using a hydrogen pre-charging procedure for Hastelloy X is investigated and discussed.

2. Experimental methods

2.1. Materials and manufacture

Commercial gas atomized Hastelloy X powder with modified chemical composition as shown in Table 1 was used as the feedstock material for the powder bed fusion laser-beam (PBF-LB) process, performed on an EOS M400 machine. Parameters were optimized to achieve 99.9%

relative density in the printed blocks, which were subsequently removed from the build-plate using a bandsaw. A commercially available wrought Hastelloy X which was hot-rolled and solution heat-treated at (1149 – 1177 °C) $\pm$ 14 °C followed by cooling in air was used for comparison. The chemical composition of the wrought Hastelloy X is listed in Table 2.

2.2. Hydrogen charging and measurement

The compact tension (CT) specimens from as-printed and wrought Hastelloy X, are given in Fig. 1a. These samples were polished along the sides of the notch over the entire length of the specimen, with a final polishing step using 1 μ m diamond suspension. Thereafter they were pre-cracked and then cleaned in isopropanol using an ultrasonic cleaner. Afterwards, a part of the specimens underwent gaseous hydrogen pre-charging in an autoclave at 34.5 MPa and 343 °C for 35 days using grade 5.0 hydrogen gas. The specimens were then cooled to room temperature in the same hydrogen atmosphere before being depressurized, removed from the autoclave, and stored in a cryogenic freezer. Hydrogen concentration was quantified in weight parts per million (wppm) using a LECO hydrogen analyser. Measurements were performed on coupons extracted via EDM after the FCGR tests from two locations: the crack extension zone of the fracture surface marked as ‘CE’ in Fig. 1b, and from the back edge of the CT specimens marked in the same figure. Additionally, 5 measurements were performed for both PBF-LB and wrought conditions on bulk material samples without hydrogen exposure to identify baseline hydrogen content from respective processing routes.

2.3. Test method for fatigue crack growth rates

The fatigue crack growth rate (FCGR) tests under constant force amplitude loading (ΔP) were performed on CT specimens in accordance with ASTM E647-2024 [25]. The specimens had an effective width, W , of 50.8 mm, a thickness, B , of 6.35 mm, and a manufactured crack starter of length $a_n = 12.7$ mm, as shown in Fig. 1. For the PBF-LB specimens, the x-direction, corresponding to the notch and crack-growth direction, was oriented parallel to the build direction (BD). For the wrought Hastelloy X specimens, the x-direction was oriented parallel to the rolling direction (RD). In both material conditions, fatigue crack propagation therefore occurred along the x-direction within the x-z fracture plane, with the y-direction corresponding to the loading direction and normal to the fracture plane. Prior to fatigue crack growth testing, each CT specimen was instrumented with direct current potential drop (DCPD) probes for in-situ crack length measurement. The specimens were then fatigue pre-cracked from the EDM notch to a crack extension of 1.27 mm at a stress ratio of $R = 0.1$ and a frequency of 20 Hz under laboratory ambient conditions (22.2 ± 1.1 °C). During pre-cracking, the maximum stress intensity factor was controlled to a nominal K_{max} of 20.9 MPa $\sqrt{m}$. Pre-cracking was terminated once the prescribed crack extension of 1.27 mm from the EDM notch was reached.

The crack growth rate, da/dN , was subsequently measured as a function of increasing stress intensity factor range, ΔK . Tests in hydrogen were conducted on specimens with and without hydrogen pre-charging, under ambient laboratory conditions (nominally at 23 °C and 50% relative humidity) in an autoclave flushed with 4.6 MPa hydrogen gas (grade 5.0). The test matrix is summarized in Table 3, with two tests performed per condition. Constant force amplitude loading with a load

Table 1
Chemical composition of modified virgin Hastelloy X powder.

Element	C	Si	Al + Ti + Hf + Nb + Ta + Zr	Cr	Fe	Mo	Co	W	B	Ni
Wt. %	0.02	0.11	0.096	21.47	18.0	8.9	1.48	0.75	0.002	Bal.

Table 2
Chemical composition of wrought Hastelloy X.

Element	C	Si	Mn	Al	Cr	Fe	Mo	Co	W	B	Cu	Ti	Ni
Wt. %	0.09	0.42	0.34	0.15	21.38	18.42	8.75	0.81	0.43	0.004	0.1	<0.01	Bal.

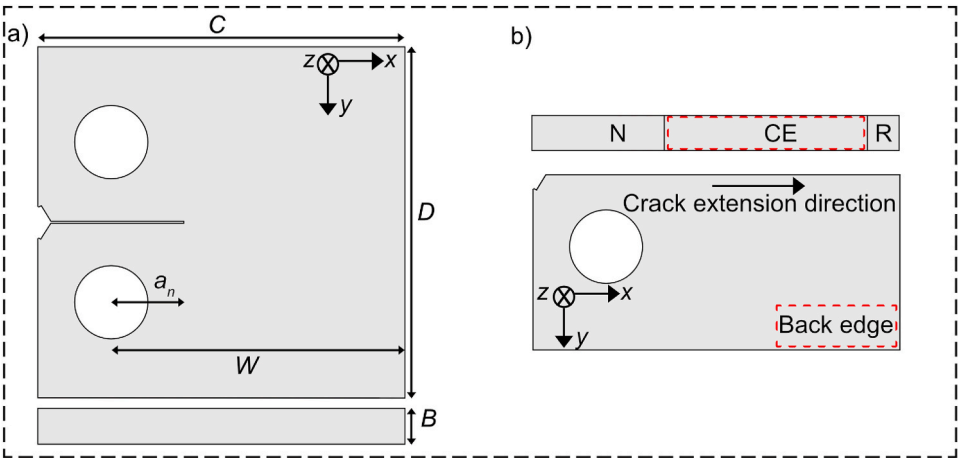


Fig. 1. Compact tension specimen used for fatigue crack propagation testing: (a) specimen geometry with dimensions ($C = 63.5$ mm, $D = 60.95$ mm, $B = 6.35$ mm, $W = 50.80$ mm, and $a_n = 12.7$ mm); (b) schematic top and side (half) views of the CT specimen showing the locations of the post-test hydrogen-concentration measurement, indicated by dashed red boxes. N denotes the notch and pre-crack region, CE the crack-extension zone, and R the residual fracture region. The side view also indicates the back-edge region. The x-direction corresponds to the crack-growth direction and is parallel to the build direction (BD) and rolling direction (RD) for the PBF-LB and wrought material respectively. The y-direction corresponds to the loading direction and is normal to the fracture plane, while the z-direction corresponds to the specimen through-thickness direction of the hot-rolled plate and PBF-LB material. Accordingly, crack propagation occurs along x within the x-z fracture plane.

Table 3
Fatigue crack growth rates test matrix.

Manufacturing route	Specimen ID	H ₂ pressure, (MPa)	Temperature (°C)	Stress ratio, R	Frequency, f (Hz)	Number of tests	Notes
Wrought	WX-1	0.0	23	0.1	10	2	Air test
	WX-2	4.6	23	0.1	0.1	2	Pre-charged
	WX-3	4.6	23	0.1	0.1	2	No pre-charge
PBF-LB	PBF-LB-1	0.0	23	0.1	10	2	Air test
	PBF-LB-2	4.6	23	0.1	0.1	2	Pre-charged
	PBF-LB-3	4.6	23	0.1	0.1	2	No pre-charge

ratio of $R = 0.1$ and a sinusoidal waveform was used to characterizing Region II (Paris regime) of the fatigue crack growth curve. To capture the effect of hydrogen on fatigue crack propagation, a test frequency of 0.1 Hz was used for the hydrogen tests, whereas reference tests in air were conducted at 10 Hz.

2.4. Microstructural characterization

To characterize the initial microstructure, coupons were extracted from the test specimens away from fracture process zone. The samples were then hot mounted and ground using silicon carbide paper with grit sizes P800, P1000, P1200, P2000, P4000. Subsequent polishing was carried out using 3 μm and 1 μm diamond suspensions, followed by 0.05 μm colloidal silica to minimize surface deformation. All fracture surfaces were cleaned via ultrasonication in ethanol for 15 min prior to microscopy. Electrolytic etching was performed using 10% oxalic acid at 5 V for 20 s.

A Zeiss Gemini 450 field emission gun scanning electron microscope (FEG-SEM) was used for imaging and microanalysis of coupons. The imaging was performed using secondary electrons utilizing an accelerating voltage of 5 kV and probe current of 500 pA. Chemical analysis was conducted using energy dispersive X-ray spectroscopy (EDS) (Oxford Analysis), employing an accelerating voltage of 5 kV and probe current

of 1.5 nA. Electron backscatter diffraction (EBSD) (Oxford, Symmetry) was employed for crystallographic texture analysis. Acquisition utilized an accelerating voltage of 20 kV, a probe current of 12 nA, and a step size of 0.2 μm . The EBSD data was subsequently processed using the open-source MTEX toolbox in MATLAB® [26]. Electron channelling contrast imaging (ECCI) was performed on electropolished sample surfaces (Struers A3) using 20 kV accelerating voltage, probe current of 5 nA, and working distance of 6 mm.

3. Results

3.1. Initial microstructure

3.1.1. Powder bed fusion-laser beam manufactured Hastelloy X

The as-printed Hastelloy X has an austenitic microstructure, without detectable carbide precipitation. A weak $\langle 110 \rangle$ crystallographic orientation parallel to the build direction (BD) as shown in Fig. 2a, was observed. The average grain size was $159 \pm 45 \mu\text{m}$, with a grain aspect ratio of 3.5, indicating elongated grains along the build direction. The presence of low-angle grain boundaries with 2.5° misorientation is shown in Fig. 2b. The microstructure also contained cellular dendritic solidification structures and melt pool boundaries, characteristic of the PBF-LB process, as shown in Fig. 2c. Repeated thermal cycling during

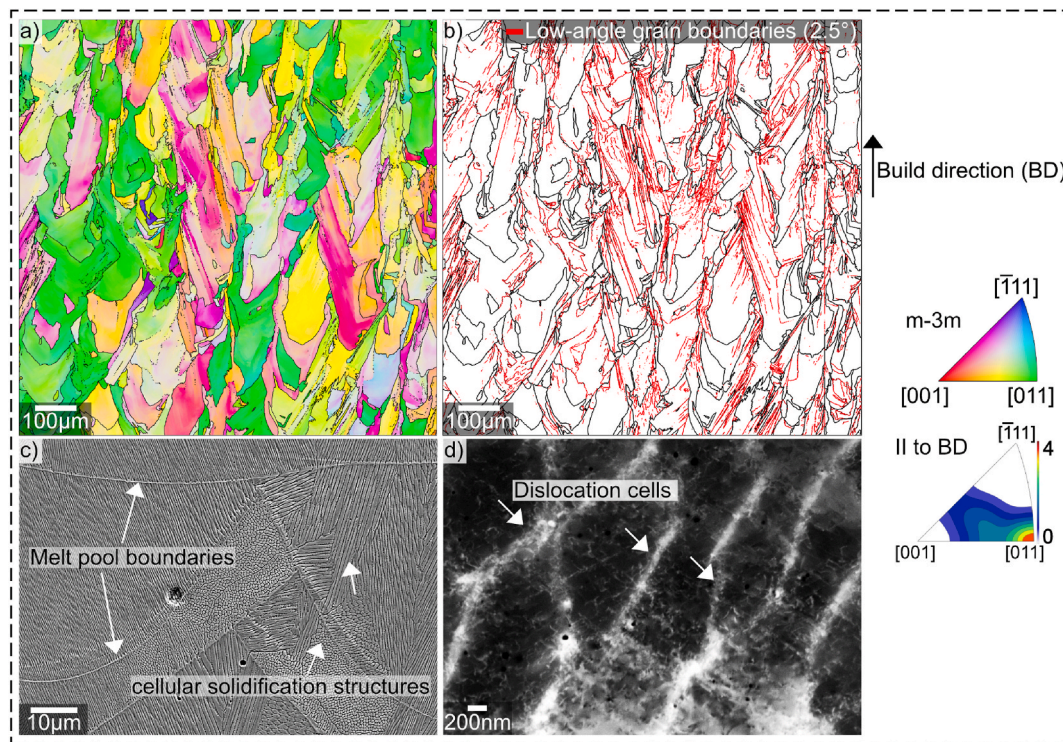


Fig. 2. (a) EBSD orientation maps along the build direction showing weak $\langle 110 \rangle$ texture; (b) EBSD grain boundary map parallel to build direction showing high-angle grain boundaries overlaid with 2.5° low-angle grain boundaries; (c) Micrograph from etched PBF-LB Hastelloy X; (d) ECCI micrograph showing presence of dislocation cells in as-printed state.

layer-by-layer processing introduced strains and promoted dislocation accumulation, resulting in decoration of the cellular solidification structures by dislocations, as shown in Fig. 2d.

3.1.2. Hot-rolled and solution annealed Hastelloy X

The microstructure of the hot-rolled and solution heat-treated (wrought) Hastelloy X consisted of an austenite matrix with carbide precipitates, which were observed as streaks aligned along the rolling direction (see Fig. A1 in Appendix). As shown in Fig. 3a, the carbides were located both inside the grains and along the grain boundaries, where they appeared both as discrete particles and as continuous carbide film.

The carbides were characterized using EDS and are found to be enriched in chromium, molybdenum, carbon as shown in Fig. 3a1. Based on their chemical composition, the observed carbides are likely to consist of a mixture of molybdenum rich M_6C and chromium rich $M_{23}C_6$ phases, in agreement with chemical and thermodynamic predictions reported in Ref. [27]. Fig. 3b shows the EDS line-scan profile acquired across the grain boundary marked as 'L1' in Fig. 3a. The line scan shows local enrichment of molybdenum and chromium at the grain boundary, further supporting the presence of M_6C and $M_{23}C_6$ carbide precipitates. In addition, the solution heat treatment promoted the formation of annealing twin boundaries, $\Sigma 3$, within the austenite grains, as shown in Fig. 3c. After excluding $\Sigma 3$ twin boundaries from the grain reconstruction, the average grain size of the wrought Hastelloy X was measured to be $107 \pm 40 \mu m$, with a grain aspect ratio of 1.6.

3.2. Fatigue crack propagation

The normalized crack propagation rate, da/dN , is plotted as a function of the stress intensity factor range, ΔK , for PBF-LB and wrought Hastelloy X tested in air and hydrogen, as shown in Fig. 4. Here, $\Delta K = K_{max} - K_{min}$, where K_{min} and K_{max} are the minimum and maximum stress intensity factors, respectively, with unit of $MPa \sqrt{m}$. The crack

propagation rates measured in air for PBF-LB-1 and WX-1 are shown in Fig. 4a. Here, the fatigue crack growth rate (FCGR) is found to be almost linear on the double logarithmic graph and nearly coincident with each other, following the Paris law [28]. The crack propagation data for WX-2 and WX-3 test specimens measured in 4.6 MPa hydrogen gas for wrought Hastelloy X is shown Fig. 4b. The two curves overlap. The FCGR of WX-2 and WX-3 are approximately three times higher than that of WX-1. Clearly, hydrogen uptake accelerates crack growth. However, pre-charged hydrogen has limited influence on FCGR when tested in H_2 . The hydrogen-induced acceleration remains nearly constant across the investigated ΔK range, with no additional amplification at higher ΔK . This behaviour contrasts with the more commonly reported trend, where hydrogen-induced acceleration is modest at low ΔK and becomes more pronounced at higher ΔK [29,30]. For additively manufactured Hastelloy X, as shown in Fig. 4c, when $\Delta K < 30 MPa \sqrt{m}$, hydrogen-induced acceleration is noticeable (PBF-LB-2 and PBF-LB-3) in comparison to tests in air (i.e., PBF-LB-1). With further increase in ΔK , i.e., $\Delta K > 30 MPa \sqrt{m}$, crack propagation rate coincides with FCGR in air. It is interesting to note here that irrespective of manufacturing route, no significant difference in crack propagation rate is observed between specimens with and without pre-charged hydrogen when specimens are tested in H_2 . A comparison between PBF-LB and wrought Hastelloy X in H_2 environment is made in Fig. 4d. It can be observed that the initial acceleration observed in H_2 for the PBF-LB-2 and PBF-LB-3 specimens are very similar to the WX-2 and WX-3 specimens, while higher FCGR is observed for wrought Hastelloy X at higher ΔK .

3.3. Hydrogen uptake

Hydrogen quantification was performed by using a LECO hydrogen analyser on material extracted from the crack extension zone and back edge, as described in section 2.2. The results are presented in Table 4. A baseline hydrogen concentration was 3.14 ± 1.6 wppm and 0.65 ± 0.13 wppm for the wrought and, PBF-LB alloy, respectively. These values

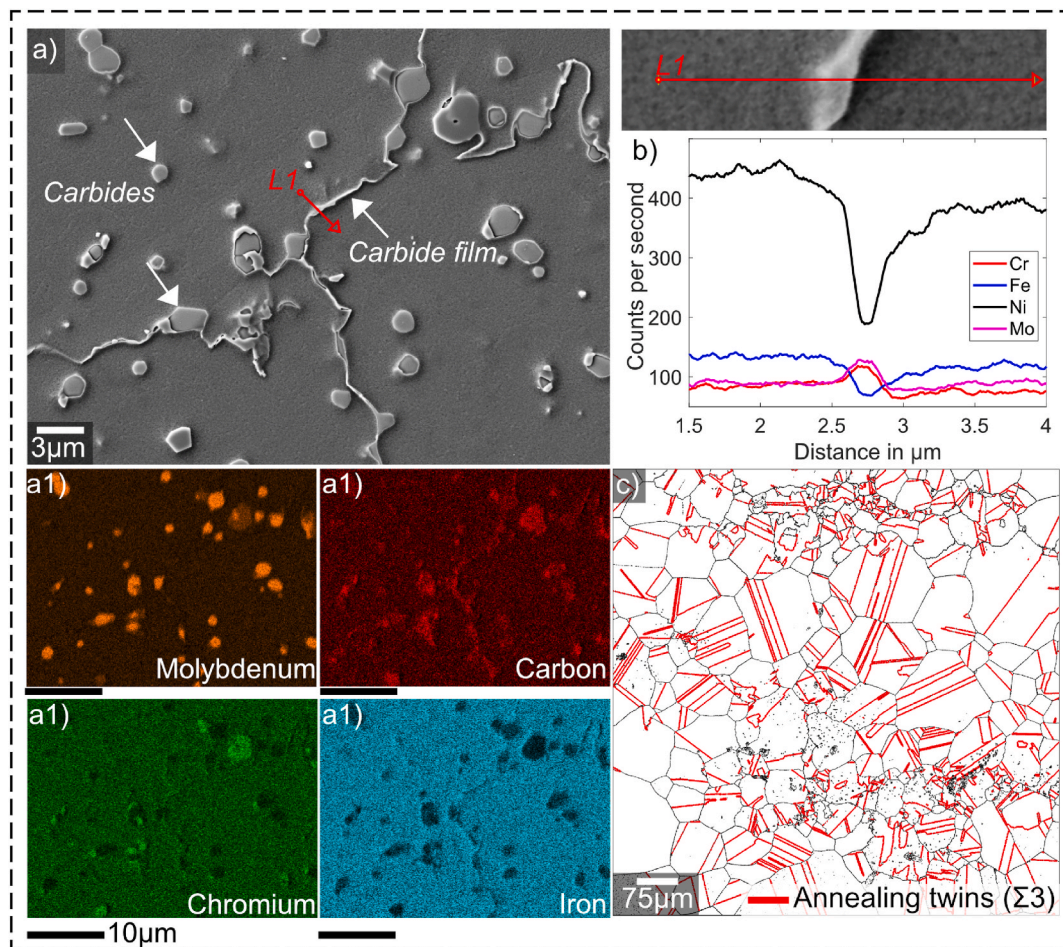


Fig. 3. (a) & (a1) SEM and EDS micrograph showing the presence of M_6C rich in molybdenum and $M_{23}C_6$ carbides rich in chromium; (b) EDS line scan of grain boundary carbide film; (c) Presence of annealing twin boundaries ($\Sigma 3$) in addition to high-angle grain boundaries (15°) misorientation.

provide a reference for the subsequent hydrogen uptake comparison. After hydrogen pre-charging, the hydrogen content increased to approximately 38 wppm and 40 wppm in the WX-2 and PBF-LB-2 specimens, respectively. Owing to the limited number of measurements (to preserve fracture surfaces for fractographic examination) and the inherent scatter in hydrogen measurements, this difference between the differently manufactured specimens should be regarded as negligible and within experimental scatter. The crack extension zone was expected to contain a higher hydrogen concentration because deformation may enhance hydrogen uptake. This region was therefore compared with the back edge, which experienced no deformation. However, the results presented in Table 4 indicate that hydrogen uptake in the crack extension zone is very similar to that at the undeformed back edge. Only a small difference in hydrogen uptake (2–3 wppm) was observed between the crack extension zone and back edge of PBF-LB-3 and WX-3 specimens. In addition, the influence of the manufacturing route on hydrogen uptake appeared to be limited.

For specimens without pre-charged hydrogen, the in-situ FCGR tests resulted in hydrogen uptake, with the hydrogen content in the crack extension zone increasing by approximately 4 wppm for PBF-LB-3 and 3 wppm for WX-3. As expected, specimens subjected to hydrogen pre-charging exhibited significantly higher hydrogen levels, reaching approximately 40 wppm in PBF-LB-2 and 38 wppm in WX-2. Despite the significantly higher hydrogen concentrations crack propagation rates were very similar to those of PBF-LB-3 and WX-3. This point will be discussed further in Section 4.

3.4. Fractography

Fatigue crack growth in wrought Hastelloy X was accelerated by approximately a factor of three in hydrogen relative to air (Fig. 4b). For PBF-LB specimens, an acceleration is evident at $\Delta K < 30 \text{ MPa } \sqrt{m}$ in hydrogen, whereas at higher ΔK the rates converge with those measured in air (Fig. 4c). To examine whether fractography reflects hydrogen exposure, fracture surfaces of all specimens were inspected. Owing to the similar fracture surface features observed for samples tested in hydrogen with and without hydrogen pre-charging (Fig. A2 in Appendix), representative fracture surfaces from WX-1, WX-2, PBF-LB-1, and PBF-LB-2 are presented in sections 3.4.1 and 3.4.2.

3.4.1. Wrought Hastelloy X

Fracture surface morphologies for wrought Hastelloy X are examined for three ΔK regimes sampled along the crack-extension region (schematic in Fig. 5): zone 1 ($\Delta K \sim 20 - 35 \text{ MPa } \sqrt{m}$), zone 2 ($\Delta K \sim 35 - 50 \text{ MPa } \sqrt{m}$), and zone 3 ($\Delta K \sim 55 \text{ MPa } \sqrt{m}$). When tested in air (WX-1), zone 1 (Fig. 5a) shows an overall ductile, transgranular appearance. The inset in Fig. 5a reveals finely spaced fatigue striations that are present both in the matrix and within annealing twins, while carbide cracking is rare. Zone 2 (Fig. 5b) exhibits a similar morphology, with clear striation markings in both matrix and twins. In zone 3 (Fig. 5c), carbide cracking becomes more frequent, and the striation spacing increases as crack propagation rates are higher (Fig. 5a). The fracture surface also becomes more undulating (“wavy”), consistent with increasingly unstable crack propagation.

When tested in hydrogen (WX-2), zone 1 (Fig. 5d) contains faceted

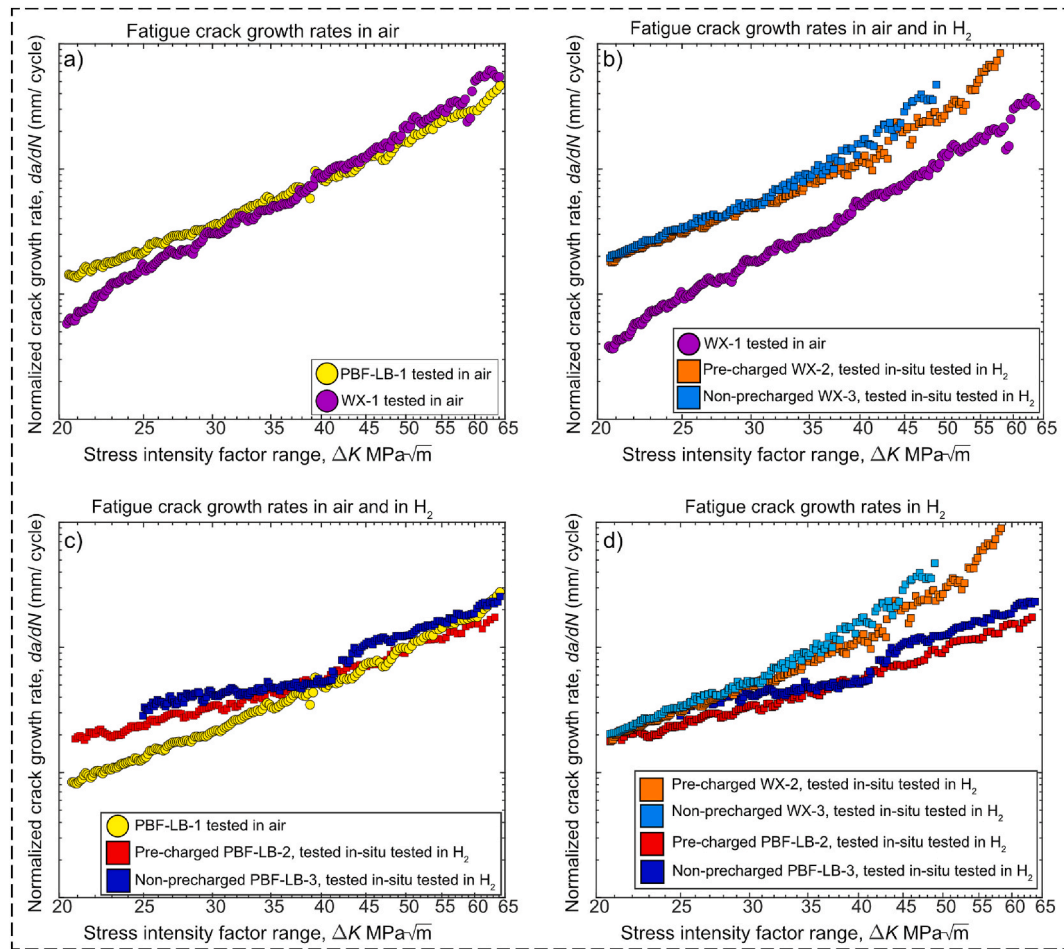


Fig. 4. Fatigue crack propagation rates: (a) in air for PBF-LB and wrought Hastelloy X; (b) in air and hydrogen for wrought Hastelloy X; (c) in air and hydrogen for PBF-LB Hastelloy X; (d) in hydrogen for PBF-LB and wrought Hastelloy X.

Table 4
Measured hydrogen concentration in wppm

Specimen id	Hydrogen quantification - location and content (wppm)	
	Crack extension zone	Back edge
WX-1 air tested	-	3.14 ± 1.6
WX-2 pre-charged	40	38
WX-3 no pre-charge	6	6
PBF-LB-1 air tested	-	0.65 ± 0.13
PBF-LB-2 pre charged	40	40
PBF-LB-3 no pre charge	5	2

grains with a textured surface appearance, as seen in the insert. No visible striations, slip traces, or surface steps was observed in these facets. The flat appearance with no fatigue striations strongly suggests cleavage fracture. These regions coexist with neighbouring areas that retain the ductile, striated morphology similar to that observed in air. In zone 2 (Fig. 5e), a large flat facet with size in the same order with grains is observed, accompanied by ‘zig-zag’ secondary cracks. The surrounding regions remain predominantly ductile with striations. The magnified view in the insert highlights the textured faceted region and again shows no evidence of slip markings. Zone 3 shown in Fig. 5f is similar to zone 2, comprising a mixture of ductile striated regions and cleavage-like facets. Notably, cleavage facets are also observed in grains containing annealing twins, where the twin regions fracture without striations, slip markings or surface steps (in contrast to the striated twins in air, Fig. 5a and b). This suggests separation across the twin structure as observed in Ref. [31], exposing the twins on the cleavage plane. Similarly, carbide

cracking is observed at longer crack lengths, with a frequency comparable to that in air. No discernible effect of hydrogen on carbide cracking is observed.

3.4.2. PBF-LB Hastelloy X

Fracture surfaces of PBF-LB-1 (tested in air) and PBF-LB-2 (tested in H_2) are shown in Fig. 6. A similar approach as for wrought alloy is used, with fracture morphologies reported for three ΔK regimes along the crack extension zone (see the schematic in Fig. 6).

For PBF-LB-1 tested in air, zone 1 (Fig. 6a) shows predominantly transgranular fracture with striations. Zone 2 (Fig. 6b) contains secondary cracks, and in zone 3 (Fig. 6c) the striation spacing increases with ΔK . For PBF-LB-2 tested in hydrogen, zone 1 (Fig. 6d) shows striations together with secondary cracks. Zones 2 and 3 (Fig. 6b and c) display fracture features qualitatively similar to those in air, namely transgranular regions with striation markings and secondary cracking. Overall, no systematic differences between air and hydrogen are observed for the PBF-LB specimens in zones 2 and 3. However, in zone 1, PBF-LB-2 exhibits a qualitatively higher number of secondary cracks than PBF-LB-1. Whether this is associated with the observed acceleration in fatigue crack growth rate in hydrogen at $\Delta K < 30 \text{ MPa}\sqrt{\text{m}}$ (Fig. 4c) is not known. It is also important to note that, no cleavage facets were observed in the PBF-LB manufactured Hastelloy X in contrast to the wrought material.

4. Discussion

The hydrogen embrittlement behaviour of wrought Hastelloy X has

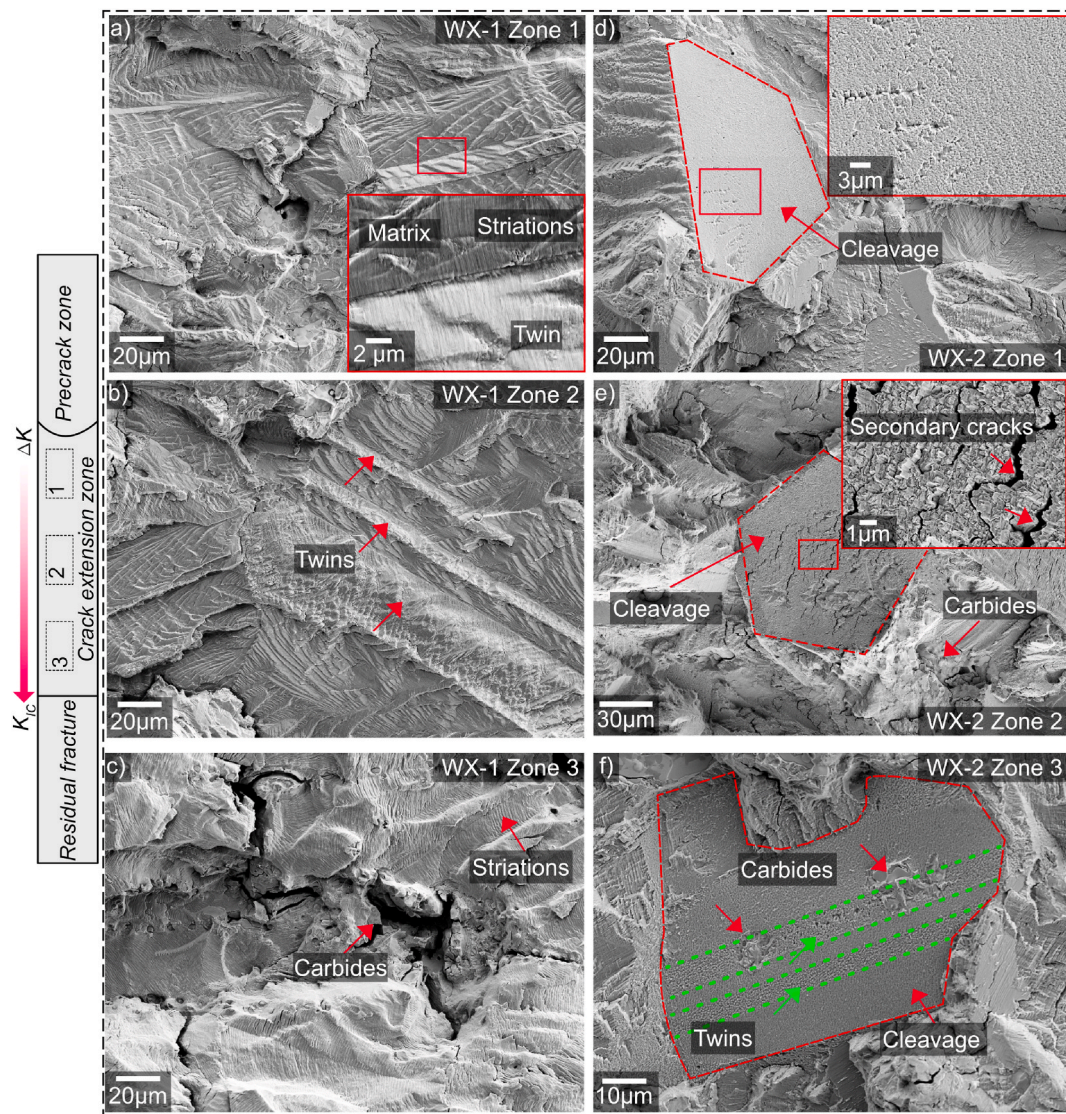


Fig. 5. (a)–(c) Fracture surfaces of WX-1 specimen tested in air. (d)–(f) fracture surfaces of hydrogen pre-charged WX-2 specimen tested in 4.6 MPa hydrogen. The schematic on the left is provided only to show the correspondence between the labelled zones and the images. It does not represent the crack-propagation direction, which is right to left.

been widely reported [10,13,23]. The observation of cleavage on the fracture surface of the wrought alloy (Fig. 5d and f) suggests that the acceleration of crack propagation (Fig. 4b) is associated with hydrogen-assisted damage. Despite growing interest in PBF-LB, the response to hydrogen uptake of Hastelloy X processed by this technique remains insufficiently explored [18]. For six-to seven-fold increase in total hydrogen concentration, as exhibited in Table 4, the hydrogen pre-charging procedure prior to testing did not lead to any measurable increase in hydrogen embrittlement susceptibility, as shown in Fig. 4b and c. These findings and their implications in both material conditions are discussed in this section.

4.1. Crack growth behaviour in air

Wrought and PBF-LB Hastelloy X possesses varied microstructure including phase constituents and grain size and shape. However, the impact of microstructure on FCGR in air has been found to be less influencing in the presently investigated stress intensity range, i.e., the Paris regime, as demonstrated by the comparable crack propagation rate of wrought and PBF-LB Hastelloy X in air (see Figs. 2, 3 and 4a). This is because at large stress intensity ranges (i.e., Paris regime), the crack

length is larger than the local microstructural features, resulting in similar crack propagation rates. Consistent with this, Shaji et al. [32] showed neither printing induced anisotropy, residual stresses nor solution heat treatment influence fatigue crack growth in the Paris regime for PBF-LB Hastelloy X. This may explain the similar FCGR in air, since the influence of local microstructural features diminishes as the crack length increases and crack growth becomes governed mainly by the applied stress intensity range.

4.2. HE mechanisms and fracture behaviour

Hydrogen exposure altered the fracture behaviour of wrought Hastelloy X, promoting cleavage fracture features that are consistent with the observed acceleration in FCGR. Although the magnitude of acceleration is modest compared with the severe hydrogen-assisted crack growth reported for highly susceptible alloys such as high strength steels [10,33], the combined FCGR response in Fig. 4d and the fractographic evidence in Fig. 5 provide evidence of hydrogen embrittlement. However, interpretation of the observed FCGR acceleration requires consideration of the different loading frequencies used for the air and hydrogen tests. The air tests were conducted at 10 Hz, whereas the

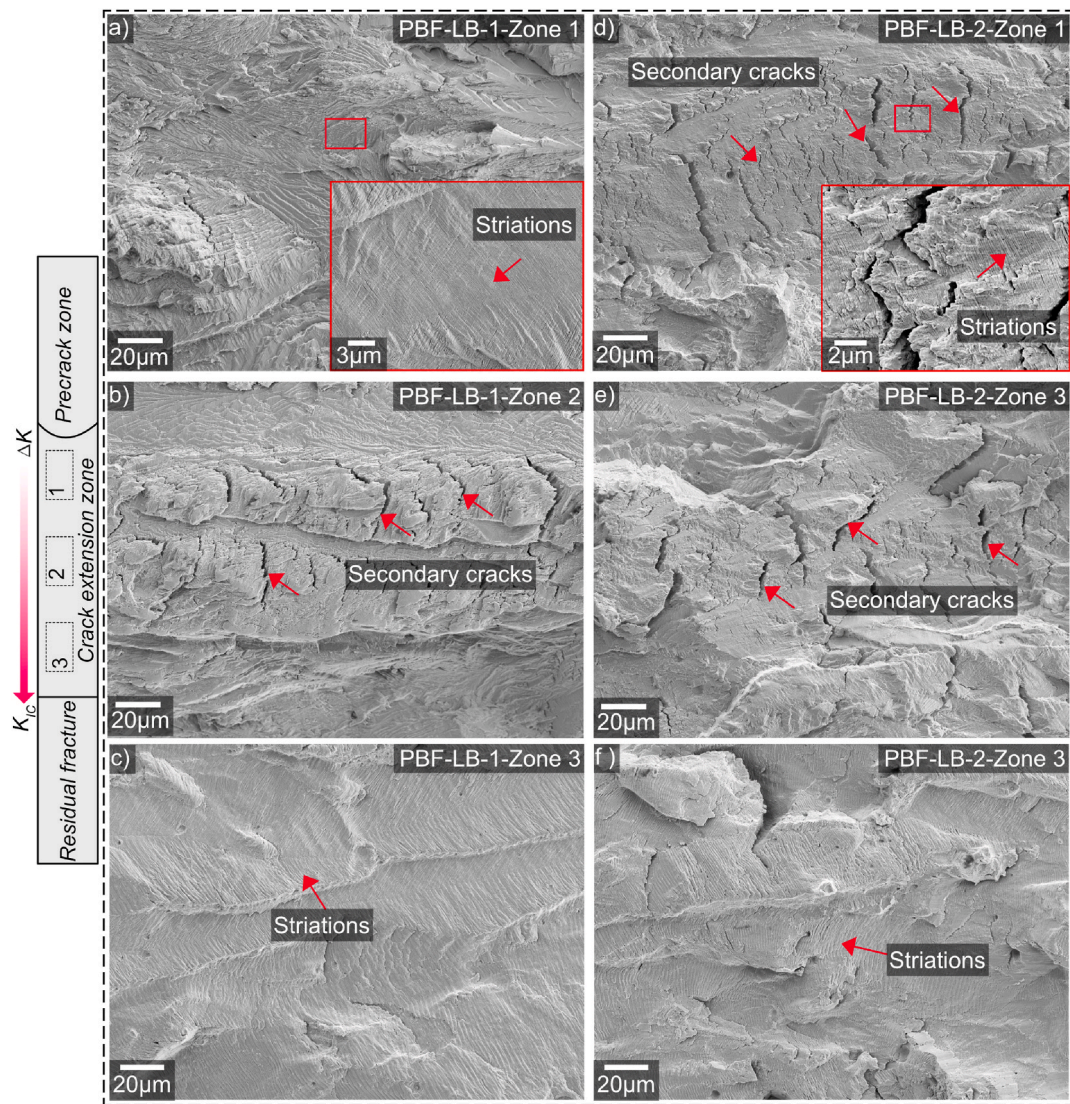


Fig. 6. (a)–(c) Fracture surfaces of PBF-LB-1 specimen tested in air. (d)–(f) fracture surfaces of hydrogen pre-charged PBF-LB-2 specimen tested in 4.6 MPa hydrogen. The schematic on the left is provided only to show the correspondence between the labelled zones and the images. It does not represent the crack-propagation direction, which is right to left.

hydrogen tests were performed at 0.1 Hz. Consequently, the effects of loading frequency and hydrogen environment were not independently isolated, and any potential contribution from the frequency difference to the observed FCGR acceleration cannot be completely excluded. Previous studies have reported that Hastelloy X exhibits only limited strain-rate sensitivity during room-temperature tensile testing [34]. Significant time-dependent deformation or oxidation-assisted effects are not expected at room temperature. The influence of the frequency difference on the fatigue crack growth rates is expected to be limited.

Hydrogen embrittlement in austenitic alloys is commonly interpreted in terms of two interacting mechanisms: hydrogen-enhanced localized plasticity (HELP) and hydrogen-enhanced decohesion (HEDE) [6,35,36]. Localised deformation along favourable slip planes can generate strain incompatibilities at microstructural obstacles, such as grain boundaries, phase boundaries, and twin boundaries, leading to stress concentrations that increase with applied strain. Hydrogen can further enhance this strain localization by facilitating dislocation motion, thereby accelerating stress concentration at these interfaces. Concurrently, hydrogen accumulation at the interfaces can reduce their cohesive strength, increasing the likelihood of interfacial separation. From a fractographic perspective, slip traces or surface steps on cleavage

facet are often considered indirect evidence of HELP mechanism, whereas interfacial decohesion, such as intergranular cracking or phase boundary separation, is more commonly associated with HEDE mechanism [37]. A coupled HELP-mediated HEDE sequence is more frequently reported, in which hydrogen-promoted slip localization elevates stress concentration and local hydrogen accumulation, together with pre-existing hydrogen along these interfaces, subsequently facilitates decohesion [6,36,38,39].

In the present study, textured cleavage facets were observed in wrought specimens tested in hydrogen, without clear evidence of slip traces or surface steps. These facets occurred both in grains containing annealing twins and in grains without annealing twins. These fracture features are comparable to those previously reported for wrought Hastelloy X tested under tensile loading in hydrogen and at high temperatures in air [5,34], where multiple grains exhibited similar facets with no evidence of slip or surface steps. However, based on the reported fracture surface observations, it was not possible to determine whether the faceted grains contained annealing twins.

The absence of visible slip markings does not exclude the involvement of HELP mechanism, since HELP describes a hydrogen-assisted deformation process rather than a distinct fracture morphology [40].

Hydrogen may reduce the stacking fault energy in FCC alloys, increase the separation between partial dislocations, suppress cross-slip, and thereby promote planar slip localization on crystallographic planes ahead of the crack tip [41,42]. Such localized deformation may contribute to crack advance without necessarily leaving pronounced slip traces or surface steps on the final fracture surface. The textured appearance of cleavage facets, in contrast to classical flat cleavage facets, suggests the separation may have involved a limited degree of plastic deformation. Furthermore, the localization of cleavage to specific grains may reflect crystallographic-orientation-dependent hydrogen activity. For example, in alloy 718, hydrogen has been reported to promote strongly localised slip on close-packed {111} planes [31,38]. However, the small number and scattered distribution of the cleavage facets across the fracture surface limited reliable identification of the operative crystallographic plane using techniques such as EBSD, and the absence of pronounced slip traces or surface steps in the present study limits the direct fractographic evidence for HELP.

In addition, hydrogen accumulation along the susceptible crystallographic planes may reduce interplanar cohesion through HEDE mechanism [43]. The textured appearance of the cleavage facets suggests that fracture occurred by rapid separation, potentially following localized deformation. In this interpretation, hydrogen first promotes localized deformation through HELP, while the resulting stress concentration, together with reduced cohesive strength, facilitates rapid separation through HEDE. Even though the observed fracture features are correlated to HELP-HEDE mechanisms, other operative HE mechanisms may also exist and should not be disregarded. The twins are revealed on the fracture surface, but there is no clear evidence of preferential cracking along twin boundaries (refer to Fig. 5f). The texture within the twins resembles early-stage dimple formation resulting from the monotonic separation. In addition, the lack of boundary-linked damage along the twin boundaries indicates these were not the dominant fracture path.

Comparable facet morphologies have been reported in other Nickel-base alloys. Zhang et al. [31] observed flat facets in grains associated with annealing twin boundaries in alloy 718. The boundaries remained pristine with no signs of void formation, even though severe slip band intersection commonly linked to HELP mechanism was observed. The crack propagated along slip bands due to intense slip localization. Additionally, the texture observed within the reported twins differed from that observed in the twins in the present study. However, unlike the present study of Hastelloy X, slip markings were observed on facets which was associated with slip localization along the plane. For austenitic steels, Nibur et al. [40] reported flat facets exhibiting surface steps, attributed to hydrogen-enhanced slip activity affecting annealing twin boundaries. In another study on alloy 718, Ogawa et al. [37] observed faceted fracture characterized by separation along {111} dislocation slip bands or annealing twin boundaries, leading to altered fracture morphology from ductile striations to facets.

Secondary cracks with a ‘zig-zag’ morphology were observed on some facets, as shown in Fig. 5e. In contrast, the secondary cracks in the surrounding regions appeared to be associated with striation separation or carbide cracking. The observed morphology resembles the ‘zig-zag’ micro-fracture reported in hydrogen charged alloy 718 [38], where cracking was associated with alternating intersecting {111} slip planes. In that study, crack growth on {100} planes near <110> directions were also reported, producing relatively straight cracks with less pronounced deflection. Such behaviour was not observed in the present study. Therefore, the ‘zig-zag’ cracks in the present study may reflect crack propagation between intersecting {111} planes. However, without crystallographic confirmation (e.g., slip traces and crystal orientation), this interpretation remains tentative.

Interestingly, a higher total hydrogen content did not produce an obvious change in the prevalence of decohesion features. Cleavage facets were also present in the specimens without pre-charged hydrogen tested in hydrogen gas (refer to Table 4 and Fig. A2). This suggests that, under the present loading mode, the supply of hydrogen from the newly

formed surface during crack propagation could be more important than supply from the bulk, promoting coupled HELP-HEDE mechanism ahead of the crack tip. Grain-boundary carbides have been reported to reduce hydrogen embrittlement resistance in stable austenitic stainless steels [44]. In the present study, carbides were observed along some facets. However, facets were also found in grains without evidence of carbide related separation (cf. Fig. 5c and f). In addition, no intergranular cracking was observed, even though continuous carbide film was observed along the grain boundaries, refer to Fig. 3a. Together, these observations suggest that carbides were not the primary factor controlling the FCG rates in hydrogen for the WX-2 and WX-3 specimens. Finally, the cleavage of certain grains may be associated with crystallographic orientation-specific hydrogen activity, but this hypothesis requires further analysis for confirmation.

4.3. Impact of manufacturing route on HE

The PBF-LB and wrought material exhibit fundamentally different microstructures, as shown in Figs. 2 and 3. These differences originates from their distinct processing routes, namely layer-wise rapid solidification during PBF-LB and hot rolling followed by solution annealing in the wrought condition. Since hydrogen embrittlement is strongly sensitive to microstructure [7,45–48], different hydrogen-assisted fatigue crack growth response is expected. In agreement with this, the present results show an approximately three-fold higher FCGR in wrought Hastelloy X. In addition, compared with the test in air, the PBF-LB specimens exhibited an acceleration in FCGR in H₂ at lower stress intensity factor ranges, with a magnitude comparable to that observed in the wrought alloy. However, cleavage facets were observed only in wrought Hastelloy X and not in the PBF-LB specimens.

The difference in FCGR and fracture morphology is likely controlled by several concurrent factors. One possible contributing factor is the cellular dislocation structure in the PBF-LB material, which contributes to its higher yield strength and thereby reduces the estimated cyclic plastic zone size. Under plane-stress conditions, which are considered relevant for the specimen dimensions used in the present study, and assuming small scale yielding, the cyclic plastic zone size can be estimated using Irwin approximation according to equation (1) [49]:

$$2r_p = \frac{1}{4\pi} \left(\frac{\Delta K}{S_y} \right)^2 \quad (1)$$

where, $2r_p$ = plane-stress cyclic plastic zone size, ΔK = stress intensity factor range, S_y = yield strength. The monotonic yield strength of PBF-LB and wrought Hastelloy X materials, together with the estimated cyclic plastic zone size, are listed in Table 5.

The PBF-LB material develops a smaller estimated crack-tip cyclic plastic zone, approximately 0.35 mm at $\Delta K = 40 \text{ MPa } \sqrt{\text{m}}$, which is roughly about one-fourth of that estimated for wrought Hastelloy X (Table 5). This suggests that plastic deformation in front of crack-tip is more confined in the PBF-LB material. Such confinement may limit the region over which HE mechanisms can develop. In contrast, the larger estimated cyclic plastic zone in the wrought material may provide a larger crack-tip process region for the operation of HELP-HEDE mechanisms.

The acceleration in FCGR in H₂ for the PBF-LB material at $\Delta K < 30 \text{ MPa } \sqrt{\text{m}}$ may be related to the interaction between the cyclic plastic zone size and the columnar grain structure. At lower ΔK , the cyclic plastic

Table 5

Monotonic yield strength and estimated cyclic plastic zone size of Hastelloy X used in the present study.

Material	Yield strength [MPa]	$2r_p$ at $\Delta K = 40 \text{ MPa } \sqrt{\text{m}}$
Wrought HX	306	~1.36 mm
PBF-LB HX	600	~0.35 mm

zone is smaller and may be comparable to the axial grain length of the PBF-LB material, approximately 0.3 mm. Under these conditions, crack-tip deformation and hydrogen-assisted deformation process may be more strongly influenced by individual grains. This may also contribute to the secondary cracking observed at shorter crack lengths, as shown in Fig. 6d. As ΔK increases, the cyclic plastic zone becomes larger than the axial grain length, making the crack-tip less sensitive to individual grain-scale features.

In addition, low-angle grain boundaries from local misorientations between cellular regions in the PBF-LB material may disrupt the continuity of crystallographic fracture planes, making cleavage fracture along a single plane less favourable. By contrast, the recrystallized grains in the wrought material are expected to contain fewer intragranular orientation gradients, which may allow HELP-HEDE mechanisms to operate along favourable crystallographic planes. Additionally, the wrought material initially contains a lower dislocation density than the PBF-LB material. The cyclic hardening during fatigue may promote dislocation multiplication ahead of the crack tip. Together with the larger cyclic plastic zone, this may enhance HELP-HEDE mechanisms in the wrought material.

These effects are partly linked to the higher yield strength of the PBF-LB material, which is associated with its cellular dislocation structure. However, this interpretation requires caution. Cyclic loading of austenitic materials can promote dislocation rearrangement into more stable cell structures [41], meaning that fatigue-induced dislocation cells may develop in the wrought material during crack growth. Therefore, the difference in FCGR and fracture behaviour between PBF-LB and wrought Hastelloy X in H_2 cannot be attributed solely to the presence of dislocation cells. Instead, the contribution of the cellular structure in PBF-LB Hastelloy X is likely related to its as-built characteristics, including cell size, cell-wall chemistry, local orientation gradients, and possible hydrogen-trapping behaviour [7].

A limitation of the cyclic plastic zone estimation in the present study is that it is based on monotonic yield strength and does not account for possible cyclic hardening or cyclic softening during fatigue crack growth. Cyclic hardening in the wrought material [50], would reduce the effective plastic zone size and therefore decrease the apparent difference between the wrought and PBF-LB conditions. Additionally, the potential influence of differences in processing history, particularly heat treatment, as well as compositional differences between the PBF-LB and wrought Hastelloy X, should be considered when interpreting their HE behaviour. Accordingly, the observed differences should not be attributed solely to the manufacturing route.

Overall, the higher FCGR and the occurrence of cleavage facets in wrought Hastelloy X are likely associated with the combined effects of a larger estimated cyclic plastic zone, greater volume of material for the operation of HELP-HEDE mechanisms, and a microstructure more favourable for locally continuous crystallographic fracture. In contrast, the absence of cleavage facets in PBF-LB Hastelloy X may be related to the specific characteristics of as-built cellular dislocation structure which results in a higher yield strength causing a smaller estimated cyclic plastic zone and local orientation gradients. Dedicated future work is required to clarify why PBF-LB Hastelloy X does not exhibit hydrogen-assisted cleavage fracture in the same manner as its wrought counterpart.

5. Conclusion

Hydrogen embrittlement susceptibility in Hastelloy X produced by powder bed fusion-laser beam (PBF-LB), and in the hot-rolled and solution-annealed condition, hereafter referred to as wrought Hastelloy X, was investigated by examining fatigue crack growth rates in air and in 4.6 MPa hydrogen gas. In addition, the effect of prior long-term hydrogen charging at 343 °C and 34.5 MPa for 35 days on fatigue crack propagation behaviour was also assessed. The main conclusions are as follows:

1. When tested in air, PBF-LB and wrought Hastelloy X exhibited comparable fatigue crack growth rates, with both materials following a Paris-law behaviour over the investigated ΔK range.
2. Testing in 4.6 MPa hydrogen gas resulted in accelerated fatigue crack growth. The wrought alloy showed an approximately three-fold increase in crack growth rate relative to air, whereas the PBF-LB alloy exhibited an increase primarily at lower ΔK values.
3. Fractography of the wrought alloy tested in hydrogen revealed localized cleavage facets, providing clear evidence of hydrogen-assisted damage. No comparable hydrogen-assisted cleavage features were observed in the PBF-LB material.
4. The combined fatigue crack growth and fractographic results indicate that hydrogen embrittlement was more pronounced in the wrought Hastelloy X than in the PBF-LB material under the investigated conditions.
5. Long-term hydrogen charging prior to testing did not produce a measurable change in fatigue crack growth behaviour or in the observed fracture characteristics. This indicates that, under fatigue crack propagation conditions in Hastelloy X, the supply of hydrogen from the surrounding atmosphere through newly formed surface could be more critical than the pre-charged hydrogen.

CRediT authorship contribution statement

Vishnu Anilkumar: Conceptualization, Investigation, Visualization, Writing-original draft, Writing-review and editing. **Santosh Narasimhachary:** Investigation, Writing-review and editing. **Fassett Hickey:** Investigation, Writing-review and editing. **Lars Nyborg:** Writing-review and editing. **Johan Ahlström:** Writing-review and editing, Supervision. **Frans Palmert:** Conceptualization, Investigation, Writing-review and editing, Supervision. **Yu Cao:** Conceptualization, Investigation, Writing-review and editing, Supervision, Funding acquisition.

Data availability

The data supporting the findings of this study cannot be shared due to restrictions on data disclosure.

Declaration of generative AI and AI-assisted technologies in the writing process

This work utilized ChatGPT, a large language model developed by OpenAI, to assist with refining the language and grammar of certain sections, generating alternative phrasing, and improving the structure and organization of the text. The authors retain full responsibility for the accuracy and originality of the research and its findings.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Yu Cao reports financial support was provided by Swedish Energy Agency. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix

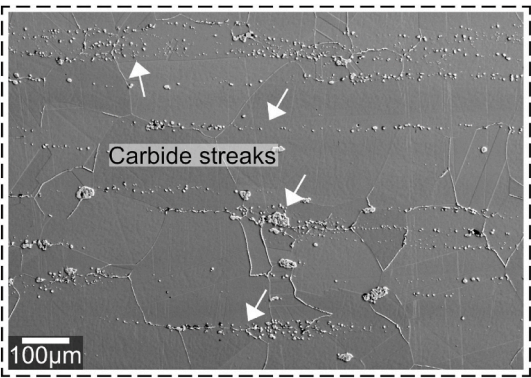


Fig. A1. Etched micrograph of wrought Hastelloy X showing presence of carbide streaks aligned along the rolling direction.

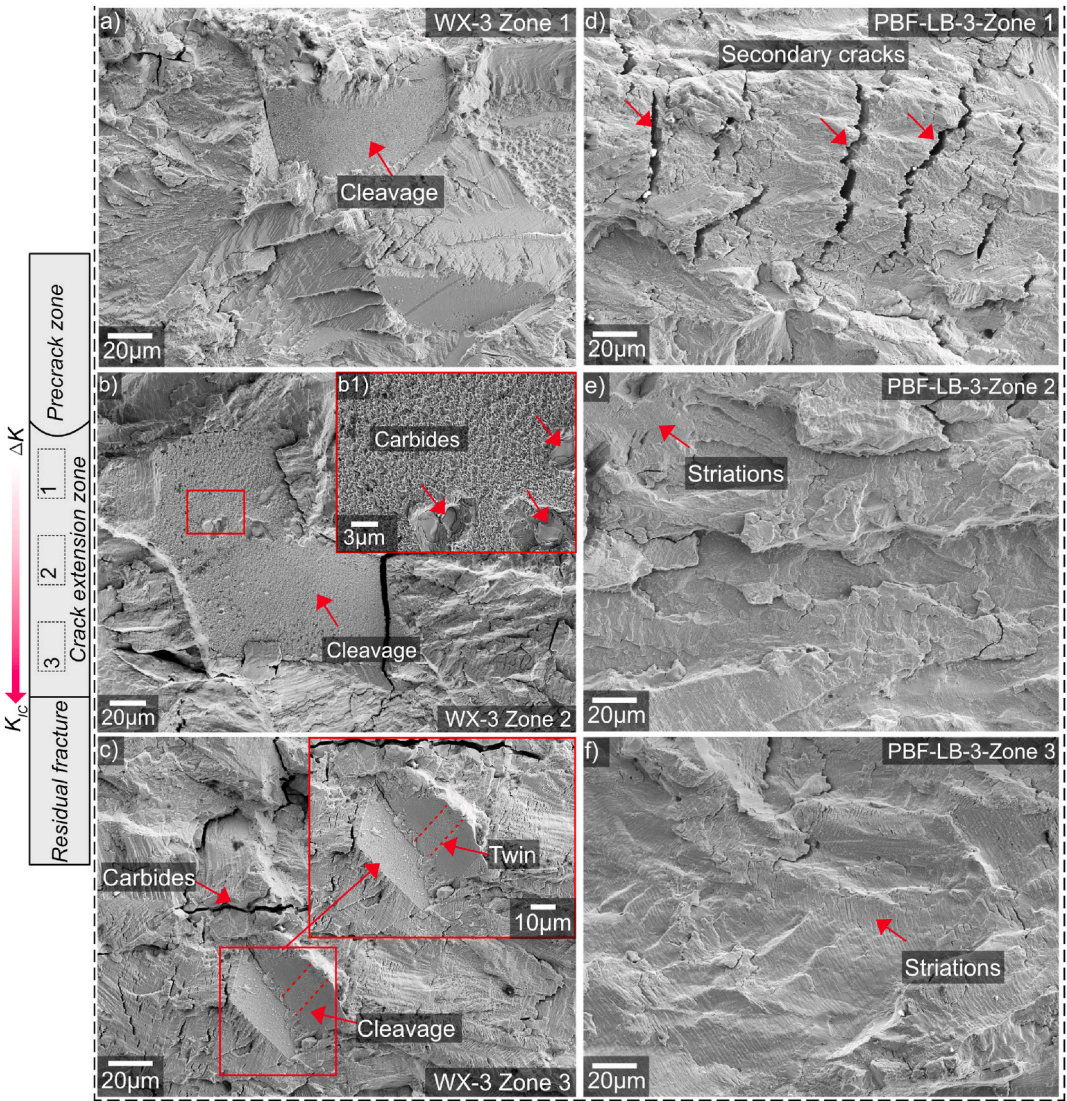


Fig. A2. Fracture surfaces from (a-c) WX-3 specimen and (d-f) PBF-LB-3 specimen. The schematic is provided only to show the correspondence between the labelled zones and the images; it does not represent the crack-propagation direction, which is right to left.

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