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Hydrogen Embrittlement of AISI 316L steel produced by Selective Laser Melting

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Abstract

Selective laser melting (SLM) is one of the common methods of additive manufacturing and it can be employed to customize components with complex geometry expected to work in hydrogen atmospheres. However, more studies are still needed to characterize the behavior of printed mechanical components intended to work in contact with hydrogen. In this study, smooth and notched tensile samples were precharged in an acid solution with 8 mA/cm^2 for 24h time. Hydrogen damage was more marked in the notched samples, especially at 0.005 mm/min, where fracture micromechanism changed from ductile in the absence of hydrogen to quasi-brittle in the presence of internal hydrogen. The role of the strain-induced martensite is also highlighted.

Keywords: 316L, SLM, tensile, hydrogen embrittlement, strain-induced martensite

1. Introduction

Nowadays, there is a continued interest in developing alloys able to work in hydrogen applications at high pressure hydrogen gas. In this regard, austenitic stainless steels such as 316 or 316L are good candidates because of their low hydrogen diffusivity and high ductility [1–3]. In the context of hydrogen technologies, the production of connector

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and elbows requires specific geometries that can be optimized through additive manufacturing. It has been reported that additively manufactured 316L boasts superior mechanical properties than those of conventional state [1]. However, greater absorption of hydrogen can be induced in the SLMed 316L [2] because of its higher density of dislocations, induced due to the rapid cooling rates during solidification. In addition, the influence of SLM defects and surface finishing in hydrogen diffusion must be better understood [2,3]. Accordingly, new production techniques, especially additive manufacturing, require re-evaluation of hydrogen susceptibility of austenitic stainless steels. In this work, smooth and notched tensile samples have been electrochemically precharged. Tensile behavior, with internal hydrogen, was studied at different strain rates and hydrogen embrittlement is discussed in terms of the operative fracture micromechanisms.

2. Experimental procedure

2.1 Material and processing

AISI 316L was additively manufactured with an ALBA 300 machine, following parameters given in Table 1. The chemical composition of the printed material is shown in Table 2. Hot extraction samples to determine hydrogen concentration and tensile samples were printed simultaneously on the same platform of the ALBA 300 machine.

Table 1. Printing parameters in ALBA 300 machine

Laser power (W)	Scanning speed (mm/s)	Hatching distance (mm)
225	650	0.1

Table 2. Chemical composition in weight percent (wt. %)

Fe	C	Cr	Ni	Mo
balance	0.02	18	12	2

2.2 Hydrogen precharging and hot extraction

Hydrogen was introduced in samples with a geometry of 10 mm x 10 mm x 1 mm. A current density of 8 mA/cm^2 was applied for 24 hours at room temperature (RT) in 1M H_2SO_4 solution, added with 0.25 g/l As_2O_3 (pH≈1). Hydrogen absorption was analyzed by means of the hot extraction technique. Samples were heated at 1100ºC for 300 s in a LECO DH603 hydrogen analyser.

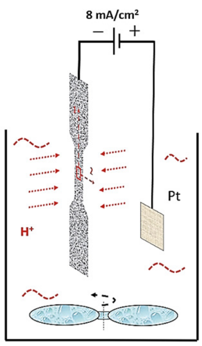


Figure 1. Hydrogen precharging in tensile samples. Experimental set up

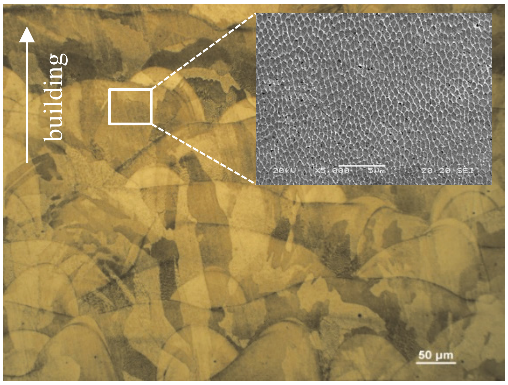
2.3 Tensile tests and fracture surfaces.

Tensile tests were carried out in a MTS Series 40 machine. In order to study the influence of hydrogen at different strain rates, tensile tests were done from 0.1 to 0.005 mm/min. Smooth and notched samples were used with the tensile direction parallel to the printing direction of the additively manufactured 316L samples. The width and thickness of tensile samples were 5 and 1 mm, respectively. The gauge length of samples was 20 mm. In the case of the notched samples, the geometry of the notch can be seen in Figure 5. Smooth and notched samples were electrochemically precharged, following conditions previously described. The experimental set up is illustrated in Figure 1. After hydrogen precharging, tensile samples were tested in air at RT. Fracture surfaces were examined in a scanning electron microscope JEOL JSM-6460LV, using an acceleration voltage of 10 kV.

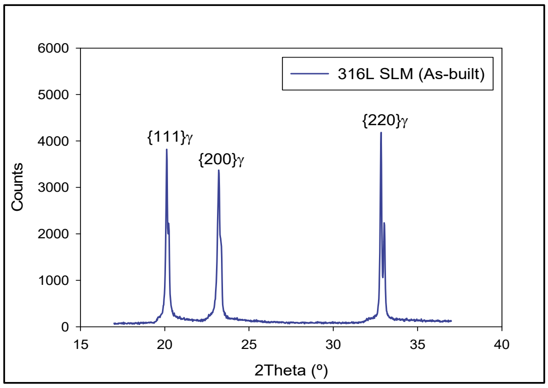
3. Results

3.1 Microstructure and hydrogen uptake

Additively manufactured 316L microstructure is showed in Figure 2(a). Microstructure shows a layered structure stacked with arc shaped melt pools. Columnar austenite grains, forming epitaxially along the building direction, can be observed. The cellular structure is also observed in the grains because of the rapid cooling [3]. After the 3D printing process, the XRD analysis revealed there are only diffraction peaks corresponding to the austenite phase, Figure 2(b). On the other hand, from hot extraction tests, hydrogen concentration was determined to be ≈37 wt ppm. However, due to the low hydrogen diffusivity of austenitic steels, additively manufactured samples are not saturated after 24h, and this concentration is only reached at the subsurface level.



(a)



(b)

Figure 2. (a) Microstructure of AISI 316L (etched with Kalling's No. 2 for 125s) after 3D printing. (b) XRD analysis, after 3D printing, conducted with a Seifert XRD 3000 TT diffractometer

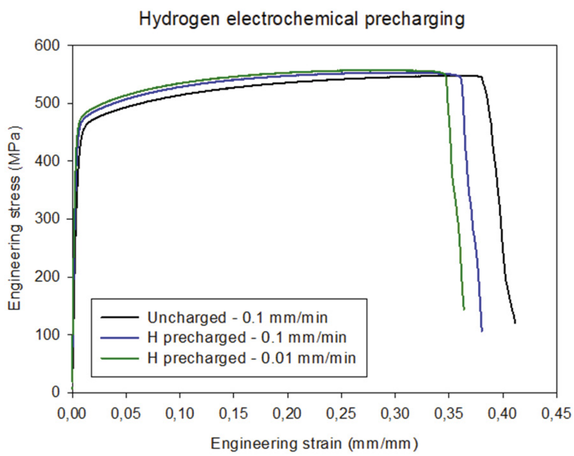


Figure 3. Tensile curves on smooth samples

Table 3. Tensile properties on smooth samples. CHS: cross-head speed

	CHS (mm/min)	σ_{ys} (MPa)	σ_{uts} (MPa)	ε (%)	RA (%)
Uncharged	0.1	477	546	39	30
H precharged	0.1	490	553	36	28
H precharged	0.01	496	557	35	27

3.2 Tensile test on smooth samples

Tensile results on smooth samples are shown in Figure 3, while tensile properties are given in Table 3. Fracture surfaces are compared in Figure 4, for the uncharged and hydrogen precharged condition (0.01 mm/min).

Hydrogen embrittlement susceptibility was evaluated by comparing slow strain rate tensile curves of uncharged and hydrogen precharged samples. In the presence of internal hydrogen (≈37 wt ppm), yield strength and ultimate tensile strength increased. This fact might be associated to hydrogen dislocation pinning mechanism in austenitic stainless steels [4]. Besides, a decrease of ductility was also observed in the presence of internal hydrogen. Dimples indicating ductile fracture were observed in the uncharged condition, Figure 4(a). However, after hydrogen precharging and especially at 0.01 mm/min, stretched and shallow dimples were observed near the surface region, Figure 4(b). The stretched and shallow dimples in a H-charged 316L steel have also been experimentally found by previous authors [5]. Stretched and shallow voids are attributed to hydrogen accelerated nucleation of voids that triggers local shearing between voids. This mechanisms has also been numerically captured [6]. Based on the low diffusivity of hydrogen in austenitic stainless steels, it is expected that high hydrogen concentration (≈37 wt ppm) was introduced at the subsurface level what slightly modified the fracture micromechanism in the presence of internal hydrogen, as can be seen in Figure 4.

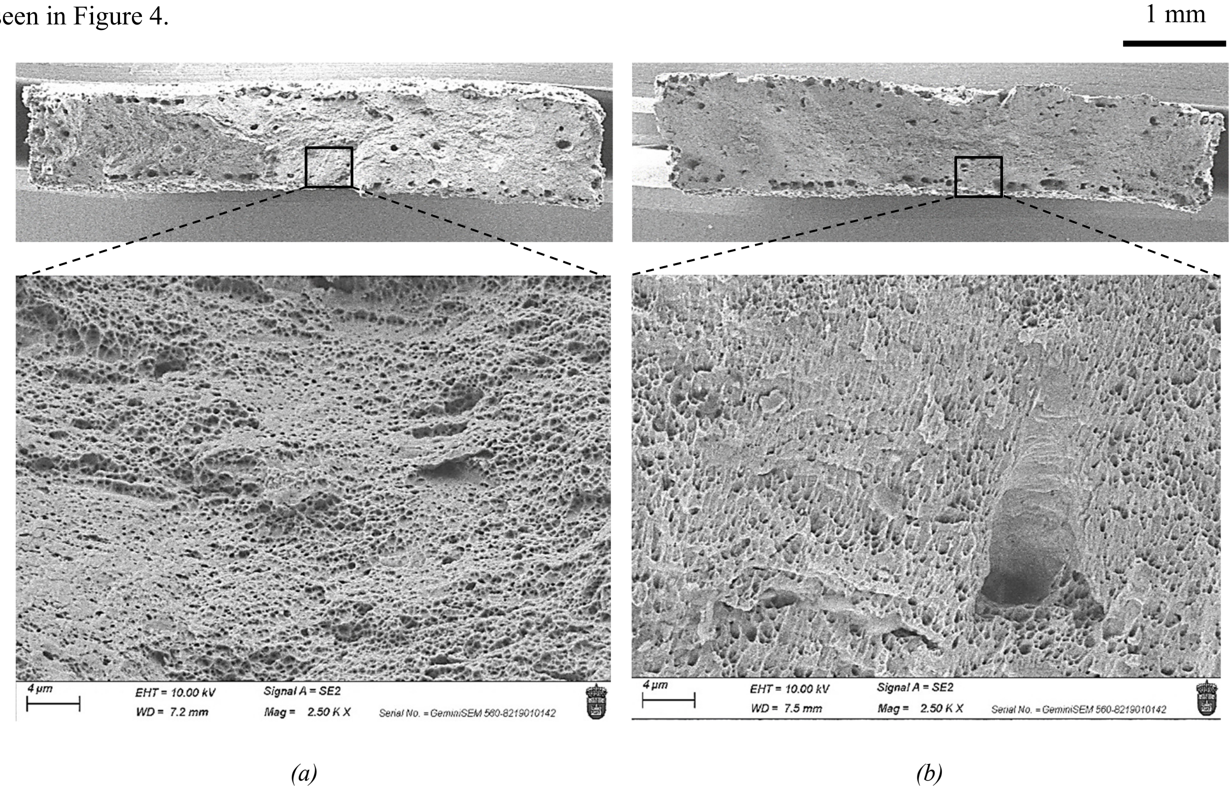


Figure 4. Fracture surfaces. (a) Uncharged. (b) Hydrogen precharged and tested at 0.01 mm/min, with stretched and shallow dimples produced by local shear strain

3.4 Tensile test on notched samples

Hydrogen influence on tensile behavior was also studied on notched samples. Tensile results corresponding to the uncharged and hydrogen precharged condition are displayed in Figure 5. The obtained mechanical properties and the embrittlement indexes are summarized in Table 4 and Table 5, respectively. On the other hand, fracture surfaces are shown in Figure 6. Fracture surfaces observations were done near the notched region. In the uncharged sample, dimples were observed, Figure 6(a). Nevertheless, fracture micromechanism was modified in the presence of hydrogen. In sample tested with hydrogen at 0.005 mm/min, secondary cracks and quasi-cleavages were noted, Figure 6(b). This fact contributes to justify the loss of strength and ductility (Table 4 and Table 5) for tensile tests conducted especially at 0.005 mm/min after hydrogen precharging (8 mA/cm^2 for 24h). Additionally, after tensile tests, XRD analysis was performed on the fracture surfaces. XRD analysis revealed the presence of strain-induced martensite near the notched region (Table 6). It is important to highlight, this phase transformation was not previously found after the 3D printing process (Figure 2b).

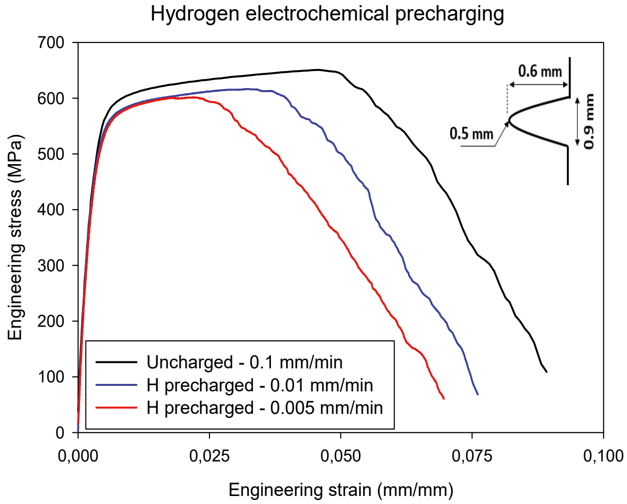


Figure 5. Tensile curves on notched samples

Table 4. Tensile properties on notched samples. NTS: notch tensile strength and RA: reduction of area

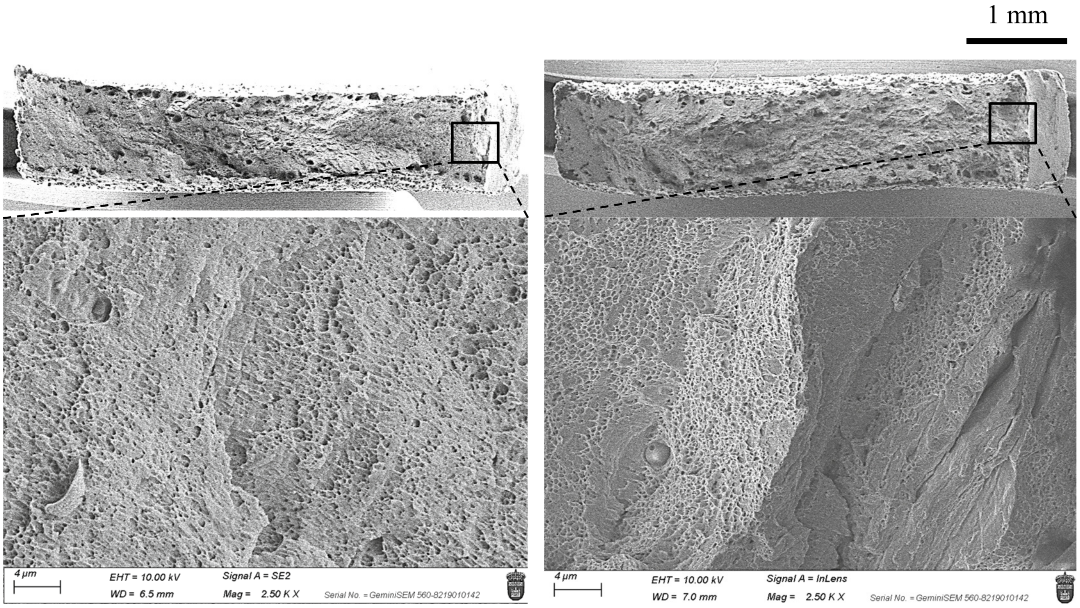
	CHS (mm/min)	Test duration (min)	σ_{NTS} (MPa)	RA (%)
Uncharged	0.1	24	645±8	7.1
H precharged	0.01	180	619±4	5.8
H precharged	0.005	360	601±1	4.9

Table 5. Embrittlement indexes on notched samples

	CHS (mm/min)	EI σ_{NTS} (%)	EI RA (%)
Uncharged	0.1	-	-
H precharged	0.01	4	18
H precharged	0.005	7	31

Table 6. Strain-induced martensite in {211} with 2θ=156.4° and {200} with 2θ=106.1°. XRD results near the notched region after the tensile tests

	CHS (mm/min)	Strain-induced martensite (%)
Uncharged	0.1	3.5
H precharged	0.01	4.0
H precharged	0.005	4.0



(a) (b)

Figure 6. Fracture surfaces. (a) Uncharged. (b) Hydrogen precharged and tested at 0.005 mm/min

Since martensite is known to be very sensitive to hydrogen embrittlement [7,8], this can be an important factor explaining the increased hydrogen embrittlement susceptibility of the sample tested at 0.005 mm/min in the presence of hydrogen (≈37 wt ppm). Thus, in the presence of internal hydrogen, it is important to highlight that the observed fracture micromechanisms near the notched region (Figure 6b) can be promoted by the strain-induced martensite formation. It is well known that hydrogen atoms are driven by the high hydrostatic stress existing in the vicinity of the notch and simultaneously, the strain-induced martensite acts as a diffusion path for hydrogen [7] due to the higher diffusivity through that phase; this short-circuit diffusion induced the formation of quasi-cleavages and secondary cracks.

4. Conclusions

Hydrogen ex-situ tensile tests were employed to evaluate hydrogen embrittlement susceptibility in an additively manufactured 316L by selective laser melting. Based on smooth tensile results, significant hydrogen hardening (yield strength and ultimate tensile strength increased) was observed because of the high internal hydrogen (≈37 wt ppm). Additionally, ductility slightly decreased. On the other hand, in the presence of a notch, the notch tensile strength and especially, the reduction of area were affected by hydrogen. In this case, fracture micromechanism changed from ductile (without hydrogen) to quasi-brittle (with hydrogen) near the notched region. Hydrogen damage is also explained because of the strain-induced martensite formation near the notch tip region.

Acknowledgements

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