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Hydrogen diffusivity and tensile properties degradation in SLMed Inconel 718

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Abstract. Besides elevated temperature applications, Inconel 718 is widely chosen for components subjected to heavy loads operating in aggressive environments, which can promote the production of hydrogen on the metal surface. Selective Laser Melting (SLM) is an emerging technology for the production of structural components in Inconel 718 or Inconel 625 alloys, allowing to create functionally optimized shapes and overcome the traditional manufacturing limitations. However, the SLM process introduces a peculiar microstructure and severe residual stresses that can affect hydrogen migration and accumulation. While the mechanical properties were deeply investigated in recent years, the resistance of SLMed Inconel 718 to the Hydrogen Embrittlement (HE) is still an open issue. The present work deals with a preliminary assessment of the hydrogen embrittlement susceptibility of the SLMed In718 in the as-built condition. Slow strain rate tests were carried out on unnotched specimens that were pre-charged by electrochemical cathodic charging. Fractographic analyses, carried out with a Scanning Electron Microscope (SEM), were used to identify the effects produced by the hydrogen intake on the material microstructure. The material resulted to be susceptible to hydrogen embrittlement, featuring a significant ductility reduction in presence of extremely elevated hydrogen concentrations. The hydrogen content was measured by employing the Hot Extraction Method (HEM), for each mechanical test. Due to a low diffusivity value, hydrogen penetrates only within a thin external layer of the specimen. Therefore, to identify the concentration profile, the effective hydrogen diffusion coefficient was identified and the hydrogen diffusion was simulated using Fick's equation. The loss in mechanical resistance was then correlated to the hydrogen concentration near the fracture onset.

1. Introduction

Nickel-based superalloys feature excellent mechanical properties, combining high strength and elevated ductility, both at room and high temperature, coupled with outstanding resistance to corrosion phenomena. These properties match the requirement of a wide range of industrial applications, from aero engines, power generation plants, to chemical plants and Oil&Gas applications.

Inconel 718 alloy is a widespread equiaxed nickel-based alloy characterized by high strength and excellent corrosion resistance, within a working temperature range between -250 °C and 800 °C. However, it is known to be susceptible to hydrogen embrittlement effects when exposed to elevated hydrogen concentrations [1, 2, 3, 4, 5]. Hydrogen can penetrate into the material during manufacturing processes or during service exposure to hydrogen-rich environments



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the surrounding environment during service exposure. In the case of aerospace applications, hydrogen can arise from fuel in rocket engines and its embrittlement effects were deeply studied [5].

Even if the Inconel 718 was developed for use in aircraft gas turbines, its combination of room-temperature strength and aqueous corrosion resistance made it a candidate for oilfield fasteners, valves, drill tools, and completion equipment, where hydrogen can arise from the conveyed fluids, [6]. Hydrogen embrittlement of the conventional Inconel 718 alloy was deeply investigated through SSR (Slow Strain Rate) tensile tests [2, 7, 8], along with the measurement of the hydrogen diffusivity coefficient [4, 9, 10]. Selective Laser Melting (SLM) is an emerging technology to produce structural components featuring complex geometries and it is particularly suitable for high-strength materials. Inconel 718 is the most widespread nickel-based superalloy produced by SLM, thanks to its wide range of applications and weldability properties. Even if the mechanical and fatigue properties of SLMed Inconel 718 are deeply investigated, the hydrogen diffusion properties and the resistance to hydrogen embrittlement phenomena are still open questions.

In the present paper, it is presented a preliminary investigation of the effects produced by cathodic hydrogen intake in SLMed Inconel 718 in the as-built condition.

The hydrogen diffusivity properties are conventionally evaluated by means of the Devanathan-Stachurski test method. However, in the case of a material featuring a very low diffusivity value, as the wrought Inconel 718, this test method requires a very thin metallic foil, namely featuring a thickness of about $50\div 100\text{ }\mu\text{m}$, to have experiment duration within two or four weeks at temperatures of $50\text{ }^{\circ}\text{C}$, [9]. Producing a so thin foil by SLM is unfeasible, as its dimensions are comparable with the power size or laser beam diameter. On the other hand, machining a bar produced by SLM would alter the surface roughness, introducing a bias between the measured value and the behavior of a component produced by SLM and exposed to a hydrogen-rich environment. As a matter of fact, exploiting the potential of AM means producing complex geometries, where successive machining could be unfeasible or cost-ineffective. The hydrogen diffusion coefficient was thus measured indirectly by measuring the hydrogen intake of specimens exposed to the same surface hydrogen concentration for different durations.

Slow Strain Rate (SSR) tensile tests were carried out along with hydrogen concentration measurements and fractographic analyses were carried out to investigate the effects produced by the hydrogen intake. The results are analyzed within the framework of the hydrogen diffusion models developed in the literature. Further investigations will be performed based on the results of this preliminary investigation, studying the effects of the aging treatments and surface roughness.

2. Materials and methods

Specimens were produced by using the Renishaw RenAM 500E Selective Laser Melting (SLM) machine present in the *Metal Additive Manufacturing* laboratory of the University of Pisa.

The chemical composition of the Inconel 718 alloy powder, provided by Renishaw plc, is reported in Table 1.

The process chamber was filled with gas argon, reaching an oxygen concentration lower than 10 ppmw during the printing phase, guaranteeing to avoid oxidation phenomena. It was adopted a standard process parameters set, featuring a layer thickness of $60\text{ }\mu\text{m}$. All the specimens were built in the vertical direction, namely with the longitudinal direction aligned with the direction of motion of the built plate, in order not to have traces of the support structures in the area of interest of the specimen and have uniform residual stresses on the specimen surfaces. The specimens were measured by a coordinate-measuring machine to check the occurrence of distortions due to the printing phase or the built plate removal. It resulted that the specimens presented no significant deviation, with values that are comprised in the typical range of accuracy

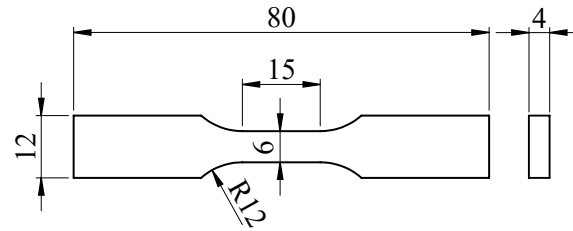


Figure 1. Specimen geometry and nominal dimensions. Dimensions in mm.

of the SLM process, namely 0.05 mm.

Table 1. Chemical composition of Inconel 718. Percentage mass values.

Ni	Cr	Fe	Nb + Ta	Mo	Ti
50÷55	17÷21	balanced	4.75÷5.5	2.80÷3.30	0.65÷1.15
Co	Al	Mn	Si	Cu	C
≤1.00	0.20÷0.80	≤0.35	≤0.35	≤0.30	0.02÷0.05

The specimen geometry, designed in agreement with the ASTM E8 standard, is characterized by a gauge length of 15 mm and by a rectangular cross-section, whose nominal dimensions, in the gauge section, are 4 mm × 6 mm, Figure 1. The specimens were tested in the as-built condition, preserving both the surface roughness and the microstructure produced by the SLM process. The effective cross-section dimensions of each specimen were measured before the mechanical tests.

Slow Strain Rate (SSR) tensile tests were carried out in air at room temperature on a servo-hydraulic machine having a load capacity of 50 kN (MTS Systems Corporation, Eden Prairie, MN USA). The machine was equipped with an extensometer having a gauge length of 10 mm (MTS 634.21-F25) and with a data acquisition system and control unit (RT3, Trio Sistemi e Misure S.r.l., Dalmine, Italy). SSR tests were performed by imposing a cross-head displacement of 1 $\mu\text{m/s}$ and thus obtaining an almost constant strain rate of $1.5 \cdot 10^{-4} \text{ s}^{-1}$, comprised within the range provided by the G129-00 ASTM standard.

Microstructural investigations were carried out both in longitudinal and transverse directions by analyzing sections extracted from the gauge length of two specimens featuring the same geometry and SLM process history of the tested ones. Test samples were prepared following the ASTM E3-11 standard and etched with Kalling's II etchant (n.94 ASTM E407). The material porosity was characterized in terms of average material density and pore size and shape distribution by using the software ImageJ.

Scanning Electron Microscopy (SEM) analyses were carried out at the "Centro per l'Integrazione della Strumentazione Scientifica - Università di Pisa" (CISUP) using a FEI Quanta 450 FEG-SEM equipped with an EDX spectrometer Bruker QUANTAX XFlash Detector 6|10. Fractographic analyses were carried out by stereo microscope as well.

2.1. Hydrogen charging and measurement

The specimens were pre-charged before the SSR test by electrochemical cathodic charging, where a constant current density is imposed between the specimen and a platinum electrode. Before

charging, all the specimens were cleaned by ultrasonic cleaning with acetone and rinsed with distilled water.

For the first three specimens, it was employed a 0.5 M aqueous solution of sulfuric acid (H_2SO_4), with the addition of 1 g/L of thiourea ($\text{CH}_4\text{N}_2\text{S}$) to increase hydrogen adsorption on the surfaces of specimens, [7, 11]. Hydrogen charging was performed at room temperature with a constant current density of 60 mA/cm² for durations between 45 and 66 hours. The room temperature, measured by a temperature data logger (Lascar EL USB-2) was 19 ± 3 °C.

To increase the hydrogen diffusivity coefficient and thus the hydrogen introduced into the material, a second batch of four specimens was charged at 70 °C by using a 3% aqueous solution of NaCl, containing 4 g/L of ammonium thiocyanate (NH_4SCN) to increase the hydrogen adsorption, [1, 12]. A constant current density of 20 mA/cm² was imposed. The hydrogen charging time ranged between 60 and 200 hours.

After each SSR test, the hydrogen content C_{HEM} was measured by Hot Extraction Method (HEM) employing the LECO® hydrogen determinator DH 603 (LECO, St. Joseph, MO, USA). The instrument measures the hydrogen flux that desorbs from the specimen heated at 265 °C and thus provides the average hydrogen content present in the analyzed coupon.

2.2. Diffusion coefficient determination

In the case of an ideal homogeneous material, where the distribution of hydrogen possible sites in the lattice is uniform, and a component without external loads and residual stresses, the hydrogen diffusion in metallic materials is known to be governed by Fick's law of diffusion:

$$\vec{J} = -\mathbf{D} \nabla C + \vec{v} \cdot C, \quad (1)$$

$$\frac{\partial C}{\partial t} = -\nabla \cdot \vec{J} + G, \quad (2)$$

where $\vec{J}$ is the diffusion flux density, $\mathbf{D}$ the diffusivity tensor, C the atomic concentration, $\vec{v}$ the transport velocity vector, and G the diffusing substance generation rate per unit volume. The presence of microstructural defects (such as dislocations, vacancies, grain boundaries, and precipitates) creates trapping sites, which provide an energetically favored environment for occupancy by the hydrogen, [13]. In the case of low occupancy in the reversible traps, Fick's first law can be used to model the diffusion of hydrogen in the alloy [13]. In this case, for an isotropic material, the diffusion equation takes the following form:

$$\frac{\partial C}{\partial t} = D_{\text{eff}} \frac{\partial^2 C}{\partial x^2}, \quad (3)$$

where D_{eff} is the effective diffusion coefficient. It varies with the temperature with an Arrhenius function:

$$D_{\text{eff}} = D_{\text{eff},0} \cdot \exp\left(-\frac{E}{R T}\right), \quad (4)$$

where, in the present case, $D_{\text{eff},0}$ is the pre-exponential parameter, and E is the activation energy equal to 48630 J/mol, [4].

Since conventional Inconel 718 alloy is known to present a low hydrogen diffusion coefficient, a direct measurement of D_{eff} is difficult due to unfeasible test durations. For this reason, D_{eff} was identified by measuring the H content (C_{HEM}) at different charging durations. The value of C_{HEM} as a function of time depends on the surface concentration and the effective diffusion coefficient D_{eff} . Therefore, it is possible to obtain the D_{eff} value through an optimization procedure.

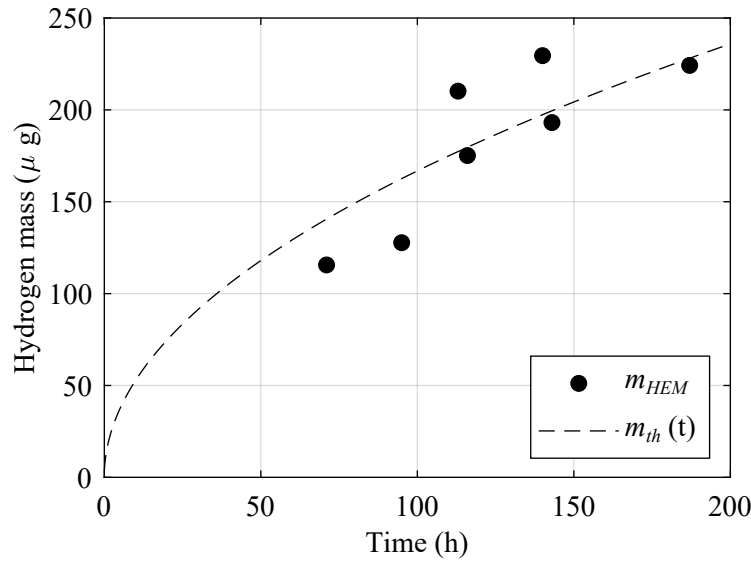


Figure 2. Effective diffusion coefficient identification. Measured hydrogen mass m_{HEM} , and theoretical trend m_{th} obtained using Equation 5 with the fitted parameters.

The theoretical value of the hydrogen mass m_{th} , at time t , as a function of the surface concentration C_s and D_{eff} , is obtained by solving Equation 3 and integrating the concentration profile in the volume:

$$m_{th}(C_s, D_{eff}, t) = \int_0^{l_p} C(C_s, D_{eff}, t) A dx, \quad (5)$$

where l_p and A are the specimen thickness and surface area, respectively. The D_{eff} value can be obtained by minimizing the following cost function:

$$F(C_s, D_{eff}) = \sum_{i=1}^7 [m_{th}(C_s, D_{eff}, t_i) - m_{HEM}(t_i)]^2. \quad (6)$$

Figure 2 shows the measured hydrogen mass m_{HEM} obtained from the electrochemically charged specimens as a function of charging times. The theoretical trend of the hydrogen mass m_{th} , obtained from Equation 5 using the fitted parameters, is reported with a dashed line.

Seven rectangular specimens having dimensions of 40 mm × 40 mm × 1.7 mm were electrochemically charged in the same conditions. It was adopted an electrolyte solution of NaCl 3.0% + NH₄SCN 0.4% (w/w), held at a constant temperature of 50 °C. The specimens were charged for different durations, with charging times (t_i) comprised between 60 and 200 hours. At the end of each charging time, the hydrogen mass was measured by HEM, by using the LECO® hydrogen determinator DH 603 (LECO, St. Joseph, MO, USA).

By solving Equation 6 for a fixed value of the effective diffusion coefficient D_{eff} , its value resulted to be equal to $5 \cdot 10^{-15}$ m²/s at the test temperature of 50 °C. This value is very similar to the value obtained by Jebaraj [9] on the conventional wrought Inconel 718 in the solutionized condition, which can be considered as the most similar to the not heat-treated SLMed material.

2.3. Hydrogen profile within the specimens

The hydrogen concentration profile, reached in the specimen after the electrochemical charging, can be obtained by solving Fick's law, assuming a uniform initial concentration, in this case,

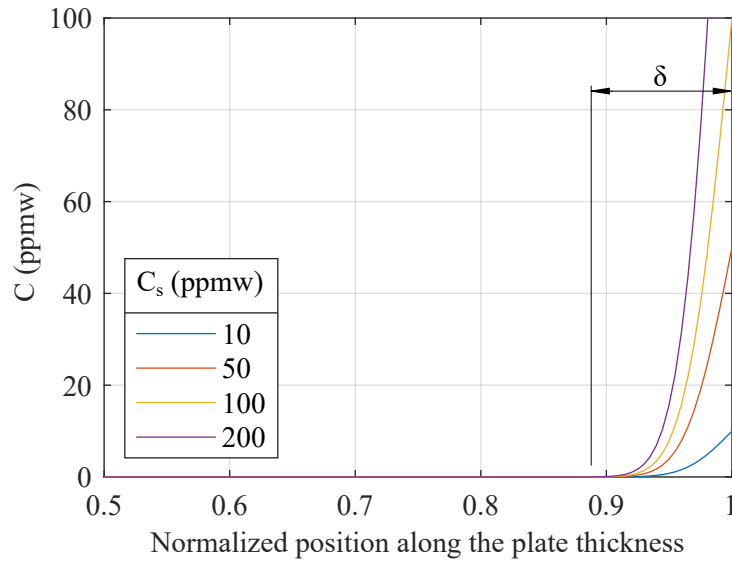


Figure 3. Sensitivity to surface concentration: hydrogen concentration profile within the specimen (charging time: 90 hours).

null, and imposing a boundary condition of constant and equal hydrogen concentration on the specimen walls.

In the case of a one-dimensional diffusion in a plane sheet, the solution is presented by Crank, [14]. Under these hypotheses, the hydrogen concentration profile depends only on the diffusivity concentration value, temperature-dependent, and on the hydrogen concentration on the specimen walls, which depends on the charging current and solution. Due to the low diffusion coefficient D_{eff} , the hydrogen concentration within the specimen does not reach a saturation value even after cathodic charging durations of hundreds of hours. The hydrogen penetration depth, calculated by using the approximate formulation derived for the case of a semi-infinite medium and presented by Crank [14], Equation 7, and employing the measured value of the effective diffusion coefficient ($5 \cdot 10^{-15} \text{ m}^2/\text{s}$ at the test temperature of 50°C), results to be about 0.26 mm after 150 hours of cathodic charging,

$$\delta = 4 \cdot \sqrt{D t} \quad (7)$$

The sensitivity of the concentration profile to the hydrogen concentration on the specimen surface at equal charging duration is reported in Figure 3. An increase in the hydrogen surface concentration produces only an increase in the average hydrogen content in the outer part of the specimen, while the penetration depth is influenced only by the charging duration and temperature. As shown in the schematic representation presented in Figure 4, after the cathodic charging, hydrogen is present only in the outer shell (black-filled area) of the specimen, while the middle part remains unaffected. Due to the non-uniform hydrogen concentration at the end of the cathodic charging, it is not possible to have a direct measure of the hydrogen concentration. As the HEM provides the average hydrogen concentration (C_{HEM}) present in the specimen, its measurement is strongly lower than the real value present near the specimen surfaces. However, based on the measured average hydrogen concentration content and the measured value of the hydrogen diffusivity D_{eff} , it is possible to calculate the hydrogen concentration on the specimen surface, C_s , and thus the average value, $C_{avg, s}$, within the penetration depth through

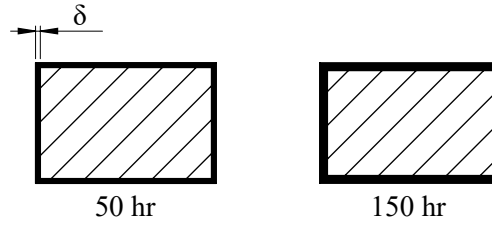


Figure 4. Hydrogen penetration within the specimen at increasing charging duration.

the integration of the hydrogen concentration profile, using the following equations:

$$C_{\text{HEM}} = \frac{1}{l_p} \int_0^{l_p} C(C_s, x) dx, \quad (8)$$

$$C_{\text{avg,s}} = \frac{1}{\delta} \int_{l_p-\delta}^{l_p} C(C_s, x) dx, \quad (9)$$

where l_p , and δ are the specimen thickness and width, and the hydrogen penetration depth, respectively. By applying this approach, an average hydrogen concentration of 10 ppmw, measured by HEM, corresponds to a surface concentration of 200 ppmw and an average concentration, within the hydrogen penetration depth, of 55 ppmw. It is worth noticing that it was neglected the contribution of the hydrogen desorption that happens between the end of the charging phase and the HEM measurement after the SSR test, thus underestimating the hydrogen content at the end of the cathodic charging.

3. Results and discussion

3.1. SSR test results

In Figure 5 the engineering stress-strain curves are reported as a function of the averaged hydrogen concentration measured by HEM. For the uncharged specimens it is reported only the average curve resulting from three tests carried out in identical conditions. In agreement with the results obtained on the conventional Inconel 718 alloy [1, 7, 11], it can be seen that the macroscopic effect of the hydrogen intake is an almost monotonic reduction of the material ductility, represented by the percentage elongation at fracture. For the lower values of the hydrogen content, the tensile strength of the material results to be unaffected by the hydrogen content as the crack happens after the occurrence of a necking phenomenon. As the hydrogen content increases, also the tensile strength decreases monotonically with the percentage elongation at fracture, due to a more consistent reduction of ductility. Within the investigated range, the hydrogen intake resulted not have an impact on the material elastic and strain-hardening phases, where the uncharged and charged curves are almost indistinguishable.

The embrittlement effect can be summarized by introducing a Hydrogen Embrittlement Index (HEI):

$$\text{HEI} = \frac{\varepsilon_{f,\text{uncharged}} - \varepsilon_{f,\text{charged}}}{\varepsilon_{f,\text{uncharged}}}, \quad (10)$$

where ε_f is the engineering fracture strain of the hydrogen-uncharged and charged specimens, respectively. As reported in Table 2, the HEI index reaches a value of 0.5 only for an average hydrogen concentration of 28 ppmw, corresponding to an estimated average value in the hydrogen affected part of the specimen of about 100 ppmw.

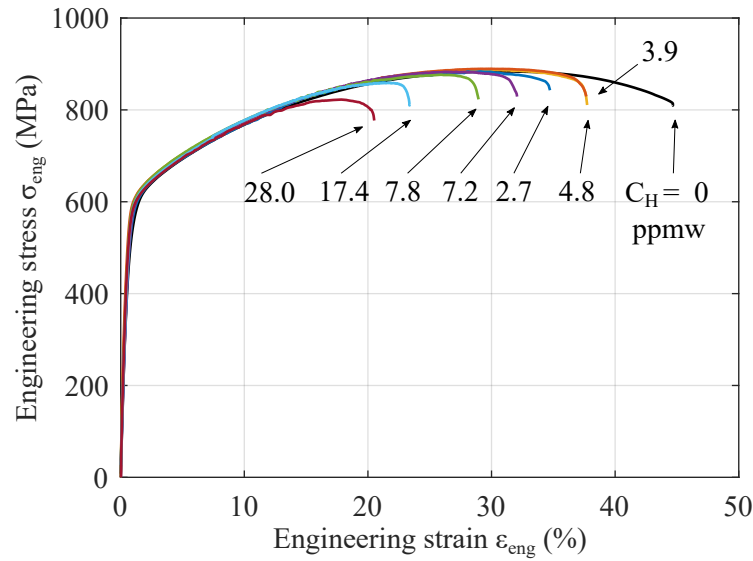


Figure 5. SSR tensile tests. For each curve it is reported the average hydrogen content measured by HEM, in ppmw.

Table 2. Hydrogen penetration depth, engineering fracture strain, and hydrogen embrittlement index values as a function of the hydrogen content.

C_{HEM} (ppmw)	0	2.7	3.9	4.8	7.2	7.8	17.4	28.0
δ (mm)	-	0.05	0.06	0.06	0.18	0.22	0.29	0.30
$C_{avg,s}$ (ppmw)	-	70	85	100	50	45	70	115
ε_f (%)	44.6	34.7	38.0	38.1	32.0	29.0	23.5	20.5
HEI	-	0.22	0.15	0.15	0.28	0.35	0.48	0.54

3.2. Fractographic investigations

The fractographic analysis, carried out by SEM, showed that the fracture surface can be divided into three regions: a central region, an intermediate region oriented at about 45° degrees to the loading axis, and a thin surface layer, Figure 6 (a). In the central region it is possible to see the traces of the SLM laser scanning path, suggesting that the final fracture took place by decohesion on the melt pool boundaries. Looking into detail to the outer part of the fracture surface, Figure 6 (b), it can be seen a clear difference between the part nearest to the specimen surface, a strip featuring a width of about 50 μm , and the more internal one. As the width of the outer strip is almost equal to the calculated hydrogen penetration depth in the investigated specimen, it suggests that the different appearance of the fracture surface is due to the effects of the hydrogen intake.

Moreover, it can be seen that there are no blisters or other cavities that can act as sites for the formation of gaseous hydrogen, suggesting that these phenomena do not affect a fully dense additively manufactured Inconel 718.

Fractographic analyses carried out by stereo microscope confirmed that the major difference between the hydrogen-free and charged material is represented by the external part of the fracture surface, within a thickness almost equal to the hydrogen penetration depth, Figure 7.

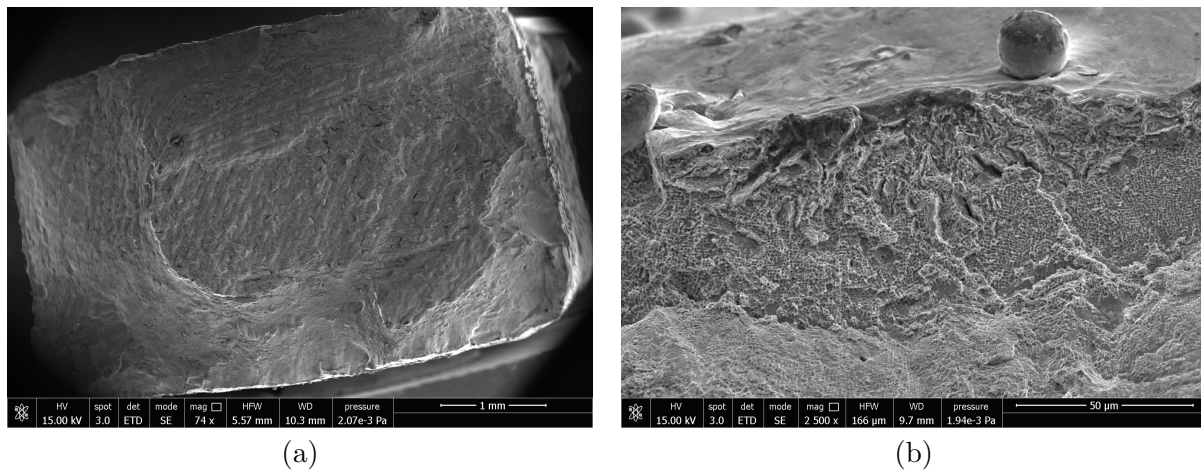


Figure 6. SEM image of the whole fracture surface (a), and of detail near specimen wall (b).

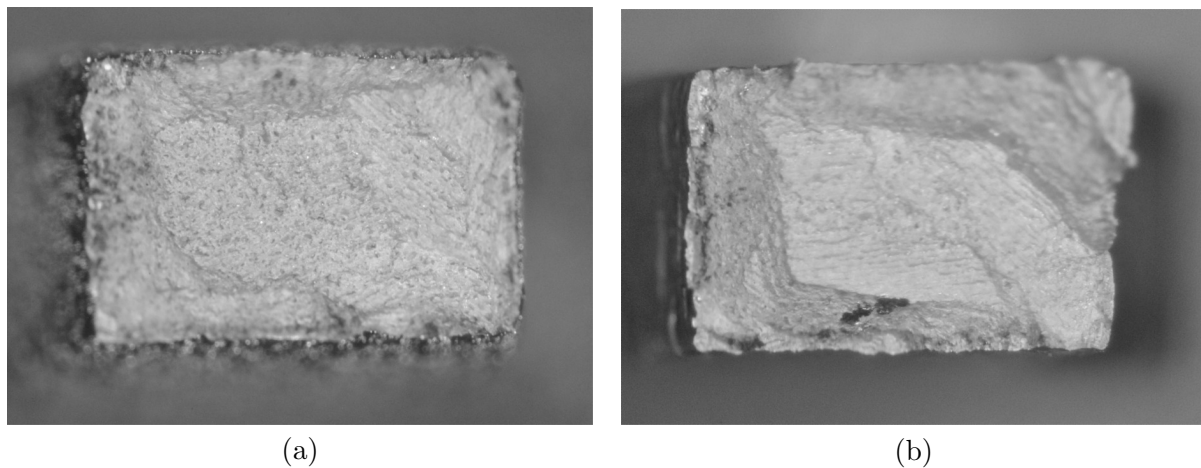


Figure 7. Fracture surfaces: specimens, not hydrogen charged (a), and with an average hydrogen concentration of 7.2 ppmw (b).

In the case of hydrogen charged specimens, the edges of the fracture surface appear to be sharper than the base material. In both cases in the central region, traces of the AM scan path are visible, slightly more marked for the hydrogen charged specimens.

While in the uncharged condition a visible necking occurs, charged specimens reached the fracture condition before the occurrence of necking, in particular at higher hydrogen concentrations, as shown in Figure 8.

As it is possible to observe in Figure 9, traces of layer delamination appeared along the gauge length of the specimen, confirming that the crack onset occurred on the specimen surface, and thus in the area affected by the hydrogen intake. The same traces can be observed also in Figure 7, in the case of the hydrogen-charged specimen. This effect can be considered peculiar of a SLMed material, which is built via a superposition of stacking layers.

3.3. Microstructural investigations

Microstructural investigations showed that the specimen microstructure is strongly influenced by the SLM process, with visible traces of the SLM scan paths, highlighted by segregations on the melt pool boundaries, and columnar grains, Figure 10, as it is typical for the as-built condition.

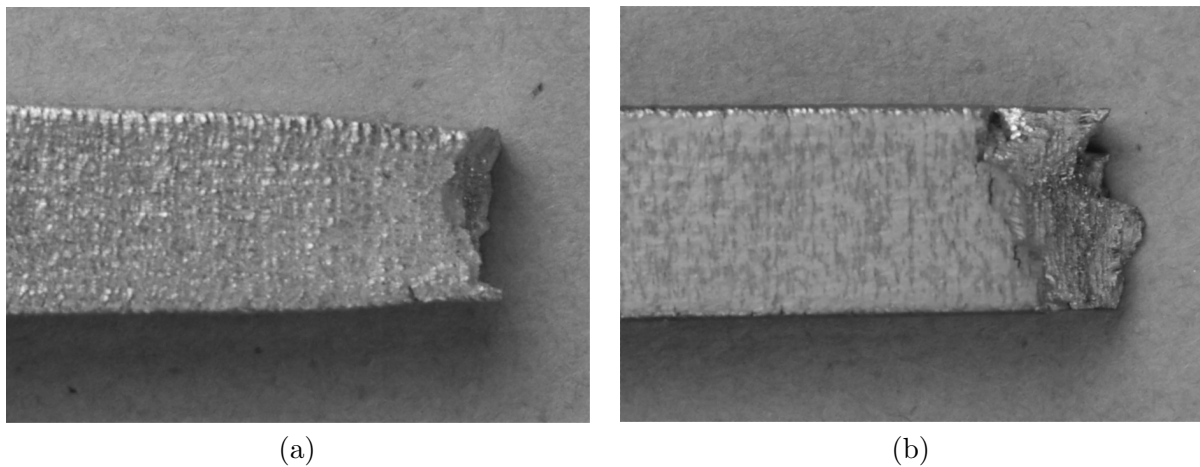


Figure 8. Side view of the specimen fracture region: not hydrogen charged (a), and featuring an averaged hydrogen concentration of 17 ppmw (b).

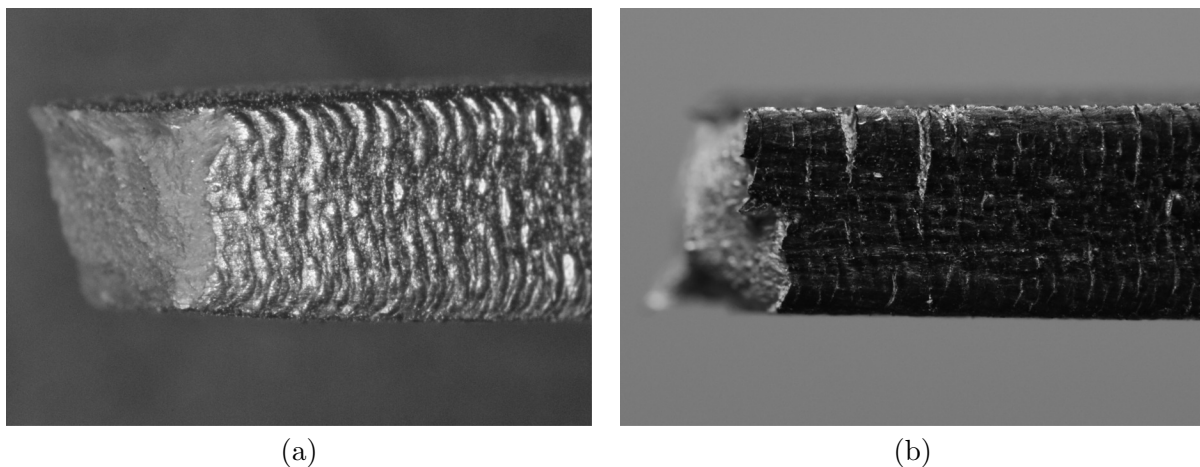


Figure 9. Detail of the surface of an uncharged specimen (a), and of a specimen with an average hydrogen concentration of 7.8 ppmw (b), featuring visible traces of layer delamination.

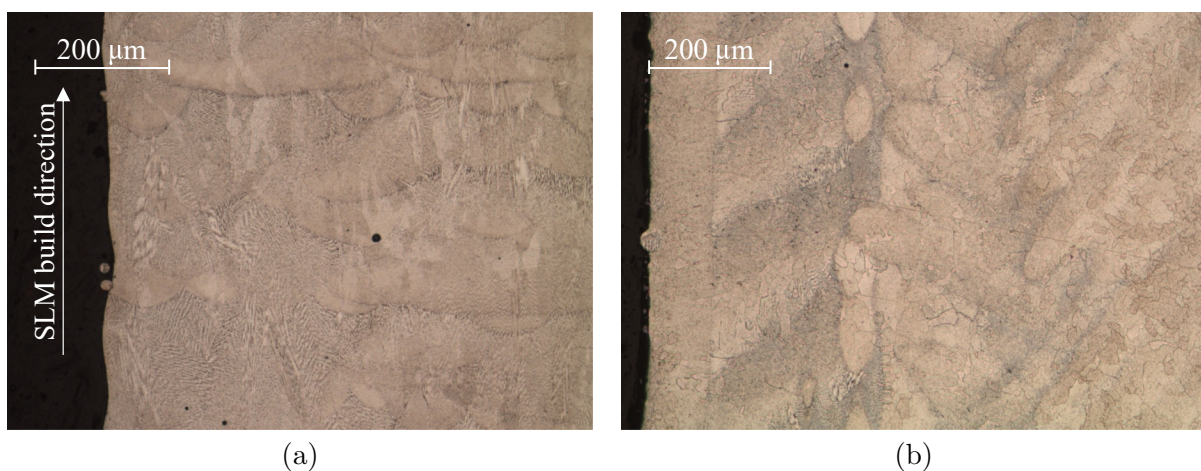


Figure 10. Specimen microstructure in the longitudinal (a) and transverse direction (b), etchant Kalling's II.

The adopted SLM process parameters resulted to produce an almost full-dense material, having a density of 99.96%, if examined from sections extracted in longitudinal directions, and 99.94% in the transversal direction. The pores are round and small, the maximum length of the major axis is less than 60 μm , suggesting that they arise from gas bubbles entrapped during the SLM process.

4. Conclusions

A preliminary investigation of the hydrogen embrittlement effect on the Inconel 718 alloy produced by SLM was carried out. The effective hydrogen diffusivity within the material was indirectly evaluated by measuring the hydrogen content at different cathodic charging durations. It was obtained a value of $5 \cdot 10^{-15} \text{ m}^2/\text{s}$ at a temperature of 50 °C, in line with the conventional wrought material in the solutionized condition. The tensile properties resulted to be degraded by the hydrogen intake, featuring a ductility reduction of about 50% in presence of average hydrogen content of 28 ppmw. As the hydrogen penetration depth is limited to the outer part of the specimen, the actual value of hydrogen concentration in the crack onset region is expected to be significantly greater. The susceptibility to hydrogen embrittlement of the SLMed material in the as-built conditions resulted to be in line with the conventional wrought Inconel 718.

Acknowledgments

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