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Effect of trapping and temperature on the hydrogen embrittlement susceptibility of alloy 718

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Ni-based alloy 718 is widely used to manufacture structural components in the aeronautic and nuclear industries. Numerous studies have shown that alloy 718 may be sensitive to hydrogen embrittlement. In the present study, the susceptibilities of three distinct metallurgical states of alloy 718 to hydrogen embrittlement were investigated to identify both the effect of hydrogen trapping on hydrogen embrittlement and the role of temperature in the hydrogen-trapping mechanism. Cathodic charging in a molten salt bath was used to saturate the different hydrogen traps of each metallurgical state. Tensile tests at different temperatures and different strain rates were carried out to study the effect of hydrogen on mechanical properties and failure modes, in combination with hydrogen content measurements. The results demonstrated that Ni-based superalloy 718 was strongly susceptible to hydrogen embrittlement between 25 °C and 300 °C, and highlighted the dominant roles played by the hydrogen solubility and the hydrogen trapping on mechanical behavior and fracture modes.

1. Introduction

Alloy 718 is a Ni-based superalloy that is widely used in high-temperature applications, particularly for structural components in the aeronautic and nuclear industries, due to its good mechanical properties and good resistance to stress corrosion cracking. The alloy is strengthened both by structural hardening due to the precipitation of stable γ' (Ni_3(Ti,Al)) and metastable γ''(Ni_3Nb) precipitates and by solid solution hardening [1]. Under the conditions required by certain fabrication processes, the precipitation of a stable form of (Ni_3Nb) corresponding to the δ phase may occur [2]. Under severe operating conditions, corrosion processes may cause local hydrogen enrichment at the material surface. Consequently, a hydrogen-assisted stress corrosion cracking phenomenon can occur under complex stress and strain states [3].

The hydrogen embrittlement of Ni-based alloys is generally exacerbated when the alloys are submitted to mechanical loading, suggesting that hydrogen diffusion occurs along stress gradients and hydrogen transport by dislocations during plastic deformation [4–14]. Hydrogen transport by mobile dislocations can lead to local hydrogen enrichment in the dislocation pile-ups close to precipitates, which favors crack initiation. The fracture modes due to hydrogen embrittlement (HE) are varied, ranging from brittle intergranular to transgranular modes with cleavage facets or ductile character.

Fournier et al. [8] studied the HE susceptibility of strengthened alloy 718 cathodically precharged at room temperature. Due to the observation of planar cleavage microfacets on the fracture surfaces of hydrogen-embrittled specimens, the authors proposed that HE probably occurs by strong hydrogen-deformation interactions, i.e., hydrogen transport by dislocations. In this case, HE would be correlated with hydrogen segregation toward moving dislocations and hydrogen transport in the form of a Cottrell atmosphere around these dislocations. Consequently, at slow strain rates, the dislocation sweeping of hydrogen takes place and HE is more pronounced. In contrast, at a strain rate of $5\times10^{-3}\,s^{-1}$, the dislocation velocity is too high to induce significant hydrogen segregation, and thus, HE is reduced.

Some authors have explored the role of precipitates in HE in alloy 718 and particularly the contribution of these precipitates to the hydrogen-trapping mechanism depending on their unique characteristics [5,12–15]. Liu et al. [12] demonstrated that both δ phase and γ'' phase play significant roles in altering the HE sensitivity of alloy 718. Hydrogen-induced cracking occurs at δ or γ'–γ''/matrix interfaces and promotes the formation of planar cleavage microfacets on the fracture surfaces of hydrogen-embrittled specimens. Young and Scully [15] studied the role of carbides in hydrogen trapping in Ni–17Cr–8Fe alloys. The authors demonstrated that mill annealing ($<1000\,°C$) produces strong trap sites (≈ 55 KJ/mol binding energy) capable of retaining

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relatively large amounts (≈ 1.8 wt ppm) of hydrogen, whereas sensitization (600 °C/24 h) produces weaker traps (19–28 KJ/mol binding energy) that dissolve less hydrogen (≈ 1 wt ppm) relative to the amount dissolved by the mill-annealed material. The difference in the trap sites is related to the predominant type of carbide (M_7C_3 in mill-annealed material vs. $M_{23}C_6$ in sensitized material). Finally, it was observed that hydrogen trapping on grain boundary carbides promoted the intergranular stress corrosion cracking (SCC) of Ni–Cr–Fe alloys. In alloy 718, carbides that are essentially primary NbC carbides and ((Nb,Ti)C,N) carbonitrides are considered as irreversible traps. Furthermore, Robertson indicated that solubility is strongly affected by the presence of age-hardening precipitates and dissolved elements in the matrix of alloy 718 [16].

Concerning the effect of temperature on the HE of Ni-based alloys, the results of different studies vary widely. Some authors have reported only HE for specimens mechanically loaded at relatively low temperature (below 150 °C) [7–9,17]. However, others have shown that the effect of HE could be extended at high temperatures. Fukuyama et al. [10] reported that alloy 718 was still HE-susceptible at 500 °C under high hydrogen pressures ranging from 1.1 MPa to 19.7 MPa. The deleterious effect of hydrogen on the tensile properties of the alloy was enhanced with the δ phase volume fraction and decreased with temperature. Cracks were initiated at the carbides and then crack propagated along the interface between the δ phase and γ matrix of the alloy or along the grain boundaries of the alloy without δ phase. Wei et al. [11] studied the effect of gaseous hydrogen on the tensile properties of alloy 718 for high-temperature applications. It was demonstrated that the ductility of a solution-treated and aged compressor disc material was reduced in a hydrogen atmosphere at 300 °C and 600 °C and to a lesser extent for a solution-annealed plate material.

The aim of this study was to characterize the effect of hydrogen trapping on the mechanical resistance of precipitates/matrix interfaces in relation to fracture mode for different metallurgical states of alloy 718. The hydrogen cathodic charging procedure was optimized to saturate the characteristic traps of each metallurgical state with hydrogen. This approach, which suppresses the hydrogen enrichment of traps by a transport mechanism, allowed for the intrinsic resistance of hydrogen-saturated interfaces to be tested. Moreover, the effect of temperature on the reversibility of hydrogen trapping was studied by desorption heat treatments in combination with hydrogen content measurements.

2. Experimental

2.1. Material

Alloy 718 is a Ni-based superalloy with high contents of iron, chromium and niobium and small amounts of aluminum, titanium and molybdenum. The material tested in the present study was fashioned from a 0.64-mm-thick strip provided after high temperature recrystallization heat treatment at 1080 °C. The chemical composition of the studied alloy is reported in Table 1.

The typical strengthening heat treatment applied to alloy 718 consists in a dwell of 8 h at 720 °C, followed by cooling at 50 °C/h and a final dwell of 8 h at 620 °C. This process leads to the precipitation of γ″ (metastable and coherent with the matrix, tetragonal $D0_{22}$ structure, Ni_3Nb composition, disc-shaped (20 nm diameter × 10 nm thickness)) and γ′ (stable and coherent with the matrix, cubic $L1_2$ structure, $(Al,Ti)Ni_3$ composition, spherical shape (20 nm)) phases embedded in a γ matrix (volume fraction of γ′–γ″ = 16% and γ′/γ″ ratio = 1/4). Semi-incoherent δ particles (stable form of γ″ phase, globular or needle-shaped (> 1 μm)) may also be observed, depending on fabrication processes employed.

Because all precipitates and inclusions may potentially act as hydrogen traps, three "model" metallurgical states were synthesized to distinguish the effect of each population of traps on the hydrogen embrittlement susceptibility of alloy 718:

- The first metallurgical state corresponded to the as-received alloy 718 obtained after high temperature recrystallization heat treatment (RHT) at 1080 °C. In this state, the alloy presented only two phases, the solid solution and primary carbides NbC and ((Nb,Ti)C,N) (Fig. 1a)
- The second metallurgical state, called HT, corresponded to a material strengthened by the previously described aging heat treatment. Representative TEM micrographs of the metallurgical state are presented in Fig. 1b and c. No δ phase was observed in the grain boundaries. γ′/γ″ precipitates were clearly observed (Fig. 1c) and were homogeneously distributed. Primary carbides precipitates were still present.
- The last metallurgical state was obtained by a preliminary heat treatment at 960 °C for 48 h to precipitate intragranular and intergranular platelets and globular δ phases (Fig. 1d). This preliminary heat treatment was followed by the standard aging treatment previously described. Therefore, in addition to γ′/γ″ and carbide precipitates, δ phase was observed. For this metallurgical state, called δ-HT, the δ phase surface fraction was increased to 9%.

For the three metallurgical states RHT, HT and δ-HT, corresponding samples presented a homogeneous grain size of ASTM 9-10. Table 2 gathers the different microstructural details of each metallurgical state.

2.2. Hydrogen charging

Hydrogen was introduced into the material at 150 °C by an electrochemical charging method in a eutectic mixture of molten salts ($NaHSO_4 \cdot H_2O$ 53.5 wt%–$KHSO_4$ 46.5 wt%) as described by Larignon et al. [18]. This electrochemical technique consists in the electrolysis of water in a molten salt bath using a three-electrode system:

$$\text{Anodic reaction}: H_2O + 2SO_4{}^{2-} \rightarrow 2HSO^- + \frac{1}{2}O_2 + 2e^- \quad (1)$$

$$\text{Cathodic reaction}: HSO_4{}^- + e^- \rightarrow H_{ads} + SO_4{}^{2-} \quad (2)$$

The potential of the sample (working electrode) is measured with respect to a silver electrode situated close to the sample and maintained at a constant cathodic voltage of −1 V. The reference electrode composed of Ag/Ag^+ in Pyrex, consisting of an Ag wire immersed in a small volume of molten salts and separated from the bath by means of a Luggin capillary, was connected to a potentiostat to monitor any change in the potential of the working

Table 1
Chemical composition of studied alloy 718 (wt%).

Ni	Cr	Fe	Nb	Mo	Ti	Al	Mn	Si	C	Cu	Co	Ta	P	B	S
53.66	18.39	18.31	4.94	3.00	0.95	0.56	0.06	0.04	0.033	0.02	0.02	0.01	0.005	0.002	0.0002