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Hydrogen assisted crack initiation and propagation in a nickel-based superalloy

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ABSTRACT

To understand the mechanism for hydrogen-induced embrittlement in a nickel-based superalloy, detailed electronmicroscopy characterisation has been employed on the UNS N07718 (Alloy 718) after hydrogen charging and slow strain rate testing to investigate the strain localisation and damage accumulation caused by hydrogen. Transmission Electron Microscopy analysis demonstrates that the microstructure of the material after tension is characterised by planar dislocation slip bands (DSBs) along $\{111\}\gamma$ planes. Consistent results from Electron Channeling Contrast Imaging (ECCI) reveal that cracks always propagate along planar DSBs in the presence of hydrogen. This phenomenon is rationalized by the evident nucleation of nanoscale voids along the DSBs, especially at the intersections between nonparallel DSBs. The proposed mechanism, confirmed by both the ECCI analysis and fractographic study by Scanning Electron Microscopy, indicates that the interaction between the hydrogen and dislocations along the DSBs leads to void nucleation. Furthermore, the results suggest that coalescence and widening of voids via the dislocation process promote the crack propagation along the DSBs in hydrogen charged Alloy 718.

1. Introduction

Structural materials used in the oil and gas deep wells face extremely aggressive environments, including low pH (H_2S), high temperature (>250 °C) and high pressure (>100 MPa) [1,2]. High strength nickel-based superalloys are promising structural materials for application in these challenging conditions, because of their capability to operate at the high temperature and high pressure conditions providing outstanding corrosion resistance. One longstanding challenge, however, is the hydrogen-induced embrittlement when these alloys are exposed to hydrogenating conditions such as the sour environments of deep wells: adsorption of hydrogen leads to changes in the fracture mode of nickel-based superalloys from ductile to brittle [3–6].

A number of mechanisms have been proposed to account for the hydrogen embrittlement in various materials under different hydrogen charging conditions [7–14]. For the metallic materials that do not form hydrides, such as Fe and Ni, the most commonly invoked embrittlement mechanisms are hydrogen-enhanced decohesion (HEDE) [12,13] and hydrogen-enhanced localised plasticity (HELP) [8,9]. According to the HEDE mechanism, the hydrogen can reduce the cohesive strength of the atomic bonding. Consequently, grain boundaries and precipitate/matrix interfaces that accumulate hydrogen beyond a critical concentration will fracture when the material is stressed. The HEDE mechanism was raised to interpret the load relaxation and intergranular failure caused by hydrogen. Although atomic scale simulations [15–17] support it, the lack of direct experimental evidence places some doubt on the relevance of this mechanism. The HELP mechanism was first suggested by Beachem [8], and was then underpinned by in-situ transmission electron microscopy (TEM) analysis, where the mobility of dislocations was evidently enhanced by the presence of hydrogen [9]. The theoretical basis for the HELP mechanism was rationalized by the hydrogen shielding effect on dislocations [18,19]. In this framework, the dislocation slip mode and dislocation-dislocation interaction are modified and specifically the equilibrium distance between dislocations is decreased [20,21]. Furthermore, dislocation slip planarity is promoted due to the dragging of hydrogen [19]. Accordingly, extensive dislocation slip is expected, and dislocation motion and dislocation-dislocation interaction are largely constrained to the initial slip plane due to limited cross slip in the presence of hydrogen.

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Phenomenologically, hydrogen embrittlement in metallic materials is generally characterised by intergranular cracking with quasi-cleavage fracture surface. Experimental studies have demonstrated that extensive dislocation activities and plasticity are associated with hydrogen embrittlement [11,22–27]. Martin et al. studied the microstructure beneath the quasi-cleavage fracture surface of a ferritic steel [22,23] and pure Ni [24], and found a good correlation between the features on the fracture surface and the underneath dislocation substructure, which indicates the importance of dislocation activities on the hydrogen embrittlement. More importantly, they flagged up that the dislocation substructure in the hydrogen containing material is finer than in the one without hydrogen [24,27,28], which provides further support for the HELP mechanism. Besides the HEDE and HELP mechanisms, the hydrogen enhanced vacancy stabilization mechanism has also been proposed [10,11,29]. In this mechanism, hydrogen is supposed to stabilize vacancies generated by the dislocation activities and promote the formation of voids by the vacancy agglomeration [29]. Although the central aspect of this mechanism is different from that of the HELP mechanism, conceptually, in both cases hydrogen induced fracture originates from, and is promoted by, dislocation activities and therefore the crack nucleation is associated with the regions of extensive dislocation slip activities.

To date, most studies dedicated to understand the mechanism of hydrogen embrittlement of non-hydride forming metallic materials have been conducted on pure nickel [9,24,30] and some steels [22,25–27,31–33]. For nickel-based superalloys, investigations have mainly focused on the fracture mode and the effect of different heat treatments on mechanical properties in regard to hydrogen embrittlement [3–5,34–37]. Since in precipitation-hardened nickel-based superalloys the interfaces between the precipitates (γ', γ'', δ and carbides) and γ matrix are often assumed to be the trapping sites for hydrogen, studies have focused on modifying the size and volume fraction of the various precipitates by specific heat treatments and compare hydrogen embrittlement susceptibility of the resulting microstructures [4,5,36,37]. Even though the activity of dislocations is demonstrated to be of crucial importance for hydrogen embrittlement in several materials, to the authors' knowledge, there is no work carried out to clarify the mechanism on this aspect in nickel-based superalloys.

In the present study, hydrogen-induced embrittlement was studied for the precipitation-hardened nickel-based superalloy UNS N07718 (Alloy 718), which is a widely used structural material for deep well application in the oil and gas industry. The correlation between the microstructural features, especially the dislocation substructure developed during straining, and the crack initiation and propagation was explored in detail by extensive Scanning Electron Microscopy (SEM), high resolution Electron Channeling Contrast Imaging (ECCI), Electron Backscattered Diffraction (EBSD) and TEM characterisations. The observations are discussed in context of previous studies related to hydrogen-induced embrittlement and a mechanistic framework is proposed for precipitation-hardened nickel-based superalloys involving dislocation slip localisation in conjunction with hydrogen promoting vacancy production.

2. Experimental

Cylindrical blanks with a diameter of 8 mm and a length of 60 mm were machined from the Alloy 718 ingot (commercially heat-treated to aerospace specifications) and solution treated at 1040 °C for 1 h followed by the furnace cooling (cooling rate is approximately 15 °C/min). The material was subsequently aged at 774 °C for 6 h followed by cooling in air. To minimise the surface oxidation, all heat treatments were carried out in an argon atmosphere. The purpose of this heat treatment was to generate a condition that is more typical for oil and gas applications.

Tensile samples with a gauge length of 25.0 mm and a diameter of 3.8 mm were machined from the heat-treated cylinders by means of electro discharge machining (EDM). The machined samples were then ground with fine silicon carbide paper to remove the recast layer from the EDM process.

Cathodic hydrogen charging was conducted at 80 °C in a NaCl (1 mol/L) solution. The samples were charged with a constant electric current density of 7.7 mA/cm^2 for 168 h. The penetration depth of the hydrogen can be estimated according to the diffusion equation:

$$x = \sqrt{2Dt} \tag{1}$$

here, t is the charging time, and x is the penetration depth and D is the diffusion coefficient of hydrogen in Alloy 718. D can be calculated based on the equation [38]:

$$D = 4.06 \times 10^{-7} e^{\left(\frac{-48.63\times10^3}{RT}\right)} \tag{2}$$

here, R is the gas constant and T is the charging temperature, i.e. 353 K in the present case. Accordingly, the hydrogen penetration depth in the sample after charging for 168 h is estimated to be ~140 μm.

Both the charged and non-charged samples were slow strain rate (SSR) tested in tension with initial strain rate of 10^{-6} s^{-1} at the room temperature using an Instron machine with a 50 kN load cell. An extensometer was employed to measure the instant strain of the sample during tensioning. The fracture surface of the sample after failure was characterised using a Zeiss EVO60 SEM operating at 20 kV.

A FEI Magellan 400 field emission gun SEM equipped with a concentric backscatter electron detector was employed, which provides ideal imaging conditions for the ECCI analysis with very high resolution and excellent contrast. The gauge section of the hydrogen charged sample strained to failure was mid-sectioned longitudinally, followed by careful grinding and polishing. The sample for ECCI analysis was finished by polishing with 40 nm colloidal silica suspension (OPS, Struers). ECC imaging was conducted on the regions both close to the sample surface (where hydrogen had diffused to) and the sample interior (where hydrogen had not enough time to diffuse to), with a low accelerating voltage (3 kV–5 kV) and a short working distance in the range of 3.5–4 mm to enable a high resolution. EBSD analysis was performed using a FEI Quanta650 field emission gun SEM equipped with a HKL system operating at 20 kV. Orientation maps from the samples were obtained prior to and after the tensile testing. In addition, Kernel average misorientation (KAM) maps, i.e. average misorientation between the each data point and its nearest neighbours, were computed from the EBSD data of the sample after tensioning.

A FEI Tecnai G20 TEM operating at 200 kV was used to characterise the γ' distribution in the initial material and the dislocation substructure after the tension experiment (from the region without hydrogen). TEM foils were prepared by twin-jet electropolishing (TENUPOL5, Struers), in a solution of 10% perchloric acid and 90% methanol at a temperature of −35 °C.

3. Results

The initial microstructure of Alloy 718 after the heat treatment described above is shown in Fig. 1. The grain morphology, which was mapped out by EBSD, indicates an average grain size of about