Hydrogen stress cracking and crack initiation in precipitation hardened Ni-alloys

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A B S T R A C T

The two different precipitation hardened nickel alloys, Alloy 718 and Alloy 725, were tested in its as received condition. Their susceptibility to hydrogen stress cracking was examined by the use of stepwise increasing load testing. The mechanical properties of the two alloys and the effect of hydrogen were studied and compared. Both alloys were proven to be susceptible to hydrogen stress cracking, however it was not possible to distinguish the susceptibility of the two alloys. Investigation of the fracture surfaces revealed a change from pure micro void coalescence failure in the samples tested in air, to a more complex fracture surface for the samples tested under cathodic polarization. Here the area closest to the edge had brittle intergranular and transgranular feature, while the centre was dominated by ductile dimples. This was attributed to the hydrogen concentration gradient through the sample and how this affects which mechanism for hydrogen degradation is dominating. In addition, crack initiation in the presence of hydrogen was investigated through tensile testing in a micro-load cell. This test showed that the cracks initiated near the grain boundaries, and propagated in a transgranular manner. This was related to an observed non-uniform stress distribution in the grains.

1. Introduction

The need for high-strength and corrosion resistant alloys for subsea applications in the oil and gas industry is increasing with the development of new and more demanding oil reservoirs. With a wide range of mechanical properties and good corrosion resistance, precipitation hardened nickel alloys are suitable candidates for such extreme conditions. Several authors have, however shown that these alloys are susceptible to hydrogen embrittlement (HE) [1–14].

Alloy 718 (N07718) and Alloy 725 (N07725) are both examples of precipitation hardened nickel alloys. Common for both is the face-centred cubic (fcc) matrix, giving the alloy a high ductility and high solubility of elements increasing the corrosion resistance, such as Cr and Mo. In addition, small additions of Nb, Ti and Al give, in combination with proper heat treatment, precipitation of finely dispersed phases contributing to the strength of the alloys. In these alloys, the main strength contributing phase is the γ'', which precipitates in the matrix as discs with a diameter of about 70 nm (sensitive to the heat treatment) [5,15–17]. This phase has a Ni_3Nb composition and a body centred tetragonal (bct) $D0_{22}$ structure. The other strength contributing phase is the γ'. This phase has a composition of $Ni_3(Nb, Ti, Al)$ and a L_{12} structure [18–21]. Due to the difference in composition of the two phases, an increase in the Nb to Ti/Al fraction, will also increase the γ'' to γ' fraction. A high Nb content, and prolonged heat treatment will promote the

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formation of the stable δ-phase. This phase has the same composition as the γ″, but it gives no strength contribution to the alloy [22,23].

Alloy 725 and Alloy 718 are closely related in the nickel alloy family. The main difference between them is the higher amount of Cr and Mo in Alloy 725. The increased Cr and Mo content gives a more corrosion resistant alloy. This is reflected in the pitting resistance equivalent (PRE_N) calculated from Eq. (1), where a higher PRE_N represents an alloy with a higher resistance to localized corrosion [24]. The compositions used for the two alloys tested in this work give a PRE_N of 27 and 46 for Alloy 718 and Alloy 725, respectively.

$$PRE_N = \text{wt\%Cr} + 3.3 \times (\text{wt\%Mo} + 0.5\text{wt\%W}) + 16 \times \text{wt\%N} \tag{1}$$

Another important difference is the increased Ti and lower Nb content in Alloy 725 which increases the γ′ to γ″ fraction in the material [25].

As previously mentioned, these alloys have proven to be susceptible to HE [1,2,5–7,12,13]. In published work, slow strain rate testing (SSRT) with and without hydrogen, has been the main method to investigate the alloys susceptibility to hydrogen stress cracking (HSC) and hydrogen assisted cracking (HAC) [26]. This revealed a severe reduction in ductility for both alloys in the presence of hydrogen. In addition, a shift from a complete microvoid coalescence (MVC) fracture with a fracture surface covered in dimples to a mixed fracture mode with brittle transgranular (TG and intergranular (IG) features was observed. In addition, "quasi-cleavage (QC)" are often observed of the fracture surfaces. This term is used for features that cannot be distinguished as pure MVC, TG or IG [27]. The features on the fracture surface of samples tested in hydrogen are often related to the mechanism for hydrogen embrittlement. Early on, the observation of IG features on the fracture surface was considered as a pure decohesion mechanism, and as proof for a hydrogen enhanced decohesion (HEDE) mechanism [28,29]. Later, the observation of plasticity and dislocation activity in these fracture shows that the hydrogen enhance localized plasticity (HELP) mechanism is also playing a role. Either alone, or in combination with the HEDE mechanism [27,30–33]. Although the existing work is very thorough and conclusive, the parameters used for the testing differ from one article to another, making it hard to compare the results. The differences in strain rate, temperature, hydrogen precharging and heat treatment, affects the hydrogen concentration and thereby the results severely [12,34,35]. The change in mechanical properties is a function of the hydrogen concentration in the material, where an abrupt change in these properties is observed when a threshold concentration is exceeded [31]. This abrupt change is considered as the critical hydrogen concentration where HEDE is domination the fracture mechanism [27,30,36]. Different hydrogen contents can therefore provoke different fracture mechanisms, making it very important to keep the hydrogen content constant when comparing the alloys. In order to compare these two alloys directly, comparable tests are done under the same conditions and with the same parameters. In this current work, stepwise increasing load testing of precharged samples with in situ hydrogen charging, were used to test and compare the HSC susceptibility of Alloy 718 and 725. This procedure has previously been used to test the HSC susceptibility of super duplex stainless steel [37]. By using the same parameters for testing both alloys, they can be directly compared with respect to their susceptibility to HSC. In addition, the test procedure itself can be compared to the more established SSRT.

In the present work, the crack initiation in the presence of hydrogen in Alloy 718 is also further investigated. A specially designed loading cell with an environmental chamber is used to load tensile samples while simultaneously observing the sample surface with a microscope. The samples were later investigated by scanning electron microscope (SEM) to further investigate how the different microstructural features played a role in the crack initiation and propagation in the alloy. As already mentioned, the crack initiation and mechanism of hydrogen embrittlement in Ni-alloys are often linked to the HELP and HEDE mechanisms. By using this setup, we hope to directly observe one or both of these mechanisms in action. Fracture surface investigation of SSRT samples tested together with observation of secondary cracks on the outer surface of the samples, gives good indication of the fracture mechanism and crack path [12]. Contributing to this, Seita et al. investigated the effect of hydrogen on the crack initiation and propagation in Alloy 725 [38]. They showed that coherent twin boundaries (Σ3) were susceptible to cracking when hydrogen was present, however, these cracks had a lower likelihood of propagating further. It was therefore proposed that the crack initiation and propagation are governed by two different mechanisms. They linked the crack propagation along grain boundaries to what the call grain boundary connectivity, while the crack initiation was proposed to be linked to hydrogen increasing plastic flow [38]. This seems to be related to the HEDE and HELP mechanism, where they propose that crack propagation along grain boundary is controlled by HEDE, while the initiation is controlled by HELP. Understanding how hydrogen and different microstructural effects affect these mechanisms could give valuable information of how the manufacturing process could be optimized in order to give a more HSC resistant material. Jothi et al. overlaid EBSD maps on SEM micrographs of a hydrogen charged SSRT sample. From this, they showed that low angle grain boundaries did not experience cracking, while grain boundaries with a misorientation between 15° and 50° did [39].

2. Experimental

2.1. Stepwise increasing load tests

The chemical composition of the alloys used in the stepwise increasing load testing is presented in.

Table 1. The tested materials were of commercial qualities, with standard solution annealing and ageing for the specific alloys. For each alloy, three samples were used for standardized tensile testing. In addition, ten samples were used for the stepwise increasing load testing. The samples for tensile testing were produced and tested according to a standardized testing procedure [40]. A modified geometry of that in NACE TM0177 [41] was used for the stepwise increasing load testing. As shown in Fig. 1, the diameter was reduced to 3 mm compared to 3,81 mm in the standard.