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Effect of different microstructural features on the hydrogen embrittlement susceptibility of alloy 718

Gaute Stenerud^{a,*}, Sigurd Wenner^d, Jim Stian Olsen^{b,c}, Roy Johnsen^a

^a Department of Mechanical and Industrial Engineering, NTNU, Richard Birkelands Vei 2b, 7491, Trondheim, Norway

^b Department of Structural Engineering, NTNU, Richard Birkelandsvei 1A, 7491, Trondheim, Norway

^c Aker Solutions, Fornebu, Oslo, Norway

^d Department of Physics, NTNU, Høgskoleringen 5, 7491, Trondheim, Norway

ARTICLE INFO

Article history:

Received 22 June 2017

Received in revised form

17 October 2017

Accepted 14 February 2018

Available online xxx

Keywords:

UNS N07718

Hydrogen embrittlement

Slow strain rate testing

Fractography

Dimples

Intergranular fracture

ABSTRACT

Due to its high strength and acceptable corrosion resistance, Alloy 718 is a viable material for the use in subsea applications. The alloy's susceptibility to hydrogen embrittlement is however limiting its viability. In this work the effect of microstructure on the hydrogen embrittlement susceptibility of Alloy 718 was examined by the use of slow strain rate testing on three different heat treatments. Cathodic pre-charging and polarization during testing were used to introduce hydrogen into the samples. A severe reduction in the ductility of the alloy due to the precipitation phases γ'' and γ' were found. In addition, one of the heats had a continuous film of a Nb and C rich phase on the grain boundary giving an intergranular fracture mechanism. This intergranular fracture mode was further enhanced by the presence of hydrogen. In the solution annealed condition, the presence of hydrogen reduced the dimple size on the fracture surface.

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Introduction

The exploration of new and more demanding oil reservoirs has increased the need for high performing alloys for subsea applications. Precipitation hardened nickel alloys are examples of such alloys, having a wide range of favourable mechanical properties and corrosion resistance. One example is Alloy 718, which was first developed for the aerospace industry, but has lately been used for both nuclear and subsea oil and gas applications [1,2].

The chemical composition of Alloy 718 gives an acceptable corrosion resistance and a potential for high strength [2]. High amounts of Nb, Ti and Al are supersaturated in the face-centred cubic (fcc) matrix after standard solution annealing. Prolonged heat treatment in the temperature range 600–900 °C will cause these elements to precipitate in the form of coherent dispersoids, producing coherency strains in the matrix. These strains act as obstacles to dislocation movement and increase the strength of the alloy [3,4]. The degree of hardening obtained by the precipitates is dependent

* Corresponding author.

E-mail address: gaute.stenerud@ntnu.no (G. Stenerud).

<https://doi.org/10.1016/j.ijhydene.2018.02.088>

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on the amount of precipitates, the size, their mismatch with the matrix and the hardness of the precipitates [3]. The size and amount can easily be controlled by heat treatment, making these alloys very versatile [5]. Solution annealing will give a ductile material with low strength, while ageing the material in the above-mentioned temperature range will increase the strength and decrease the ductility of the alloy. The strength will reach a maximum when an ideal compromise between precipitate size and quantity is obtained [6]. After this, the precipitates will grow on the expense of each other, resulting in fewer, larger precipitates less able to pin the dislocation movement. Another possible result of over-ageing is transformation to a stable, incoherent phase with no strength contribution to the alloy [7].

There are mainly two precipitating phases contributing to the strength of Alloy 718. The main strength contributing phase is the γ'' , a body centred tetragonal (bct) DO_{22} phase with a composition of $\text{Ni}_3(\text{Nb}, \text{Ti}, \text{Al})$ [4,6–14]. This phase precipitates as discs with a $(001)_{\gamma''} \parallel [001]_{\gamma}$ and $[100]_{\gamma''} \parallel \langle 100 \rangle_{\gamma}$ orientation relationship with the matrix (γ) [15]. In addition, the γ' phase will precipitate during heat treatment. The γ' phase has a cubic L_{12} structure with a composition of $\text{Ni}_3(\text{Al}, \text{Ti})$ and precipitates with a cube to cube relationship with the matrix [16]. Due to the composition of the alloy, there will be more γ'' than γ' in the alloy [17]. Other important phases present are the δ -phase (Ni_3Nb , DO_a), primary carbides (MC) and carbonitrides. The stable δ -phase is generally unwanted in the material due to its negative effect on the ductility, however it can be deliberately introduced during thermo-mechanical treatments due to its grain boundary (GB) pinning abilities [18–20].

The above-mentioned properties of Alloy 718 make it a viable option where more conventional alloys fail. This alloy has, however, proven to be susceptible to hydrogen embrittlement (HE) [21–28]. An alloy's susceptibility to HE is closely related to its microstructure. In the case of Alloy 718 it is interesting how the strengthening phases are affecting this susceptibility. From the literature, it is known that certain precipitates or microstructural features can act as hydrogen traps in the material [29–32]. A trap site can be considered as a low energy site for the hydrogen in the material. The lower energy the hydrogen has in this site, the stronger is the trap. It is normal to differ between reversible and irreversible traps. An irreversible trap is a defect where the hydrogen has sufficiently low energy to be trapped irreversibly under the conditions of the test [29,33]. The threshold between a reversible and irreversible trap will therefore vary with the conditions of the experiments. A theory proposed by G.M. Pressouyre connects irreversible traps to the materials susceptibility to HE [33]. He proposed that uniformly distributed irreversible traps with high saturation level (amount of hydrogen that can be trapped) are beneficial to the HE resistance by reducing the amount of hydrogen that can diffuse to critical sites. Reversible traps, on the other hand, can store hydrogen until it is energetically favourable for the hydrogen to diffuse further. For example, applying a tensile load on a system, will cause stress raisers at potential flaws. Hydrogen from nearby reversible traps can diffuse to such stress raisers, and facilitate crack nucleation [33–36]. In such a case the reversible traps act as hydrogen sources, increasing the HE susceptibility

of the material. As described earlier, the microstructure of Alloy 718 contains several heterogeneities that may act as traps. In Alloy 718, the primary MC carbides and the carbonitrides are considered to be strong, irreversible traps [37]. According to Pressouyre's theory, these heterogeneities should not increase the HE susceptibility of the alloy. In the case of the other heterogeneities, the γ'' , γ' and the δ -phase are all considered to be reversible traps, which again can increase the HE susceptibility of the alloy [22,38]. In these models, the traps are assumed to be uniformly distributed and the availability of hydrogen to be limited. If for instance the irreversible traps are preferably formed at grain boundaries, the effect may be reversed.

A lot of experimental work has been done to connect the precipitates in Alloy 718 to its HE susceptibility [21–24,27,37,39,40]. In all cases where the δ -phase has been under investigation, a deleterious effect on the HE resistance has been observed [21–23]. The reason for this effect is unknown but it could be related to the previously discussed trapping phenomenon. In the case of the hardening phases γ'' and γ' a similar effect has been documented, but the effect is not as pronounced as for the δ -phase. Being the main hardening phases in the alloy, high strength heats will contain more γ'' and γ' compared to the heats with lower strength. Increased strength may therefore increase the HE susceptibility as well. Effects of other microstructural features such as grain size, grain boundary segregation and elements in solid solution may also affect the HE susceptibility [41]. This has not been thoroughly investigated, but work by Sjöberg and Cornu indicates that a smaller grain size increases the HE susceptibility of the alloy [40]. The effect of grain size is however a controversial point. Both improved and reduced HE resistance with grain refinement has been found in other materials [42].

The motivation for the present work was to increase the insight of the combined effect of hydrogen and certain microstructural features on the HE susceptibility of Alloy 718. This was done by slow strain rate testing (SSRT) of parallels with different heat treatments. A SSRT setup, with a pre-determined strain rate and reproducible conditions, gives results from the different parallels that can be directly compared. Tests were performed in air, or submerged in electrolyte with cathodic polarization to promote hydrogen evolution. Finally, high resolution microscopy techniques such as scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were used to examine both the microstructure and the fracture surfaces.

Experimental and material

For this work an alloy with composition within the UNS N07718 (Alloy 718) standard was used. The actual composition of the tested alloy is shown in Table 1. As received from the supplier, the alloy was aged for 8 h at 720 °C and 8 h at 620 °C before air cooling. This is a standard heat treatment procedure and results in a good compromise between strength and ductility [43]. From the received material three dog-bone shaped pieces were cut by electric discharge machining (EDM). These three pieces were subject to different heat

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