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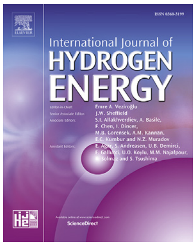

Hydrogen embrittlement of super duplex stainless steel – Towards understanding the effects of microstructure and strain

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ABSTRACT

The effects of austenite spacing, hydrogen charging, and applied tensile strain on the local Volta potential evolution and micro-deformation behaviour of grade 2507 (UNS S32750) super duplex stainless steel were studied. A novel in-situ methodological approach using Digital Image Correlation (DIC) and Scanning Kelvin Probe Force Microscopy (SKPFM) was employed. The microstructure with small austenite spacing showed load partitioning of tensile micro-strains to the austenite during elastic loading, with the ferrite then taking up most tensile strain at large plastic deformation. The opposite trend was seen when the microstructure was pre-charged with hydrogen, with more intense strain localisation formed due to local hydrogen hardening. The hydrogen-charged microstructure with large austenite spacing showed a contrasting micro-mechanical response, resulting in heterogeneous strain localisation with high strain intensities in both phases in the elastic regime. The austenite was hydrogen-hardened, whereas the ferrite became more strain-hardened. SKPFM measured Volta potentials revealed the development of local cathodic sites in the ferrite associated with hydrogen damage (blister), with anodic sites related to trapped hydrogen and/or micro voids in the microstructure with small austenite spacing. Discrete cathodic sites with large Volta potential variations across the ferrite were seen in the coarse-grained microstructure, indicating enhanced susceptibility to micro-galvanic activity. Microstructures with large austenite spacing were more susceptible to hydrogen embrittlement, related to the development of tensile strains in the ferrite.

Introduction

Duplex stainless steels (DSS) provide exceptional corrosion resistance with high mechanical strength, with the material often employed in demanding industrial applications such as energy, marine, petrochemical, and oil and gas. There is a synergic behaviour believed to exist between ferrite and austenite in duplex microstructures, which lead to superior electrochemical properties to most ferritic and austenitic

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counterpart steels. However, DSS's are prone to hydrogen infusion, in particular under cathodic protection, causing hydrogen embrittlement (HE) and/or hydrogen-induced stress cracking (HISC).

Hydrogen in steels has been reported to increase strength [1,2] as a compromise of severe loss of toughness [3]. Hydrogen diffusion in body-centred cubic (BCC) structures occurs faster than in face-centre cubic (FCC) lattices, but the solubility is larger in the austenite than in the ferrite [2,4,5]. Embrittlement due to hydrogen has been reported due to an increase of the dislocation density in both austenite and ferrite phases in DSS microstructures [6]. The cause of embrittlement has also been ascribed to phase transformation from austenite to martensite [1,4,7,8]. ε-martensite was observed in 21Cr-1Ni and 22Cr-5Ni DSS after electrochemical cathodic charging, which transformed to α'-martensite due to strain releasing effects and elastic stresses during hydrogen desorption (outgassing) [4,7]. Lattice expansion of up to 2% was observed in the austenite occurring after hydrogen charging, resulting in surface damages [4]. These findings questioned the immunity of austenite to HE. Thus, the austenite in super duplex stainless steel (SDSS) may also be susceptible to martensite transformation, ultimately resulting in HE/HISC.

The superior mechanical properties of DSS's are due to a well-balanced interplay between ferrite and austenite grains. The austenite in SDSS's is usually harder due to high nitrogen contents, and therefore plastic deformation typically sets in on ferrite grains [9–12]. Dislocations, however, can easily jump over interface boundaries to austenite grains which can more easily slip due to more close-packed systems [10]. The deformation process is alternating between plastic deformation of ferrite and austenite until failure, and therefore the load share among the grains of both phases has utmost important, which is decisive in determining the toughness as well as the strength of DSS's. Hydrogen may affect the load partitioning among ferrite and austenite, causing embrittlement and premature failure of structural components [13].

Load partitioning among the phases in DSS microstructures is a well-known phenomenon due to the different thermal expansion coefficients and deformation characteristics of ferrite and austenite [10,14–16], leading to internal micro-stresses during quenching [17]. Internal stresses are unavoidable and require the need for optimisation of (final) heat treatments [18,19]. The interaction of the microstructure with hydrogen further complicates the matter as hydrogen can alter the stress triaxiality in both phases, which often results in large variations in macro- and micro-stresses over the entire microstructure [11,18,20,21]. Hydrogen can further cause residual damage such as blister formation [22] or twinning [23] in the ferrite and dislocation multiplication [13] and local amorphisation [24] in the austenite, often resulting in crack nucleation due to local embrittlement [13]. The microstructure performance of DSS's is usually improved by a sequence of hot and cold rolling, resulting in a layered structure of austenite and ferrite, both having equiaxed and texture-free grains [25,26].

Hydrogen interaction with DSS microstructures varies depending on the austenite spacing, grain size, and phase ratio of ferrite to austenite [13,20,27,28]. A reason for the pronounced microstructure dependence of HE is the difference in the degree of hydrogen trapping and localisation [29]. Quantification of local hydrogen content and association of hydrogen with the microstructure have key importance to understand and characterise the effect of HE or HISC. However, the interaction of hydrogen and strain and the resulting effects on the load sharing between ferrite and austenite as well as the electrochemical nobilities have more practical relevance, since hydrogen-enhanced cracking is not simple mechanical embrittlement only, but also related to electrochemical reactions, such as localised corrosion (anodic metal dissolution) and the cathodic hydrogen evolution.

There is a permanent diffusion and redistribution of hydrogen in the microstructure, and diffusible hydrogen has been known to cause embrittlement in steels [2]. There are, however, also a wide range of potential trapping sites such as vacancies, dislocations, precipitates, grain boundaries, phase interfaces, triple points, micro- and nano-cracks, voids, and other defects that bind hydrogen and/or accelerate its transport [2,29]. Hydrogen segregation depends on the stress triaxiality, the diffusion distance between inner and outer surfaces, and microstructural features that lie hidden underneath the surface [29]. The interaction of hydrogen can become more complicated if mechanical load is superimposed on the material. Therefore, the interaction between hydrogen and strain needs to be assessed in order to understand microstructure susceptibility to HE and/or HISC.

The aim of this work was to study the effects of austenite spacing in grade 2507 SDDS on load partitioning between ferrite and austenite. Key aim was also to correlate the local Volta potential development in microstructures with small and large austenite spacing using an in-situ mechanical loading technique coupled with Digital Image Correlation (DIC) to evaluate the change of the corrosion tendency (nobility) caused by hydrogen. The microstructures were mechanically loaded and imaged, in-situ, using optical microscopy to obtain information about micro-strain development in both phases. DIC was used to visualise and quantify the strain development in both phases. SKPFM was applied to measure the local Volta potential during mechanical loading. Strain localisation and Volta potential evolution on fine- and coarse-grained microstructures, both with and without prehydrogen charging, were investigated and are discussed in light of better understanding of microstructure susceptibility to HE/HISC.

Experimental

Materials used

Two grade 2507 (UNS S32750) SDSS's were used in this study. The wrought materials had different austenite spacing, and were supplied in the solution-annealed microstructure condition. The material with small austenite spacing was obtained as plate material with 7 mm and 10 mm thicknesses, whereas the materials with large austenite spacing was a cylinder ingot (bar material) with a diameter of 70 mm. The nominal chemical composition of the materials is given in