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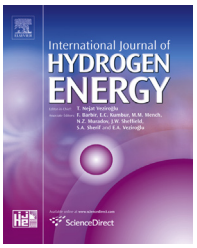

Role of lattice strain and texture in hydrogen embrittlement of 18Ni (300) maraging steel

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ABSTRACT

Hydrogen embrittlement causes engineering components to fail unexpectedly. Maraging 300 steel was hydrogen charged and subjected to slow strain rate tensile test until fracture. Electron backscatter diffraction analysis of fractured specimen revealed that cracks initially propagated intergranulary along prior-austenite grain boundaries. When cracks faced martensitic $\{111\}\alpha$ planes parallel to normal direction (ND) they were deflected and continued to propagate transgranulary through $\{001\}\alpha//$ND planes. Finally, cracks were arrested by $\{111\}\alpha//$ND planes. Crystallographic planes on which cracks propagate/are arrested, correlate well with planes that exhibit highest/lowest magnitude of lattice strain determined during tensile loading using *in situ* synchrotron X-ray diffraction.

Introduction

High-strength maraging steels are extensively used in the nuclear power, chemical processing and aerospace industries, in applications where they are susceptible to hydrogen embrittlement (HE) [1–11]. The HE is typically characterised by a significant loss of tensile ductility and causes engineering components to fracture unexpectedly. It can be affected by microstructure, hydrogen content, strain rate, temperature, level of applied stress in addition to magnitude of residual stress [8,12–15]. The most common hypotheses that are used to explain the HE mechanism include (i) hydrogen-enhanced vacancy clusters formation [16], (ii) hydrogen-induced decohesion [17] and (iii) hydrogen-enhanced localised plasticity (HELP) [18]. In case of martensitic maraging steels, the later mechanism is the predominant failure mode [19]. This mechanism is based on the accumulation of hydrogen at

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dislocations, precipitates and grain boundaries [20]. Dissolved hydrogen enhances dislocation mobility and reduces number of available slip systems because the cross slip is hindered [12,21]. Since the deformation becomes more localised, in that region the propensity for cracking is enhanced [21]. When exposed to hydrogen, high-strength steels may fail either by intergranular separation along the prior-austenite grain boundaries [22] or by transgranular fracture along planes which traverse the prior-austenite grains [23]. Intergranular, quasi-cleavage, or microvoid coalescence fracture modes can operate depending upon the microstructure, the crack-tip stress intensity, the concentration of hydrogen and dislocation interactions with grain boundaries [8,12]. Intergranular decohesion can be caused by the grain boundary segregation of impurity elements and hydrogen [24]. The transgranular fracture in body-centred cubic (BCC) crystals involves the separation of atomic bonds along low-index {001} crystallographic planes which are considered energetically favourable due to their low surface energy [25]. According Qiao and Argon [26], as cleavage crack propagation occurs in two adjoining grains a crack propagates in the first grain and is arrested by the grain boundary. With the increased applied stress cleavage microcracks are induced in the second grain along the {001} facets. Then, microcracks propagate along the cleavage facets of the second grain until bridging the cleavage crack in the first grain and break the grain boundary. Finally, the crack continues to propagate in the second grain with its perturbed crack front.

Although crystallographic texture is likely to reduce hydrogen-induced cracking because it can determine availability of low resistance paths for crack propagation [27–31] a limited work has been conducted to date. An understanding of crystallographic texture and load interaction effects at a crack-tip can play a key role in improvement of HE resistance of critical engineering components.

Recently, Venegas et al. [28,29,32] demonstrated that high resistance to HE can be achieved through tailoring of texture. In these studies, a low carbon steels with crystallographic texture dominated by $\{001\}\alpha//ND$ fibre was prone to HE due to the availability of low resistance cleavage paths. In contrast, texture composed of $\{112\}\alpha//ND$, $\{111\}\alpha//ND$ and $\{011\}\alpha//ND$ fibres was less susceptible to HE.

When a polycrystalline material is subjected to deformation, slip occurs initially in grains orientated favourably with the load axis. The neighbour grains, aligned in unfavourable orientation for slip, will experience local load increase generating intergranular microstrain [33]. Neutron or synchrotron X-ray diffraction can be used to study lattice strains in a polycrystalline material from the shift of the diffraction peaks [34,35].

This work examines the role of lattice strain accumulation in individual crystallographic planes on hydrogen-induced crack propagation in maraging steel. We measured the lattice strain in maraging steel using synchrotron X-ray diffraction during *in situ* tensile loading and found correlation between lattice strain and crack propagation. The results obtained can be used to better understand the damage mechanism caused by hydrogen and for improvement of the material's resistance to hydrogen-induced crack propagation.

Experimental

The studied material was commercial maraging steel grade 300 containing 18.7% Ni, 9.6% Co, 4.8% Mo, 0.9% Ti, balance Fe (wt.-%). Cylindrical tensile test samples with a gauge length of 28 mm and diameter of 4.1 mm were machined from a forged bar 300 mm in diameter. The specimens were subjected to solution annealing at 820 °C for 1 h followed by air cooling to ambient temperature and then ageing treatment at 480 °C for 3 h and air cooling. The heat treated samples were then grounded using SiC paper with mesh size up to 1200, followed by polishing with 6, 3 and 1 μm diamond paste. Finally, they were etched with Marble's reagent consisting of 10 g $CuSO_4$ + 50 ml HCl + 50 ml distillated water [36] and examined using Olympus® BX-51M optical microscope. X-ray diffraction measurements were carried out on the heat treated samples using a Philips® X'Pert Pro diffractometer. Step scan mode with step size 0.013°, time per step 100 s and angular interval 10–120° was applied. Cu K_α radiation (wavelength of 0.154 nm) was used in addition to operating voltage and current of 40 kV and 45 mA, respectively. In order to minimize the texture effect a spinner holder was used.

The heat treated samples were cathodically charged for 24 h prior to loading and during the on-going slow strain rate test in aqueous 0.6 M NaCl electrolyte. For the hydrogen charging, a potential of $-1.2\ V_{SCE}$ according the ASTM G129-00 and ASTM F1624-09 standards [37,38] was applied. The slow strain rate tests were conducted using a strain rate of $1.0 \times 10^{-6}\ s^{-1}$ in a Cortest® tensile test machine. Further experimental details of this test can be found in work carried out by Santos et al. [39]. Relative strength and ductility losses were chosen to identify HE susceptibility.

Synchrotron X-ray diffraction was used to measure the lattice strain accumulated in different crystallographic planes during tensile loading. This test was performed on specimens without hydrogen charging, which were subjected to identical heat treatment as shown in the previous section. For *in situ* determination of the lattice strain, samples with cross-section of $4 \times 4\ mm^2$ were deformed to failure at room temperature in tension in the Gleeble Thermomechanical Simulator integrated within the XTMS beamline at the Brazilian Synchrotron Light Laboratory, in Campinas. The Gleeble system operated in stroke control mode, with macroscopic force applied to the sample recorded using a 44 kN load cell. The tensile specimens were positioned perpendicular to the diffraction beam, continuously loaded to a selected stroke using a strain rate of $1 \times 10^{-3}\ s^{-1}$ and held for ~300 s. A monochromatic X-ray beam with dimensions at the slit system of $2.0 \times 0.5\ mm^2$ and wavelength of 1.0332 Å (12 keV) was used to illuminate the sample during loading. Then a scan in angular range of 21–83° (covering an interplanar *d*-spacing range of 0.78–2.83 Å) was acquired before a subsequent stroke step was applied. The diffraction patterns were acquired using two silicon microstrips MYTHEN one-dimensional detectors. The instrument parameters were obtained using Al_2O_3 powder standard. A laser dilatometer was used to record changes in sample cross section at gauge centre during straining.

The measured data consisted of a series of diffraction patterns as function of applied load and strain. The positions

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