



# The influence of hydrogen on the mechanical and fracture properties of some martensitic advanced high strength steels studied using the linearly increasing stress test



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## ABSTRACT

The influence of hydrogen on the mechanical and fracture properties of four martensitic advanced high strength steels was studied using the linearly increasing stress test and electrochemical hydrogen charging. The hydrogen influence increased with steel strength, decreasing charging potential, and decreasing applied stress rate. Increased hydrogen influence was manifest in (i) the decreased yield stress attributed to solid solution softening by hydrogen and (ii) the reduced macroscopic ductility, and by the change from ductile cup-and-cone fracture to macroscopically brittle shear fracture, attributed to a dynamic interaction of hydrogen with the dislocation substructure somewhat similar to the HELP mechanism.

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## 1. Introduction

Advanced high strength steels (AHSS) are new steels created for the automotive industry [1–4] to decrease vehicle weight, and an improved vehicle crash resistance [4]. AHSS differ from conventional auto steels in that they have higher strength, typically greater than 600 MPa, and often between 800 and 1200 MPa [5]. This higher strength is achieved by relatively complex metallurgy. Research on AHSS began in the early 1980s, and led to the creation of the first-generation AHSS [6,7]. These AHSS were ferrite-based, and examples include dual-phase (DP), martensitic (MS), complex phase (CP) and transformation-induced plasticity (TRIP) steels [8]. Subsequent research is developing the third-generation AHSS [8–10].

The martensitic AHSS (MS–AHSS) are the strongest, but exhibiting the lowest ductility [11]. Strength and hardness increase with increasing carbon content, whereas the ductility and toughness decrease with increasing carbon content. The lack of ductility also limits the formability of these steels, which is important, because auto bodies are mechanically shaped from sheet steel. Nevertheless, MS–AHSS are important because they have the highest strength-to-price ratio among the AHSS [8]. MS–AHSS find applications in the parts of the vehicle which require good crash resistance,

such as bumper beams and reinforcements, door intrusion beams and reinforcements, windscreen upright reinforcements, and B-pillar reinforcements [1,12–14].

Hydrogen embrittlement (HE) has long been the bane of high-strength steels [15–17]. HE is a failure mode caused by the presence of a relatively small amount of hydrogen. HE may trigger catastrophic failures at relatively-small applied loads, or may cause degradation of ductility and toughness. Recent studies have revealed some HE susceptibility for some AHSS [18–22]. However, past attempts to predict HE resistance based on the microstructure, composition and processing, have not been successful. Hence, a much deeper understanding of how hydrogen interacts with steel is essential to reduce or eliminate HE in AHSS.

Several mechanisms have been proposed for HE. For non-hydride forming metals such as steel, the following three mechanisms are the most likely: (i) hydrogen-enhanced decohesion (HEDE), (ii) hydrogen-enhanced local plasticity (HELP), and (iii) adsorption-induced dislocation emission (AIDE) [16]. The HEDE mechanism proposes that hydrogen causes a reduction in the cohesive bond strength between metal atoms, leading to weakness under tensile load, and causing brittle crack propagation [23,24]. The HELP mechanism proposes that the presence of hydrogen increases dislocation mobility, causing highly localized plastic deformation [25]. Since the plastic deformation is concentrated in a small volume, the total macroscopic ductility is low. The HELP mechanism has considerable support [15,26,27], even though, at first blush, the terms enhanced plasticity and embrittlement appear

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contradictory. The AIDE mechanism is similar to the HELP mechanism, in that AIDE also involves localized plasticity. The key difference is that AIDE proposes that the localized plasticity occurs close to the surface. The AIDE mechanism proposes that hydrogen is adsorbed at the surface, at regions of stress concentrations like crack tips [28], and that the hydrogen triggers the release of dislocations from the advancing crack tip, causing crack growth, and intense deformation in the crack vicinity.

The linearly increasing stress test (LIST) was developed by Atrens et al. [29] for the study of HE and stress corrosion cracking (SCC). The LIST subjects a smooth specimen to a linearly increasing load until the specimen fractures [30]. The specimen may be tested in air, or when exposed to an embrittling environment. The LIST is load controlled, whereas the related constant extension rate test (CERT) is strain controlled. Both tests are identical until yielding, or the initiation of sub-critical cracking. Thereafter, the LIST is concluded more rapidly, whereas a considerable length of time is taken in a CERT, whilst cracks open up relieving the applied strain, and the specimen extends. Winzer et al. [31] showed that both tests can provide similar information, if instrumented, to evaluate the threshold stress for crack initiation by HE or SCC, although CERTs are rarely so instrumented. The LIST has been successfully applied to different types of steels, such as plain carbon [32,33], alloy [33,34], micro-alloyed [33,35], high strength [33,36], and medium strength steels [37,38]. To the knowledge of the authors, there has been no investigation of the influence of hydrogen on the mechanical properties of MS–AHSS using the LIST.

In summary, LIST has the following advantages. The LIST allows measurement of the yield stress and the threshold stress for crack initiation by hydrogen or by stress corrosion cracking. This quantity is potentially a design allowable, which could be used in the assessment of structural integrity of a component in service. This also allows quantification of the degree of hydrogen influence in terms of the reduction of the threshold stress, as well as in terms of the decrease in ductility. Furthermore, the LIST is considerably quicker than a CERT at the same applied stress rate.

The aims of the present work were: (i) to investigate the influence of hydrogen on the mechanical properties (particularly the threshold stress for crack initiation and the ductility) and fracture processes of four commercial martensitic AHSS, (ii) identify the mechanisms of HE involved in each fracture process, and (iii) determine the environmental limits of the usability of these steels.

## 2. Experimental methodology

### 2.1. Steels

The four martensitic AHSS were commercially produced, in the form of rolled-sheet. The four steel grades were given the following designations: MS980, MS1180, MS1300, and MS1500. The mean sheet thicknesses were 1.2 mm for MS1300 and MS1500, 1.5 mm for MS980, and 1.8 mm for MS1180. Table 1 presents the chemical and mechanical properties of the steels, as provided by the steel producer. From MS980 to MS1500, the yield and tensile strengths increased, the ductility decreased, there was a slight increase in the carbon content, and the concentration of the other alloying elements was essentially constant.

### 2.2. Microstructure analysis

Representative samples were metallurgically mounted using a conductive polymer resin; ground using a series of emery or silicon carbide (SiC) papers starting from 300 grit, followed with 600 grit, and finishing with 1200 grit paper; rough polished using 3 and 1  $\mu\text{m}$  diamond; final polished using colloidal silica; etched with 2%

nital, and viewed with a light microscope (LM), and with a scanning electron microscope (SEM). The microstructure of each steel consisted mainly of ferrite and martensite. The relative amounts of each phase were evaluated using point analysis as described in ASTM E562 [39]. The grain size was estimated using the microscope calibration bar.

### 2.3. Linearly Increasing Stress Tests (LISTs)

Fig. 1 presents a typical LIST specimen. The specimen thickness corresponded to the steel sheet thickness. These specimens were machined from the steel sheet, and polished with 1200 grit SiC paper. The long direction of the specimen coincided with the rolling direction of the steel sheet.

Fig. 2 presents a schematic of the LIST apparatus [29]. The loading train for the specimen was on the left hand side of the lever beam. This allowed the specimen to be exposed to the environment. A 14 kg weight, on the other side, was moved, starting at the equilibrium position, along the lever beam by a synchronous motor, and resulted in the application of a linearly increasing (engineering) stress to the specimen. The applied (engineering) stress on the specimen was given by [29]:

$$\sigma = \frac{(13720)d}{A} \quad (1)$$

where  $d$  (m) was the position of the movable weight from the zero load condition, and  $A$  was the specimen cross-section area ( $\text{mm}^2$ ) of the gauge section. The numerical factor in Eq. (1) depended on the mechanical advantage, the mass of the movable weight (14 kg) and  $g$ , the acceleration due to gravity. The applied stress rate could be specified by the choice of the synchronous motor driving the weight from the equilibrium position.

LISTs were carried out in laboratory air, and with electrochemical cathodic charging at  $-1.200$ ,  $-1.500$  and  $-1.800 \text{ V}_{\text{Ag}/\text{AgCl}}$  in a 0.1 M NaOH (pH 12.6) solution. Increasingly negative potentials represent increasing hydrogen fugacity. The electrochemical charging was carried out using a typical three-electrode cell as shown schematically in Fig. 3. The LIST sample was the working electrode, the counter electrode was a platinum mesh, and the reference electrode was Ag/AgCl saturated with KCl. The applied cathodic potential was controlled using a Bank Elektronik MP81 potentiostat. The solution was prepared using analytic grade reagent and distilled water.

Before each LIST, to ensure an equilibrium and constant hydrogen content throughout the specimen, the specimen was hydrogen pre-charged at the charging potential in the 0.1 M NaOH solution for 24 h, with a static stress of 20% of the yield strength applied to the specimen.

The applied stress rates on the samples varied according to the speed of the synchronous motor. Three motors were used with speeds of 3, 30 and 300 revolutions per hour (rph). The 300-rph motor produced the fastest applied stress rate, the 30-rph motor produced the intermediate rate, and 3-rph motor produced the slowest rate. The applied stress rates depended also on the specimen dimensions. The 300-rph motor produced applied stress rates of 0.54, 0.64, and 0.8  $\text{MPa s}^{-1}$ , the 30 rph motor produced applied stress rates of 0.054, 0.064, and 0.08  $\text{MPa s}^{-1}$ , and the 3 rph motor produced applied stress rates of 0.0054, 0.0064, and 0.008  $\text{MPa s}^{-1}$ . An applied stress rate of 0.8  $\text{MPa s}^{-1}$  corresponds to a strain rate of  $3.9 \times 10^{-6} \text{ s}^{-1}$  in the initial elastic part of the Linearly Increasing Stress Test (LIST), whereas an applied stress rate of 0.0054  $\text{MPa s}^{-1}$  corresponds to a strain rate of  $2.6 \times 10^{-8} \text{ s}^{-1}$  in the initial elastic part of the LIST.

The specimen designation indicated the type of steel, the environment, and the speed of the synchronous motor, which provided an indication of the applied stress rate. Thus, the sample designa-

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