



Evaluation of the hydrogen embrittlement susceptibility in DP steel under static and dynamic tensile conditions

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ABSTRACT

Hydrogen (H) induced mechanical degradation is studied in DP steel by performing tensile tests under static and dynamic conditions. Tensile specimens were electrochemically H charged and tensile tests were done ex-situ after charging. Different H contents were charged into the samples by modifying the current density. The strain rate is increased from static (1.67×10^{-2} and 1.67 s^{-1}) to dynamic (450 and 900 s^{-1}) conditions to verify the effect of H diffusivity during the tensile tests on the hydrogen embrittlement (HE) susceptibility. Therefore, a reproducible methodology was established by using a standardized tensile machine for static testing and split Hopkinson bar experiments for dynamic conditions. The HE degree increased with current density due to higher amount of H, as confirmed by melt extraction. The HE% also increased with slower strain rates since H was able to diffuse to a crack tip, hence accelerating failure. Even at the highest strain rate (900 s^{-1}), the material lost about 10% of its ductility due to H present in the sample and not because of H diffusion during the test. This was concluded since H induced brittle failure initiated at the edges of the samples at low strain rates. Though at a strain rate of 1.67 s^{-1} , fracture initiated in a ductile way from the center similarly as for tests performed without charging. Fractographic visualization of the fracture surfaces revealed an embrittled central line when charged with H, which evolved into a major crack. MnS inclusions were found in this central line accounting for the H induced crack initiation.

1. Introduction

In the automotive industry, advanced high strength steels (AHSS), such as dual phase (DP) steels, are widely used due to their outstanding combination of both formability and strength. The use of these AHSS has been stimulated since these materials can both guarantee an increased safety together with the required weight reduction to meet the rigorous CO₂ emission regulations. DP steel has a ferrite-martensite microstructure with low yield strength, but a high work hardening exponent at low and moderate deformation levels leading to a high ultimate tensile strength. The typical characteristics of DP steel are determined by its thermo-mechanical processing which incorporates a volume fraction of austenitic islands transforming into martensite. Hence, the corresponding strength level is predominantly dependent on the volume fraction and the hardness of the martensite phase in the DP structure. Additionally, a volumetric expansion of martensite into the ferrite matrix during production results into residual stresses at the interface between martensite and ferrite. Therefore, a high density of mobile dislocations is present in the soft ferrite contributing to excellent

continuous yielding. This means that there is no yield point phenomenon and no formation of Lüders bands.

Although the mechanical properties of AHSS are excellent, they are unfortunately considered to be prone to hydrogen (H) induced mechanical degradation. Moreover, further applicability of AHSS has been reported to be hindered by the H phenomenon [1]. Essentially, in the presence of H, these materials undergo a large ductility loss, mainly referred to as hydrogen embrittlement (HE). Amongst others, DP, transformation induced plasticity (TRIP) and high strength low alloyed (HSLA) steels are widely used grades in the automotive industry and their interaction with H has therefore already been subject of numerous researches [2–7]. The detrimental effect of H on the mechanical properties was specifically considered by Depover et al. [8]. A rather low HE sensitivity of about 8% was found for a HSLA steel which was correlated to the beneficial presence of Ti and Nb based carbo-nitrides. A DP and TRIP steel, however, lost about 54% and 60% of their initial ductility, respectively. Duprez et al. [9] also performed tensile tests on these materials immediately after electrochemical H charging and on samples that were atmospherically discharged for one week. Their main

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conclusion was that the H induced ductility loss for DP and TRIP steel was mainly reversible since a large part of the ductility was recovered after discharging the tensile specimen for one week, whereas for the HSLA steel, the difference between immediate and after one week testing was limited. These observations were further correlated with the findings by Pérez Escobar et al. [10], who performed a thermal desorption spectroscopy (TDS) study on these high strength steels. Different times in vacuum, before the TDS measurement started, were applied to evaluate the H effusion. They detected that H present in TRIP and DP steel was mainly diffusible, leaving the material easily when kept in vacuum, whereas the opposite observations were made for HSLA. Additionally, the obtained activation energies for the different traps in these AHSS were in the same range, indicating the complex correlation between a certain microstructural feature and a specific obtained desorption peak. Only the TRIP steel showed a high temperature peak with an E_a of 90 kJ/mol which was proved to come from hydrogen trapped in the retained austenite [11]. Furthermore, a detailed microstructural analysis was performed on this material by Laureys et al. [4, 5] showing that crack initiation occurred in the martensitic islands, mainly along the martensitic lath interfaces, and that crack propagation was mainly stress driven.

Nevertheless, the significant contrast in mechanical behavior between the ferrite and martensite phase in DP steels induces certain jeopardies, especially in the presence of H [12]. Sun et al. [13], attributed the relative high sensitivity of DP steels to HE to sub-structural changes, because cracks initiated along the interface and lath boundaries when H charging was applied. However, Gu et al. [14] considered the delayed fracture properties of a H charged 1500 MPa bainite-martensite DP steel and revealed that its crack growth rate is less than that of conventional quenched and tempered high strength steels. They ascribed this to the fine DP microstructure and the lath boundaries which may act as beneficial H traps, slowing down segregation and diffusion of H to the crack tip. Recently, Koyama et al. [15] studied the micro-cracking behavior in martensite-ferrite DP in the presence of H. They demonstrated that H affected the damage evolution process considerably. Both the H-enhanced decohesion (HEDE) and H-enhanced localized plasticity (HELP) mechanisms were considered to contribute to the damage evolution in DP steels leading to an unpredictable HE failure. The impact of H diffusion was also recently visualized in DP steel by studying the fracture surfaces of an in-situ charged tensile specimen [16]. The calculated H diffusion distance matched perfectly with the observed transition between H induced brittle and ductile fracture.

AHSS are often used in body-in-white vehicle structures due to their weight reducing capacity and increased safety guarantee. Crashworthiness is a key parameter of their applicability and many mechanical parameters are strain rate dependent. Therefore, the effect of strain rate on hardening and fracture of AHSS has been studied extensively. Static and dynamic tensile tests on two DP and two TRIP steels performed by Huh et al. [17] showed an increase of the elongation at fracture for one of the TRIP steels, though strain rate independent values for the other steels. Similar results were obtained by Curtze et al. [18] who concluded that over a wide range of strain rates relatively constant values of the fracture elongation are obtained for a TRIP and DP steel. However, the combination of H induced mechanical degradation and dynamic high strain rate conditions has not been given much attention so far. Therefore, the aim of this work is to investigate the effect of H on the impact properties of a specific DP steel grade. This H influence was evaluated by modifying both the H content in the tensile specimens and the applied strain rate going from static to dynamic conditions.

Table 1

Chemical composition of the used materials in wt%.

Material/Element	C	Mn	Si	Other
DP	0.07	1.50	0.25	0.4%–0.8% Cr + Mo

2. Experimental procedure

2.1. Material characterization

The sensitivity of DP600 steel to H induced mechanical degradation was investigated in this work. Hot and cold rolling was done till a final thickness of 1.2 mm, which was followed by subsequent annealing via industrial annealing parameters required to attain the desired microstructure. The chemical composition is presented in Table 1. The microstructure was characterized using light optical microscopy (LOM) for which the material was grinded, polished and finally etched using a 2% Nital solution. The samples were immediately cleaned using methanol and acetone. The DP600 steel is a ferritic-martensitic dual phase steel with approximately 23.6% of martensite. The corresponding grain sizes were about 7 μm for the ferritic phase and about 2 μm for the martensitic phase, as shown in Fig. 1. Tensile samples were machined with their tensile axis parallel to the rolling direction. A dogbone-shaped specimen geometry was used, as illustrated in Fig. 2. The small size of the samples is required for the dynamic tests [19]. However, in order to exclude effects related to sample geometry, the same sample geometry is used for the static tests as well.

2.2. Determination of total hydrogen content

The total H content was determined by melt extraction at 1600 °C. Samples were H charged electrochemically in 1 g/l thiourea 0.5 M H_2SO_4 electrolyte for two hours, which guaranteed saturation without H induced damage, as shown in [16]. The applied current density was modified to induce different amounts of H into the tensile samples. For absorption from an aqueous solution, a relation is found between H solubility and applied current density during electrochemical charging, i.e. the solubility is proportional to the square root of the current density [20]. Therefore, the current densities increased from 0.8 over 5 to 25 mA/cm². The time between H charging and H detection was kept constant at 2 min due to the experimental procedure used to perform both the static and dynamic tensile tests, which also required 2 min between H charging and tensile testing. The system to determine the H content includes a pulse furnace in which a pre-weighted sample is

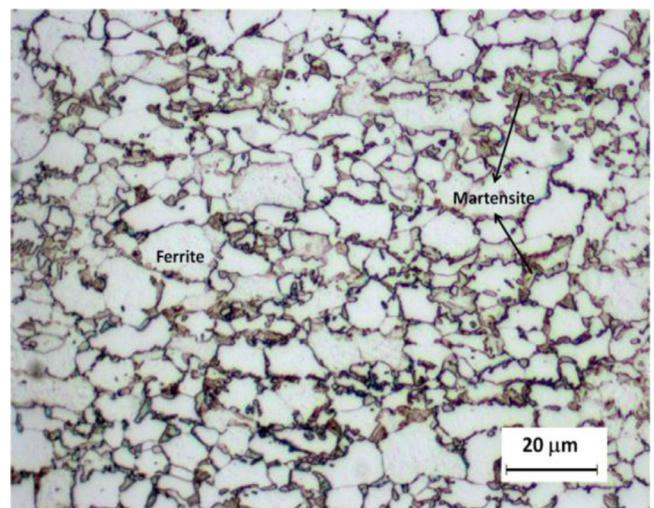


Fig. 1. Light optical microscopic image of DP600 steel.

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