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Effect of Change in Current Density on Hydrogen Embrittlement of Advanced High-Strength Steel S960MC during Hydrogenation

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Hydrogen embrittlement involves the interaction between hydrogen and the microstructure of metals, which can lead to an alarming loss of mechanical properties. For advanced high-strength (AHS) steel S960MC grade, which finds application in fields ranging from heavy machinery to construction, understanding this phenomenon is important. The material's complex crystalline lattice, carefully engineered to maximize strength, becomes vulnerable in the presence of hydrogen. The sources of hydrogen that can lead to embrittlement of steel are various. From the exposure of steel to hydrogen during production processes to the absorption of hydrogen from the environment. After the absorption of hydrogen into the material, hydrogen atoms diffuse in the microstructure and look for places with high stress concentration (cracks, inclusions, grain boundaries, etc.). In these regions, atomic hydrogen disrupts interatomic bonds, weakening the material and making it susceptible to embrittlement and subsequent complete failure of the component. This research is focused on how the change in current density affects the hydrogen embrittlement of AHS steel S960MC during hydrogenation. It was found that the mechanical properties of steel decrease at a lower current density, but not to the same extent as at a higher current density. Thus, it can be said that the change in current density influences the hydrogen embrittlement of S960MC steel.

Keywords: Hydrogen Embrittlement, Current Density, Advanced High-Strength Steel, Hydrogenation, S960MC

1 Introduction

Steel is used in many industries due to its favourable properties and low cost compared to other materials. It is a globally dominant and key material of the world economy. The automotive industry is one of the largest consumers of steel. A lot of advanced high-strength (AHS) steels have been developed, and are still being developed, which are well formable and have an excellent combination of strength, ductility and strain hardening. Currently, a lot of effort is being paid into designing lightweight steel structures, as well as reducing CO_2 and increasing energy efficiency and resource efficiency. For this reason, the use of high-strength steels is increasingly required. AHS steels are not lighter than conventional steels, but their high strength allows thinner sheets to be used to reduce vehicle weight. Steels with a yield strength of 690 MPa - 1100 MPa are mainly used in the construction of buildings, factories, bridges, cranes, wagons, cars, etc. In recent years, AHS steels with a yield strength of 1300 MPa have been developed [1-7].

However, with increasing yield strength, the susceptibility to degradation of mechanical properties in the presence of atomic hydrogen is higher. Steels can absorb hydrogen during manufacturing processes such as galvanic coating, welding, heat treatments, and painting as well as under specific service conditions [2]. This leads to a reduction in toughness and deformation properties, or to hydrogen-assisted cracking (HAC) in the case of mechanically loaded components [8, 9].

Hydrogen in atomic form easily diffuses into the steel structure and passes through the metal lattice even at room temperature, reducing formability and causing brittleness of the material. Accumulation of recombined hydrogen (H_2 – molecular state) on crystal lattice defects, pores, cracks, etc. (Fig. 1) generally leads to cracking and embrittlement of metallic materials [1,10,11].

In the world of materials science, the interaction between mechanical properties and electrochemical processes is a captivating area of study, and the interaction between hydrogen and metals has long captured the attention of researchers and engineers alike [13]. The issue of hydrogen embrittlement (HE) has persisted for a while and has an impact on the durability and safety of crucial parts in sectors ranging from aerospace to the automotive. Despite significant research efforts, the mechanisms of hydrogen embrittlement are still unclear. This is true even when putative mitigating measures have been developed. Regarding the fundamental mechanisms and associated experimental evidence that supports each of these theories, there are numerous opinions in the literature [14-16].

When parameters of electrochemical processes like current density are involved in the hydrogenation process, which frequently involves the diffusion of hydrogen atoms into the steel's lattice structure, the process becomes even more difficult. For this reason, in case of usage galvanic technologies it is necessary to analyse how the current density affect the hydrogenation of structural materials [17].

This research is focused on the hydrogen embrittlement of advanced high strength steel S960MC, which is frequently electroplated by zinc and how it is affected by the change in current density during hydrogenation. Since S960MC steel has a fine-grained martensitic microstructure and hydrogen is trapped at the grain boundaries during hydrogenation, this steel becomes susceptible to hydrogen embrittlement. Samples from experimental material with two different surface treatments were subjected to the electrolytic hydrogenation. This was supposed to simulate the penetration of hydrogen into the material in an aggressive environment. Samples were hydrogen charged at three different current densities, namely 1 mA/cm², 0.5 mA/cm² and 0.2 mA/cm². Then, the samples were tested using tensile tests to determine the changes in mechanical properties after hydrogenation. With increasing current density, a significant decrease in mechanical properties and a change in the fracture character were noted.

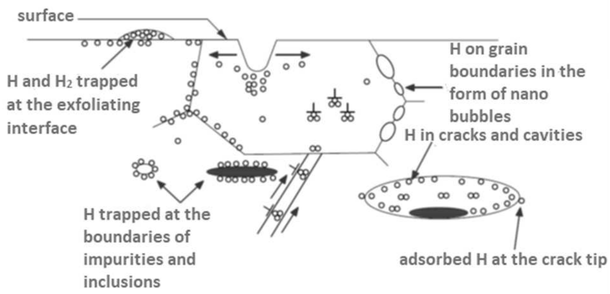

Fig. 1 *Schematic representation of hydrogen traps in materials [12]*

2 Experimental material and methods

2.1 Material

The material used for this research was a thermomechanically treated (hot rolled) advanced high-strength (AHS) steel S960MC grade in the form of sheet with a thickness of 3 mm. Chemical composition of investigated steel was analysed using optical emission spectroscopy (OES) and together with mechanical properties is presented in Tab. 1 and Tab. 2.

Tab. 1 *Chemical composition of S960MC steel [wt. %]*

C	Si	Mn	P	S	Al	Nb
0.085	0.200	1.150	0.014	0.011	0.026	<0.004
V	**Ti**	**Mo**	**Cu**	**Cr**	**Ni**	**N**
0.014	0.018	0.116	0.019	1.110	0.049	0.012

Tab. 2 *Mechanical properties of S960MC*

Yield Strength [MPa]	Tensile Strength [MPa]	Ductility [%]
1014	1162	4

The material which was examined in this research was microalloyed steel with a low carbon content and an increased chromium content. Thermomechanical treatment caused grain refinement, which resulted in tensile strength values up to 1150 MPa. As a result, a fine martensitic microstructure (Fig. 2 and 3) was created. The samples surface treatment also had no effect on the resulting microstructure. The microstructure of S960MC steel was studied by Olympus IX70 inversion metallographic microscope.

The surface states of the as-received and grinded experimental steel are documented in Fig. 4 and 5.

In general, it can be assumed that hydrogen should diffuse more easily into steel with a ground surface than into steel with a surface covered with oxide residues after hot rolling. The surface oxide layer of this steel in the as-received state is so thin that it cannot be identified on the transverse metallographic cut even at 500x magnification, see Fig. 6.

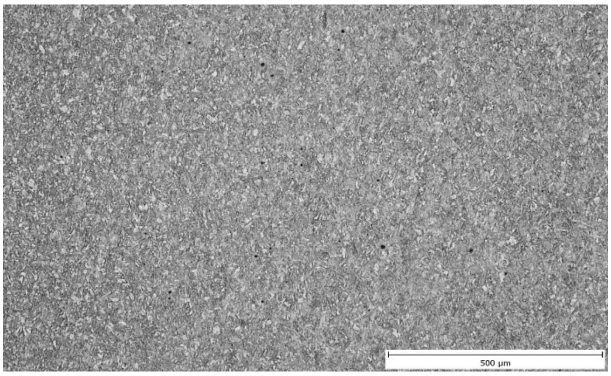

Fig. 2 *Microstructure of S960MC steel (100x, Nital etch.)*

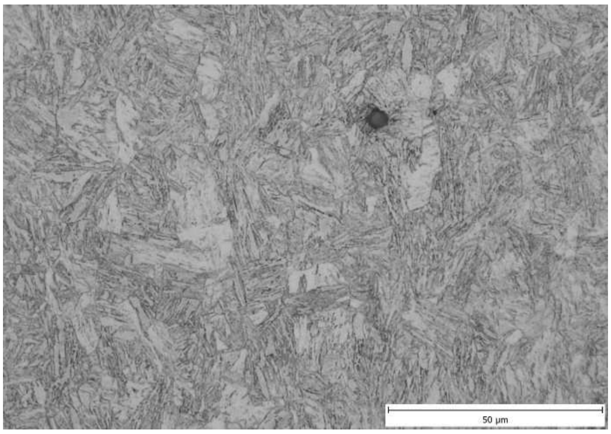

Fig. 3 *Microstructure of S960MC steel (1000x, Nital etch.)*

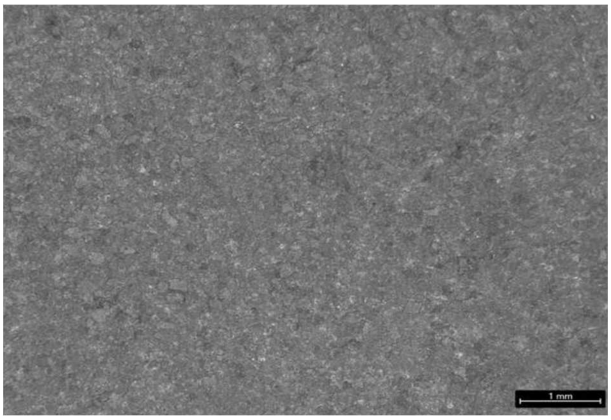

Fig. 4 *As received surface of S960MC steel*

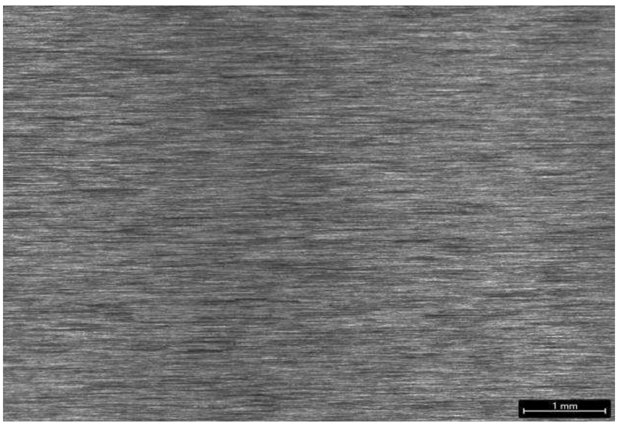

Fig. 5 *Grinded surface of S960MC steel*

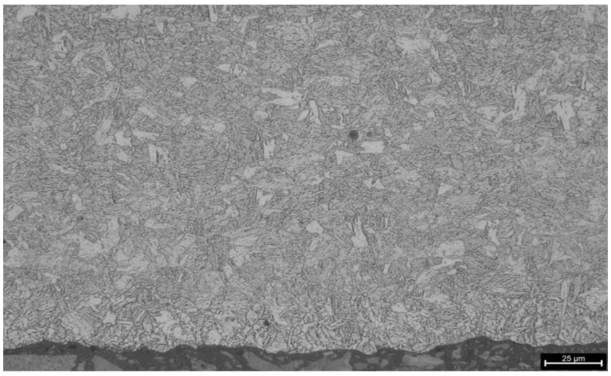

Fig. 6 *Character of the surface of S960MC steel in as-received state – transversal cut (500x, Nital etch.)*

Very thin surface oxide layer does not represent a significant barrier for diffusing hydrogen. Therefore, in this study, no observable difference was noted between hydrogenation characteristics of the as-received and grinded steel. In the case of continuous thick oxide layers, observable differences could already appear. In addition, due to the very small size of diffusing hydrogen atoms, it is known that hydrogen atoms, which very easily penetrate into steel, the surface of which is covered not only with a layer of oxides, but also through various intentionally applied protective coatings (PVD, galvanic coatings, hot sprayed coatings, …).

2.2 Methods

The samples from studied material (advanced high-strength steel S960MC) were electrolytically hydrogen charged in their as-received state (AR) with the surface oxide layer and after removing the oxide layer by grinding (G). Experimental sample was connected as a cathode and platinum plated wolfram mesh was connected as an anode. Samples were hydrogen charged in solution of 0.05 M sulfuric acid (H_2SO_4) with the addition of 1 g of potassium thiocyanate (KSCN) per one liter of distilled water. The hydrogen charging was carried out for 4 hours at the temperature of 20 ± 2 °C and at three different current densities (1 mA/cm^2, 0.5 mA/cm^2, 0.2 mA/cm^2). After undergoing hydrogenation, the samples were subsequently dried and tensile tests were conducted within a 5-minute timeframe. These tensile tests were carried out utilizing a multifunctional LFV 100 kN servohydraulic testing machine.

A fractographic analysis of fractures after the tensile test was performed on a JEOL 6490LV scanning electron microscope in secondary electron mode.

3 Results and discussion

As mentioned, after hydrogenation a tensile test was performed on each sample. Three measurements were performed for each state of the surface of the samples and also for all three current densities. Subsequently, average values were calculated from these measurements. The results of tensile tests can be seen in Tab. 3 and also from the graphs (Fig. 7). The observed differences between the AR and G states of the samples are negligible in the case of tensile tests and may be measurement errors. Research has shown that the higher the current density, the greater is the embrittlement of steel due to hydrogenation. The greatest embrittlement was demonstrated at a current density of 1 mA/cm², mainly due to a decrease in ductility. The decrease in yield strength and tensile strength was also significant. With decreasing current density during hydrogenation, the mechanical properties of the studied steel S960MC increased. The resulting fractures after tensile tests of individual samples were fractographically evaluated (Fig. 8, 9 and Fig. 10).

Tab. 3 *Mechanical properties of samples according to current density during hydrogenation*

Surface condition	**Current density** [mA.cm-²]	**Yield Strength** [MPa]	**Tensile Strength** [MPa]	**Ductility** [%]
AR	1	702.62	802.77	0.21
	0.5	1009.36	1029.02	0.79
	0.2	1060.42	1169.66	2.25
G	1	970.40	989.79	0.76
	0.5	1060.01	1149.47	0.93
	0.2	1070.68	1174.61	3.51

It can also be clearly seen from the graphs that with decreasing current density, the material achieves higher values of mechanical properties. During the tensile test, samples that were hydrogenated at a current density of 1mA/cm² broke at lower stress and strain comparatively with the samples that were hydrogenated at a lower current density. Those samples were less affected by the hydrogen and thus failure occurred at higher stress and strain values. Arniella et al. [18] measured the hydrogen concentration in 42CrMo4 steel using the electrolytic method of hydrogenation in a solution of 1 M H_2SO_4 + 0.25 g/l As_2O_3 for 3 hours at a current density of 1 mA/cm² and 0.5 mA/cm². They found that at a higher current density, the penetration of hydrogen into the sample also increases, which caused significant embrittlement of tested material. So, we can say that the same interaction of hydrogen with the material also occurs in our research on S960MC steel during hydrogenation using upper mentioned current densities. Álvarez et al. [17] in their research describes, that the greater cathodic current density was applied, the greater the hydrogen concentration at the specimen surface, thus, embrittlement is increased.

The fracture surfaces of the samples in their as received state and after removing the oxide layer by grinding obtained after tensile tests do not differ significantly from each other. So, it can by stated that the surface treatment of the samples has no significant effect on the fracture character. The changes in the fracture character were affected by the effect of the change in the current density during the hydrogenation of the samples.

The higher the current density used during hydrogenation, the more brittle fracture occurs (Fig. 8-a, 9-a and 10-a). At low current densities, less hydrogen embrittlement occurs, and the character of the fracture is manifested by shallow pits and small fish-eyes. As the current density increases, the fish-eyes become larger. At even higher current densities, there is a greater influence of hydrogen, and the fracture manifests itself as an intercrystalline fracture with large fish-eyes. Fracture surfaces are also formed by quasi-cleavage fracture, mainly in the area around secondary cracks (Fig. 8-b, 9-b), which is a manifestation of hydrogen embrittlement.

Fish-eyes, which are characteristic of hydrogen embrittled steels, occurred on the fracture surface of all hydrogenated samples (Fig. 8-e, f, 9-c and 10-c, d). Fish-eyes form mainly around larger inclusions, around which quasi-cleavage fracture spreads and passes into transgranular ductile fracture.

Váňová et al. [3,14] achieved similar fracture surface characteristics during the hydrogenation of, in their case, TRIP steels. In the research carried out by Lovicu et al. [4] also describe the resulting fracture surfaces of the samples after hydrogenation by brittle fracture. Whereas samples with lower hydrogen concentration showed mixed transgranular-intergranular fracture, while at higher hydrogen content intergranular fracture prevailed, as expected due to increased hydrogen activity.

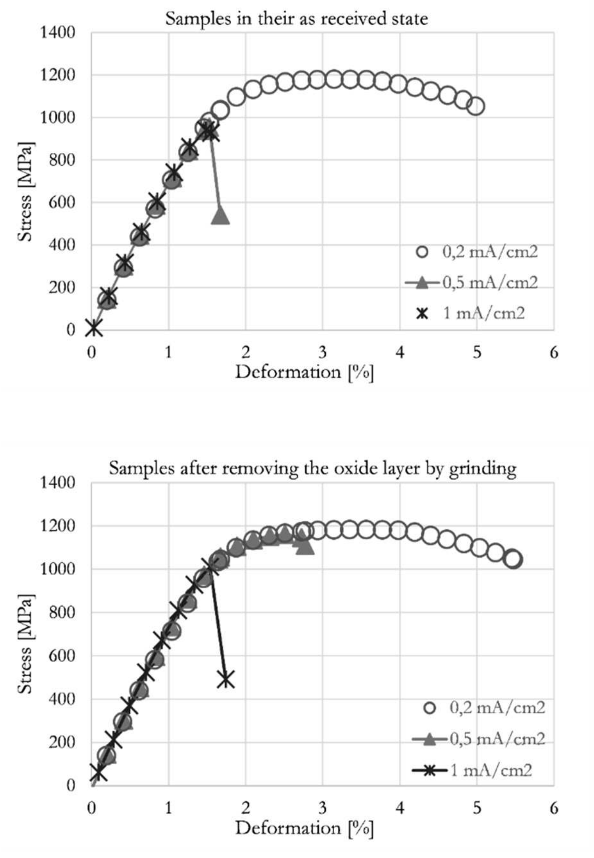

Fig. 7 *Tensile test diagrams of samples after hydrogenation using three different current densities*

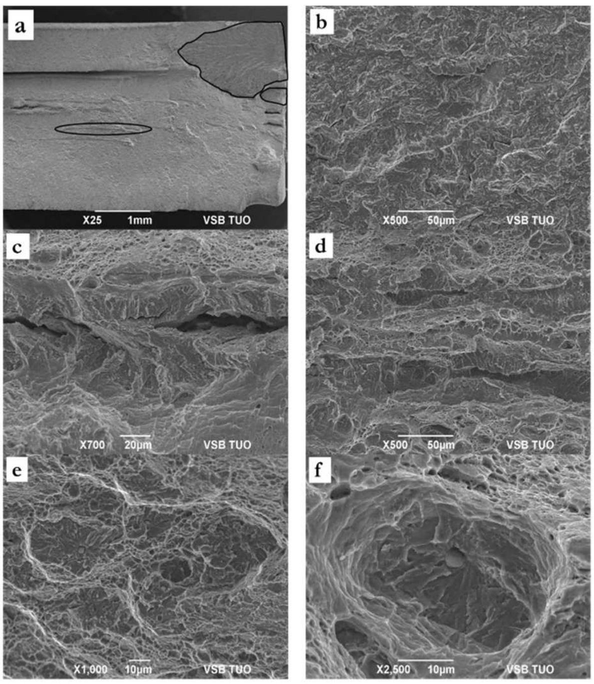

Fig. 8 *Fractographic analysis of the fracture surface after hydrogenation at a current density of 1 mA/cm² a) brittle fracture surface; b) quasi-cleavage fracture around secondary cracks; c) brittle fracture in the area of the crack; d) brittle area towards the edge of the sample; e) fish-eyes around inclusions, shallow pits; f) fish-eye around the MnS inclusion*

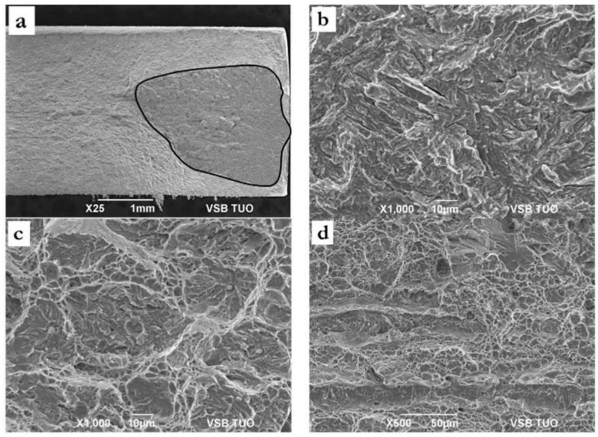

Fig. 9 *Fractographic analysis of the fracture surface after hydrogenation at a current density of 0.5 mA/cm² a) brittle fracture; b) quasi-cleavage fracture and network of secondary cracks; c) fish-eyes around MnS inclusions, shallow pits; d) fracture around segregation belt*

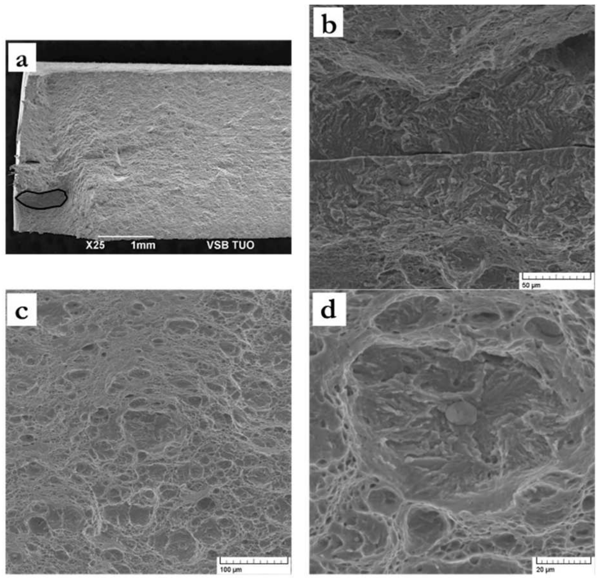

Fig. 10 *Fractographic analysis of the fracture surface after hydrogenation at a current density of 0.2 mA/cm² a) brittle fracture; b) brittle fracture in the area around segregation belt; c) fish-eyes around MnS inclusions, shallow pits; d) fish-eye with quasi-cleavage fracture*

4 Conclusion

Hydrogen in AHS steel S960MC grade has changed many steel properties. It can be said that hydrogen reduced the tensile strength and significantly reduced the ductility. It is clear from the results that hydrogen acted worse on samples that were subjected to higher current density during hydrogenation. The samples showed an area of quasi-cleavage fracture mainly around the cracks. The fractographic analysis also showed fish-eyes, which are typical features caused by hydrogen. Based on this research it can be stated that AHS steel S960MC grade is highly susceptible to hydrogen embrittlement. We also figured out that the level of current density has an impact on the hydrogenation of this steel, which significantly impairs its mechanical properties.

Acknowledgement

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