THE INFLUENCE OF HYDROGEN ON THE MECHANICAL PROPERTIES OF TRIP 780 STEEL

VÁŇOVÁ Petra[1], SOJKA Jaroslav[1], KULOVÁ Taťána[1], KONEČNÁ Kateřina[1],
BRUCHÁČEK Adam[1], MATÝSEK Dalibor[1]

[1]*VSB - Technical University of Ostrava, Czech Republic, EU*

Abstract

The susceptibility to the hydrogen embrittlement of commercial heat of TRIP 780 steel of the type C-Mn-Si was studied on previously hydrogenated samples by a slow strain rate tensile tests (SSRT) in the initial state and in the state after 5% deformation in the longitudinal and transverse directions relative to the rolling direction. Samples were hydrogenated in 0.1N sulfuric acid solution with the addition of KSCN. Due to the transformation of retained austenite to deformation-induced martensite after 5% deformation, an increase of yield strength of 230 MPa, a tensile strength of 30 MPa and elongation at fracture decrease by 6.5 % occurred in contrast with the initial state without hydrogen. The presence of hydrogen in the steel in the initial state caused a decrease of steel strength from 900 MPa to 753 MPa, and a significant reduction in elongation at fracture from 34 % to 6 %. Hydrogen present in the samples after the 5% deformation had an effect on an increase of the yield strength by 60 MPa, a decrease in the tensile strength by 100 MPa, while the elongation at fracture reached only 2.5 %. There were no differences found in the mechanical properties of the samples taken in the longitudinal and transverse direction relative to the rolling direction. Fracture surfaces of the tensile test bars were subjected to fractographic analysis. For hydrogenated samples, the proportion of brittle fracture and quasi-cleavage fracture increased.

Keywords: Hydrogen embrittlement, TRIP steel, slow strain rate test (SSRT), retained austenite

1. INTRODUCTION

Nowadays, research, development or production of automotive components and technology development belong to one of the fastest growing and developing industry sectors. Bodywork construction requirements, such as high firmness, stiffness or low weight, which is connected to vehicle emissions, are closely related to the development of new materials. Despite the increased usage of non-ferrous alloys, steel accounts for approx. 62% of the total weight of a modern car. The bodywork itself consists of many different types of sheet metal that require many complicated forming operations. This implies that these materials need not only high strength but also good formability. These requirements are met by Advanced High Strength Steels (AHSS). This group includes, among other steels, also TRIP steel. [1-2]

TRIP steels are multi-phase steels, whose resulting properties depend on the optimization of the chemical composition. As to microstructure, they consist of ferrite (α), bainite (α_B), retained austenite (RA), and martensite (M) under certain conditions. The whole principle of TRIP is based on a change of retained austenite to the deformation-induced martensite, which gives the original steel with ferritic-bainitic structure higher strength and desired formability. However, in order to reach the TRIP effect, it is necessary to maintain 10 - 15 % of retained austenite in the structure. The carbon content is increased and can reach up to values of 0.2 %. Higher manganese content influences the stabilization of the non-transformed austenitic matrix. Aluminum or silicon, again, if they are present in the steel in higher amounts, they prevent the formation of carbides. The strength limit of these steels ranges from 500 to 1000 MPa, which, along with their excellent formability and energy absorption capability at higher deformation rates, predetermines them to be used in demanding applications in the automotive industry. At present, the greatest attention is paid to the development of TRIP steels with a strength limit of 500 to 800 MPa that led to the improvement of the microalloying process using Ti, V, and Nb. These materials are also very often processed by hot dip galvanizing to increase the corrosion resistance. Technologies, linked to the surface processing of sheets or their welding in humid environment, are potential sources of hydrogen that can penetrate to the matrix and cause changes of mechanical properties or, in case of exceeding the critical hydrogen concentration, also the crack initiation and crack growth. [1-8]

This work is devoted to the study of hydrogen embrittlement in steel TRIP 780. Selected samples were subjected to the electrolytic hydrogenation, which was supposed to simulate the penetration of hydrogen into the material in an aggressive environment. Samples were studied by the SSRT (slow strain rate test) method that is based on a slow sample deformation. Thanks to this method it is possible to describe the influence of hydrogen on the mechanic characteristics of the steel in detail. The work also contains a description of fracture surfaces before and after the hydrogenation.

2. EXPERIMENTAL PART

2.1. The description and the microstructure of the material

The experimental part is focused on steel TRIP 780, specifically on the commercially manufactured type C-Mn-Si. This material was delivered in a state after two-stage cold rolling and after one hot rolling. The surface is often processed by hot dip galvanizing or by galvanic plating. The strength limit should be according to the producer in the range of 780 - 900 MPa, yield strength in the range of 450 - 550 MPa, the ductility is greater than 23 %. This steel is characteristic by perfect plastic properties and by good energy absorption ability. The very good energy absorption is the reason of the usage of this material for the production of bodywork security parts, in particular transverse or longitudinal crossbeams, B pillars and bumper reinforcements. The maximum content of aluminum in the TRIP 780 steel should be 2.5 wt. %, the maximum content of manganese and silicon should be 2.0 wt. %.

The chemical composition of delivered samples was found out by GDOES (glow discharge optical emission spectroscopy) method on Spectruma GDA 750 device and carbon and sulfur contents by combustion elemental analysis on the Eltra CS 2000 device (**Table 1**).

Table 1 Chemical composition of TRIP 780 in wt. %

C	**Mn**	**Si**	**P**	**S**	**Cr**	**Ni**	**Mo**
0.20	1.57	1.65	0.015	<0.001	<0.001	<0.001	<0.001
Cu	**Ti**	**Co**	**B**	**Pb**	**V**	**W**	**Al**
0.007	0.002	0.003	<0.001	<0.001	0.001	<0.001	0.058

The microstructure of steel TRIP 780 was studied in polished and etched condition by Olympus IX70 inversion metallographic microscope (abbr. OM) and by the JEOL 6490LV scanning electron microscope (SEM) in secondary electron (SE) mode. In **Figure 1** and **Figure 2** is showed the microstructure in the longitudinal direction relative to the rolling direction, etched in the Nital solution. The microstructure was formed mostly by ferrite and bainite. With larger magnification, it is possible to partly identify M/A constituent (martensite/retained austenite).

In the microstructure of steel TRIP 780 there occurred: round aluminum oxides, elongated manganese sulphides and sharp-edged, often occurring in rows, titanium nitrides. The size of individual inclusions was around 5 μm, the length of rows reached up to 35 μm.

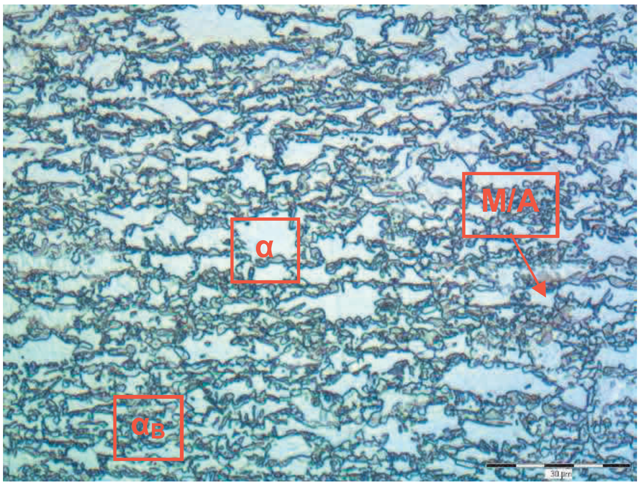

Figure 1 The microstructure of steel TRIP 780, longitudinal direction, etched in Nital (OM)

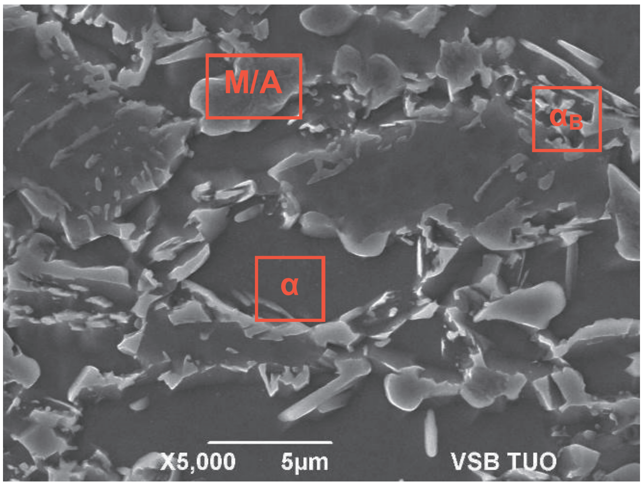

Figure 1 The microstructure of steel TRIP 780, longitudinal direction, etched in Nital (SEM-SE)

2.2. Mechanical properties

From a 1.17 mm sheet, samples for the tensile test in the transverse direction (marked P) and the longitudinal direction (marked L) were taken in relation to the rolling direction of the length of L_c = 60 mm by electrospark cutting technology. For the reason of high corrosion rate, the samples were sharpened on planer grinding machine, which decreased the sheet thickness to 1.12 mm. Tension test bars were loaded with a slow strain rate 10^{-4} s^{-1}. A part of samples was subjected to 5 % plastic deformation, and then again loaded until the fracture. Tensile test took part on the multifunctional servo-hydraulic LFV 100 kN test machine. Values of the ultimate tensile strength R_m, yield strength $R_{p0.2}$ and elongation at fracture A_{60} of the studied material in the initial state and in a state of 5% deformation are reported in **Table 2**. It follows from the results of mechanical properties of steel TRIP 780 that the direction of rolling unambiguously didn't influence the basic mechanical properties. Due to the deformation, the cross section was reduced from 11.18 mm^2 to 10.70 mm^2. The specific attribute of TRIP steels is the transformation of retained austenite into martensite induced by plastic deformation, which leads to strengthening the steel while maintaining good plastic properties. The influence of retained austenite transformation on deformation induced martensite caused an increase in the yield strength by 230 MPa, a strength limit by 30 MPa and a drop in the ductility by only 6.5 %.

Electrolytic hydrogenation consisted in saturation of the test samples with hydrogen, when the formed test bars were dipped into the electrolyte and connected as a cathode. The anode was formed by a quenched tungsten grille located around the hydrogenated sample. The electrolyte was a solution of 0.1N sulfuric acid with the addition of one gram of KSCN per 1 litre of H_2SO_4. The hydrogenation process was carried out for 4 hours at a current density of 1 mA·cm^{-2}. Hydrogenated samples were clamped to the tensile testing machine immediately after finishing the hydrogenation and were subjected to load at a deformation strain rate of 10^{-4} s^{-1}. The degree of hydrogen embrittlement was expressed by so called hydrogen embrittlement index, denoted as F [%] (eqn.1).

$$F = \frac{A_H - A_0}{A_0} \cdot 100 \qquad \text{eqn. 1}$$

where A_0 is the ductility of samples without hydrogen [%] and A_H is the ductility od samples with hydrogen [%].

When comparing the results from the previous hydrogen-free tensile tests, it can be seen that the presence of hydrogen in the steel caused a drop of strength from 900 to 753 MPa, a yield strength drop by 30 MPa, and a decrease in the elongation at fracture from 34 % to about 6 %, which corresponds to the hydrogen embrittlement index 83.3 %. A similar hydrogen embrittlement index was also found in laboratory produced steel TRIP 800 (variant A) but with current densities 15 mA·cm^{-2} [9], or at work [10] with current densities of 1 and 5 mA·cm^{-2} in solutions 0.1N H_2SO_4 + KSCN (or As_2O_3). For samples with a previous 5 % plastic deformation and after electrolytic hydrogenation, the effect of hydrogen on the yield strength was an increase by 60 MPa and the strength limit drop was by 100 MPa. The response of the material to the tensile load in dependence on the stress-elongation for all four studied states is shown in **Figure 3**. From the curves, the material strengthening due to the phase transformation of retained austenite to the deformation-induced martensite is evident, and in the case of hydrogenated samples after the previous plastic deformation also the increase of the yield strength. However, hydrogen in both states (initial and state after 5 % deformation) caused significant embrittlement, even when using a low current density. The introduced deformation resulted in almost complete transformation of retained austenite in the sample loaded from the initial state without the presence of hydrogen. For the other states, regardless of the height of the total deformation of retained austenite, it was stabilized to about 6 vol. % (**Table 2**).

Table 2 Results of mechanical properties in the initial state and after the 5 % deformation

Sample indication	$R_{p0.2}$ [MPa]	R_m [MPa]	A_{60} [%]	F [%]	RA [vol. %]
Initial state without H	521 ± 45	900 ± 47	33.8 ± 1.7	-	2.2
After 5 % deformation without H	754 ± 24	931 ± 17	27.2 ± 2.0	-	6.1
Initial state with H	559 ± 15	753 ± 28	5.7 ± 0.7	83.3 ± 2.2	6.7
After 5 % deformation with H	814 ± 9	837 ± 9	2.4 ± 0.3	91.1 ± 1.1	5.9

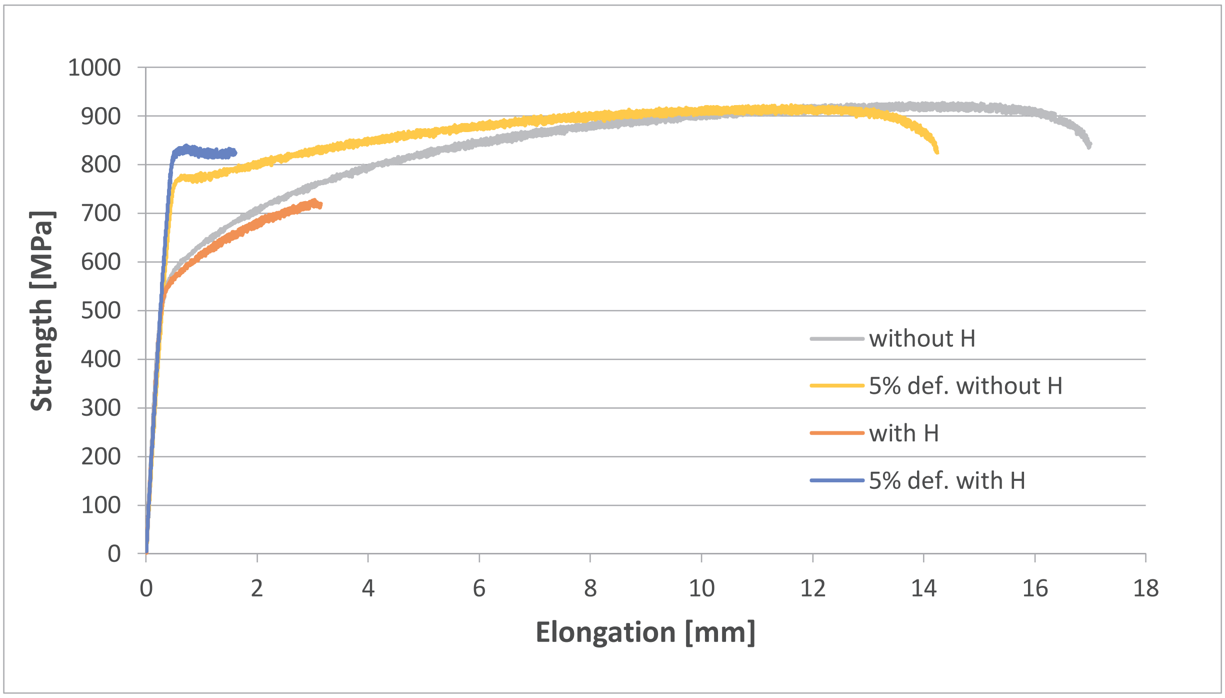

Figure 3 Dependence of the strength on the elongation of selected steel TRIP 780 samples

2.3. Fractographic analysis

The fractographic analysis of fracture surfaces was carried out by using the JEOL 6490LV scanning electron microscope in secondary electron mode. Samples that were not subjected to hydrogenation exhibited mixed fracture. At the centre of the sample (in location marked a) a transgranular cleavage fracture can be observed, the edge of the sample (marked b) shows signs of transgranular ductile fracture with a dimple morphology (**Figure 4**). On the fracture surface of the sample after 5% deformation and subsequent rupture with the absence of hydrogen the significant difference in fracture morphology wasn't observed compared to the initial state.

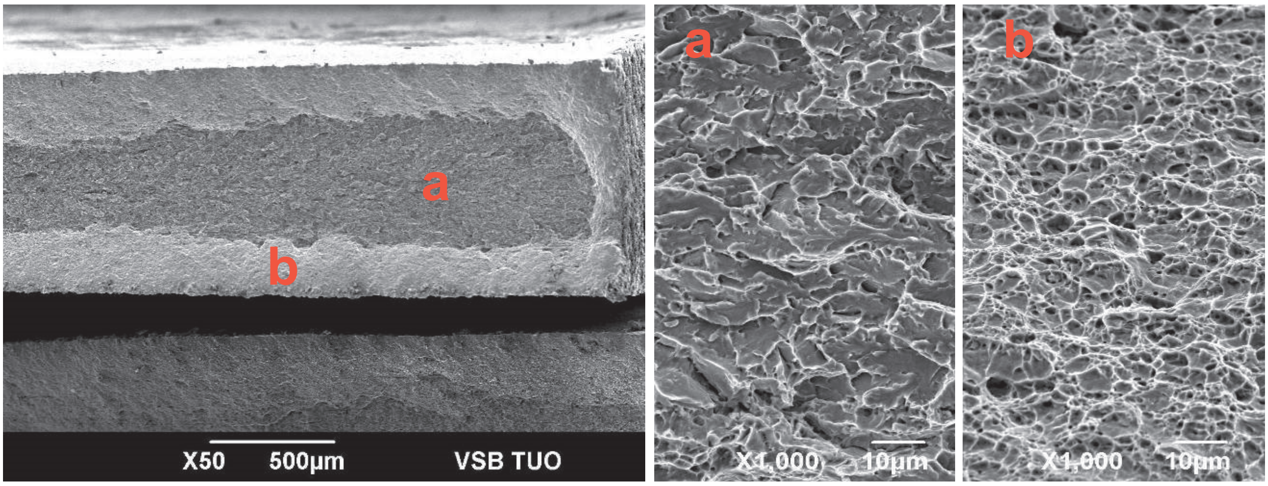

Figure 4 View of the sample fracture surface in the initial state without hydrogenation, location a transgranular cleavage fracture (centre), location b transgranular ductile fracture (edge)

The change of fracture mechanism occurred after hydrogenation. Quasi-cleavage fracture, which is typical for hydrogen embrittlement, is apparent on the fracture surface. Soft quasi-cleavage facets are visible in *c* detail (**Figure 5**) and a transition of quasi-cleavage fracture through bridges with transgranular ductile fracture (marked d). Quasi-cleavage fracture is mostly on the edges of the sample and around prolonged non-metallic inclusions (**Figure 5**). The morphology of sample fracture surface without and with 5 % deformation was similar.

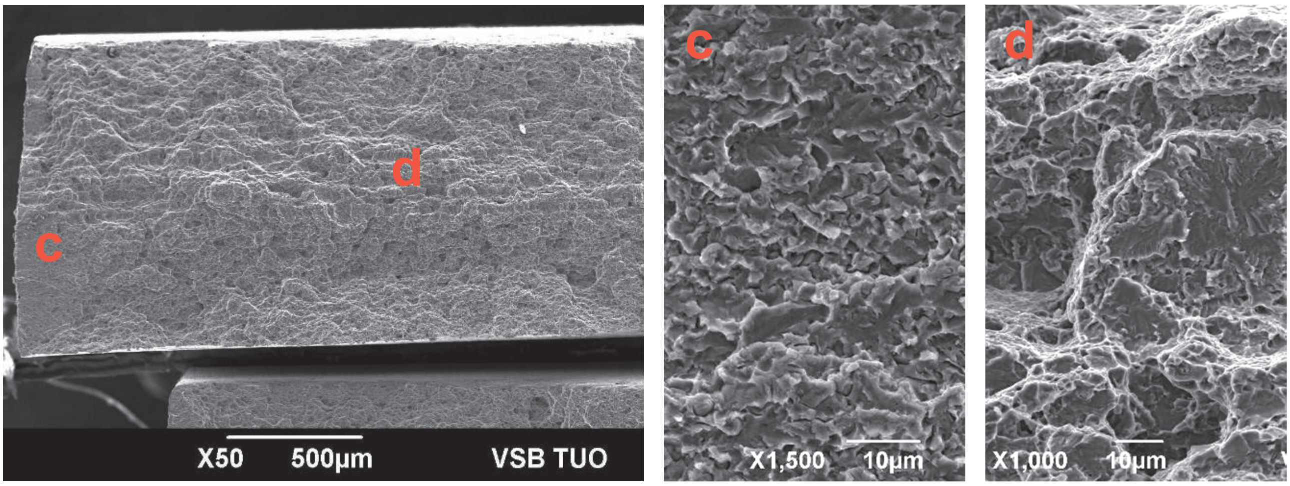

Figure 5 View of the sample fracture surface in the state after hydrogenation, location c quasi-cleavage fracture (edge), location d mix of quasi-cleavage fracture and transgranular ductile fracture (centre)

3. CONCLUSION

The aim of this work was to describe the influence of hydrogen on mechanical properties and failure mechanisms of the commercially produced steel TRIP 780. In the absence of hydrogen and after 5 % plastic deformation, the TRIP effect was manifested by transformation of retained austenite into deformation-induced martensite, which led to an increase in strength properties, specifically yield strength by 230 MPa, and a decrease in plastic properties by only 6.5 %. Samples subjected to electrolytic hydrogenation showed a high value of the hydrogen embrittlement index *F*, expressed as a relative change in the ductility of the material, namely 83 %, or 91 %, in a state of absence or with 5 % of pre-deformations. The hydrogen embrittlement also manifested itself in the character of the fracture surface, which showed a high proportion of quasi-cleavage failure. Nevertheless, the electrolytic hydrogenation proceeded at a low current density. It is, therefore, necessary to pay increased attention to the production processes where the risk of interaction of hydrogen with the material is possible.

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REFERENCES

[1] Advanced High-Strength Steel Application Guidelines [online]. 2014 [cit. 2017-05-10 http://309fbf2c62e8221fbaf0-b80c17cbaf20104b072d586b316c6210.r88.cf1.rackcdn.com/AHSS_Guidelines_V5.0_20140514.pdf

[2] DE COOMAN, B. C. Structure-properties relationship in TRIP steels containing karbide-free bainite. *Current Opinion in Solid State and Material Science,* 2004, vol. 8, pp. 285-303.

[3] ZAEFFERER, S., OHLERT, J., BLECK, W. A study of microstructure, transformation mechanisms and correlation between microstructure and mechanical properties of a low alloyed TRIP steel. *Acta Materialia*, 2004, vol. 52, no. 9, pp. 2765-2778.

[4] WANG, X. D., et al. Microstructures and stability of retained austenite in TRIP steels. *Materials Science and Engineering,* 2006, vol. 438A, pp. 300-305.

[5] LEE, Ch. G. et al. Effects of volume fraction and stability of retained austenite on formability in a 0.1 C-1.5 Si-1.5 Mn-0.5 Cu TRIP-aided cold-rolled steel sheet. *Materials Science and Engineering,* 2004, vol. 371A, no. 1, pp. 16-23.

[6] ZHANG, M.Y., ZHU, F.X., ZHENG, D.S. Mechanical properties and retained austenite transformation mechanism of TRIP-aided polygonal ferrite matrix seamless steel tube. *Journal of Iron and Steel Research International*, 2011, vol. 18, no. 8, pp. 73-78.

[7] ZHU, L.J., DI, W. U., ZHAO, X.M. Effect of silicon content on thermodynamics of austenite decomposition in C-Si-Mn TRIP steels. *Journal of Iron and Steel Research International*, 2006, vol. 13, no. 3, pp. 5773-60.

[8] MUKHERJEE, M. et al. Strain-induced transformation behaviour of retained austenite and tensile properties of TRIP-aided steels with different matrix microstructure. *ISIJ international*, 2006, vol. 46, no. 2, pp. 316-324.

[9] SOJKA, J. et al. Effect of hydrogen on the properties and fracture characteristics of TRIP 800 steels. *Corrosion Science*, 2011, vol. 53, no. 8, pp. 2575-2581.

[10] VÁŇOVÁ, P., SOJKA, J., KONEČNÁ, K., WENGLORZOVÁ, A. Study of Hydrogen Embrittlement of the TRIP 800 Steels by Means of Tensile Tests at Simultaneous Hydrogen Charging. In *METAL2012 : 21st International Conference on Metallurgy and Materials.* Ostrava : Tanger, 2012, s. 570-575.