THE INFLUENCE OF SURFACE CHARACTERISTICS ON HYDROGEN EMBRITTLEMENT OF CARBON STEELS

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VLIV POVRCHU NA VODÍKOVÉ ZKŘEHNUTÍ UHLÍKOVÝCH OCELÍ

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Abstrakt

Tato práce prezentuje výsledky tahových zkoušek uhlíkových ocelí typu A333 Gr. 6 za přítomnosti vodíku při pomalých rychlostech zatěžování. Oceli byly zkoušeny v dodaném stavu od výrobce a po tepelném zpracování 870 °C / 40 min / voda + 600 °C / 2h / vzduch. Mikrostruktura dodaných vzorků byla feriticko-perlitická a po tepelném zpracování obsahovala směs bainitu a feritu. Cílem práce bylo ukázat vliv vodíku na degradaci materiálu v závislosti na mikrostruktruktuře a při pomalých rychlostech zatěžování (rychlost 10^{-5} s^{-1}). Při testech bylo také přihlédnuto k vlivu drsnosti povrchu na vodíkové zkřehnutí. Zkušební vzorky byly rozděleny na dvě skupiny. První reprezentovala zkušební vzorky s běžnou drsností povrchu. Druhá skupina zahrnovala vzorky s vyleštěným povrchem. Takto upravený povrch měl eliminovat potencionální zárodky defektů na povrchu a tím snížit riziko předčasného porušení zkoušených vzorků. Lomové plochy byly zkoumány fraktografickou analýzou za pomocí stereomikroskopu a řádkovacího elektronového mikroskopu (SEM). V obou případech, jak v dodaném stavu, tak i po tepelném zpracování, byly lomové plochy číškového charakteru a z fraktografického pohledu měly transkrystalický křehký lom. Detailnější informací o charakteru porušení poskytl řádkovací elektronový mikroskop, který ukázal převládající transkrystalický štěpný lom s občasnými malými lokalitami kvasištěpného lomu, který se také označuje jako tzv. „rybí oka“. Při testech nebyl pozorován vliv lepší povrchové úpravy (resp. nižší drsnosti), který by vedl k zvýšené odolnosti proto vodíkové křehkosti.

Abstract

Presented paper summarizes results of slow strain rate (tensile) test in the presence of hydrogen of the carbon steel of type A333 Gr.6 according to ASTM. Steel was tested in as-received state and after quenching and tempering in accordance to the following regime: 870 °C / 40 min / water + 600 °C /2 hours / air. Microstructure of steel in as-received state was ferritic-pearlitic and after heat treatment– quenching and tempering was a mixture of tempered bainite and ferrite. The aim of this work was to demonstrate the influence of hydrogen on the degradation of material with different microstructure by slow strain rate test (tensile), where the strain rate was 10^{-5} s^{-1}. The impact of surface quality to resistance of steels against hydrogen embrittlement was also evaluated. Tensile specimens were divided into two groups. The first groups represented tensile specimens with a standard surface roughness. The second groups represented specimens with finely ground surface to eliminate potentional sites for defect initiation and subsequently to decrease a risk of early failure of specimens. Specimens were submitted to the fractographic analysis of fracture surfaces after tensile test using the stereomicroscope and scanning electron microscope In both cases, in initial state and after heat treatment fracture has a cup character and from the fractographic viewpoint it is transgranular ductile fracture with dimple morphology. More detailed determination of fracture character was obtained by SEM. There is mainly transgranular cleavage fracture, however, there are small areas of quasi cleavage fracture (QCF), reminding so-called "fish eyes". As well, surface quality improvement of tested specimens didn't lead to increasing of material resistance against hydrogen embrittlement.

Keywords: slow strain rate test, hydrogen embrittlement, fish eyes, carbon steel

1. Introduction

Many structural materials, including steels, are exposed during their exploitation to impacts of aggressive environment. In other to ensure safety and long-term reliability of structures made of these materials, it is necessary to design these materials in such a manner that they resist to influences of aggressive environment. One of the methods evaluating susceptibility of steels to hydrogen embrittlement is slow strain rate test. Elementar hydrogen is collected in steel by means of cathodic absorption during hydrogen embrittlement [1]. Decreasing of ductility and reduction in area indicates hydrogen embrittlement. This phenomenon can be approved by tensile test with not very high strain rate. Effects of hydrogen embrittlement were not observed during Charpy impact test or tensile test for a very high rate of deformation. One of the effects of hydrogen embrittlement during a low rate of deformation is also the formation of so-called "fish eyes" [2]. Fish eyes appear on fracture areas as light round facets, orientated perpendicularly to the direction of applied stress. Initiation sites for fish eyes formation are mainly non-metallic inclusions, from which cracks spread with quasi cleavage fracture.

2. Experimental procedure

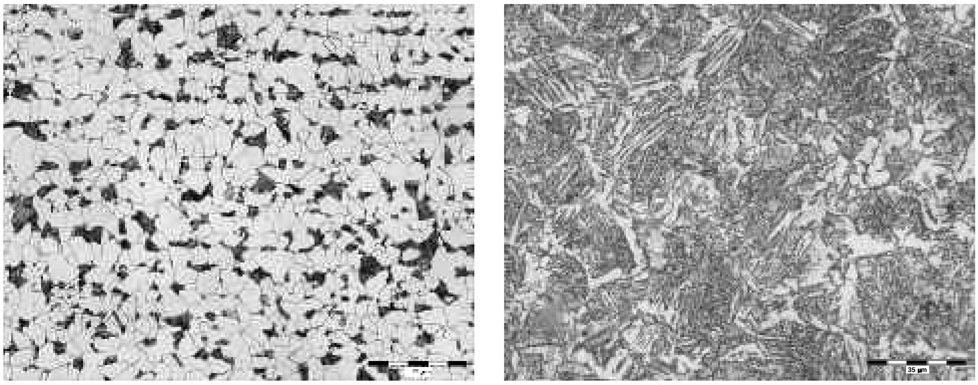
The testing was made on carbon steel of type A333 Gr. 6 according to ASTM (tube 508/15,09 mm) in as-received state (AR) and after quenching and tempering according to the following regime: 870°C/40 min/water + 600°C/2 hours/air (QT). Chemical composition of the studied steel is given in Table 1.

Table 1 Chemical composition (mass %)

C	Mn	Si	P	S	Cr	Ni	Cu	Mo	V	Ti	B
0.18	0.86	0.21	0.009	0.016	0.14	0.10	0.23	0.03	0.001	0.02	0.002

Microstructure of steel in as-received state (AR) was ferritic-pearlitic and it is shown in Fig. 1a. Microstructure of specimens after heat treatment– quenching and tempering (QT) was a mixture of tempered bainite and ferrite. It is presented Fig. 1b.

The specimens were tested by slow strain rate test (tensile), where the strain rate was 10^{-5} s^{-1}. Tensile specimens were divided into two groups. The first groups represented tensile specimens with a standard surface roughness. The second groups represented specimens with finely ground surface to eliminate potentional sites for defect initiation and subsequently to decrease a risk of early failure of specimens.



a) as-received state (AR) b) after heat treatment (QT)

Fig.1 Microstructure of steel A333 Gr. 6

Table 2 Mechanical properties of studied steels, in the state without presence of hydrogen

Specimens	$R_{p0,2}$ (MPa)	R_m (MPa)	A_{35} (%)	Z (%)
AR	309.5	457.5	29.6	71.4
AR_L	312.5	458.0	33.0	72.4
QT	414.5	539.5	24.5	78.4
QT_L	454.5	566.0	22.1	78.3

These specimens were marked by index L. Mechanical properties of the studied steel are summarised in Table 2. Fine grounding manifested itself by increase of mechanical properties after QT. Heat treatment also resulted in increase of the yield strength R_m and reduction in area Z.

3. Results and discussions

The influence of hydrogen on degradation of material properties was tested with method of a slow strain (tensile) test. Specimen was placed in the cell with 0.1 N solution of H_2SO_4 during tensile test and cathodically hydrogenated for the current density 5 mA/cm^2. Hydrogenated specimens were marked by index H. Mechanical properties of the studied steel after electrolytical charging are summarised in Table 3 and Fig. 2. It is evident, that hydrogen charging has slight impact on yield limit $R_{p0,2}$ and strength limit R_m. A decreasing of ductility occurred after hydrogen charging. After reaching the strength limit the applied force came to a rapid decline comparing to state without hydrogen, which is apparent from Fig. 2.

Table 3 Mechanical properties of studied steels, with presence of hydrogen

Specimens	$R_{p0,2}$ (MPa)	R_m (MPa)	A_{35} (%)	Z (%)
AR_H	313.5	463.5	20.5	21.6
AR_L_H	306.7	463.0	19.9	21.6
QT_H	427.5	554.5	17.2	22.3
QT_L_H	430.0	551.5	18.7	26.7

Much more expressive is the influence of hydrogen on the reduction in area of steel. This factor can be easily described by index of hydrogen embrittlement F, which shows proportional change of reduction in area in the material without present of hydrogen and after hydrogen charging (equation 1).

$$F = \frac{Z_0 - Z_H}{Z_0} \cdot 100 \text{ in (\%)}, \qquad (1)$$

where Z_0 is reduction in area in as-received state in %,
Z_H is reduction in area in hydrogen charged state in %.

Table 4 Index of hydrogen embrittlement F

Specimens	Z_0 (%)	Z_H (%)	F(%)
AR	71.4	21.6	69.7
AR_L	72.4	21.6	70.2
QT	78.4	22.3	71.6
QT_L	78.3	26.7	65.9

It is clear from the Table 4, that electrolytic charging of material during tensile test induced embrittlement of material both in AR and QT state. Microstructure change didn't result in decreasing or increasing of index of hydrogen embrittlement F. Index of hydrogen embrittlement was 70 % for specimens in initial stage (AR), and 69 % for specimen after heat treatment (QT). During evaluation of resistance of similar steels against sulphide stress cracking (SSC) it was found, that quenching and tempering (QT) led to the increasing of steel resistance against this kind of degradation [3]. As well, surface quality improvement of tested specimens didn't lead to increasing of material resistance against hydrogen embrittlement. Nevertheless, it is generally known, that steel sensitivity against hydrogen embrittlement rise with rising surface roughness [4].

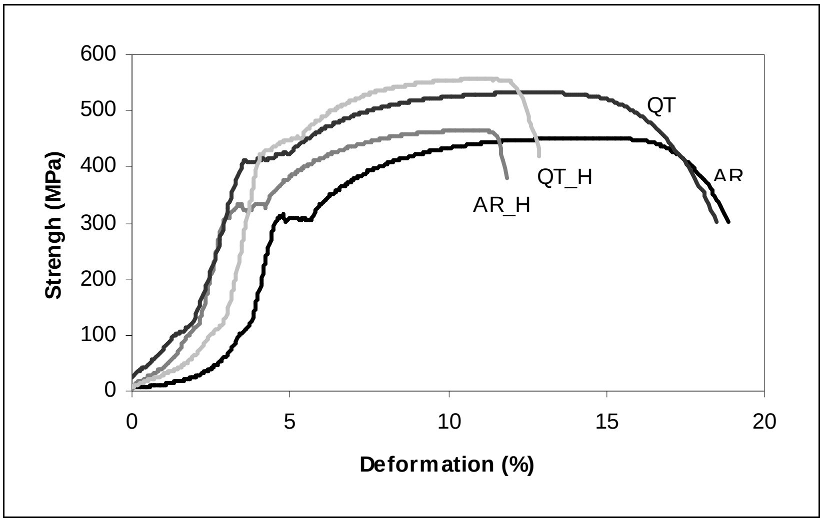
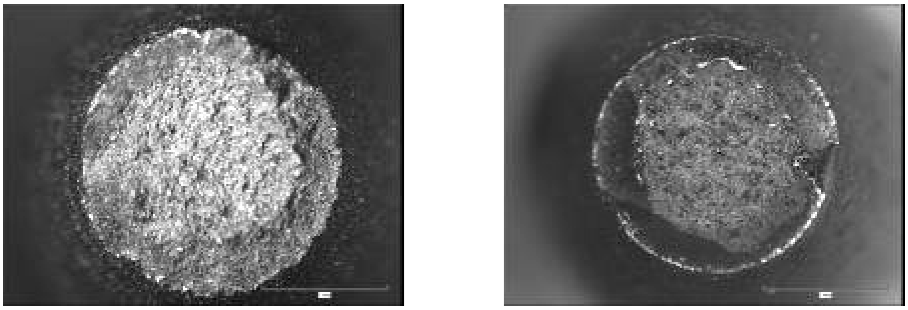


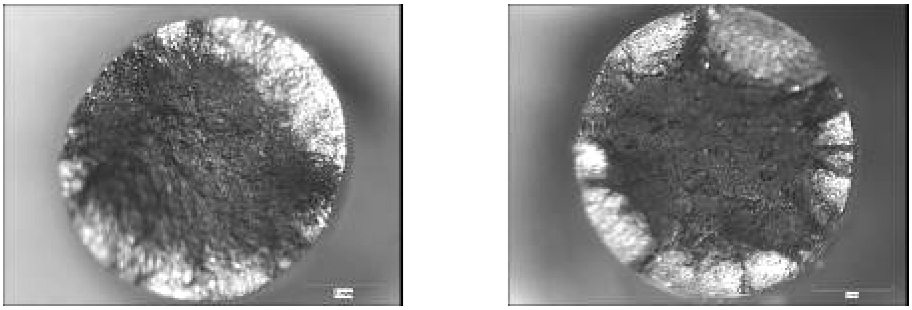
Fig.2 Tensile diagrams of different states of steel A333 Gr. 6

Specimens were submitted to the fractographic analysis of fracture surfaces after tensile test using the stereomicroscope and scanning electron microscope (SEM). Fracture areas of tested specimens can be seen in as-received state using the stereomicroscope in Fig. 3. In both cases, in initial state (Fig. 3a), and after heat treatment (Fig 3b), fracture has a cup character and from the fractographic viewpoint it is transgranular ductile fracture with dimple morphology. A higher reduction in area of material after heat treatment can be seen in the Fig. 3. Hydrogen charging took effect on fracture area of tensile specimens as a shear fracture (Fig. 4). Light facets occurred on specimen borders in initial state (Fig. 4a) and also in state after heat treatment during top (vertical) (Fig. 4b) lighting on stereomicroscope.



a) as-received state (AR) b) after heat treatment (QT)

Fig.3 Examples of fracture surface of steel A333 Gr. 6, without presence of hydrogen, stereomicroscope

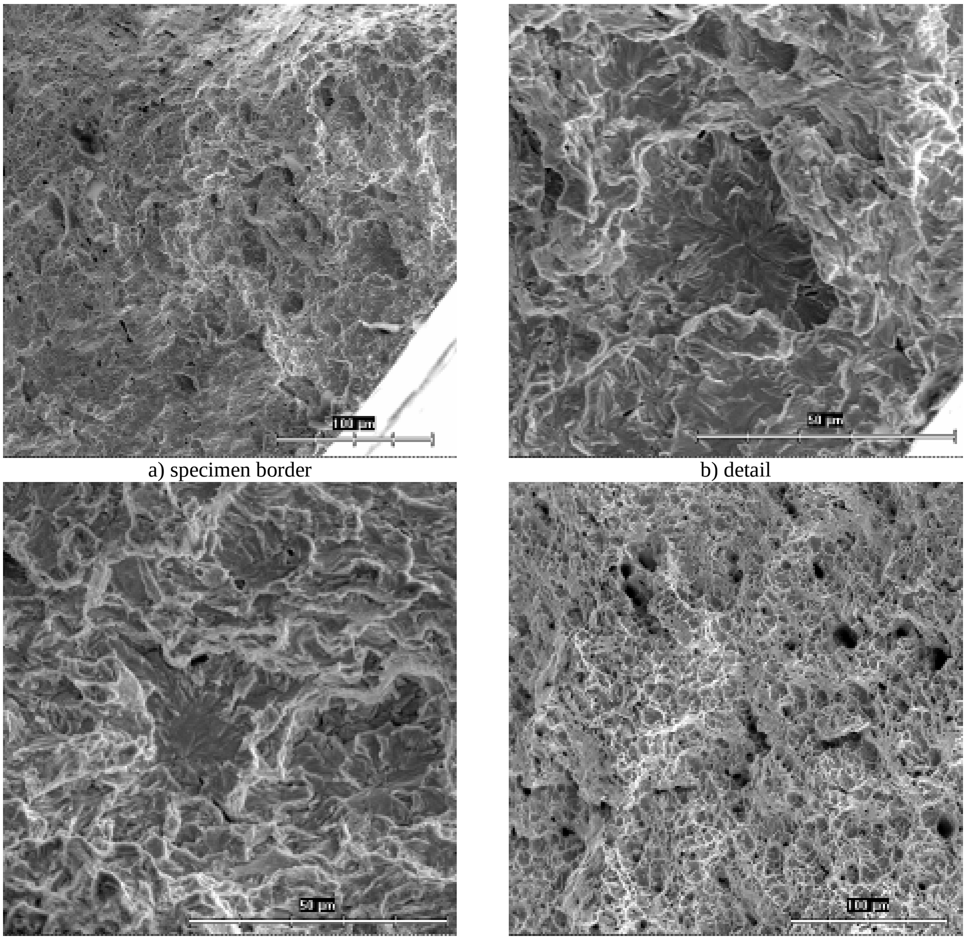


a) as-received state (AR) b) after heat treatment (QT)

Fig.4 Examples of fracture surface of steel A333 Gr. 6, with presence of hydrogen, stereomicroscope

More detailed determination of fracture character was obtained by SEM. Fig. 5 shows state of fracture area of hydrogenated material in initial state (AR). There is mainly transgranular cleavage fracture (Fig. 5a – 5c) in the border part of specimen. However, there are small areas of quasi cleavage fracture (QCF), reminding so-called “fish eyes”, which was found in materials observed in works [1-5]. Non-metallic inclusions were found in the centre of “fish eyes”, e.g. type MnS (Fig. 5b). There was also an area of transgranular ductile fracture with dimple morphology in the middle part of fracture surface (Fig. 5d).

Character of fracture surface, after hydrogen charging for heat-treated specimens (QT), is shown in Fig. 6. The borders of fracture surface are mainly consisting of quasi cleavage fracture (QCF) (Fig. 6a – 6c). Fracture partly reminds of fatigue damage as it was initiated on specimen surface. Specific initiation sites were not found, from which the fracture could start (Fig. 6b). The middle of fracture area consists of transgranular ductile fracture with dimple morphology. Nevertheless, there are some deep “holes” comparing to specimens in initial state. Morphology of fracture areas of hydrogenated specimens after slow strain rate test is similar to morphology of fracture areas after SSC tests of micro alloyed steels according to API, namely in initial state, and after heat treatment [4].



a) specimen border b) detail

c) specimen border d) centre of specimen

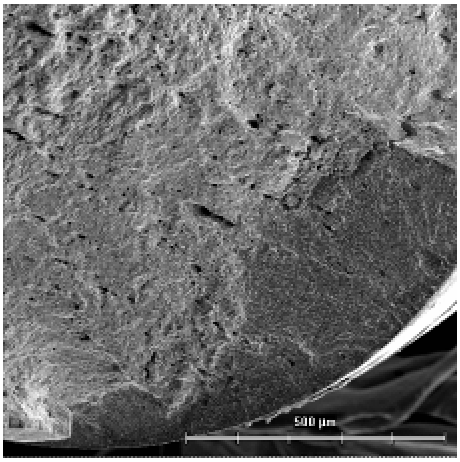
Fig.5 Examples of fracture surface of steel A333 Gr. 6, as-received state after hydrogen charging (AR), SEM

4. Conclusion

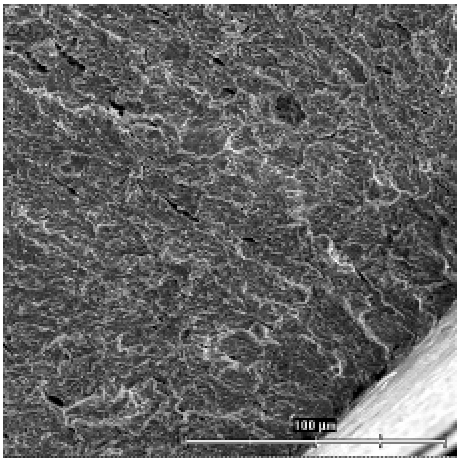
The obtained results can be summarised in the following way:

- Presence of hydrogen during slow strain rate (tensile) test led to embrittlement of material.
- Embrittlement of material manifested itself by decreasing of ductility and mainly by decreasing of reduction in area.
- The influence of hydrogen on mechanical properties of material, and reduction in area, was described by index of hydrogen embrittlement.
- Although the material shows higher strength after heat treatment, it didn't have an important impact to change of hydrogen embrittlement index.

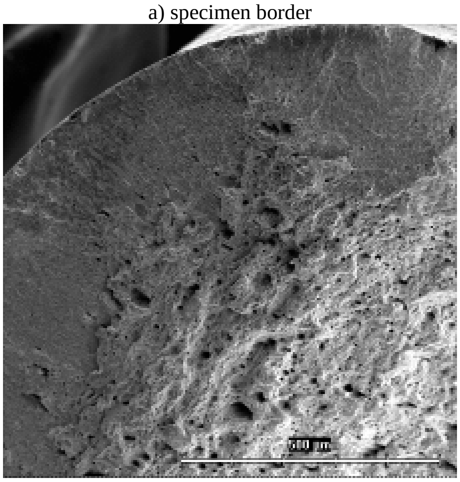
- Improving of specimen surface quality by fine grinding didn't have any impact to material properties.
- Embrittlement of material due to hydrogen, was observed also on the character of fracture surface.
- There occurred areas of transgranular cleavage fracture and small areas reminding of “fish eyes” in the border of fracture surface in initial state of hydrogenated material.
- Borders of fracture surface consisted solely of quasi cleavage fracture for hydrogenated specimens after heat treatment.



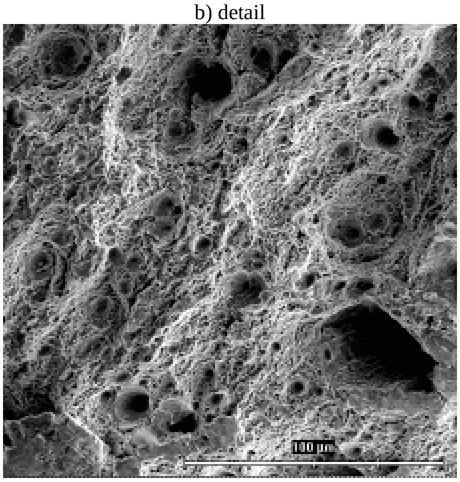
a) specimen border



b) detail



c) specimen border



d) centre of specimen

Fig.6 Examples of fracture surface of steel A333 Gr. 6, after heat treatment and hydrogen charging (QT), SEM

Acknowledgements

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