ROLE OF TESTING CONDITIONS IN SULPHIDE STRESS CRACKING OF X52 AND X60 API STEELS

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VLIV PODMÍNEK ZKOUŠENÍ NA SULFIDICKÉ PRASKÁNÍ POD NAPĚTÍM OCELÍ X52 A X60 DLE API

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Abstrakt

V rámci příspěvku byla hodnocena odolnost ocelí X52 a X60 dle API vůči sulfidickému praskání pod napětím, a to pomocí testů za konstantního napětí pod mezí kluzu v souladu s předpisem NACE TM 0177 a také pomocí tahových zkoušek při pomalé rychlosti deformace. Oceli byly zkoušeny jednak ve stavu po válcování, jednak po laboratorním kalení ve vodě a následném vysokoteplotním popouštění při teplotě 600 °C. Při testech za konstantního napětí závisela odolnost ocelí vůči sulfidickému praskání pod napětím na strukturním stavu a byla výrazně lepší po kalení a popouštění podobně jak je pozorováno v případě vodíkem indukovaného praskání. Při testech pomocí tahových zkoušek při pomalé rychlosti deformace byla odolnost ocelí vůči sulfidickému praskání pod napětím na struktuře více méně nezávislá. Na druhé straně, při hodnocení odolnosti ocelí pomocí tahových zkoušek při pomalé rychlosti deformace hrála důležitou roli orientace zkušebních těles vzhledem ke směru tváření. Degradace ocelí byla mnohem výraznější u zkušebních těles orientovaných ve směru přes tloušťku, zatímco u zkušebních těles odebraných ve směru podélném nebo příčném byla míra degradace nižší. Nejvýznačnějšími iniciačními místy pro vznik trhlin byly při testech pomocí tahových zkoušek při pomalé rychlosti deformace nekovové inkluze. U zkušebních tyčí orientovaných ve směru podélném nebo příčném byly trhliny iniciovány přednostně na ásticích globulárních oxidů. U zkušebních tyčí orientovaných ve směru přes tloušťku byla iniciace trhlin pozorována výhradně na protvářených částicích sulfidů manganu. Toto chování lze vysvětlit pomocí rozdílných geometrických charakteristik různých typů nekovových vměstků. Sulfidy manganu se jeví jako spíše neškodné inkluze při testech v podélném nebo příčném směru díky své malé tloušťce. Ve směru přes tloušťku působí velmi škodlivě, neboť se zde projeví jejich velká plocha a zároveň i ostrost.

Abstract

Resistance of X52 and X60 API steels to sulphide stress cracking was tested by means of tensile tests at a constant load below the yield strength in accordance to NACE standard TM 0177 and also by means of slow strain rate tensile tests. Both steels were tested in two different states, after hot rolling and after laboratory water quenching and high temperature tempering at 600 °C. The results showed that steel resistance to sulphide stress cracking depended strongly on the microstructure but only when tests at the constant load were performed. In this case, quenching and tempering increased steel resistance to sulphide stress cracking considerably, in a similar way as in the case of hydrogen induced cracking. The results of slow strain rate tensile tests were different: they were similar regardless of the heat treatment. On the other hand, degree of degradation depended strongly on specimen orientation. Degradation of steel properties was much more pronounced for tests performed in through thickness orientation of tensile specimens while it was less pronounced for tests performed in longitudinal or transversal orientation of tensile specimens. Non-metallic inclusions seemed to play an important role as crack initiation sites during slow strain rate tensile tests. For tests performed in longitudinal or transversal direction cracks initiated on globular oxides predominantly while in through thickness orientation cracks initiated on elongated manganese sulphides only. This behaviour could be related to geometric characteristics of different kinds of non-metallic inclusions. Manganese sulphides seem to be rather harmless inclusions for specimens in longitudinal or transversal directions thanks to their low thickness. They become very noxious for specimens taken in through thickness direction because of their high area and sharpness.

Key words: API steel; sulphide stress cracking; slow strain rate test; microstructure.

1. Introduction

Steels that are used in the oil industry and that are in contact with crude oil or natural gas may succumb to severe degradation due to hydrogen sulphide, which is nearly always present, to some extent, in the above-mentioned substances. The main kinds of steel degradation that can be encountered in environments containing hydrogen sulphide are known as hydrogen induced cracking (HIC), sulphide stress cracking (SSC) and stress oriented hydrogen induced cracking (SOHIC) and they have been described in the literature in details [1]. In the case of HIC it is usually recognised that microstructure, rather than anything else, is the key parameter that influences the resistance of steels. On the contrary, resistance of steels to SSC is usually related to their strength level. Microstructure is considered only indirectly, by the performed heat treatment. Resistance of steels to SSC is tested by several kinds of tests. In the majority of cases, tests with constant applied loads, below the yield strength of the steels, are used . In some cases, slow strain rate tests (SSRT) are also used, mainly to shorten exposure time. It is assumed implicitly that both kinds of SSC tests, with constant loads below the yield strength as well as slow strain rate tests, give the same steel classification [2,3].

The purpose of this paper is the study of resistance of X52 and X60 API steels to SSC. The main objective is the role of testing conditions, i.e. comparison of results obtained by means of constant load tensile tests and slow strain rate tests.

2. Experimental procedure

The materials used in this study were tubes made of X52 (255/25 mm) and X60 (500/25 mm) steels meeting the requirements of the API 5L standard. The chemical composition of the steels is given in Table 1.

Table 1 Chemical composition of the steels (mass %)

Steel	C	Mn	Si	P	S	Cr	Ni	V	Nb	Ti
X52	0.09	0.92	0.28	0.007	0.010	0.02	0.01	0.004	0.03	0.01
X60	0.21	1.52	0.19	0.012	0.003	0.16	0.15	0.05	0.03	0.01

Steels were studied in two different states:

- in as-received state (AR) - after rolling;
- after laboratory quenching and tempering (QT) 870°C/40 min/water + 600°C/2 hours/air.

Structure analysis was performed by means of optical metallography (OM) and scanning electron microscopy (SEM). Resistance to sulphide stress cracking was tested using two different methods: constant load tests (CLT) in accordance with NACE TM 0177 Standard, Method A and slow strain rate tests (SSRT). The testing solution was a water solution consisting of 5.0 wt. % NaCl and 0.5 wt. % glacial acetic acid saturated by H_2S in both test types.

Slow strain rate tests were performed on smooth cylindrical specimens with diameter 3 mm. Specimens were taken in longitudinal (L), transversal (T) and through-thickness (TT) directions of the tubes. The gauge length was 15 mm in L and T directions. Very short specimens had to be used in a TT direction. Tests were carried out at ambient temperature and at a strain rate of 5.10^{-6} s^{-1}.

3. Results and discussion

3.1 Microstructure and mechanical properties

For each of the studied steels, microstructure in as-received state consisted predominantly of ferrite with some portion of pearlite, presented in the form of more or less pronounced bands. In X60 API steel, bands containing bainite or martensite were also observed. Laboratory quenching and tempering did not result in a fully martensitic or bainitic structure in the mid-thickness of X52 API steel. The microstructure corresponded rather to tempered bainite with a non-negligible amount of ferrite. For X60 API steel, no ferrite was observed and the microstructure was fully a tempered martensite. Banded structure, presented in as-received state, disappeared for both studied steels. Microstructure of the steels was presented in details elsewhere [4].

The mechanical properties of the studied steels are summarised in Table 2. It is obvious that both steels met standard requirements. Quenching and tempering increased yield strength and tensile strength considerably without any particular loss of plastic properties (elongation) or notch toughness. For X52 steel, the difference between YS in as-received and quenched and tempered state was approximately 100 MPa; for X60 API steel it was 140 MPa.

Table 2 Mechanical properties of the studied steels (L direction)

Steel	**R_e (MPa)**	**R_m (MPa)**	**A_5 (%)**	**$KV_{0°C}$ (J)**
X52/AR	390	515	24.5	196
X52/QT	486	610	22.6	218
X60/AR	593	770	21.8	148
X60/QT	733	792	20.2	165

3.2 Constant load SSC tests

The results of SSC tests performed at constant loads in accordance with NACE TM 0177, Method A are presented in Fig. 1.

Presented results of CLT clearly show the same behaviour as usually observed in the case of hydrogen induced cracking (HIC) tests, confirming the important role of the microstructure in SSC process. Laboratory quenching and tempering, resulting in tempered martensitic structure or in a mixture of tempered bainite and ferrite, considerably increased steel resistance to SSC. Very similar findings were presented in [2].

Examples of fracture surfaces of broken CLT specimens are shown in Fig. 2 a and b for AR and QT states, respectively. Fracture surfaces consisted predominantly of quasicleavage fracture (QCF) with some large cracks perpendicular to the fracture surface in as-received state. Only a small portion of fracture surface corresponded to a transgranular ductile fracture. A rupture probably occurred as a consequence of HIC and SSC. In some smaller quasicleavage regions, which were circular or elliptical, some "holes" were observed in their centres. These holes could correspond to the presence of non-metallic inclusions in steels, as SSC initiation sites.

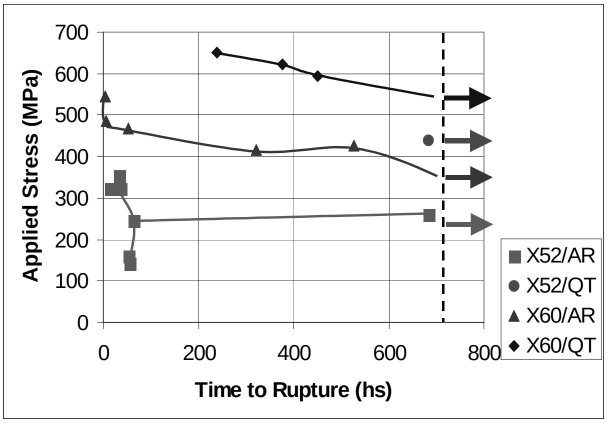
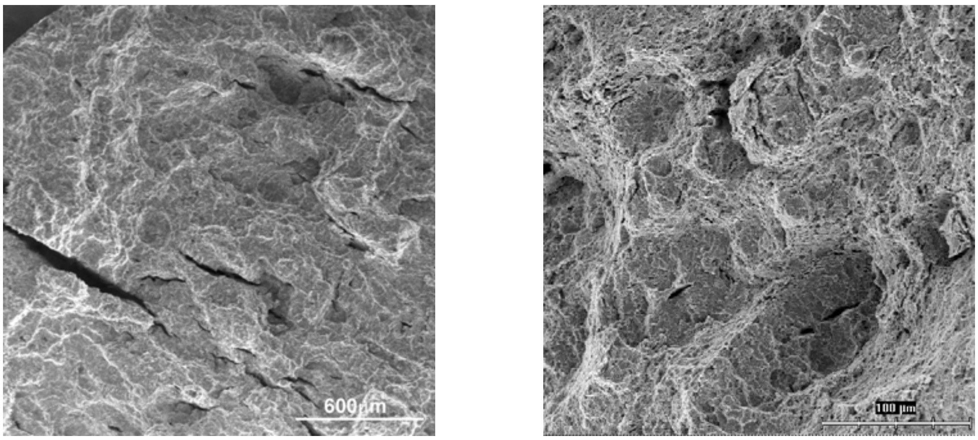


Fig.1 Results of SSC tests – Time to rupture as a function of applied stress for the studied steels



a) General view of fracture surface – X52 API steel/AR state.

c) Quasicleavage areas with "holes" in their centres – X60 API steel/QT state.

Fig.2 Fracture surfaces of CLT specimens

After laboratory quenching and tempering, fracture surfaces of broken CLT specimens were similar to those in the as-received state. Nevertheless, no large cracks perpendicular to fracture surface were observed after quenching and tempering. It can be assumed that there was no important contribution of HIC during CLT after quenching and tempering as a result of microstructural changes.

3.3 Slow Strain Rate Tests

Tests performed without hydrogen (in air) revealed that material properties were quite isotropic with no significant changes depending on specimen orientation. The presence of hydrogen was mainly seen in a decrease of plastic properties - elongation and reduction in area (RA). High anisotropy of tensile properties, especially anisotropy of reduction in area, was found in the presence of hydrogen, as the decrease in RA was much greater in the TT direction. It should be pointed out that in the case of SSRT the beneficial effect of quenching and tempering was not as great as in the case of CLT results. In Fig. 3 reduction in area is plotted as a function of the specimen orientation for as-received state and quenched/tempered state of X52 API steel. It is obvious that the material response to hydrogen was similar, regardless of its structure state.

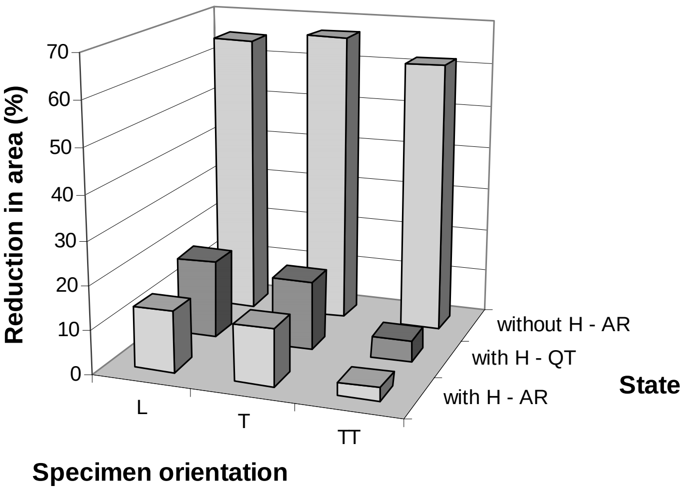
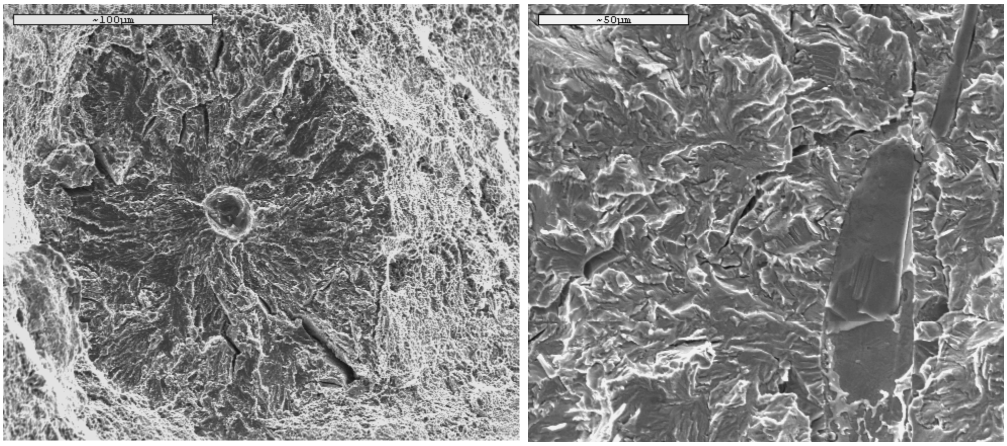


Fig.3 Results of SSRT – Reduction in area as a function of specimen orientation for AR and QT state of X52 steel

Fracture surfaces were entirely transgranular ductile in the absence of hydrogen. On hydrogenated specimens, fracture surfaces were mixtures of quasicleavage and ductile fractures. There were numerous quasicleavage regions on fracture surfaces, either circular or elliptical. In many of them it was possible to identify non-metallic inclusions as the crack initiation sites. The inclusions were predominantly globular oxi-sulphides in L and T directions and exclusively elongated manganese sulphides in TT direction. Examples of fracture surfaces of SSRT specimens are given in Fig. 4.



a) Globular oxide acting as crack initiation site – L direction

b) Elongated MnS acting as crack initiation site – TT direction

Fig.4 Examples of fracture surfaces of SSRT specimens

A question arises why material resistance to SSC tested by SSRT does not depend on microstructure as is the case of CLT. It seems that results depend significantly on the degree of plastic deformation. If SSC is tested by CLT, specimens are generally loaded in the elastic domain. Although some plastic deformation is possible in the regions of stress concentration, it can never be a bulk plastic deformation.

In the case of SSRT, the situation is quite different. It is probably a result of bulk plastic deformation during these tests, which enables extensive hydrogen deformation interaction, as proposed by Magnin [5]. It is well known that non-metallic inclusions act as hydrogen traps and a higher hydrogen concentration can be expected in their vicinity, even without external stress. During plastic deformation, hydrogen concentration around inclusions can increase considerably due to hydrogen transported by dislocations. As dislocations are blocked in their movement by non-metallic inclusions, they release transported hydrogen in the vicinity of the inclusions. Local hydrogen concentration can thus reach a sufficient value to provoke crack initiation. The presented results indicate that the role of non-metallic inclusions in SSC is enhanced by those tests that enable high hydrogen-deformation interaction. It is obvious that this behaviour is closely related to the strain rate that has to be sufficiently low.

4. Conclusions

The results obtained applying constant load and slow strain rate SSC tests on X52 and X60 API steels having different mechanical properties can be summarised as follows:

- High dependence of SSC resistance on microstructure was observed when constant load tests were performed. Quenching and tempering resulted in higher mechanical properties but also gave considerably higher SSC resistance in comparison with as-received state.
- In the case of SSRT, the resistance of steels did not seem to depend on microstructure. Non-metallic inclusions represented the only crack initiation sites. The enhanced role of non-metallic inclusions could be attributed to an important hydrogen-deformation interaction occurring during these tests.

- The degree of susceptibility to SSC varied as a function of specimen orientation for SSRT. The worst situation was observed for the through-thickness orientation of the specimens. This behaviour could be related to the different geometric characteristics of globular oxides and elongated manganese sulphides presented in the tested steels.

Acknowledgements

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Literature

[1] Timmins P. F.: Solutions to Hydrogen Attack in Steels, ASM Int., Ohio, 1997.
[2] Carniero R. A., Ratnapuli R. C., Cunha Lins V. F.: Mater. Sci. Eng., A357, 2003, 104.
[3] Contreras A., Albiter M., Salazar R., Perez V.: Mater. Sci. Eng. A407, 2005, 45.
[4] Sojka J. et al.: Acta Metallurgica Slovaca, 11, 2005, 323.
[5] Magnin T.: Acta Mater., 44, 1996, 1457.
[6] Tsay L. W., Chi M. Y., Chen H. R., Chen C.:Mater. Sci. Eng. A416, 2006, 155.