



The effect of hydrogen concentration on fracture of pipeline steels in presence of a notch

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ARTICLE INFO

Article history:

Received 19 May 2010

Received in revised form 13 October 2010

Accepted 18 October 2010

Available online 25 October 2010

Keywords:

Hydrogen charging of metal

Hydrogen concentration

Cathodic polarisation

Current density

Acoustic emission

Work of local fracture initiation

Critical hydrogen concentration

ABSTRACT

The comparative assessment of local fracture initiation at notches in API grade pipeline steels: X52, X70 and X100 have been done for conditions of electrochemical hydrogenating. The factor of hydrogen concentration in metal was taken into account. The relationship between hydrogen concentration in metal and work for initiation of the local fracture emanating from the notch has been derived. The existence of some critical hydrogen concentration, which causes the significant loss of local fracture resistance of material, was also shown and discussed.

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1. Introduction

General reason, which increases of attention to the problem hydrogen degradation of pipeline steels, is fact that hydrogen will play a decisive role in a future energy system, when fossil fuels have become scarce and thus expensive and/or unsuitable from ecological reasons. The number of aspects related to the technical feasibility and economics of developing a hydrogen energy infrastructure are presented and discussed in literature during last decades [1,2]. The possible use of existing pipeline networks for mixtures of natural gas and hydrogen offers a unique and cost-effective opportunity to initiate the progressive introduction of hydrogen as part of the development of a full hydrogen energy system [3].

From this reason, the safety, durability and integrity issues related to hydrogen/natural gas mixtures in the existing natural gas system become actual and important. First of all, there is a potential problem of so-called “hydrogen embrittlement” of pipeline material – the effects of transported hydrogen on material mechanical properties [4].

Moreover, the specific long-term exploitation of pipelines promotes the hydrogenating of steel. The external environmental conditions cause free corroding processes, where hydrogen can evaluate on metal surface as result of cathodic counterpart of the anodic dissolution reaction. This fact has been proved by several studies [5–7].

External interference incidents are characterised by potentially severe consequences: leaks and ruptures. In majority, these types of incidents are caused by initial damaging of the pipe external surface as result of third party interference e.g. by scratches and gouges and also pitting corrosion. Such defects can be considered as typical stress concentrators and consequently the privileged sites where the further damaging and failure processes occur. Here, it should be noted that

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Nomenclature

D	outer diameter of pipe in mm
t	wall thickness of pipe in mm
σ_Y	yield stress in MPa
σ_U	ultimate stress in MPa
E	potential of cathodic polarisation in mV
i	density of cathodic current in mA/cm ²
τ	time exposition in h
Q_H^{eV}	total quantity of evaluated hydrogen on metal surface in mA s
Q_H^{abs}	total quantity of absorbed hydrogen by metal in mA s
C_H	hydrogen concentration in metal in mol/cm ³
P	load in N
Δ	displacement in m
U_i	work for fracture initiation from the notch N m
U_f	total work for specimen fracture N m

environmental effects on localised stressed volumes of material are principally different from the effects on the bulk of material [8].

Nowadays, the pipe diameter has been significantly increased due a strong needs in energy carriers. The pipelines by diameter of 48 in. are now operating without a proportional increasing in thickness from technological (formability) reasons. At the same time, service pressure is expected to grow until 120 bars and, consequently, the constitutive yield stress of steels was increased. In prevail majority the existed pipelines are made with steel X52. The modern pipelines are made with steel X70 and steel X100 serves for designing of the advanced pipeline systems.

In the present study the comparative assessment of local strength at notches of three API grade pipeline steels: X52; X70 and X100 has been done for conditions of hydrogen charging. Here, the factors of cathodic hydrogen charging, time of exposition, hydrogen concentration in metal and applied load were taken into account. The relationship between hydrogen concentration and work of fracture initiation has been derived and discussed.

2. Object of study and experimental procedure

The objects of study were three API grade pipeline steels, namely: X52; X70 and X100. Nowadays, the steel X52 is the most usable in existed gas pipelines and another two steels are the objects of implementation into new pipeline systems.

The specimens for tests were machined from real pipes (Table 1). The chemical composition of steels and their mechanical properties in air are given in Tables 2 and 3 respectively. The structural specificity of steels can be seen in Fig. 1. The steel X52 has a microstructure, which consisted predominantly of ferrite and pearlite under average grain size about 40–60 μm (Fig. 1a). The microstructure of steel X70 is polygonal ferrite (size about 20–30 μm) with small martensitic–austenitic particles at grain boundaries (Fig. 1b). The resulting microstructure of steel X100 is mainly ferritic–bainitic with grain size in the range 5–10 μm (Fig. 1c).

For test the special “Roman tile” [9] specimens were used. The specimens were notched for modelling of the longitudinal external defects under operating internal pressure. The specimen view and notch geometry are given in Fig. 2.

The study was conducted in special soil solution NS4 with pH = 6.7 [10]. Chemical composition of this environment is given in Table 4.

The specimens were coated by special dielectric isolation, excepting the small area at the notch (Fig. 3) [11]. The size of auxiliary (counter) electrode was 20 × 40 mm. In this case its square is significantly larger than the working electrode area, which to be polarised. During testing the specimens were immersed into the cell with NS4 solution (Fig. 3) and exposed under constant potential of cathodic polarisation $E_{cath} = \text{const}$. For this experimental procedure the Potentiostat VMP has been used. During all test the level of pH was controlled and regulated between 6.6 and 6.7 by bubbling of CO₂ gas. The nitrogen gas was also used for stabilization of pH and removing the dissolved oxygen from solution. The gas bubbling was set in the following proportion: 80% of N₂ gas and 20% of CO₂ gas.

Table 1
Size of pipes.

Steel grade	Outer diameter D , mm	Wall thickness t , mm
X52	610	11.0
X70	710	12.7
X100	950	16.4

Table 2

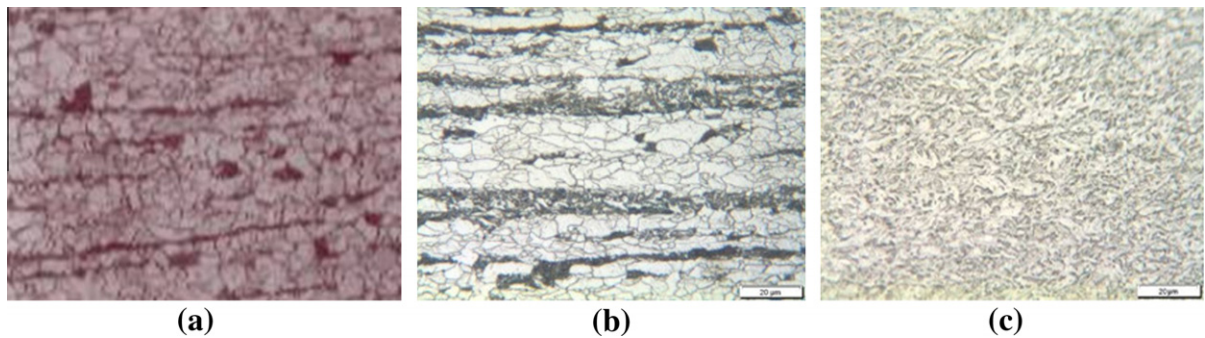
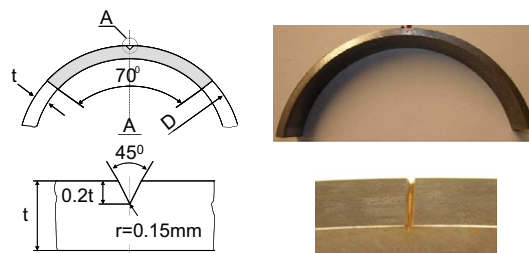
Chemical compositions of steels (wt.%).

Steel grade	C	Mn	Si	Cr	Ni	Mo	S	Cu	Ti	Nb	Al
X52	0.206	1.257	0.293	0.014	0.017	0.006	0.009	0.011	0.001	<0.03	0.034
X70	0.125	1.68	0.27	0.051	0.04	0.021	0.005	0.045	0.003	0.033	0.038
X100	0.059	1.97	0.315	0.024	0.23	0.315	0.002	0.022	0.022	0.046	0.037

Table 3

Mechanical properties of steels in air at ambient conditions.

Steel grade	σ_U , MPa	σ_Y , MPa	Elongation %	Reduction in area %
X52	528	410	30.2	–
X70	712	590	18.3	–
X100	890	866	6.75	76

**Fig. 1.** Structural specificity of pipelines steels in transversal direction (X500): (a) X52; (b) X70 and (c) X100.**Fig. 2.** "Roman tile" specimen and geometry of notch.**Table 4**

Chemical composition of NS4 solution (g/l).

KCl	NaHCO ₃	CaCl ₂ ·2H ₂ O	MgSO ₄ ·7H ₂ O
0.122	0.483	0.181	0.131

Here, it should be noted that specific feature of presented study is considering of hydrogenating conditions, which are very similar to real operating pipelines, therefore received data are more realistic for prediction of hydrogen embrittlement phenomenon than recorded in literature studies that were conducted with using of cathodic charging of hydrogen under high density of polarisation current [12–15].

In given tests, the specimens were hydrogen charged at some constant potential of polarisation, which is slightly negative than free corrosion potential for the given steels. Taking into account that for studying steels the free corrosion potential is about $E_{corr} = -800$ mV(SCE), it has been chosen: $E_{cath} = -1000$ mV(SCE) = const.

Moreover, for simulating of the service conditions, the specimens were loaded during the hydrogen-charging process. The level of load was defined as the gross hoop stress σ_{gross} , which corresponds of the internal pressure in pipe under exploitation $p_{exp} = 70$ bar (see Table 5).

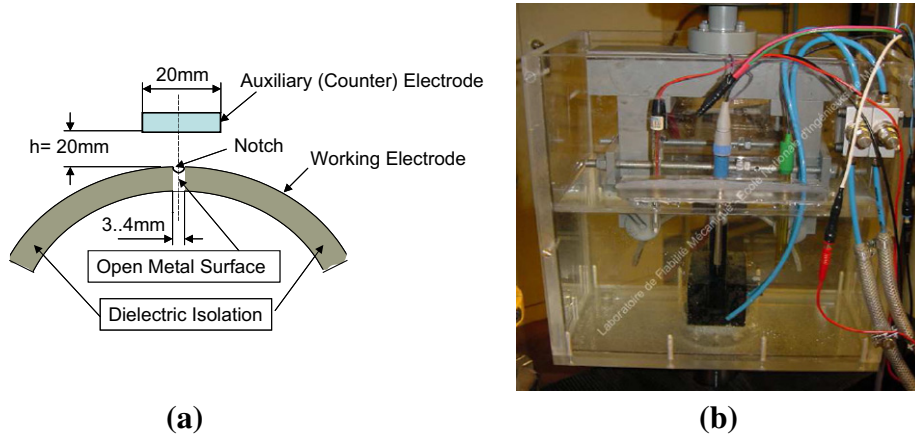


Fig. 3. Mutual location of working electrode (specimen) and auxiliary (counter) electrode (a) and general view of corrosion cell (b).

The hydrogen-charging process was controlled by registration of the cathodic polarisation current $I_{cath}(\tau)$. The total quantity of evaluated hydrogen on metal surface during time of exposition τ_{exp} can be found as:

$$Q_H^{ev} = \int_0^{\tau_{exp}} I_{cath}(\tau) d\tau \quad \text{under } E_{cath} = \text{const.} \quad (1)$$

Hydrogen concentration in metal has been determined on the base of hydrogen discharging process under anodic polarisation with using of hydrogen electrochemical oxidation method proposed in work [16]. For this aim, the standard three-electrode electrochemical cell has been used. The hydrogen discharging of specimen was carried out in 0.2 M NaOH (pH = 12.4) solution under anodic polarisation $E_{anodic} = +168$ mV(SCE) during some defined time τ_{dis} . The total quantity of absorbed hydrogen by metal can be defined as:

$$Q_H^{abs} = \int_0^{\tau_{dis}} [I_H(\tau) - I_{ref}(\tau)] d\tau \quad \text{under } E_{anodic} = \text{const.} \quad (2)$$

Here $I_H(\tau)$ is anodic polarisation current for hydrogen charged specimen and $I_{ref}(\tau)$ is anodic polarisation current for specimen without hydrogen (reference curve). Calculation of hydrogen concentration was done according to formula:

$$C_H = \frac{Q_H^{abs}}{zFv}. \quad (3)$$

where z is the number of electrons take in reaction; F is the Faraday constant; v is the effective volume of specimen: C_H (mol/cm³); Q_H^{abs} [A · s]; $z = 1$; $F = 9.65 \times 10^4$ C/ mol.

After assigned exposition under hydrogenating conditions, all specimens were tested to failure under increasing static loading, according to 3-point bending scheme. Testing machine INSTRON [17] was used under the constant rate of displacement of grips $d\Delta/d\tau = 0.02$ mm/s. The “load–displacement” diagram and acoustic emission (AE) signals were simultaneously registered by PC during the tests. The start of fracture process has been defined by acoustic emission method [18].

The detailed description the given test procedure and specificity of experiments can be found in our work [10,22].

3. Results and discussion

Process of hydrogen charging of pipeline steels at the given conditions of cathodic polarisation was characterised by following parameters: averaged meaning of cathodic current density i_c ; total quantity of evaluated Q_{ev} and absorbed Q_{abs} hydrogen; coefficient of efficiency of hydrogen permeation in metal $k = Q_{abs}/Q_{ev}$ and hydrogen concentration in metal C_H . The numerical values of these parameters are given in Table 6.

Table 5
Values of applied tensile stress σ_{gross} .

Steel grade	D , mm	t , mm	σ_{gross} , MPa
X52	610	11.0	194.1
X70	710	12.7	195.7
X100	950	16.4	202.7

Table 6 contains the direct experimental data of hydrogenation of pipeline steels, which were received from single test for each time of exposure τ . It should be noted that hydrogen charging of metal is complicated process and some scatter of the results is always inherent for such tests.

Taking into account the scatter for received data, the power equation for description of dependence $C_H = F(\tau)$ was chosen for further considerations. This equation allows the continuous growth of hydrogen concentration in metal with increasing of exposure time that is expectable for given process:

$$C_H = A \times 10^{-6} \times \tau^m \text{ (mol/cm}^3\text{)}. \quad (4)$$

where A and m are constants (see Table 7).

This relation and data in Table 7 can be used for forecasting of hydrogen concentration in the given steels of API grade during of long-term exploitation of pipelines. This is important for preventing of possible brittle failure of pipes, which can occur at some critical concentration of hydrogen in metal [10,19,20].

Based on Eq. (4), the summarised result of comparative assessment of electrochemical hydrogen absorption by pipeline steels with different strength is given in Fig. 4. It can be seen that for assigned testing conditions, i.e. in deoxygenated, near-neutral pH NS4 solution and under loading by tensile stress, which simulates of operating conditions, the resistance to hydrogen absorption is highest for steel X100 and it decreases with decreasing of steel strength.

As example of comparative assessment of electrochemical hydrogen absorption by stressed pipeline steels was demonstrated for some conventional fixed time of exposition $\tau = 200$ h (Table 8). It has been found that hydrogen concentration in studied pipeline steels is practically linear function of yield stress σ_Y or ultimate stress σ_U (Fig. 5).

The study of local strength of notched specimens in presence of hydrogen was resulted in the dependencies of work for fracture initiation from the notch U_i and total work for specimen fracture U_f . The determining of the points on “load–displacement” diagram, which correspond to fracture initiation from the notch and total specimen fracture were made on the analysis of the acoustic emission (AE) signals that were simultaneously registered by PC during the tests. Here the special software was used [18]. The scheme of determination of parameters U_i and U_f is presented in Fig. 6:

and

$$U_i = \int_0^{\Delta_i} P(\Delta) \cdot d\Delta \quad U_f = \int_0^{\Delta_f} P(\Delta) \cdot d\Delta \quad (5)$$

The test results for different time of exposition of specimens in hydrogenating conditions are given in Table 9. Here the hydrogen concentration C_H in metal was calculated by using of the analytical relation (4).

The main observation based on these results (Figs. 7 and 8) is the existence of some critical time of exposition and as a consequence – some critical hydrogen concentration C_H^* , when the essential decreasing of fracture toughness value is occurred (Fig. 9). The values of C_H^* for studied steels are given in Table 10.

Here, it should be pointed on some anomaly behaviour of steel X70. In hydrogenating conditions its resistance to local fracture from notch is lowest among other studied steels. From other side, the same tendency can be seen for test in air (Table 9). Probably, this phenomenon is caused by very high sensitivity of this steel to notch (so-called “notch effect”) even in inert condition as result of its microstructural specificity (see Fig. 1b). This notch effect is quite strong and it can grade the hydrogen effect. For steel X70 is also inherent higher scattering of test results (see, for example, Fig. 9b) that shows on complicated competitive processes during hydrogen assisted local fracture.

Table 6

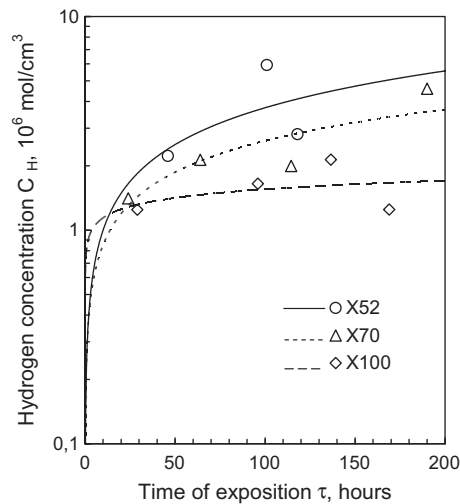
Data for determining of hydrogen concentration in stressed pipeline steels.

τ , h	Q_H^{ev} , mA s	i_c , mA/cm ²	$\tau_{dis} = 1$ h		$k = \frac{Q_H^{abs}}{Q_H^{ev}}$
			Q_H^{abs} , mA s	C_H , 10 ⁶ mol/cm ³	
API X52					
46	57,360	0.226	362.4	2.224	0.0063
101	168,798	0.240	1216.5	5.924	0.0072
118	147,522	0.524	458.6	2.814	0.0031
API X70					
24	55,189	0.276	346.1	1.408	0.0063
64	144,032	0.246	576.4	2.133	0.0040
114.5	300,806	0.315	490.7	1.997	0.0016
190	77,715	0.315	1241.1	4.593	0.0160
API X100					
29	54,562	0.015	1158.2	1.246	0.0212
96	132,289	0.036	1539.7	1.651	0.0116
136.5	290,317	0.382	1991.7	2.136	0.0069
169	357,512	0.669	1164.7	1.250	0.0033

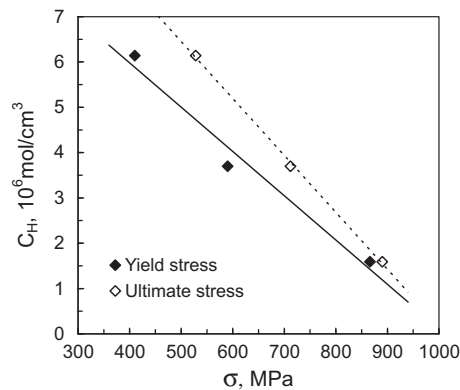
Table 7

Values of constants in formula (4).

Steel	$A, \frac{\text{mol/cm}^3}{(\text{h})^m}$	m
API X52	0.30	0.57
API X70	0.40	0.42
API X100	0.80	0.13

**Fig. 4.** Hydrogen concentration in metal versus time of exposition of stressed specimens under cathodic polarisation of pipeline steels API X52, X70 and X100.**Table 8**Hydrogen concentrations in steels API X52, X70 and X100 versus their mechanical properties ($\tau = 200$ h).

Steel	σ_Y , MPa	σ_U , MPa	C_H , 10^6 mol/cm^3
X52	410	528	6.14
X70	590	712	3.70
X100	866	890	1.59

**Fig. 5.** Assessment of hydrogen concentration in API grade pipeline steels as function of yield stress σ_Y and ultimate stress σ_U (time of exposition under hydrogenating conditions $\tau = 200$ h).

Therefore, for assigned testing conditions, steel X52 is preferable than steel X70 from the point of view of local strength at notches. The steel X100 demonstrates quite high resistance against notch effect, but within close limits of hydrogen concentration variables (Figs. 8 and 9c).

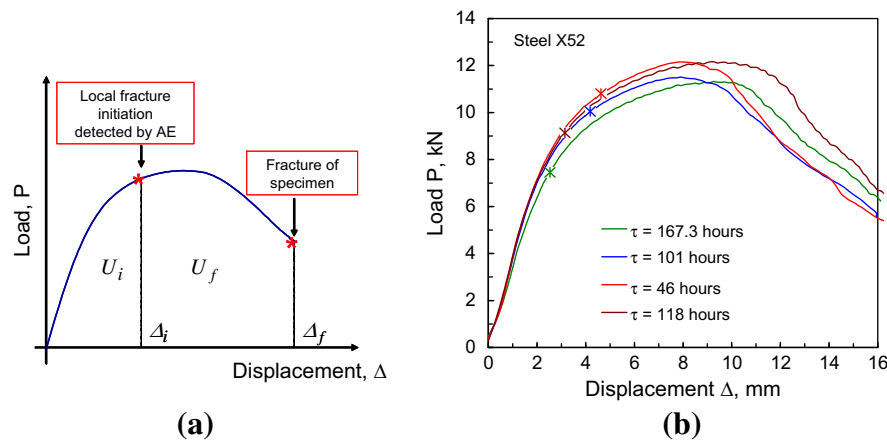


Fig. 6. Scheme of determining of parameters U_i and U_f (a) and real sample for steel X52 (b).

Table 9
Test data on local fracture from notch in roman tile specimens at hydrogenating conditions.

τ , h	U_i , N m	U_f , N m	C_H , 10^6 mol/cm ³ formula (4)
<i>API X52</i>			
0 (air)	27.23	226.47	0
46	32.12	170.84	2.660
101	26.57	126.33	4.165
118	15.53	132.98	4.551
167.3	10.47	124.90	5.553
<i>API X70</i>			
0 (air)	17.64	145.46	0
24	12.68	136.26	1.52
64	6.88	142.62	2.294
114.5	11.41	135.59	2.929
190	15.25	129.00	3.623
<i>API X100</i>			
0 (air)	119.00 ^a	318.00 ^a	0
29	114.75	312.63	1.239
96	102.86	296.78	1.448
136.5	64.29	258.46	1.516
169	12.72	241.77	1.558

^a Meanings received by extrapolation.

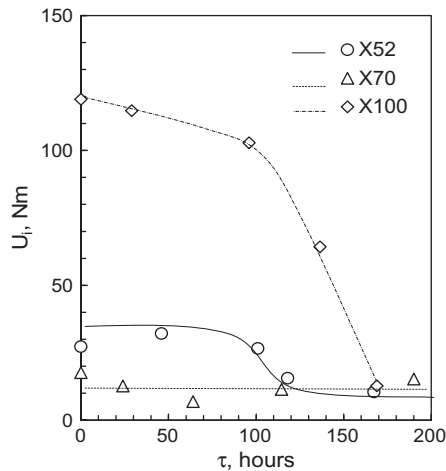


Fig. 7. Work for local fracture initiation U_i from notch as function of time of exposition of specimens in hydrogenating conditions.

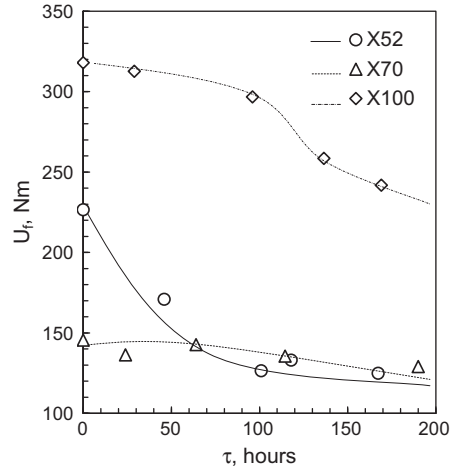


Fig. 8. Total work for notched specimen fracture U_f as function of time of exposition of specimens in hydrogenating conditions.

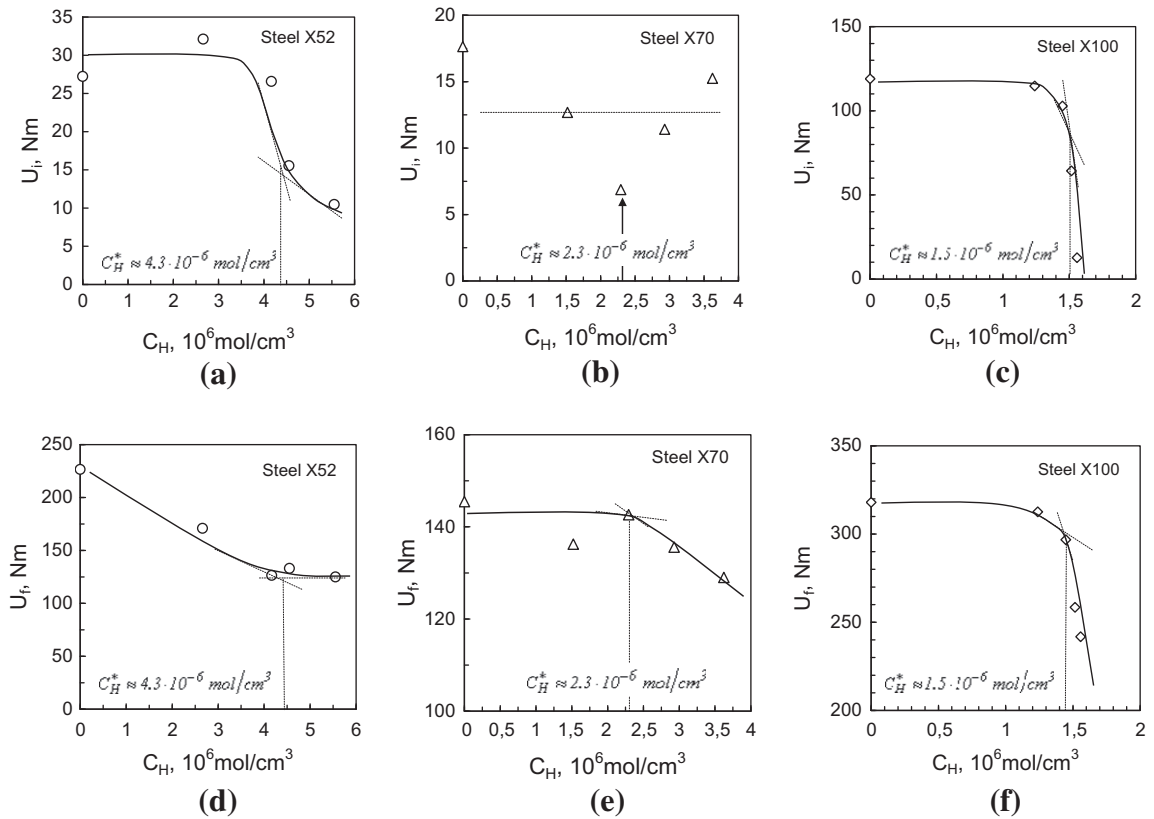


Fig. 9. Determining of critical hydrogen concentration C_H^* in steels API X52, X70 and X100: (a–c) based on U_i data; (d–f) based on U_f data.

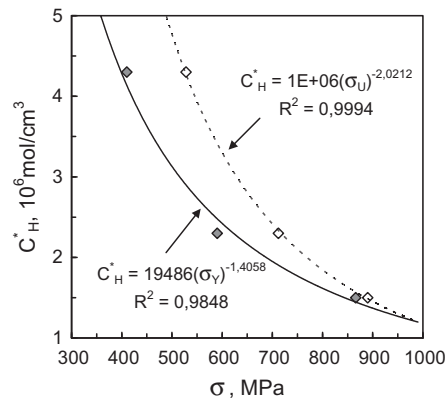
It may be stated that definition “critical concentration” is often used in studies of problems of hydrogen in metals and alloys [19–21], but with different physical sense. In given study, the critical hydrogen concentration C_H^* can be classified as concentration, which causes the significant loss of local fracture resistance of material at notches.

From this point of view, the comparative assessment of parameter C_H^* for studied pipelines steels has shown the following. There is the tendency of monotonic decreasing of C_H^* values with increasing of yield stress σ_Y or ultimate stress σ_U steel (Fig. 10). This dependency can be described by power function, like to:

$$C_H^* = B_1 \cdot (\sigma_Y)^{n_1} \quad \text{or} \quad C_H^* = B_2 \cdot (\sigma_U)^{n_2} \quad (6)$$

Table 10Assessment of critical hydrogen concentrations C_H^* for API grade pipeline steels.

Steel	$C_H^*, 10^6 \text{ mol/cm}^3$	
	Based on U_i data	Based on U_f data
X52	4.3	4.3
X70	–	2.3
X100	1.5	1.5

**Fig. 10.** Critical hydrogen concentration C_H^* in API grade pipeline steels X52, X70 and X100 versus yield stress σ_Y and ultimate stress σ_U .

where B_1, B_2, n_1, n_2 are the some constants of material's properties and testing conditions. Here, the standard deviation $R^2 = 0.98\text{--}0.99$ that is fully acceptable (see Fig. 10). As first approximation, it can be concluded that

$$C_H^* \sim \frac{1}{\sigma_U^2}. \quad (7)$$

The decreasing of hydrogen sensitivity with increasing of yield stress of steel can be explained by the fact that dislocations are more pinning by precipitates introduced for high strength steel. Consecutively hydrogen atoms have less possibility to move dragging by dislocations. The relation (7) may be useful for preliminary assessment of steel sensitivity to hydrogen embrittlement at notch.

Finally it should be noted that the diagram “work of local fracture initiation – hydrogen concentration” could be a function of many factors, especially a radius of the notch tip. Therefore, the critical hydrogen concentration may be variable and depends on specificity of hydrogen interaction with local elastoplastic zone at the notch tip. The received curves $U_i = F(C_H)$ can be used for assessment to hydrogen embrittlement of pipelines with steels of API grade under assigned testing conditions, i.e.: size of pipe and as result – geometry of specimen; notch radius, rate of loading and mode of hydrogen charging. Also, these curves are the base for determining of the critical hydrogen concentration C_H^* .

4. Conclusions

The local fracture initiation at notches in API grade pipeline steels X52, X70 and X100 has been studied for conditions of electrochemical hydrogenating. The received results have led to the following conclusions.

For assigned testing conditions, i.e. in deoxygenated, near-neutral pH NS4 solution and under loading by tensile stress, which simulates of operating conditions, the resistance to hydrogen absorption is highest for steel X100 and it decreases with decreasing of steel strength.

The diagrams $U_i = F(C_H)$ can be useful for assessment to hydrogen embrittlement of pipelines with steels of API grade under assigned testing conditions.

Steel X70 is very sensitive to notch effect even in air and the presence of hydrogen strengthens this tendency. As result, for assigned testing conditions, steel X52 is preferable than steel X70 from the point of view of local strength at notches. Steel X100 demonstrates quite high resistance against notch effect, but within close limits of hydrogen concentration variables.

It is important to note that hydrogen concentration provides a fracture resistance transition similarly to temperature, loading rate and notch acuity. This phenomenon is describes for the first time (according to our knowledge) in this paper.

Some critical hydrogen concentration C_H^* exists which depends on material, notch radius and specimen geometry through stress triaxiality. Behind this critical stress concentration and for a given stress triaxiality and steels, there is a significant loss of local fracture resistance of material at notches. The tendency of monotonic decreasing of C_H^* values with increasing of yield stress σ_Y or ultimate stress σ_U of steels has been derived.

The concept of hydrogen concentration transition C_H^* may be considered as an important engineering parameter for forecasting of strength and reliability of exploited pipelines and under designing of new pipelines for hydrogen transportation as well.

Acknowledgements

Presented study was partly performed within the frame of the European project NATURALHY (EC Contract No. SES6/2004/502661). I. Dmytrakh would like to thank to Ecole Nationale d'Ingenieurs de Metz (ENIM) and Universite Paul Verlaine Metz for the opportunity to conduct this research.

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