



# EXPERIMENTAL STUDY OF THE MECHANISM OF HYDROGEN EMBRITTLEMENT OF METALS WITH THE BCC LATTICE\*

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The study gives results of investigations of the effect of hydrogen on the mechanism of metal fracture. In metal containing diffusible hydrogen, plastic deformation leads to formation of residual hydrogen, which is connected to formed dislocations and microcracks. The presence of hydrogen connected to dislocations leads to localisation of plastic deformation of metal. Initiation of microcracks occurs by the shear mechanism, and their further growth takes place due to formation of new defects at the old crack apex and their coalescence.

**Keywords:** arc welding, low-carbon steel, hydrogen embrittlement, diffusible and residual hydrogen, hydrogen localisation of ductility

Welding of structures of high-strength low-alloy steels involves a problem of formation of hydrogen-induced cold cracks, which is caused by such a phenomenon as hydrogen embrittlement (HE) of metal. The HE mechanism is based on interactions of hydrogen with dislocations and a change in properties of dislocation clusters under the effect of hydrogen. Theoretical aspects of the HE mechanism are considered in study [1]. The present study is dedicated to experimental investigation of the HE mechanism.

Ferritic-pearlitic steel VSt3sp (killed) of the following composition, wt.%: 0.12 C, 0.14 Si, 0.42 Mn, 0.1 Ni, 0.12 Cr, 0.022 S and 0.012 P, was used as the investigation material (Figure 1). Specimens of this steel were annealed at a temperature of 600 °C.

Mechanical properties of the hydrogen-containing specimens of steel VSt3sp were determined in the first

series of experiments. The specimens were saturated with hydrogen by the electrolytic method in the 5 % sulphuric acid solution with an addition of 0.05 % sodium thiosulfate for 8–13 h, the current density being 4 mA/cm<sup>2</sup>. Re-grinding of the specimens after hydrogenation took no more than 1 min. Before the mechanical tests the specimens were stored in liquid nitrogen. The contents of diffusible and residual hydrogen were determined by the chromatographic method [2].

In the second series of experiments, the specimens of steel VSt3sp were subjected to tension to different degrees of plastic deformation (10, 15 and 17 %). After that hydrogen was removed by heating the specimens to 50 °C and holding for 7 days. After the removal of hydrogen, the specimens were tensioned to fracture. The specimens that contained no hydrogen were subjected to the identical test cycle. Mechanical tests to uniaxial tension were carried out on cylindrical specimens with a gauge length of 30 mm and diameter of 5 mm.

Mechanical tests to uniaxial tension testing were conducted by using servohydraulic machine «Instron-1251». Prior to the tests the specimens were heated in alcohol to room temperature. It took no more than 3 min to heat a specimen and then place it in the grips, as well as fix the strain gauge. The strain rate in tension of the specimens was of 1·10<sup>-3</sup> s<sup>-1</sup>.

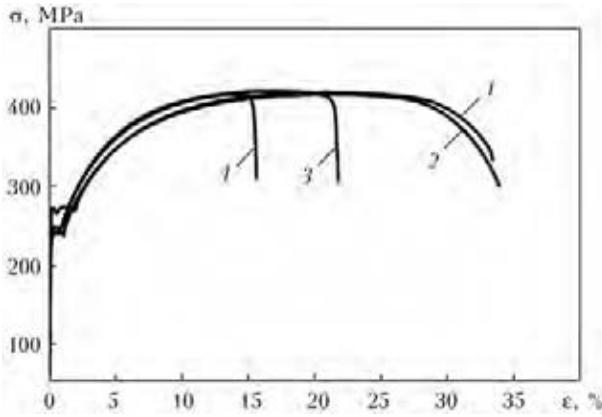
Fractographic analysis was carried out by using JEOL scanning electron microscope JSM-35CF.

Results of the uniaxial tension tests in the proof stress  $\sigma$ –relative strain  $\varepsilon$  coordinate system are shown in Figure 2. Mechanical properties of steel VSt3sp were recovered after hydrogenation and subsequent degassing (curves 1 and 2 in Figure 2). Increase in diffusible hydrogen content  $[H]_{diff}$  leads to fracture of metal at a lower degree of plastic deformation



Figure 1. Microstructure (x500) of steel VSt3sp

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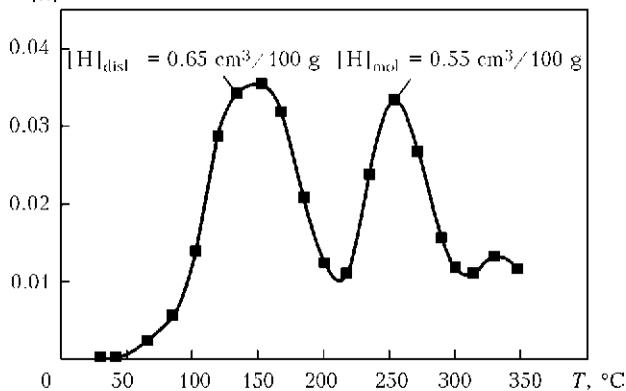
**Figure 2.** Effect of the content of diffusible hydrogen  $[H]_{diff}$  on fracture of steel VSt3sp specimens in uniaxial tension: 1 – initial state ( $\psi = 54\%$ ); 2 – after hydrogenation to  $[H]_{diff} \approx 7 \text{ cm}^3/100 \text{ g}$  and degassing ( $\psi = 62\%$ ); 3 –  $[H]_{diff} = 6.5 \text{ cm}^3/100 \text{ g}$  ( $\psi = 23\%$ ); 4 –  $[H]_{diff} = 8.5 \text{ cm}^3/100 \text{ g}$  ( $\psi = 15\%$ )

(curves 3 and 4 in Figure 2). Fracture of the hydrogen-containing metal occurs after stresses reach the value of tensile strength, i.e. at the beginning of localisation of plastic deformation in the form of a neck. The values of ductility of metal, i.e. elongation and reduction in area, are most sensitive to HE.

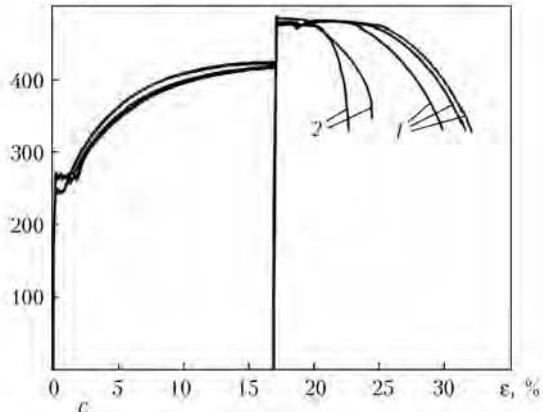
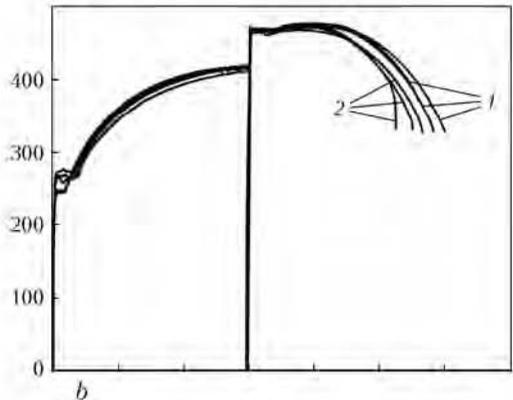
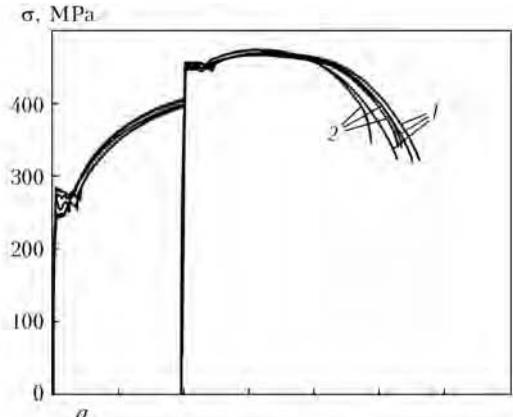
After mechanical tests the specimens were held at room temperature for 7 days. The content of residual hydrogen in fractured metal was measured by thermal desorption analysis. For this the specimens were cut out from a region with a uniform plastic deformation. The analysis results are shown in Figure 3. The first peak in the thermal desorption spectrum corresponds to hydrogen at dislocations ( $[H]_{disl}$ ), and the second one – to molecular hydrogen ( $[H]_{mol}$ ) located in microcracks, this being evidenced by its desorption temperature. New dislocations acting as hydrogen traps initiate during the process of plastic deformation. Accumulation of hydrogen at the dislocations facilitates their coalescence [1] and leads to initiation of microcracks. Molisation of hydrogen takes place after it gets into the formed defects.

To determine the effect of hydrogen on the mechanism of initiation of microcracks, the specimens of steel VSt3sp were preliminarily deformed to an elon-

$$v_{[H]}: (\text{cm}^3/100 \text{ g})/\text{min}$$

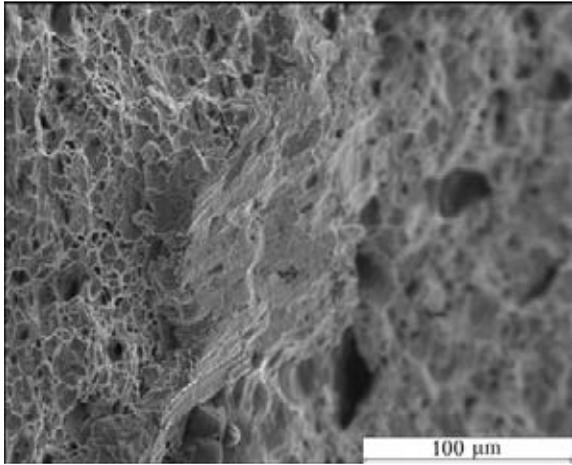


**Figure 3.** Rate of removal of residual hydrogen  $v_{[H]}$ .

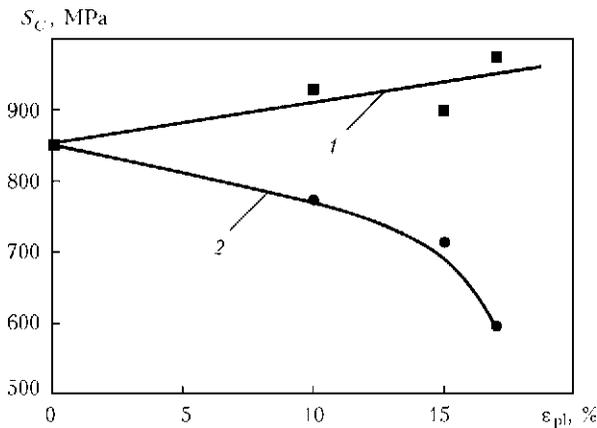


**Figure 4.** Tension diagrams for specimens of steel VSt3sp with different preliminary deformations: a –  $\epsilon = 10\%$  ( $\psi_{[H]} = 51\%$ ,  $\psi = 62\%$ ); b –  $\epsilon = 15\%$  ( $\psi_{[H]} = 49\%$ ,  $\psi = 57\%$ ); c –  $\epsilon = 17\%$  ( $\psi_{[H]} = 39\%$ ,  $\psi = 62\%$ );  $\psi_{[H]}$ ,  $\psi$  – average reduction in area of hydrogen-containing and hydrogen-free specimens, respectively; 1 – initial state; 2 – hydrogen content  $7 \text{ cm}^3/100 \text{ g}$  (the specimens were degassed after preliminary deformation)

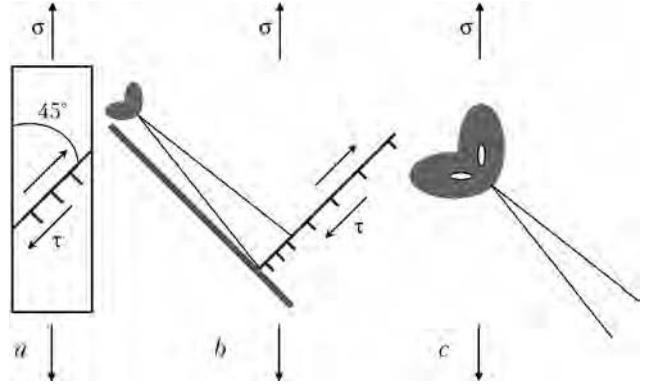
gation of 10, 15 and 17 % and then degassed, after which they were deformed to fracture. The content of diffusible hydrogen in the specimens after electrolytic saturation was  $7 \text{ cm}^3/100 \text{ g}$ . As shown by the thermal desorption analysis, after degassing of the deformed specimens of steel VSt3sp at a temperature of  $50^\circ\text{C}$  for 7 days the hydrogen was desorbed from them at a temperature above  $200^\circ\text{C}$ . Therefore, the hydrogen at dislocations was removed during degassing. The mechanical test results are shown in Figure 4. Strain ageing of metal takes place after unloading and holding at a temperature of  $50^\circ\text{C}$ . The value of strengthening does not depend on the presence of hydrogen



**Figure 5.** Microstructure of the surface of shear microcrack at the centre of steel VSt3sp specimen with a hydrogen content of  $7 \text{ cm}^3/100 \text{ g}$  after preliminary deformation of 17 %

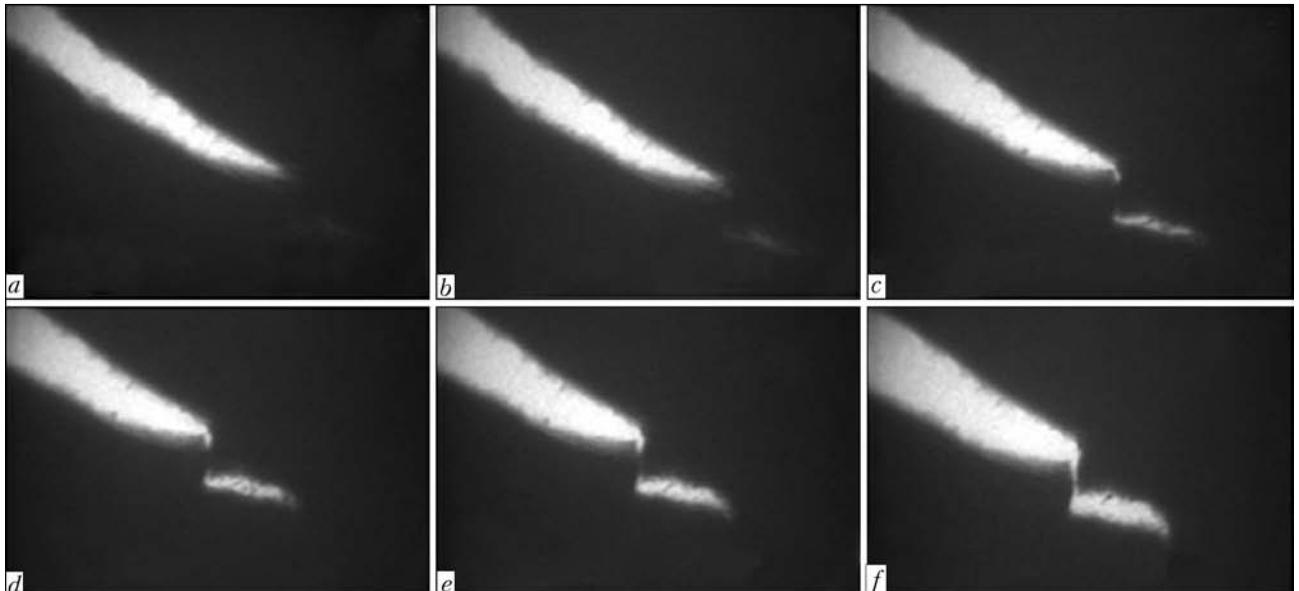


**Figure 6.** Effect of preliminary plastic deformation  $\epsilon_{pl}$  of specimens of steel VSt3sp on fracture stress  $S_C$ : 1 – initial state; 2 –  $[H]_{diff} = 7 \text{ cm}^3/100 \text{ g}$  (the specimens were degassed after preliminary deformation)



**Figure 7.** Schematics of initiation and growth of crack in hydrogen-containing metal: *a* – formation of slip systems in tension; *b* – initiation of microcrack in slip plane; *c* – growth of crack due to initiation of new microcracks at its apex;  $\tau$  – tangential stress

and equals 50 MPa. At a deformation of 10 %, the hydrogen has no substantial effect on mechanical properties of the specimens of steel VSt3sp (see Figure 4, *a*). The same takes place also at a deformation of 15 % (Figure 4, *b*). At a deformation of metal at a level of 17 %, the effect of hydrogen on mechanical properties of steel VSt3sp was much aggravated (Figure 4, *c*). A microcrack oriented at an angle of  $45^\circ$  to the specimen axis appeared on the fracture surface of the specimens with a hydrogen content of  $7 \text{ cm}^3/100 \text{ g}$  after a preliminary plastic deformation of 17 % (Figure 5). No such cracks were detected in the hydrogen-free specimens. The fracture stress was calculated to evaluate the impact of microcracks formed under the effect of hydrogen on metal fracture [3]. The Bridgeman's relationships [4], allowing for those suggested by Kopelman [5], were used to calculate the maximal stress value in the neck of a specimen at the time point of fracture:



**Figure 8.** Propagation of crack in specimens of steel IN903 without hydrogen (*a*) and by adding it after 17 (*b*), 22 (*c*), 29 (*d*), 32 (*e*) and 39 (*f*) s

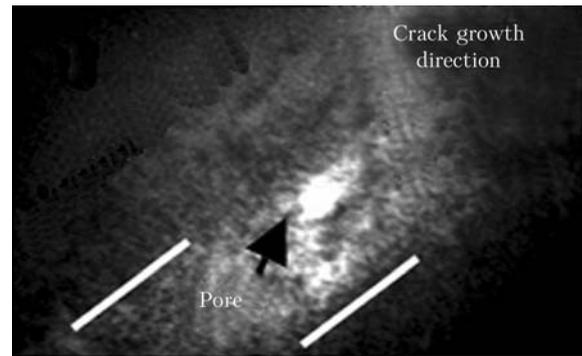


$$S_C = S_K \frac{1 + \ln(1 + \eta/2)}{(1 + \eta/2) \ln(1 + \eta/2)},$$

where  $\eta = 0.92(e - 0.1)$ ;  $e = \ln(1/(1 - \psi))$ ; and  $S_K$  is the average stress at the neck at the time point of fracture of a specimen. As seen from Figure 6, the value of fracture stress of metal in the initial state grows with increase of the preliminary plastic deformation [6]. This is related to the fact that microcracks, which form in metal as a result of plastic deformation and do not lead to fracture at the moment of their formation, become blunted with further plastic deformation [7]. At the presence of diffusible hydrogen in metal, the trend is reversed, i.e. the fracture stress decreases with growth of the preliminary plastic deformation. This is indicative of the fact that in the hydrogen-containing metal the microcracks that formed during plastic deformation do not blunt but continue growing.

Plastic deformation of metal leads to initiation of new dislocations, which act as hydrogen traps. The presence of hydrogen at dislocations leads to decrease in the force of repulsion of the dislocations and localisation of plastic deformation [1]. The key stage of the HE mechanism is coalescence of dislocations at the crack apex [8]. Schematic of initiation and growth of a crack in specimens testing is shown in Figure 7. Growth of the crack by the brittle and quasi-brittle mechanism occurs due to initiation of a new microcrack at the apex of the old one and its subsequent coalescence. The dislocation model of this process for the hydrogen-free metal is considered in study [9]. In the hydrogen-containing metal, the formed microcracks continue growing during plastic deformation due to initiation of new defects at the crack apexes (Figures 8 and 9) [10].

It can be concluded from the above-said that the HE mechanism consists in the following. Plastic deformation of metal results in formation of dislocations, which act as traps for diffusible hydrogen and lead to redistribution of the latter. The presence of hydrogen around dislocations leads to their coalescence at a lower external stress, at a macrolevel this showing up as facilitation of shear deformation and localisation of plastic deformation. Further growth of a crack occurs due to initiation of a new microcrack at the apex of the old one as a result of plastic deformation localised here under the effect of hydrogen.



**Figure 9.** Formation of micropore in slip shear line ahead of the crack apex in a hydrogen-containing specimen of steel IN903

## CONCLUSIONS

1. As established by thermal desorption analysis, the cause of hydrogen embrittlement of metal in plastic deformation is hydrogen at dislocations.

2. The presence of hydrogen at dislocations leads to facilitation of shear deformation and initiation of a microcrack at a lower external stress, compared to the hydrogen-free metal.

3. Brittle growth of a crack occurs due to initiation of a new microcrack at the apex of the old one as a result of localisation of plastic deformation at the crack apex under the effect of hydrogen.

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