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GAS EMISSION AND REDISTRIBUTION OF HYDROGEN IN AGING OF WELDED STRUCTURES FROM DIFFERENT METALLIC MATERIALS

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The results of investigations of hydrogen behavior in process of aging of welded structures from different materials were analyzed. Gradation of the processes taking place in metal at aging was carried out and reasons of reduction of crack resistances in welded joints at room temperatures were found. The mechanism was proposed, which explains spontaneous hydrogen emission from metal in process of aging, reasons and consequences of these processes. 22 Ref., 2 Tables, 11 Figures.

Keywords: *aging of welded structures, hydrogen, crack resistance, boson, mechanism, cold crack*

Cold cracks are formed in welded joints of different class steels as a consequence of phase transformations resulting in decrease of metal strength properties (for example, formation of hardening structures), presence of diffusion-mobile hydrogen (DMH) and effect of welding stresses. Cold cracks are formed in cooling stage at temperatures below 423 K as well as in aging of welded structures at room temperature during some time after welding. Considerable number of works (for example [1–6]) are dedicated to problems related with investigation of the reasons and mechanisms of formation of cold cracks in welded joints of steels and titanium alloys of different composition and development of activities on their prevention.

Concept of aging can be found in technical literature. It is implied as isothermal holding of metal after the end of welding in process of welded joint cooling at some temperature (usually T = 373–423 K in a period from 0 min to 8 h) for partial release of welding stresses and removal of excessive DMH. Such an operation results virtually in complete recovery of plastic properties of metal and, therefore, further aging of this welded joint at room temperature does not already lead to formation of cold cracks in it. Thus, this topic was investigated for different steels starting from the middle of XX century (for example, it is reflected in works [7–9]). This paper does not cover these problems. Aim of the present paper is an analysis of the processes taking place in metal of welded joints and structures during aging only at room and climatic temperatures (therefore aging is not discussed here), which are used in real operation of many steel parts and structures susceptible to formation of cold cracks under these conditions. This problem is bought up comparatively rare and it is not studied enough.

It is known that performance of some technological operations (fusion welding, rolling, heat treatment) of different steels and alloys provokes spontaneous emission at room temperatures during some time of so-called diffusion-mobile hydrogen. Its amount and distribution in the welded joint can be controlled by various methods of analysis. The most widespread of them is a glycerol method, when the welded sample is immersed into a bath with glycerol and the places and frequency of hydrogen bubbles emission on its surface is controlled [8]. It is supposed that content of remaining in metal hydrogen (so-called residual hydrogen — RH) stays constant at room temperature during long time. It is determined that DMH emission mainly takes place on fusion line, HAZ-BM boundary and grain boundaries. For high-strength steels, mainly of martensite and martensite-bainite classes, this phenomenon is related with appearance of cold cracks in the welded joints in process of their cooling to temperatures below 373 K and further aging at room temperatures (Figure 1). It is widely stated that in 3–10 days of aging amount of emitted DMH reduces to safe level and cracks are not formed in future.

It is assumed that no phase transformations take place in process of aging, only diffusion (spontaneous release of hydrogen from metal) and relaxation processes causing transfer of plastic deformations from grain boundaries to grain body [7] are observed. This results in rise of ductility and cold crack resistance. Simultaneously, there is reduction of electrical resistance of steel that indicates ordering of metal atomic structure [9].

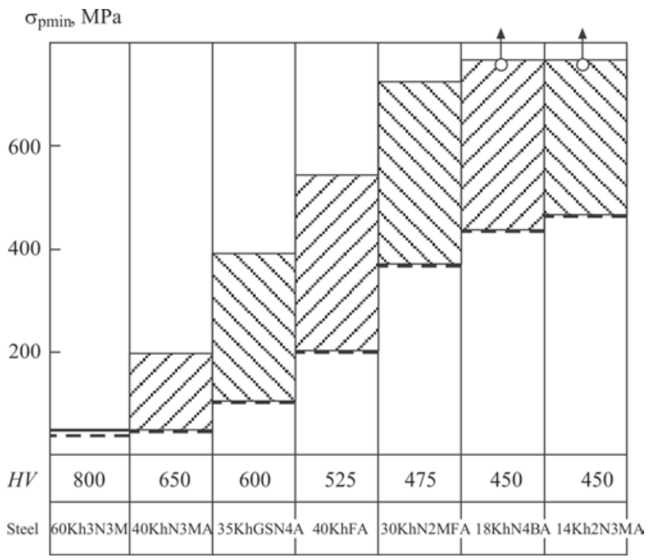


Figure 1. Effect of DMH content (boson H^-) in deposited metal on cold crack resistance of different steels. T-sample LTPP2-3, electrodes UONI 13/45 (dashed line — $[H^-] = 4$ ppm; solid line — $[H^-] = 1$ ppm (2)) [1]

Numerous empirical observations were analyzed and on their basis technological solutions were proposed. They allow eliminating appearance of cold cracks during aging of the welded joints from low-alloy high-strength steels [5, 6, 10]. However, deep systematic investigations of the processes taking place in metal of welded joint during its aging have not been carried out, the reason and mechanism of negative effect of hydrogen on cold crack formation during aging are not determined. This has become a subject of investigation in this work.

The investigations, related with metal aging after technological treatment, are usually carried out on small-size samples, i.e. the process is virtually modeled. Under real conditions after welding is finished many products are used at room and climatic temperatures for a long time (years) in media containing hydrogen in different forms, i.e. in form of moisture in air, condensate (mist), running water (rain), vapors of acids in atmosphere, working hydrogen-containing fluids etc., however, for such conditions an effect of «external» hydrogen of the medium, from which it is absorbed by metal before as well as in process of operation, is not considered.

Investigations carried by us together with I.K. Pokhodnya and colleagues [11] showed that DMH is a negative quasi-ion of hydrogen H^-. Next works [12] demonstrated that hydrogen absorbed by metal is contained in solid body simultaneously in two states, i.e. in form of H^- and H^+ quasi-ions. H^- quasi-ion has quantum properties of boson, namely ultramobility in solid body. A mass transfer coefficient of hydrogen-boson (diffusion coefficient in specific case) is more than hydrogen diffusion coefficient (fermion) in the same steel by several orders. For example, for steel 14Kh2GMR at room temperatures in 40 h after welding it reaches $D^{H-} = 1.95 \cdot 10^{-3}$ cm^2/s value and after aging during one month $5.85 \cdot 10^{-7}$ cm^2/s. This explains spontaneity of DMH emission under these conditions. H^+ hydrogen quasi-ion has the fermion properties (this is so-called residual or proton-ionized hydrogen), which is removed from metal only in process of heating or melting.

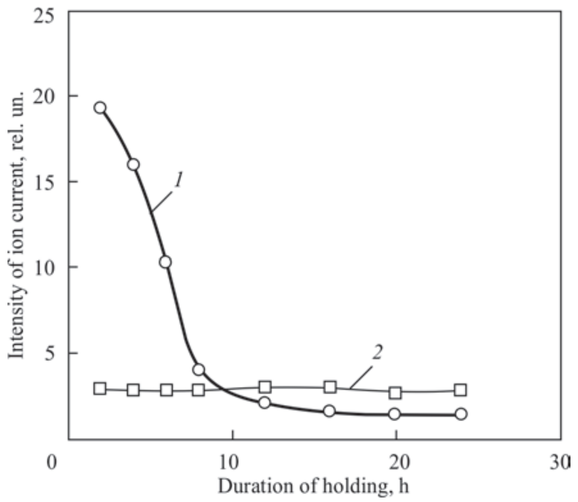


Figure 2. Dependence of intensity of secondary ions of hydrogen H^- (*1*) and H^+ (*2*) on duration of exposure in ultrahigh vacuum of preliminary hydrogen-charged samples of steel St3 [11, 12]

It is determined [12] that under certain conditions (temperature, metal composition) hydrogen-boson can transform into another form, namely hydrogen-fermion and vice versa. For structural steels such a critical temperature is 602 K.

It is found that content of DMH is higher in subsurface weld metal layers (Figure 3). Relationship between H^- and H^+ changes from the surface to weld metal depth (Figure 4) and also in variation of met-

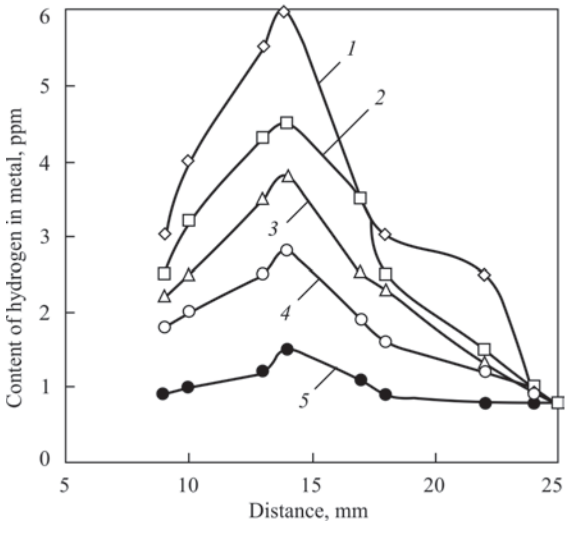


Figure 3. Change of hydrogen distribution in St3 steel sample in aging after welding in 1, 2, 3, 4 and 5 h, respectively (on *x*-coordinate — distance from weld metal surface [14]

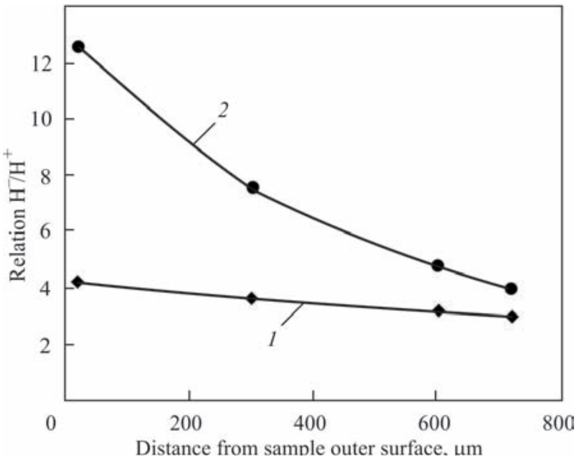


Figure 4. Change of H$^-$/H$^+$ relationship on normal section radius of cylinder sample from steel 20 along atomically pure fracture surface after heat treatment (*1* — normalizing; *2* — quenching) [12]

al composition in process of aging as well as during operation. For example, introduction of a nickel in deposit metal results in change of H$^-$/H$^+$ relationship at preservation of total content of hydrogen (boson and fermion) (Table 1). Hydrogen-boson is one of the main reason of cold cracks initiation [12].

Analysis of the results of aging process observation and experimental data on control of DMH and RH content in metal of welded joints, carried earlier by different authors, showed that all cases of aging of steels and alloys, being accompanied by spontaneous emission or redistribution of hydrogen at room temperatures can be classified on various groups that differ by nature, dynamics of process or level of hydrogen effect on metal mechanical properties:

1. In process of aging independent on its duration the structure and phase composition of metal are preserved and the changes of composition (including on hydrogen) do not reach critical values. Metal structure is energy stable. Probability of cold cracks appearance is low or completely absent.

2. In long-term aging metal of welded joint is subjected to changes of structure caused by stress relaxation and redistribution of chemical elements in the range of grains and their boundaries. Structures, formed in the welded joint, are energy non-equilibrium, unstable in time, their gradual stabilizing takes place in process of aging. There is a probability of appearance of cold cracks, in particular, in the first hours after welding.

3. In the process of long-term aging (or operation) there are phase transformations taking place in the metal, which promote changes of content of separate sections of metal of welded joint and solubility of hydrogen in them. The structures with large energy instability (hardening structures, martensite, bainite) and high welding stresses, zones with local plastic deformation, hydrogen solubility and increased local chemical inhomogeneity appear. Localizing of increased hydrogen concentration and intensity of its peaks reach and exceed the critical level for this steel, alloy. There are possible chemical reactions inside metal with formation of film iron hydrates, titanium hydrates or molecular gases (for example, hydrocarbons $CH_{2,}$ CH_4 type in high-carbon steels). Probability of cold crack appearance is very high.

4. Process of long-term aging (and operation) provokes change of charge state of hydrogen dissolved in metal and relationship between quasi-ions of hydrogen of different charge type. Probability of appearance of cold cracks depends on value of this relationship, coefficient of chemical inhomogeneity, intensity and locality of hydrogen concentration peak, possibility of chemical reaction between quasi-ions of hydrogen and steel components. Probability of cold cracks appearance is very high.

5. Duration of welded joint aging affects the intensity and parameters of final treatment of the products and their crack resistance. Formation titanium hy-

Table 1. Effect of composition of coating sprayed over on wire 09G2S on content of diffusion-mobile hydrogen (DMH) and residual hydrogen (RH) in deposit metal of steel 14Kh2GMR and on H$^-$/H$^+$ relationship [13]

No.	Wire coating composition	Content of hydrogen in deposit metal, 10^{-4} %				DMH relationship (H$^-$) to RH (H$^+$), H$^-$/H$^+$
		Separate determination		Total content		
		DMH, CA method	RH, LMA	Sum, DMH + RH	Determination by VM method	
1	Initial (base) metal	5.5	3.1	8.6	8	1.77
2	BM + 1.5 % N1	4.85	5.2	9.05	9	0.74
3	BM + 1.6 % Ni + 0.44 % REM, including 0.017 % Ce	1.9	7.2	9.1	8.5	0.26
4	BM + 0.45 % Y + 1.8 % Ni	4.6	4.2	8.8	9.5	1.09
5	BM + 2 % Ni + 0.6 % REM, including 0.26 % Ce	2.3	6.6	8.9	8.9	0.35
6	BM + 0.3 % Y	5.2	3.9	9.1	10	1.33

Designations: BM — base metal; CA — chromatographic analysis; LMS — local mass-spectrum analysis; VM — vacuum melting method; REM — rare-earth metals (mixture).

drates and (or) film iron hydrates on grain boundary is possible. There is probability of cold crack formation.

Let us consider several examples illustrating proposed gradation of aging processes for different metals and alloys after their thermal or deformation treatment in different hydrogen-containing media.

Single-phase metals (for example, copper) as well as structural steels in a layer adjacent to fusion surface do not change their structure in process of aging. These materials can be used as model ones in analysis of welded joints of steels and titanium alloys. They allow evaluating and comparing the parameters and dynamics of hydrogen absorption by metal in process of local deformation, appearing in weld HAZ metal in its cooling as a result of effect of welding stresses and desorption of hydrogen in partial relaxation of welding stresses during aging.

There is a significant effect of medium, in which researched processes take place, on these processes (in welding — humidity of welding consumables; in deformation, aging and operation of welded products — content of hydrogen-containing medium and value of oscillations of climatic temperatures).

Figures 5 and 6, *a* show the data on change of a value and rate of hydrogen absorption by copper in process of its plastic deformation in different media, and Figure 6, *b* represents a rate of hydrogen desorption from the same samples of copper for 42 months of aging at room temperature. Copper is a single-phase and ductile metal, therefore, phase and structural changes had no effect on the studied processes and internal stresses caused by deformation and hydrogen dissolution are reduced to a minimum. Hydrogen absorption from hydrogen-containing medium by deforming metal at room temperatures is provoked by influence of structure-deformation effect described in work [15], desorption of hydrogen is relaxation of stresses in long-term aging.

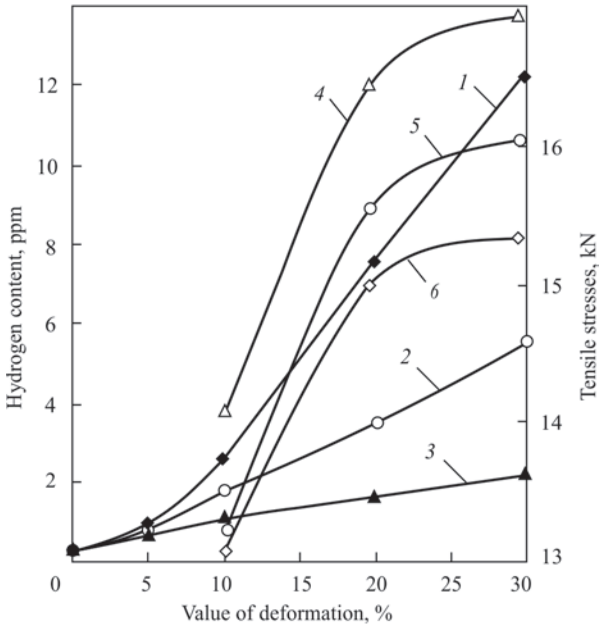
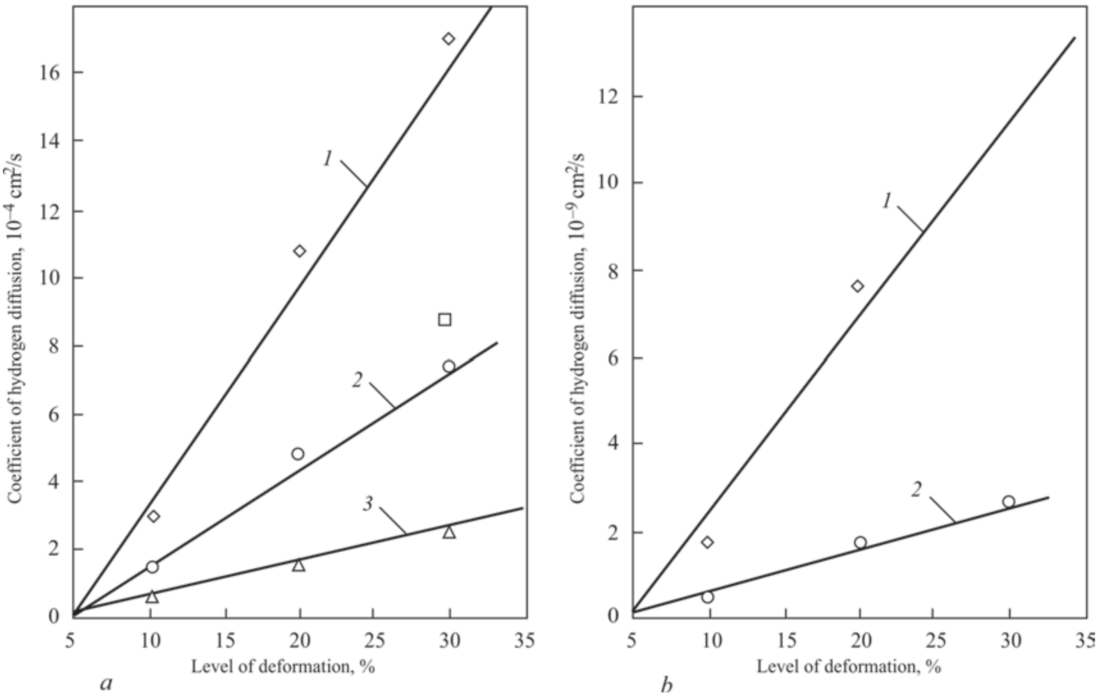


Figure 5. Dependence of hydrogen content in copper (curves *1*–*3*) and value of loading (curves *4*–*6*) on level of deformation in hydrogen gas (*1*), condensed (*2*) and running water media [16]

Content and distribution of hydrogen in the welded joint is significantly effected by content of this admixture in the medium, in which welding is carried



a *b*

Figure 6. Change of hydrogen diffusion coefficient at its absorption by copper in (*a*) process of deformation (30 min) in hydrogen gas medium (*1*), (structured) water condensate (*2*), simple (running) water (*3*) and desorption (*b*) from the same copper samples after tension and aging during 42 months (1308 days) [15–17]

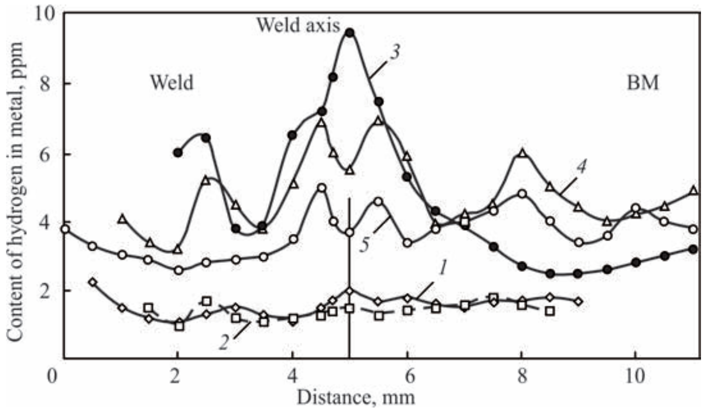


Figure 7. Distribution of hydrogen-boson in welded joint of steel 14Kh2GMR at different humidity of flux AN-17M: *1*, *2* — humidity 0.017 %; *3*, *5* — humidity 0.04 %. Aging after welding: *1* — 4 h; *2* — 5.5; *3* — 3; *4* — 4; *5* — 5. Local analysis of hydrogen content by LMA with TEE method [10]

out, and aging process. For example, change of flux humidity (as medium) in 14Kh2GMR steel welding results in increase of hydrogen content in metal (Figure 7) and to more inhomogeneous distribution of this admixture. This increases probability of crack appearance during the first hours of aging [10]. Decrease of flux humidity from 0.04 to 0.017 % is enough to make hydrogen distribution in the welded joint virtually homogeneous in 4 hours of aging and have minimal probability of crack appearance. It is typical that hydrogen removal from welded joint metal during aging is not identical in its different areas. The highest metal degassing rate is reached along the fusion surface (Table 2, Figures 8, 9) where structural changes in the process of aging have not been observed.

Investigation of process of hydrogen emission from this metal area by method of local mass-spectrum analysis with thermal electron extraction by electron-beam probe (LMA with TEE) [17] showed that hydrogen in process of aging is not desorbed from metal by continuous flow, but by the portions, pulses and curve of change of local content of hydrogen along fusion line during one month after welding represents itself damping wave (Figure 9). The averaged values of experimental measurements match with distributions calculated on Fick's formula. A measurement error made 10^{-4} ppm [17] that is considerably less than amplitude of concentration oscillations of hydrogen in the same area of the sample making several ppm units (Figure 9). Therefore, observed deviations from the curve, plotted by Fick's equation (dashed line on Figure 9) are not the measurement errors and reflect a real process of degassing of hydrogen from metal taking place in zone of surface fusion. Work [19] informs about pulsed nature of hydrogen emission from steel 9GS after rolling in the process of aging during cooling and room temperatures.

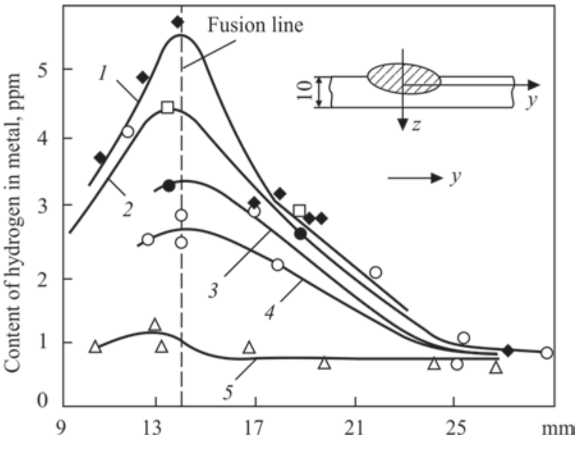


Figure 8. Distribution of hydrogen-boson in weld of St3sp (killed) steel in *Y* axis direction: *1* — directly after welding; *2* — in 1; *3* — 2; *4* — 24; *5* — 72 h after welding [20]

A wave nature of change of hydrogen content in process of long-term aging (during 10 months) was observed after multilayer argonarc welding of high-strength steel VNS-2. The parts from this steel produced by welding failed after 5–6 months in process of their operation as well as in conservation storage, i.e. without service loads. In order to find out the reasons of such behavior of material the welding coupons were made. They were used to cut out every month a strip of metal and produce the samples for the next investigation using LMA with TEE method on 0B768M unit at E.O. Paton Electric Welding Institute of the NAS of Ukraine. A discrete-spot analysis on local content of hydrogen by 5 parallel routes passing through weld and HAZ of the welded joint was carried out on each such sample. Obtained measurement results were averaged by the samples and

Table 2. Hydrogen diffusion in different areas of weld [18]

Steel, area	*T*, K	D^H, cm^2/s	Type of structure
14Kh2GMR, weld	293	$6.8\cdot10^{-5}$	Martensite-bainite
14Kh2GMR, HAZ	293	$3.6\cdot10^{-7}$	Same
14Kh2GMR, fusion surface	293	$1.8\cdot10^{-4}$	»

at the end of year by the whole series of monthly measurements. Figure 10 shows the final results of these measurements. It is determined that in process of aging the total content of hydrogen in metal for 10 months of observations has changed insignificantly (curve *1* on Figure 10), but in separate areas of the welded joints the local concentration of hydrogen has increased from 5 ppm in metal (at once after welding) to 34 ppm in 7 months of aging of the welded coupons at room temperature. This is significantly higher than the critical limit of allowable hydrogen concentration in this steel (10 ppm) and caused product failure. Periodic variation of hydrogen content in the separate areas of welded joint was found in process of long-term storage, however, a curve reflecting these changes appeared to be rising. Metallographic examination of metal structure before and after aging showed incomplete martensite tranformation in metal with formation of carbide grid, having lower strength than metal and higher hydrogen solubility. It is determined that there are migration and redistribution of hydrogen-boson in this martensite steel in the field of local internal stresses and microareas of plastic deformation at room temperature during aging. Hydrogen-boson is concentrated along the grain boundaries and in the zones of their joining where dilatons and clusters of vacancies are formed. Hydrogen mobilizes in them developing significant inner pressure. This promotes partial decay of martensite with carbide grid precipitation. Hydrogen from the surrounding metal migrates in these areas since its solubility in carbide is considerably higher than in metal. As a result process of metal degradation is accelerated. The analysis registers a rise of local concentration of hydrogen in the separate areas at preservation of its average content in metal (curve *3*, Figure 10). Hydrogen-boson reacts with carbon forming with it hydrocarbons, for the first CH_2, then CH_4. These processes (increase of hydrogen concentration in carbide grid, formation of new portions of dilatons, growth of inner pressure, formation of new portions of hydrocarbons) take place simultaneously, i.e. this is synergetic process. It can be assumed that in a period of formation of new portions of hydrocarbons there is increased consumption of hydrogen and concentration curve will show decrease of its local concentration and inner pressure, process of formation of new portions of hydrocarbons fails. Local concentration of hydrogen in a carbide grid rises again to the critical level when process of formation of hydrocarbons is reactivated and defect zones increase. There is appearance of microcracks in the carbide grid which grow in process of aging. At seven months of aging the crack in metal rises to the critical values. Local concentration of

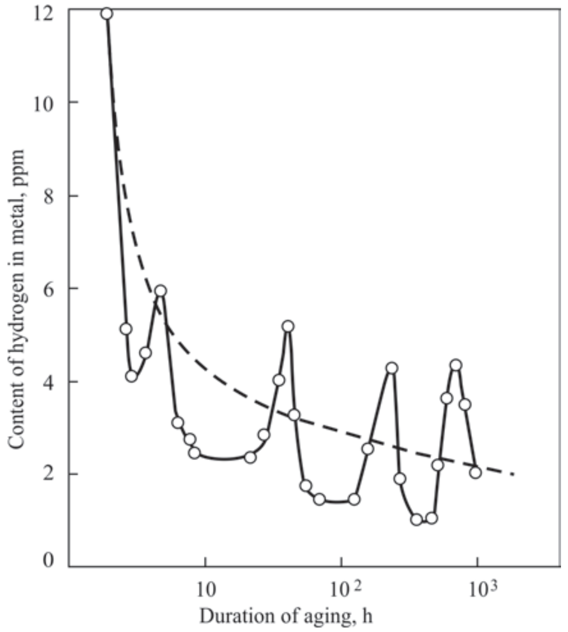


Figure 9. Change of hydrogen-boson concentration in weld-base metal fusion zone in process of aging of 14KhGMR steel samples at room temperatures. LMA with TEE method measurement of hydrogen by focused beam of electrons. Electric arc welding by ANP-1 electrodes (dashed line — change of hydrogen concentration in time on Fick's formula) [17]

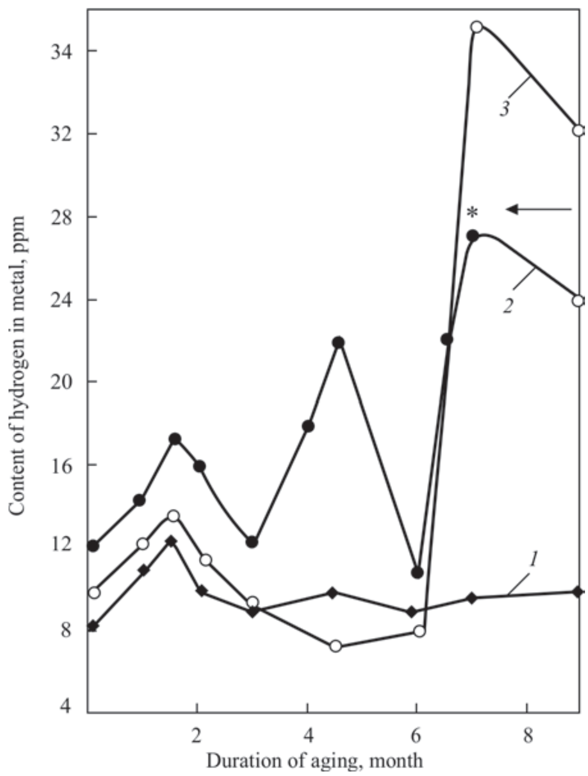


Figure 10. Change of hydrogen concentration at T = 300 K in weld of steel VNS-2 during 9 months after welding; content of hydrogen in weld metal: average (*1*); in light/ferrite/ (*2*) and dark/carbide/ (*3*) strip (on diagram marked * (indicated by arrow) — result of determination of hydrogen amount by vacuum-melting method in the sample cut from fracture zone)

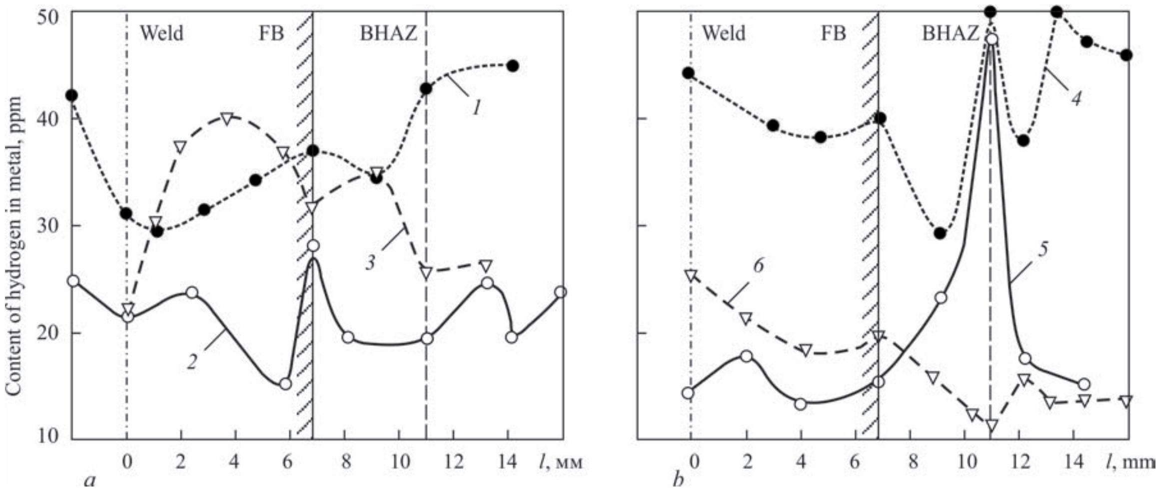


Figure 11. Hydrogen in welded joint of AT-3 alloy: without heat treatment (*1*, *4*) and after annealing at T = 823 K (*2*, *5*) and 923 K (*3*, *6*); in 3 h (*a*) and in 40 months (*b*) after welding: *l* — distance from weld axis; FB — fusion boundary; BHAZ — boundary of HAZ [21]

hydrogen in carbide grid reaches 32–34 ppm (curve *1* on Figure 10). Plastic deformation is activated in ferrite. During it the dislocation cores capture hydrogen from the medium [15] and there is also intensive increase of local hydrogen concentration in it. Ferrite with such high local content of hydrogen (28.0 ppm) (curve *2* on Figure 10) is also susceptible to hydrogen embrittlement [17]. Thus, favorable conditions for metal degradation are developed in the localized zone of welded joint of steel VNS-2 in 7 months after welding in process of aging independent on effect of external (operation) loads, which only insignificantly (less than for 1 month) accelerate failure process.

One more interesting materials science aspect of the consequences of long-term aging is a change of final technological operations, in particular, mode and nature of welded joint annealing performed before and after long-term aging (Figure 11). It is determined that if argonarc welded product of alloys AT-3 is used in structure at once after welding or during a month after, it is reasonable to use incomplete annealing (T = 823 K) and this is enough for removal of welding stresses and obtaining optimum mechanical characteristics of AT-3 alloy welded joint. There are no dangerous situations related with significant local hydrogen saturation of local areas of welded joint (Figure 11, *a*) in such metal. Moreover, complete annealing (T = 923 K) results in somewhat increase of hydrogen content in weld metal and HAZ (curve *3* on Figure 11, *a*). However, if welded product ages during three or more years and only after it is used in the structure than there is a need in carrying out complete annealing (T = 923 K) since long-term aging provokes in welded joint metal some structural changes. They are accompanied by redistribution of some chemical elements, in particular, hydrogen, namely its content on HAZ–BM boundary rises and after incomplete annealing the local concentration of hydrogen in this area reaches critical values up to 51 ppm (curve *5* on Figure 11, *b*). Work [22] informs that the welded joints of titanium-based alloys contain a narrow resolidification zone with high internal stresses on HAZ–BM boundary. The structural components of this band (plate) in process of aging are «covered» with film carbides, oxides, titanium hydrates. As a result in this area of metal the stresses rise, and strength drops.

Hydrogen segregation coefficient in these areas reaches 3.0–3.5 values [22]. In our case (Figure 11, *b*, curve *5*) this coefficient is still higher and equals 8, moreover, the concentration peak is acute, i.e. this area of metal has high probability of crack formation. The nature of hydrogen distribution curves on welded joint section in three years of aging (Figure 11, *b*, curves *5* and *6*) indicate that the high internal stresses appearing in this area of metal are not entirely eliminated, but can be removed at complete annealing (curve *6* on Figure 11, *b*). This fact shall be taken into account during manufacture of welded products and structures.

Conclusions

The mechanism was proposed, which explains the process of spontaneous gas emission of hydrogen in the process of aging at room temperatures, redistribution of this admixture in the products volume and change of tendency of welded joint metal to crack formation.

It is determined that aging at room temperatures results not only in loss by metal of part of earlier absorbed hydrogen as a consequence of relaxation of welding stresses in the welded joint, but redistribution of hydrogen inside metal if there are processes of phase (as in steel VNS-2) or structural as in alloy AT-3) transformations related with different solubility of hydrogen in separate phases or structures. This leads to increase of chemical inhomogeneity, appearance of the areas with local hydrogen concentration exceeding the critical values for this steel or alloy and provoking cold crack formation.

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