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ROLE OF HYDROGEN EMBRITTLEMENT IN THE CORROSION CRACKING OF ALUMINUM ALLOYS

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Although the corrosion cracking of high-strength aluminum alloys has been investigated for more than 50 years, hydrogen embrittlement as one of the mechanisms or a stage in the mechanism of corrosion cracking has been intensely studied for only the last 10 years. This may be explained by the fact that in the basic medium for corrosion cracking tests (3-3.5% NaCl aqueous solution) cathodic polarization stops corrosion cracking [1]. In dry hydrogen subcritical crack growth does not occur [2] and the solubility of hydrogen in aluminum is very low [3]. However, at the present time there has been accumulated significant experimental and theoretical material related to the hydrogen embrittlement of aluminum alloys which in this work is systematized for the purpose of obtaining a qualitative picture of the role of hydrogen embrittlement in corrosion cracking and determination of the influence of metallurgical factors on both phenomena.

1. Conditions of Hydrogen Absorption. Hydrogen absorption of aluminum alloys at a temperature close to room occurs in water and humid air [4-7] and in aqueous solutions of salts and acids and is strengthened in cathodic polarization [8-10] and ion implantation [11]. In the case of high temperatures, hydrogen absorption occurs from the hydrogen of the atmosphere and, especially intensely, as the result of the reaction

$$2\,Al + 3\,H_2O \rightarrow Al_2O_3 + 3\,H_2 \qquad (1)$$

(here the H_2O is the vapor of the gaseous phase crystal hydrate water of the oxide films on the metal). At temperatures above 783°K reaction (1) occurs according to a chain mechanism [12]. For appreciable development of it with participation of crystal hydrate water at lower temperatures (~458°K) holds of seconds are sufficient [13]. Therefore, hydrogen absorption may occur not only under corrosion cracking test conditions but also before the experiments during metallurgical operations if access of humid air and the presence of hydroxide films are not excluded.

2. Reaction of Hydrogen with Crystalline Structure Defects. In accordance with the quantum-mechanical calculations [14], the passage of H^+ ions from an octahedral void of the aluminum lattice into a vacancy is accompanied by a gain in energy of 1.23 eV. From this it follows that the hydrogen in aluminum, at least partially, is dissolved according to the substitution type. The quantity of the substitutional solution depends upon the concentration of the equilibrium [15] and excess vacancies. The total quantity of dissolved hydrogen is finally determined by the hydrogen pressure and at high pressures by its volatility [16].

Existing methods of measurement of concentrations both of dissolved hydrogen and of vacancies at low temperatures are very unreliable. Their maximum values (in atomic shares) at room temperature are 10^{-2} [17] and 10^{-4} [18], respectively. There are evidences of the
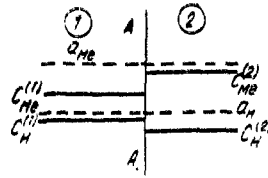
Fig. 1. Plan of the relationship of the concen-
trations (C) and the activities (a) of the com-
ponents of the solid solution providing equili-
brium of adjacent areas (1 and 2) with different
concentrations of the elements.

formation of a solid substitutional solution, that is, of "vacancy—hydrogen atom" complexes
[19-20] and the diffusion coefficient of the complexes is approximately equal to the diffu-
sion coefficient of the vacancies.

The fact that in plastic deformation of an aluminum specimen with voids filled with
tritium the latter was captured from the voids and distributed in the volume of the specimen
only in the presence of excess vacancies may, for example, serve as experimental (although
indirect) evidence of the actual significance of the "vacancy—hydrogen" complexes in the
occurence of hydrogen embrittlement [21].

3. Microstructural Effects of Hydrogen Absorption. As the result of hydrogen absorp-
tion by alloys in moist air, there are formed hydrogen bubbles [4-7], for which, in addition
to everything else, excess vacancies are necessary since it has been firmly established that
voids in hydrogen-impregnated aluminum occur only in supersaturation of it with vacancies
[21, 22]. The excess vacancies are created on the surface of the alloys as the result of
selective oxidation of magnesium and/or lithium from the solid solutions and the intermetal-
lide particles [23, 24]. In the structure of Al—8% Mg alloy with an increased hydrogen con-
centration C_H there is a higher density of intragranular precipitates [25]. Acceleration
of decomposition of hydrogen-impregnated supersaturated aluminum-base solutions has been ob-
served [26]. For an understanding of the essence of the phenomenon, which has not yet been
determined [25, 26] it is necessary, in our opinion, to use the rule known from the thermo-
dynamics of alloys [27], according to which with the addition to a binary solution of a
third component the activity of the second increases if the third changes the concentration
of valence electrons in the solution in the same direction as the second. It is easy to see
that lithium, copper, zinc, and magnesium, which are separately or in combination basic
alloy elements in modern production aluminum alloys, decrease the concentration of valence
electrons in the solid solution the same as, naturally, hydrogen must do. Therefore solu-
tion of hydrogen is responsible for the thermodynamic necessity of precipitation of the
above elements from the solid solution.

The mutual influence of the components on the activity of one another thermodynamically
allows the situation when in the vicinity areas with different concentrations of the compo-
nents are stable in time (Fig. 1). For example, in one area there is more hydrogen but less
alloy element and in another the reverse.

4. Transport through an Oxide Film. The mechanism of formation of hydrogen includes
the stage of diffusion of water or hydroxyl ions OH⁻ through the surface film to the metal—
oxide boundary, where it is liberated [12, 28, 29]. At room temperature with a relative
humidity of H = 100%, a condensate precipitates on the surface of the specimen [28, 29] and
therefore this mechanism is completely possible in solutions particularly since the corro-
sion cracking of aluminum alloys in aqueous solutions occurs in the range of potentials in
which in accordance with the Pourbaix diagram [30] conditions of formation of hydroxyls are
satisfied. In laborabory air the facts of formation of hydroxyls on 01420 alloy [31] and
also of hydrogen impregnation of this alloy and alloys of the Al—Mg and Al—Zn—Mg systems
[7, 23] have been established.

There is, in addition, convincing proof that cathodic discharge of H^+ ions may be one
of the stages of the process of hydrogen impregnation of aluminum alloys if it is assumed
that the hydrogen atom occurring in discharge diffuses through the film to the metal. To
this, in particular, is referred the influence of "hydrogen poisons," compounds of arsenic
preventing molization of the catodically reduced hydrogen and strengthening hydrogen impreg-
nation [10].

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An original hypothesis has been advanced [32, 33] of the possibility of hydrogen embrittlement of aluminum alloys only with a high permeability of the oxide films in relation to hydrogen, which occurs by damage of it by Cl^- ions together with mechanical action. In connection with this some critical stress intensity factor K_{cr} dependent upon the Cl^- ion concentration in the solution is determining. With $K < K_{cr}$ hydrogen embrittlement is absent and corrosion cracking occurs by local anodic solution. This assumption is confirmed by the fact that cathodic polarization, in principle promoting hydrogen impregnation, accelerates corrosion cracking with $K > K_{cr}$ but with $K < K_{cr}$ retards it. However, cathodic polarization retards the development of the oxide film and may change its structure, composition, and protective properties. On the basis of theoretical and experimental study of this property of cathodic polarization it was shown [34] that strong cathodic polarization and a high crack growth rate (that is, high values of K) cause local anodic solution of Al—7% Mg alloy, which is similar in the sence of corrosion cracking to Al27-1 [32, 33].

At present there is not direct data on the fact that hydrogen atoms of OH^- ions actually diffuse through the oxide film in corrosion cracking of aluminum alloys. Therefore, the question remains debateable. In the opinion of the author the latter are nevertheless true, first, because hydrogen impregnation in corrosion cracking in aqueous solutions occurs with potentials of oxygen depolarization when OH^- ions are formed and, second, because in the lattice of the hydroxyls there are obvious paths in the sublattice for diffusion of these ions.

It is very clear that such parameters of the media and the specimens as pH, the concentration of corrosion inhibitors or promoters, the humidity of the air, the electrode potential, and the chemical composition of the structural constituents influence the structure and growth rate of the oxide film, that is, its protective properties and, consequently, the kinetics of hydrogen embrittlement and corrosion cracking in general.

5. **Influence of Hydrogen Embrittlement on Mechanical Properties. Dislocation Transport of Hydrogen.** Specimens of Al—Zn—Mg alloy that are strongly (and to a considerable depth) hydrogen-impregnated in a humidity chamber at 343-393°K are embrittled. Failure sets in upon reaching the reduced tensile strnegth σ_t with a rate of deformation of $\dot{\varepsilon} = 10^{-3}$ sec^{-1} [6]. Approximately the same was observed on specimens after cathodic hydrogen impregnation in hydrochloric acid [9] but in this case embrittlement was intensified at low rates of deformation.

Specimens actively in tension ($\dot{\varepsilon} \approx 10^{-3}-10^{-4}$ sec^{-1}) in gaseous hydrogen with $p_{H_2} = 70$ MPa both without a preliminary hold and hydrogen impregnated for 524 h at 344°K did not become brittle [35]. However, with a reduction in strain rate even an 11-day hold at room temperature with $p_{H_2} = 24$ MPa was sufficient for the appearance of hydrogen embrittlement [8].

The effect of hydrogen embrittlement is a maximum at a temperature close to room but the specific value of the temperature of maximum brittleness drops with a decrease in $\dot{\varepsilon}$ (with a certain concentration C_H) and increases (with a specified rate $\dot{\varepsilon}$) with an increase in C_H [36].

There is evidence of entrapment of hydrogen by dislocations and concentration of it at the fracture surface in static tests [35], making it possible to conclude that dislocations transport hydrogen if only it is present in the solid solution. However, the increase in hydrogen embrittlement with a reduction in $\dot{\varepsilon}$ confirms not just some entrapment of hydrogen by dislocations but also movement with them of Cottrellatmospheres, the possibility of formation of which in hydrogen-impregnated aluminum alloy has been proven [8].

Of the other features of the influence of hydrogen on mechanical properties, we should note a decrease in the yield strength $\sigma_{0.2}$ [10], flow stress, and elongation of previously hydrogen impregnated 7075-T6 alloy in tension in 3.5% NaCl solution. The effects are intensified if additional hydrogen impregnation occurs during testing with the addition of $NaAsO_2$ salt to the solution.

The influence of hydrogen on the mechanical properties is reversible. In the majority of alloys the properties are restored in exposure of the hydrogen-impregnated specimens in dry air or vacuum at room temperature. For 7075-T6 [8] and Al—8% Mg [37] alloys a 1-week hold in laboratory air is sufficient for recovery. It is interesting that hydrogen bubbles are formed in such air in Al72-1 alloy (very similar to Al—8% Mg alloy) [7] and therefore the level of hydrogen impregnation at which they occur is insufficient for the appearance of hydrogen embrittlement.
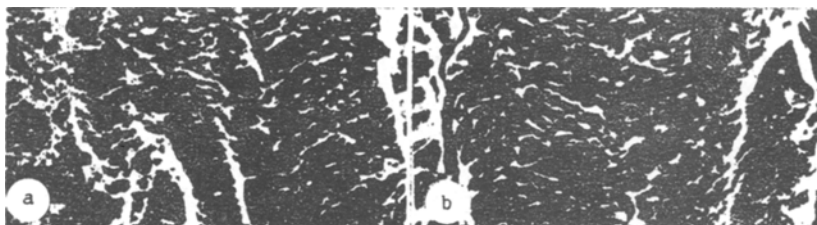
Fig. 2. Electron tractograms of the fractures of specimens
of V95T1 alloy. The surfaces of fracture are parallel to
the rolling plane: a) fracture in corrosion cracking in a
solution of 1 N H_2CrO_4 + 0.015 N NaCl [32], crack growth
direction longitudinal; b) fracture in impact loading at
77°K, crack growth direction transverse. Rolling direction
parallel to the short side of the photographs. 1250 ×.

6. **Influence of Hydrogen Absorption on Corrosion Cracking.** Preliminary hydrogen
absorption accelerates the corrosion cracking of Al—Mg [25], Al—Zn—Mg, and Al—Zn [38] alloys
and on specimens with a notch the incubation period until crack origin is shortened [38].
The latter effect is reversible (recovery in 8 days in laboratory air of a cathodically
hydrogen-impregnated specimen).

7. **Structure of Fractures in Hydrogen Embrittlement.** In static tension hydrogen ab-
sorption leads to grain boundary fracture, total or partial, of specimens of Al—Zn—Mg—Cu—(Cr)
system alloys after a hold in a humidity chamber [5], cathodic polarization and exposure in
3% NaCl solution, and gaseous hydrogen impregnation [8], of Al—Zn—Mg system alloys after a
hold in a humidity chamber [5, 6], and of Al—Mg system alloys after cathodic hydrogen impreg-
nation and corrosion cracking tests in 3.5% NaCl solution [37].

After hydrogen impregnation in humid air [6] and ion implantation [11] areas of fracture
by cleavage appear in the fracture. The extent of the zone of brittle fracture characterizes
the area of metal where the hydrogen concentration is greater than the critical [6] necessary
for embrittlement.

In the fractures of specimens of Al—Zn—Mg system alloy corrosion cracking tested at
343°K in air (H = 100%) on the surfaces of the grain boundary facets there is observed a
fragmented crust which is interpreted as former (before cracking) aluminum hydride AlH_3 [17].
This is indirect evidence of the formation of MgH_2 hydride at 343°K in a humid (H = 100%)
atmosphere in Al—Mg and Al—Zn—Mg system alloys (obtained by comparison of the kinetic curves
of decomposition of hydride in heating and curves of the liberation of hydrogen in heating
of the alloys after exposure) [39] making it possible to conclude that decomposition of MgH_2
under the action of an electron beam in the microscope causes hydrogen bubbles (it is true
that there is an opinion [17] that AlH_3 hydride is formed both in humid air and in corrosion
cracking).

After hydrogen impregnation in corrosion cracking tests [17, 37, 40] embrittlement of
the grain boundaries occurs in Al—Zn—Mg and Al—Mg system alloys and if as the result of some
propagation of the corrosion crack fracture of the specimens is completed in static tension
in air then the portion of the fracture adjacent to the corrosion portion is grain boundary.

In corrosion cracking in moist air (it is assumed according to the hydrogen embrittle-
ment mechanism) on the surface of the fracture there appear steps corresponding to the
moments of crack arrest in its jumplike subcritical growth [41]. Since for a certain alloy
the distance between the steps does not depend upon heat treatment or upon the intensity of
stresses whereas the rates of cracking change strongly, it may be assumed that these steps
are a characteristic of the structure of the grain boundaries and not of the jump itself.
We were able to confirm this experimentally on specimens of V95T1 alloy fractured by differ-
ent methods on a plane parallel to the plane of rolling (with crack growth both in the longi-
tudinal and in the transverse directions). In corrosion cracking tests and impact loading
tests at 77°K grain boundary fractures with relief steps (Fig. 2) similar to those observed
earlier [41] were obtained. Outside the relationship to the type of fracture the steps were
the same in form and orientation to the rolling direction, which proves their structural
origin. Consequently, on the fractures formed by hydrogen embrittlement or corrosion crack-
ing there are not boundaries corresponding to the jumps of the cracks in their subcritical
growth.

8. Hypotheses of the Mechanism of Hydrogen Embrittlement in Corrosion Cracking. The essence and sequence of the events in corrosion cracking in aqueous NaCl solutions with the assumption that hydrogen embrittlement is the leading mechanism of cracking are phenomenologically the following [42]: 1) the formation of pitting; 2) acidification of the medium at the pitting; 3) cathodic discharge of H^+ ions; 4) absorption of hydrogen by the metal; 5) diffusion of it into the area in front of the crack; 6) embrittlement of the metal in the prefracture zone; 7) failure of the embrittled metal by mechanical stresses. Processes 2-7 are repeated in acidification of the medium immediately after in the embryonic crack.

To this it may only be added that in corrosion cracking in humid air the origin and growth of a crack occurs without the formation of condensate [24, 43] and stages 1-4 are replaced by processes of liberation of hydrogen according to reaction (1) and its solution in the metal.

Under certain conditions each of stages 2-7 may completely control the corrosion cracking process but taking into consideration the purposes of this work the main interest is in stages 5-7 with only the difference that under stage 5 is understood not only diffusion but everything related to the transport of hydrogen within the metal.

9. Transport of Hydrogen within the Metal. For the case of cracking of aluminum alloys according to the hydrogen embrittlement mechanism there are not theoretically based values of the depth of penetration of hydrogen and its concentration in the area in front of the crack tip necessary for its further advance. The diffusion rate of the hydrogen must agree in order of magnitude with the rate of crack advance v_{cr} if the latter develops in two stages, embrittlement in the first and in the second a jump in the crack by the distance of embrittlement at the speed of sound [40].

In principle, acceleration of transport of hydrogen is possible at a crack tip [44] if the gradient in the hydrostatic component of stresses is sufficiently high [45]. However, according to improved calculations [46] the distribution of stresses is somewhat different so that the only method of acceleration of transport is transfer of hydrogen by moving dislocations (section 5). The upper determination of the rate of transfer of hydrogen by dislocations is determined by the critical rate v_c of breaking away of them from segregations (Cottrell atmospheres) [47]:

$$v_c = 4kTD_H/\beta. \tag{2}$$

Keeping in mind that dislocations effectively capture hydrogen only in the presence in the metal of excess vacancies [21] and also the formation of vacancy–hydrogen complexes [14, 19, 20] it must be taken into consideration that in essence the dislocations transport these complexes. Their specific atomic volume is approximately the same as for a vacancy. In the given situation the parameter of elastic interaction β is equal to $1.6 \cdot 10^{-30}$ J/m. In addition, in place of the coefficient D_H in Eq. (2) we may substitute the diffusion coefficient of vacancies, the best known determination of which at room temperatures is 10^{-12} m^2/sec [48]. As a result, we obtain $v_c \approx 10^{-3}$ m/sec. If the average value for all alloys of $D_H = 10^{-13}$ m^2/sec calculated on the basis of data on its values in pure aluminum [49] and in 7075-T6 alloy [8] is used as the diffusion coefficient of hydrogen, then $v_c \approx 10^{-4}$ m/sec.

10. Embrittlement of Metal. The embrittling factors are hydrogen bubbles, aluminum and alloy element hydrides, and hydrogen dissolved in the lattice or at the grain boundaries [6].

The majority of investigators who have studied hydrogen bubbles have assumed them to be harmless. Nevertheless, since hydrogen bubbles are sources of tensile stresses and stress concentration, they will obviously have some embrittling action [7, 50].

The fact of formation of hydrides (section 7) is too unconvincing to assume them to be the basic reason for embrittlement. This is indicated, above all else, by the reversibility of hydrogen embrittlement of hydrogen impregnated specimens (section 5) in laboratory air while in hydrogen bubbles forming under similar conditions the hydrogen pressure is sufficient for precipitation of hydrides [7, 16]. In addition, if it is assumed that embrittlement is caused by AlH_3 hydride, then alloys with an increased magnesium and/or lithium content must resist corrosion cracking to a greater degree. If formation of any hydride is harmful, then alloying with either element would strenghen the sensitivity to hydrogen embrittlement.

Actually, for example, in Al—Zn—Mg system alloys the corrosion cracking resistance depends not only upon the magnesium content but also upon the C_{Mg}/C_{Zn} ratio (with their total constant). A change in magnesium content in one direction or another, disturbing the optimum C_{Mg}/C_{Zn} ratio, reduces the corrosion cracking resistance [42] although the probability of formation of MgH_2 hydride increases with an increase in magnesium content.

The addition of lithium to Al—Zn—Mg system alloys increases their corrosion cracking resistance [51]. Consequently, neither a stimulating (precipitation of MgH_2 and LiH hydrides) nor a protective (retarding of formation of AlH_3 phase) role of alloy element hydrides appears and in addition an explanation is not obtained within the limits of the hydride hypothesis of the influence of alloy elements, which additionally places it in doubt.

An increased hydrogen content in aluminum alloys promotes embrittlement of them for several reasons. One of them is acceleration of dispersion hardening (section 3). This is a very serious factor in type AL27-1 alloys, which are used in the hardened condition and naturally age in service. This very reason, assuming a different C_H in the original castings, may now explain the very large dispersion in the values of $\sigma_{0.2}$ and σ_t [52] of different heats of an alloy after many years of natural aging and the sharp decrease in it after heat treatment ("recovery").

Another and more general reason is related to the reduction in flow stress caused by the high hydrogen content in the solid solution (section 5).

11. Certain Details of the Mechanism of Subcritical Crack Growth. For specificness let us consider corrosion cracking in laboratory air. Hydrogen bubbles are formed in alloys of different systems and with dissimilar corrosion cracking resistance [7], which is an indication of the high hydrogen content in the matrix surrounding them. It has been established (sections 5-7, 9) that failure with significant hydrogen embrittlement is promoted by intense transport of hydrogen. It is reasonable to assume that these same factors determine subcritical crack growth.

In uniaxial tension with $\sigma < \sigma_{0.2}$ dislocation creep of aluminum alloys occurs at room temperature [53, 54], especially intensely at the tip of a corrosion crack, where $\sigma \geqslant \sigma_{0.2}$. However, by itself it is insufficient for subcritical crack growth since in dry gases it is absent [2]. Under corrosion cracking conditions creep is eased by excess vacancies [24]. Dissolved hydrogen has a similar influence (section 5). Acceleration of creep with a high hydrogen content creates the prerequisites for transport of hydrogen at the critical rate. The remainder occurs, let us say for brevity, the same as in hydrogen embrittlement in static tests (section 5). It must also be added that the influence of the factors of the corrosive medium (section 4) amounts in the proposed mechanism to a change in the content of hydrogen and excess vacancies in the metal under the hydrogen film.

12. Role of Metallurgical Factors. The fracture of aluminum alloys in hydrogen embrittlement is primarily intergranular (section 7). In this case the content (more accurately, the activity) of magnesium (lithium) in the boundary zone, which determines the intensity of selective oxidation and generation of excess vacancies, acquires important value. Depletion of alloy elements here occurs as the result of formation of grain boundary precipitates. It is intensified with a decrease in hardening rate and in increase in aging temperature in heat treatment for the purpose of recovery of properties.

The influence of alloy element content in boundary zones on the corrosion cracking of aluminum alloys has been discussed more than once [24, 50, 55, 56] but that on the atomic-mechanical nature of corrosion cracking through the formation of excess vacancies has been little investigated [24, 50].

In addition to excess vacancies of oxidation origin, excess thermal vacancies related to Guinier—Preston zones may participate in hydrogen embrittlement in corrosion cracking. They must originate in intersection of the zones by dislocations, especially of zones the size of which after intersection becomes less than the critical. The vacancy concentration also decreases in hardening with reduced rates, in heat treatment for "recovery," and with an increase in aging temperature and time. Therefore, the above forms of treatment reduce the concentration of excess vacancies and in accordance with what has been presented (sections 2, 9, 11) must retard hydrogen embrittlement. In full agreement with this conclusion is the well-known fact that a reduction in hardening rate increases the corrosion cracking resistance of Al—Mg, Al—Li—Mg, Al—Zn—Mg, and Al—Zn—Mg—Cu system alloys and "recovery" and aging at increased temperature occur the same for alloys of the two last systems.

A secondary but very important metallurgical factor is the hydrogen content is semi-finished products or even or individual portions of them (section 3). Such hydrogen may be sufficient for subcritical crack growth (sections 2, 11). For the present only this hydrogen may be the likely explanation for the large spread in corrosion cracking resistance of specimens of different heats of a single alloy.

13. Significance of Hydrogen Embrittlement in Corrosion Cracking. It is assumed [8, 41, 42, 52, etc.] that hydrogen embrittlement is either the only or the primary mechanism of corrosion cracking of aluminum alloys. These assumptions are confirmed by the results and discussions of this work leading to the conclusion that in the case of corrosion cracking in humid air (without the formation of condensate in the cracks) hydrogen embrittlement is today the only acceptable mechanism of corrosion cracking. In corrosion cracking in solutions, especially in jumplike crack growth [8, 24, 58, 59], the initial stage of the jump is very probably determined by the hydrogen embrittlement mechanism. The metallurgical factors weakening hydrogen embrittlement also retard corrosion cracking.

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