MECHANISMS OF LIQUID-METAL EMBRITTLEMENT

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Even in the last century in soldering, mankind encountered the problem of liquid-metal embrittlement, a reduction in the ductility and strength of solid metals and alloys under the action of liquid ones. The first publications which reported on embrittlement of brass by mercury were in 1914 [1, 2]. The effects observed were related to penetration of mercury into the brass grain boundaries.

A systematic study of liquid-metal embrittlement has been made for more than 40 years, but there is still not a generally accepted point of view on the mechanism of interaction of a liquid metal with a solid one. The investigations of Soviet scientists made before 1955 were partially systematized in the monograph of Ya. M. Potak [3]. The observed strong reduction in strength and ductility of structural metals in molten low-melting metals is explained from the position advanced by P. A. Rebinder [4] of the concept of the adsorption influence of the medium. The author came to the conclusion that the reduction in surface energy of the solid body caused by adsorption leads to a reduction in its brittle strength and this aids in the formation of brittle cracks at stresses significantly less than in air. Therefore, in testing quite ductile materials in such media, when the brittle strength may not be lower than the resistance to plastic deformation, liquid-metal embrittlement does not occur. This was used to explain the selectiveness of the action of the medium. The possible influence of diffusion penetration of the medium was excluded.

W. Rostoker, J. MacCaughey, and G. Marcus [5], also seeing the reason for the observed phenomena of liquid-metal embrittlement in the reduction of interphase surface energy of the solid body, held the point of view that the role of regular and nonregular diffusion processes in liquid-metal embrittlement is insignificant. As proof a number of works were quoted where it was shown that embrittlement occurs when diffusion penetration of the medium into unstressed metal is absent.

To reveal the mechanism of liquid-metal embrittlement a large combination of investigations were made by V. I. Likhtman, E. D. Shchukin, and P. A. Rebinder on single crystals of very pure metals. Based on the Rebinder theory of the adsorption nature of embrittlement of solids in the presence of surface-active media, the authors for a quantitative rating of the influence of the medium used the known Griffiths approach on the relationship of strength to the surface energy of a solid, which under the action of liquid metals is sharply reduced. Since the medium is adsorbed on the surface, then, in the opinion of these authors, it cannot reduce the strength of ideal crystals but does reduce the strength of real ones, diffusing along the defects. The amount of the effect is related to what degree the adsorption layers of the surface-surface active substances from the embrittling medium are able to penetrate into the developing defects, covering the new surfaces of solid forming in them. For such a medium as liquid metals the possibility is assumed of internal adsorption, i.e., adsorption on the originating surfaces developing in the volume of a body being deformed. Somewhat later A. Westwood and M. Kamdar, not using concepts of internal adsorption, showed the possibility of local weakening of the bonds at the tip of a crack and on the surface of a solid metal, which is caused by surface adsorption of atoms of the medium [6].

The influence of a medium does not always include a reduction in strength and ductility of solids. Under certain conditions samples of pure metals with low-melting coatings deform by a greater amount than without the coatings [1]. The observed plastification of single crystals with low-melting coatings at temperatures above the temperature of "forced" cold brittleness (the temperature of the brittle–ductile transformation in the medium) was also explained by adsorption weakening of interaction of atoms of the solid and was considered as the most general aspect of the influence of the medium, related by a single reason with embrittlement and spontaneous dispersion [1].

Within the limits of this model easing of failure was explained by penetration of atoms of the medium into the zone of prefailure. Included in the interrelationship at the moment of rupture or reordering of the bonds, the medium influences these processes and aids in compensation of the new bonds being uncovered.

Since the understanding of the "surface energy of a solid" later received the sense of the work for formation of new surfaces, then the surface energy in the Griffiths equation was considered as consisting of two components, the interphase surface energy γ_{sl} and the work for plastic deformation γ_p, assuming the presence of a symbatic relationship between them [7]. The condition of failure in the medium may then be written as

$$\sigma_* = \mu(E/c)^{1/2} \cdot (\gamma_{sl} + \gamma_p)^{1/2}, \tag{1}$$

where E is the modulus of elasticity, c is the size of the defect, and μ is the proportionality coefficient. The value of γ_p may be determined only experimentally and therefore Eq. (1) does not make it possible for us to check the correctness of the model.

Analyzing the results of experimental work with the use of understandings of the adsorption effect of plastification, M. I. Chaevskii [8] assumed the possibility both of loss of strength of metals being deformed and of strengthening of them, assuming that plastic flow may be eased in the initial period and made more difficult in further deformation. In experiments made by him, strengthening was observed only in the case of breaking up of the grains under the action of the molten metal. The author proposed a mechanism of easing of plastic flow and origination of a crack in polycrystalline metals. Since adsorption of the medium eases outlet of dislocations to the surface, then surface sources of Frank—Reed dislocations may be activated, forming large accumulations in front of obstacles (grain boundaries) at the opposite end of a slip plane, which increases the normal and tangential stresses. If the increased tangential stresses cause plastic deformation in the neighboring grain, plastification starts but if this does not occur, then the possible doubling of local tensile stresses leads to the occurrence of a crack.

Such a mechanism does not explain embrittlement of plastic metals and therefore M. I. Chaevskii [8] used, the mechanism of internal adsorption of the molten metal, using a model of a wedge-shaped crack and the concept of a decrease in the interatomic interaction of the solid metal—medium system, assuming that brittle failure under the influence of the medium is possible only in the case of three-dimensional interaction of it with a metal being deformed. According to the model accepted [9, 10], plastic deformation at the tip of a crack causes the formation of vacant points, increasing the free energy and chemical activity of the solid metal. The medium, diffusing into the activated defective volume of the metal, changes the strength of the interatomic bonds, especially at the grain boundaries. Considering the alloy formed as a solid solution, the author rated the energy of the bond of atoms of different elements from the position of the statistical theory of strictly regular solutions [11] and came to the conclusion that if in the interaction of a solid body being deformed with a liquid low-melting metal a solution is formed, the low-melting component of which conforms to the Raoult law or has a positive deviation from it, then there will always be loss of strength of the metal being deformed. If there is a negative deviation, then according to this criterion it is impossible to judge about the change in strength [9].

M. J. Kelley and N. S. Stoloff [12] hold the point of view that liquid-metal embrittlement is caused by adsorption reduction of the failure energy of the atomic bonds at the tip of a crack in the solid metal (effective surface failure energy) and the possibility of the appearance of liquid-metal embrittlement depends upon the mutual solubility of the solid and liquid metals.

Using the conclusion of J. J. Gilman [13] that the energy of the atomic bond controls the surface failure energy even in those cases where deformation at the tip of the crack is plastic and assuming [14] that in the presence of a liquid metal the strength of an atomic bond of solid metal at the tip of a crack is decreased by the value of the energy of the bond between a solid and a liquid metal, M. J. Kelley and N. S. Stoloff [12] adopted as the criterion of change in surface failure energy the ratio of the energy of the atomic bond in a solid metal to the energy of the atomic bond of a solid and a liquid metal. To determine the energy of the bond between the atoms of a solid and a liquid metal, the Brewer—Engel approach [15, 16] was used. This takes into consideration the contribution of the electron transitions to the energy of the interaction. The authors considered the possibility of the appearance of liquid-metal embrittlement for approximately 40 pairs of metals (Fig. 1). At the same time, the influence of mutual solubility was determined by comparing the parameters of solubility determined as the square root of the density of energy of the bond [17]. Separation of the area of failure into two zones, brittle and ductile, is not very convincing since the majority of the points (pairs of metals) are grouped near the boundary and the boundary inself was chosen arbitrarily. In addition, the appearance of liquid-metal embrittlement depends upon the experiental conditions [1-3, 5, 18-20] and therefore it is not surprising that the data of these works does not agree with that given. As an example, it was observed in [18] that iron is embrittled by mercury and copper by aluminum, while in tests in air iron is embrittled by indium and not embrittled in vacuum [20]. As a result of diffusion of the medium there may be a substantial change in such characteristics of the solid metal related to ductility as deformation strengthening, defect packing energy, or the limiting failure deformation and thereby failure is influenced. Regrettably this mechanism does not take this into consideration.
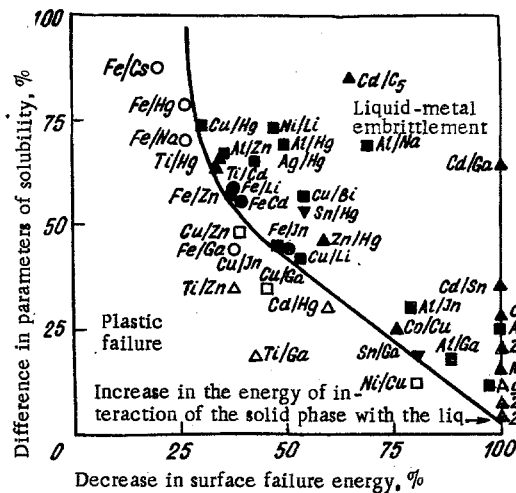
Fig. 1. The relationship of embrittlement to the strength
of the bond of the solid phase with the liquid and to solu-
bility [12]: △, ▲) close-packed hexagonal structure; ○,●)
body-centered cubic; □, ■) face-centered cubic. White
points are tests in the absence of the medium influence
and blacks ones in a medium of the second element.

The basis of the "energy of the bond—solubility" model was the inevitability of diffusion of liquid metal
into solid, and, at the same time, such systems as Al—Na, Fe—Na, Fe—Cd, and Fe—Hg, which are among the
immiscible, were considered [21]. But even for such systems which may form solid solutions a necessary
condition is diffusion of liquid metal into solid exceeding growth of the crack. Such a mechanism was used
on [22] for treating tensile-test results of high-purity zinc single crystals with a drop of mercury applied.
The authors explained the spasmodic character of failure by diffusion of the liquid mercury into the zinc, pre-
paring it for brittle failure. The calculations made, however, show that even in the initial stage of failure,
when the time of stops in the growth of a crack is tenths of seconds, diffusion of the medium is possible to a
depth of a tenth of the jump of the crack and this ratio becomes even greater by the end of failure when the
time of the stops decreases.

In some works [23-26] liquid-metal embrittlement is explained by selective solution of the solid metal.
According to this mechanism, developed in most detail by E. É. Glikman, Yu. V. Goryunov, and their colleagues
[25, 26], the origin and subcritical growth of a crack, related to the amount of interphase surface energy, oc-
curs in the grain boundaries as a result of their solution (as places with increased chemical potential). Me-
chanical stresses only strengthen the thermodynamic nonuniformity, promoting this selective solution of the
metal being deformed at the tip of the crack and deposition of dissolved atoms on its edges at a distance from
the tip of the crack approximately equal to the amount of the opening. The rate of growth of the crack v de-
pends upon the applied stresses σ but does not depend upon its length

$$v = a(\sigma - \Delta), \tag{2}$$

where a and Δ are constants not dependent upon stresses. The influence of the medium reflects the coefficient
a, which increases sharply with a decrease in the interphase surface energy γ_{sl}

$$a \sim \exp(-A\gamma_{sl}/kT), \tag{3}$$

where A is a coefficient, k is Boltzmann's constant, and T is absolute temperature.

Probably the complexity of the problem of liquid-metal embrittlement may be explained by such a variety
of proposed mechanisms of its occurrence. Apparently a common error of many authors engaged in this prob-
lem is the tendency to expand the models proposed by them to all possible cases of interaction of a solid metal
being deformed with a medium. For example, the Satiskii mechanism [24], which is true in the case of inter-
action of metals forming a eutectic, may not occur in other systems. Not only the formation of a solid solu-
tion, but even the presence of a liquid phase in the volume of the solid metal still does not predetermine em-
brittlement. It is known that leaded steel starts to embrittle at a temperature below the melting point of lead
and resumes its plastic properties soon after melting of the lead [27]. Probably it is impossible to expand

the "solution-deposition" mechanism [23, 25, 26] to those cases where failure in media at low temperatures is caused by stress concentration at the tip of a crack, under the action of which there is formation of a local plastic zone determining the basic energy expenditures for failure [28] and also if the solid metal does not dissolve in the liquid, as was noted in [6].

Under the action of liquid metal media failure may be caused by three basic processes: a) adsorption; b) uniform or selective corrosion (solution); c) the formation of new phases in the volume of the metal being deformed. Naturally the failure mechanism will depend upon which of the processes is predetermining.

Recent works on investigation of surface phenomena [29-33] make it possible to look at the mechanism of adsorption easing of deformation and failure of solids somewhat differently. Adsorption-active media may ease not only the movement and outlet of dislocations to the surface but also their origin [32-34]. As a result of this the dislocation density in the surface layers may be strongly increased [34], which is an indication of the large amount of deformation strengthening.

The model of the influence of a liquid-metal medium on the mechanical characteristics of metals and alloys being deformed, when solution of them and diffusion of the medium is not observed, may be the following (its basic assumptions are presented in [20, 35-37]):

1. The universality of the effect of adsorption-active media includes easing of the occurrence of plastic deformation in the surface layers of the metal caused by a reduction in the free surface energy at the boundary between the phases.

2. A medium, including a liquid-metal one, promotes the occurrence of plastic deformation in the surface layer not only as a result of easing the outlet of dislocations to the surface but also as a result of reducing the stress for the origin and start of dislocations. In the case of ductile materials this eliminates the barrier effect of the surface and increases their ductility.

3. The increased generation of dislocations under conditions limiting relaxation (low homologous temperature, high rate of deformation, low defect packing energy) favors an increase in their density in the surface layers of the metal, i.e., stronger deformation strengthening.

4. Strengthening of the surface layer promotes the origin in it of microcracks at deformations less than in the absence of a medium.

5. After passage by a crack of the surface strengthened layer further development of it will be retarded in the more ductile matrix but will resume after an increase in its opening to a value less than in vacuum since the influence of the medium on deformation of the local zone near the tip of the crack is similar to the influence on the free surface. As a result of this, propagation of the crack will occur with lower energy consumptions.

Within the framework of this model the possibility of appearance of the medium effect is determined by two factors: reduction in the surface energy and the properties of the solid under specific conditions of plastic deformation. At the same time, it is of no importance whether a single atom medium of liquid-metal type or multiatom organic compounds is being adsorbed. Of course, the quantitative expression of the effect will be different since the influence of the medium on the surface, the rate of migration, the possibility of penetration into narrow cracks, etc., will be different. The aggregate state of the medium does not play a role. The occurrence of a crack according to the proposed mechanism may occur if there is a solidified low-melting metal on the surface [20], although the development of such a crack, if it is not a sufficiently effective stress concentrator, may not occur since the medium will not be delivered to the tip of the crack.

The action of the medium on the surface of the solid includes, primarily, reduction in the interphase surface energy [4] and a change in the forces of representation [38] and surface relaxation [29]. In addition to adsorption of the medium, a definite contribution to this may be made by a change in the state or removal of the surface films [31, 32, 38]. Replacing the elastic oxide film by a medium with a shear modulus less than that of the matrix changes the sign of the force of representation acting on the dislocation and the latter will be forced to the surface with a force inversely proportional to the distance from it [38]. Close to the surface the field of translations will start to act on the dislocation. The amount of translation of the atoms relative to the position in the volume may be significant [30] but rapidly decreases and infringes upon five or six atomic planes. In connection with the increase in distance between atomic planes a dislocation to outlet to the surface must increase its Burgers vector, i.e., form an additional incomplete dislocation, for which it is necessary to expend the energy [30]
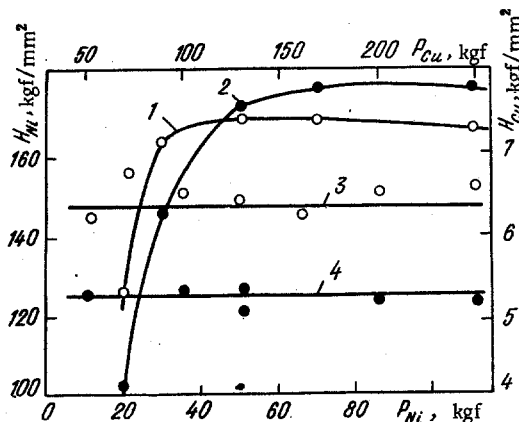
$$w \simeq \beta G \sum_{1}^{n} b_i^2, \tag{4}$$

Fig. 2. The relationship of Meier contact hardness to the force of insertion for nickel (1, 2) at 20°C in pure Vaseline oil (1) and with the addition of 2% oleic acid (2) and also for copper (3, 4) at 500°C in vacuum (3) and in Pb—Bi eutectic (4) [43].
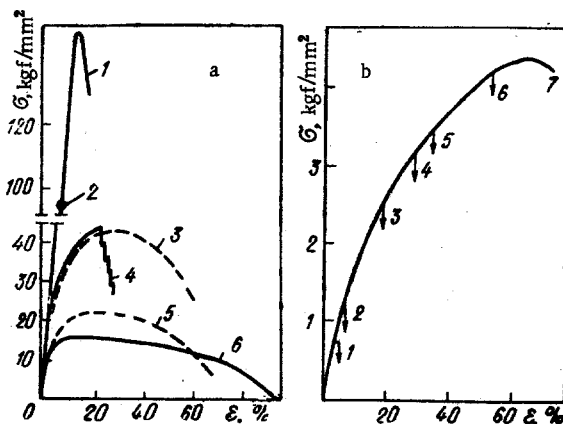

Fig. 3. Tensile curves: a) hardened 20Kh13 steel samples at 450°C (1, 2) and samples of Armco iron [19] at 300 (3, 4) and 550°C (5, 6) in vacuum (1, 3, 5), molten Pb—Sn (2), and molten Bi (4, 6); b) of pure (more than 99.999%) polycrystalline Al at 25°C in the presence of various mercury solutions [6]: 1) 3 at. % Ga; 2) 3 Zn; 3) 3 Cd; 4) 3 Ti; 5) 1.3 at.% Sn, in pure mercury (6), and in air (7).

where β is a constant, n is the number of displaced atomic planes, G is the shear modulus, and b_i is the difference between the Burgers vector in the surface layer and in the volume. The surface will either retard or ease outlet of dislocations, depending upon the difference in values of energy related to the representation effect and the field of translations. It is clear that adsorption of the medium, decreasing the field of translations [29], must ease plastic deformation. Easing of plastic deformation in a thin surface layer has been observed in the case of adsorption of gaseous nitrogen [39], organic media [40], aqueous solutions [41], and liquid metals [42]. However, as a result of adsorption of the medium the origin of dislocations is eased [31-33] and an increase in their density in the surface layer will promote its strengthening [34, 35].

Investigations made [43] showed that the character of the influence of the medium on plastic deformation by insertion of a spherical indentor depends upon the conditions under which it is done. In the initial stage the medium normally eases deformation (Fig. 2) and then may cause strong deformation strengthening if the relaxation capacity of the material is low. In those cases where the stresses are easily relaxed (such as tests of copper at 500°C)* the medium eases plastic deformation (Fig. 2, curve 4). Only with a sharp increase in

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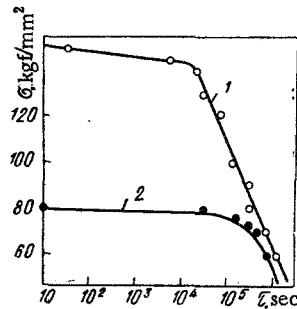*Experiments made by I. G. Dmukhovskaya.

Fig. 4. The long-term strength of hardened 20Kh13 steel samples at 450°C in vacuum (1) and in a Pb—Sn eutectic [44].

the rate of deformation is it possible to observe an increase in deformation strengthening of copper in molten Pb—Bi eutectic at 500°C [43]. The properties of the surface layer originating during deformation determine to a significant degree the behavior of the sample as a whole. At low temperatures stronger surface strengthening causes liquid-metal embrittlement [36], at temperatures above recrystallization, when the surface layer is strengthened in the medium less than in vacuum there is even plastification (Fig. 3a), while a reduction in strength (tensile strength) is caused by less deformation strengthening of the sample core (curve 6). A liquid-metal medium has such an influence on deformation of plastic metals when to obtain the strengthening (accumulation of damages) necessary for the origin of a crack significant deformation is required even in a medium and the subcritical development of a crack without a medium is impossible. In a brittle material a crack originates under the action of a molten metal at low plastic deformations or even in the stage of microplastic deformation (below the yield strength) and further propagation of it, if the solid metal is sensitive to stress concentration, without participation of the medium (curve 2). In these cases the medium has a very strong influence on the strength of metals. Failure of plastic metals also may occur at deformations less than those corresponding to the maximum on the tensile curve. At the same time, a strong influence of the medium on strength is also observed (Fig. 3b).

The adsorption character of the influence of the medium is revealed well in long-term strength tests of hardened 20Kh13 steel samples [44]. The fact that at a temperature of 450°C samples covered with a Pb—Sn eutectoid (Fig. 4) do not support long-term loads at stresses of more than 80 kgf/mm², while with a reduction in load by 2-5% the medium does not influence their life may be explained only by the adsorption effect. Such small variations in stress cannot markedly change the diffusion coefficient or the chemical potential and thereby cause the formation of a solution in a local volume [9, 12, 22] or strengthen solution [23-25, 26]. To a significant measure these processes must depend upon time, which in this case is not observed.

The only reason for the sharp bend in the long-term strength curve in tests in Pb—Sn (Fig. 4) may be a change in the mechanism of failure and since in its nature it is dislocation, then the medium must influence the formation of the dislocation structure during deformation. Similar results are observed in testing single crystals [1, 6].

In conclusion let us note that in the present state of investigations into liquid-metal embrittlement of the existing mechanism three may be distinguished as the most probable:

1) solution—deposition, which may be true at increased temperatures and when solution of the solid metal in the liquid is possible;

2) a change in the energy of the bond in local volumes as a result of the formation of a hypothetical solution, which may occur at increased temperatures and with the condition that penetration of the liquid metal into the solid occurs;

3) the adsorption influence of the medium on plastic deformation of the surface layers, predetermining subsequently the character of failure.

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