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BLISTERING AND HYDRIDE EMBRITTLEMENT

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ABSTRACT

The effects of hydrogen on the mechanical properties of metals have been categorized into several groups. Two of the groups, hydrogen blistering and hydride embrittlement, are reasonably well understood, and problems relating to their occurrence may be avoided if that understanding is used as a basis for selecting alloys for hydrogen service. This review describes both blistering and hydride embrittlement, emphasizes the understanding, and presents several techniques of materials selection and use to minimize their adverse effects.

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BLISTERING AND HYDRIDE EMBRITTLEMENT

INTRODUCTION

The effects of hydrogen on the mechanical properties of structural materials have been categorized in many ways. Terms such as hydrogen attack, blistering, delayed failure (static fatigue), hydriding, reversible embrittlement, environmental embrittlement, and hydrogen stress cracking all describe common, yet often specific, manifestations of environmental degradation of the mechanical properties of materials exposed to hydrogenous atmospheres. Such degradation has caused numerous service failures in engineering structures and thus has led to extensive research and development efforts on hydrogen effects. The volume of literature concerned with the effects of hydrogen on metals is almost overwhelming: for example in a 1951 review of hydrogen embrittlement of iron,[1] 1991 references were cited, and the December 1974 issue of *Metals Abstracts Index* cited 25 papers dealing with hydrogen in metals. It is thus apparent that any review of hydrogen metal interactions can only discuss a very small fraction of the available literature. Since January 1974, at least five reviews have been published as either conference proceedings[2-4] or individual articles.[5,6] On September 7 of this year yet another international conference on the influence of hydrogen on the properties of materials will be held (*Effect of Hydrogen on Behavior of Materials*, Moran, Wyoming).

The effects of hydrogen on metals depend on several factors including:

- Hydrogen concentration and diffusivity, and therefore, on exposure conditions such as temperature, pressure, and time.
- The possibility of reactions to form hydrides, and thus on hydrogen solubility and the properties of the hydrides.
- The possibility of reactions among hydrogen and impurities and/or alloying elements, and thus on the purity and composition of the metals.

Several proposed mechanisms by which hydrogen may affect the load-bearing capabilities of metals require the development of a critical hydrogen concentration within the metal lattice. This critical concentration is proposed to lower lattice cohesion,[7] cause localized plasticity,[8] restrict glide,[9] precipitate as gas bubbles,[10] or cause embrittlement by a combination of these and other mechanisms.[11-21] Regardless of the mechanism, however, the fact that hydrogen degrades mechanical properties is well known, and hydrogen embrittlement has been extensively studied. These studies have produced a degree of understanding which often permits design engineers to choose materials so as to minimize the probability of adverse hydrogen effects. The objective of this review is to emphasize this understanding in two areas: hydrogen blistering and hydride embrittlement.

BLISTERING

Hydrogen can be absorbed by a metal during casting, forging, heat treatment, welding, and finishing, or in service. If the external hydrogen pressure is reduced after absorption, hydrogen diffusion from the metal is required for the system to obtain thermodynamic equilibrium. The internal hydrogen concentration, C, in equilibrium with an external hydrogen pressure, p, over a hydride-free metal is

$$C = C_O p^{\frac{1}{2}} \exp(-\Delta H_S/RT) \text{ cc gas (NTP)/cc metal}$$

Hydrogen diffusion from the metal is also required for equilibrium if the temperature is reduced. If the temperature change is rapid, diffusion may be too slow to maintain equilibrium, and the metal lattice may become supersaturated with hydrogen.

Local equilibrium may be maintained within the lattice, however, by precipitation of molecular hydrogen at internal cavities (or defects). The hydrogen pressure at these cavities will depend on the initial concentration and the amount of the temperature decrease; it can easily exceed the pressure required to strain the metal plastically. Such strain opens microcavities and causes blistering. For practical purposes, however, blisters typically do not originate because of exposure to hydrogen gas but are developed by reactions between the metal and water vapor during melting and heat treatment, or from hydrogen introduced during finishing operations.

Iron and aluminum will react with atmospheric moisture at elevated temperatures according to the equations

$$Fe + H_2O \rightarrow FeO + 2H$$

$$2Al + 3H_2O \rightarrow Al_2O_3 + 6H$$

The metal lattice becomes supersaturated with hydrogen because the effective atomic hydrogen pressure during the reaction is many times the equilibrium pressure. Subsequent precipitation of molecular hydrogen leads to the development of high pressures and blisters (Figure 1a).[5] If hydrogen is absorbed while the metal is liquid, gas bubbles (blow holes) may develop because of the decrease in hydrogen solubility during solidification (Figure 1b).[22]

Precipitation of hydrogen gas at internal defects has also led to blistering after acid pickling, electroplating, and other finishing operations.

The cause of blistering is well-known: a change in environment (temperature, hydrogen overpressure) leads to precipitation of hydrogen gas from a supersaturated metal lattice. Thus, handling and finishing techniques have been developed to minimize the quantity of hydrogen in the metal. Acid pickling and other such processes that may introduce hydrogen are avoided when practical, and possible moisture sources, such as the coatings of welding electrodes, are carefully considered before use. Melts are degassed by bubbling an active or inert gas through the molten metal. Chlorination of aluminum is an effective method of removing hydrogen, and hydrogen is removed from some metals, such as magnesium, simply by holding the metal molten to allow hydrogen outgassing before casting.

Inclusions and other defects are typical sites for hydrogen precipitation. These sites can be controlled, to a limited extent, by processing techniques. For any given hydrogen content, the extent of blistering decreases when the number of precipitation sites is increased. Thus a homogenous distribution of very small inclusions will minimize susceptibility to embrittlement. In addition to this indirect effect of inclusions on embrittlement, hydrogen may diffuse into a metal and react directly with inclusions or alloy phases[23-26] to form a gaseous product. This phenomenon is often termed "hydrogen sickness."

Hydrogen sickness in copper is more correctly described by the term "steam embrittlement" and is only observed in copper which contains oxygen. It is caused by hydrogen reduction of copper oxide inclusions and/or interaction with dissolved oxygen to form water. The problem was encountered originally when hydrogen gas was used to maintain a reducing atmosphere during the heat treatment of copper. Hydrogen diffused into the metal during such anneals and reacted with oxide particles to form water. At temperatures above the critical temperature for water, the steam pressure generated by the reaction often exceeded the strength of the copper and caused plastic deformation and/or tearing. Examination of steam-embrittled copper frequently reveals grain boundary cracks or cavities (Figure 2). However, cavities are also found within the grains and along prior grain boundaries in alloys that have received thermo-mechanical treatments after oxide precipitation. Problems relating to the occurrence of steam embrittlement can be avoided if:

- Alloys chosen for hydrogen service have low oxygen contents and are free of oxide inclusions
- Alloys containing oxide are not annealed in hydrogen

Silver is also susceptible to steam embrittlement; however, in this case oxide reduction, per se, may not occur. Blisters have been found on high purity (99.99^{+}%) silver annealed in hydrogen.[25] Hydrogen diffuses into the silver and reacts with the dissolved oxygen to form water vapor, which is nearly insoluble and therefore precipitates. Detailed metallographic studies indicate that grain boundary precipitation is predominant. Individual bubbles continue to grow as the reaction proceeds until bubble agglomeration leads to grain boundary cracking.

Blistering is also often observed in steels used in the chemical and petrochemical industry because of hydrogen reaction with carbide inclusions and carbon in solid solution.[13] The reaction is termed "hydrogen attack," and high pressures are developed because of methane formation. The formation of methane reduces the mechanical properties because in addition to the formation of high pressure gas bubbles, the carbides and dissolved carbon are eliminated, thus lowering the strength. The susceptibility to hydrogen attack can be reduced by the use of steels containing titanium, vanadium, or other additions to react with carbon to form carbides that are not reduced by hydrogen.

In addition to hydrogen attack, steels are also susceptible to a phenomenon known as flaking. Hydrogen, in excess of about five parts per million will cause this phenomenon, which is manifested as internal cracks or bursts, usually occurring during rapid cooling from rolling forging. The phenomenon is more pronounced in heavy sections and in the higher carbon steels. In carbon steels, flaking may be prevented by slow cooling after rolling or forging. This slow-cooling operation presumably permits the hydrogen to diffuse out of the steel. Such a controlled slow-cooling operation after rolling is now standard practice in the manufacture of rails, and this practice has practically eliminated the occurrence of flaking.[26]

Hydrogen sickness, hydrogen attack, blistering, flaking, and other forms of damage caused by hydrogen reaction with alloy or impurity elements are generally described as irreversible hydrogen embrittlement. Although some ductility may be restored by heat treatments, the removal of strengthening phases, such as carbides, cause strength losses which are not generally recovered. Furthermore, it is doubtful that the microvoids, pores, blisters, or fissures developed during the attack can be healed effectively by thermomechanical treatments. Therefore, for design or fabrication considerations, prevention is a much more effective measure than subsequent attempts to recover strength and ductility losses.

Prevention of hydrogen attack was perhaps best described in the classic work of Nelson in 1949.[27] The Nelson Diagrams, first developed in that paper, resulted from extensive study of empirical data for high temperature hydrogen attack as a function of alloy composition, hydrogen partial pressure, and exposure temperature. These correlations (Figure 3) are still used in the selection of steels for systems which are to be exposed to hydrogen. The Nelson Diagrams provide indications of safe operating limits for a wide variety of steels under a broad range of exposure conditions. As stated by Troiano[28] in a 1973 keynote lecture for a *Hydrogen in Metals* conference, "it (the Nelson Diagram) is the major source of relevant engineering information available, and I know of no one who whould dare defy its indicated limitations."

HYDRIDE EMBRITTLEMENT

Zirconium, titanium, niobium, vanadium, uranium, and other such metals and their alloys form hydrides if the external hydrogen pressure is high enough. The titanium-hydrogen phase diagram (Figure 4) [29] is typical of titanium, zirconium, and hafnium, and shows that below the eutectoid temperature, hydrogen solubility decreases rapidly with decreasing temperature and is practically nil at room temperature. The other hydride formers show similar solubility decreases. Because of this decrease in hydrogen solubility, hydrides often precipitate even when the hydrogen content of the metal is very low. In some respect, this is similar to blistering; a hydrogen-rich phase is formed, and this phase may affect the mechanical properties of the metal.[11] Under severe hydrogen charging conditions, the strains developed by precipitation of the less-dense hydride phase can produce stresses sufficient to cause failure even when no load is applied.[13]

Susceptibility to hydride embrittlement is dependent on numerous variables[30-31] including hydrogen content, hydride distribution and morphology, temperature, and strain rate. The metal hydrides are not only brittle and less dense than the metal matrix, but precipitate at dislocations,[32] at grain and twin boundaries,[33] and at other defects. This combination of hydride-matrix properties influences both the initiation and growth stages of fracture.[33]

Hydride embrittlement generally increases with increasing hydrogen content (Figure 5).[34] This increased susceptibility is due to an increase in the number of hydrides per unit volume, a corresponding decrease in the interhydride spacing, and a change in actual hydride distribution.[35] Hydride distribution (orientation), which also affects interhydride spacing, is of such major importance in some cases that the role of hydrogen content is suppressed. Tensile specimens of Zircaloy-2 containing hydrides oriented parallel to the stress axis failed after 10% elongation; whereas comparable specimens containing hydrides perpendicular to the stress axis, but with only 0.05 times the total hydrogen, exhibited no macroscopic ductility.[36]

Increasing the temperature decreases the susceptibility to hydride embrittlement because, for any given hydrogen content, the volume fraction of hydride decreases. However, there is some evidence that above a critical temperature, the hydride may become ductile,[37] and thus be less likely to enhance crack initiation. Ductile-to-brittle transitions have been observed in hydrided zirconium alloys, and the transition temperature has been shown to increase with increasing hydrogen content (Figure 6).[35] This phenomenon was shown to be consistent with variations in inter-hydride spacing and led to the conclusion that the embrittling effect of hydride precipitation is due to cracking along the hydride.[35] The shape and distribution of the hydrides strongly depend on the heat treatment prior to precipitation and on the cooling rate during precipitation.[38] Because cracks propagate along metal-hydride interfaces (Figure 7),[39] cooling rates also influence embrittlement. Slow cooling apparently promotes the formation of thin platelets primarily at grain boundaries and on specific crystallographic planes, whereas rapid quenching precipitates highly dispersed particles. Correspondingly, the toughness (resistance to hydride embrittlement) is higher in hydrided titanium when the hydrides are compact, than when thin hydride platelets are present.[34]

The existence of hydride precipitates before testing is not a necessary condition for hydride embrittlement. Vanadium alloys with hydrogen contents less than the apparent terminal solid solubility are embrittled because the test stresses (strains) cause hydride precipitation and subsequent embrittlement.[40] In such case, the apparent solubility may be decreased by an applied stress because precipitation of the low-density hydride phase is accompanied by nucleation of numerous dislocations at hydride-matrix interfaces.[38] Test stresses aid hydride nucleation and thus lower the apparent solubility. This effect is markedly different from the thermodynamically predicted increase in actual hydrogen solubility through the application of elastic tensile stresses.[41]

Surface hydrides also affect the mechanical properties of hydride-forming metals. Failure of tensile specimens with surface hydride layers is initiated in the hydride and occurs at very low strains. Most of the conditions for surface hydride formation are met by exposure of hydride-forming metals to gaseous hydrogen, even at very low pressures. For example, calculations show that for alpha-phase titanium at room temperature, the equilibrium pressure above which hydrides will form is 5×10^{-14} torr. However, titanium alloys can be exposed to much higher hydrogen pressure with no adverse effects because of the protection afforded by the oxide film typically present on such alloys.[42] Surface hydride formation can be predicted from a knowledge of hydrogen transport rates in both the oxide film and the metal matrix.[42] Furthermore, surface hydride formation, per se, was shown to degrade the mechanical properties and may (without the application of stress) cause disintegration of the specimen (Figure 8).[42]

Hydride embrittlement, in most cases, is a maximum at high strain rates. Studies with hydrided titanium alloys show that impact and notch specimen testing are effective methods for determining the susceptibility to hydride embrittlement (Figure 9).[43]

The combination of stress orientation, strain-induced hydride precipitation, hydrogen diffusion along a stress gradient, and hydride properties apparently led to failure of a welded Ti-6Al-4V pressure vessel.[44] The initially uniform hydrogen distribution changed during the 1-5 years service at room temperature, and an almost continuous line of hydrides precipitated at a weld-base metal interface (Figure 10).[44] The failure was consistent with many of the ideas proposed by the various investigators. First the hydrogen distribution changed because hydrogen diffused preferentially to a high stress region.[19] The high hydrogen concentration lowered the stress for dislocation motion[8] causing plastic deformation at the tip of the defect. The plastic deformation further concentrated the hydrogen[10] and initiated hydride precipitation[40] with the hydrides oriented nearly perpendicular to the effective tensile stress at the crack tip.[36] Failure then occurred through the hydride or along the hydride-metal interface. A subsequent evaluation[45] of this type of "delayed failure" demonstrated the feasibility of such a mechanism.

Metallographic evidence for progressive hydride growth is shown in Figure 11, and a similar effect is observed in Figure 12.[45] This type of "delayed failure" is in agreement with the generalized model for hydrogen embrittlement proposed in Reference 11. In fact, both blistering and hydride embrittlement, which require either a hydrogen gas phase or a hydride phase, are almost by definition compatible with the proposal[11] that "the hydrogen embrittlement of any metal can be explained in terms of localized formation of a phase whose mechanical properties differ from those of the matrix because of hydrogen enrichment." However, as will be discussed in other papers in this session, evidence for the formation of new phases is not nearly as well established for either classical delayed failure or environmental embrittlement.

CONCLUSIONS

This discussion attempted to describe two forms of hydrogen embrittlement of metals:

- Embrittlement resulting from blister formation
- Embrittlement resulting from hydride formation

Clearly, hydrogen embrittlement is not a simple phenomenon, and many aspects of hydrogen-metal interactions remain unknown. However, within the above classifications, a basis for selecting materials for hydrogen service may be found. Only a small number of metals are hydride formers, and these should not be used in hydrogen atmospheres. Blister formation can be avoided by proper control of process variables, heat treating, and finishing operations. Selection of materials resistant to other forms of hydrogen embrittlement is more difficult and is the topic of the last paper in today's session.

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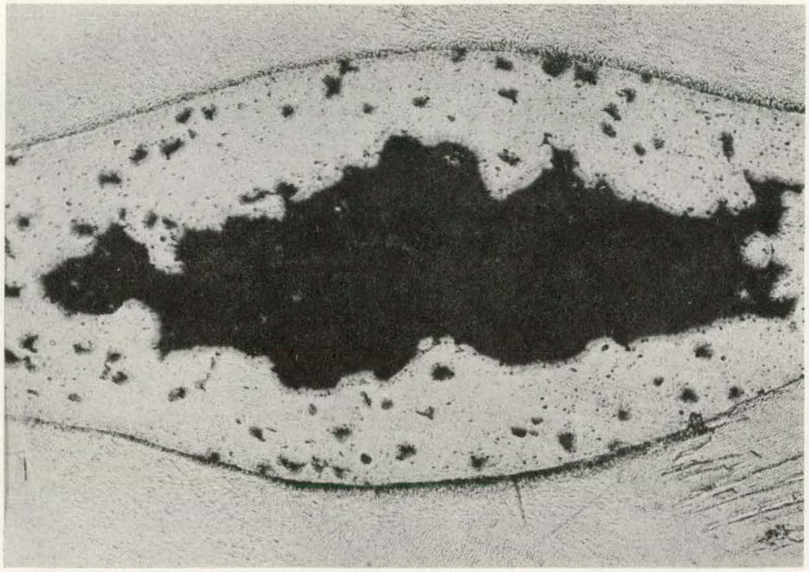

FIGURE 1a Hydrogen Blister in Aluminum. Blister formed by exposing 1100 Al to room air in muffle furnace at 773°K. *(From Reference 5)*

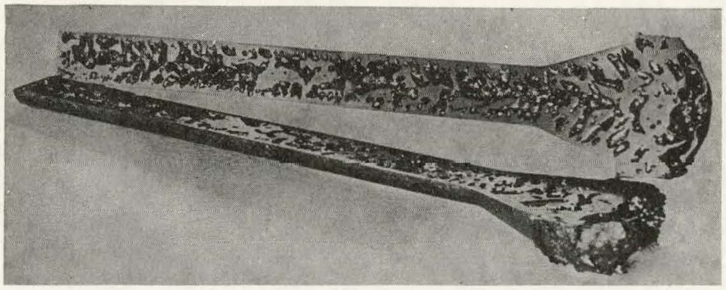

FIGURE 1b Hydrogen Blow Holes in Iron Casting. Iron was melted and cast in one atmosphere hydrogen; porosity caused by hydrogen precipitation due to a large drop in solubility upon solidification. *(From Reference 22)*

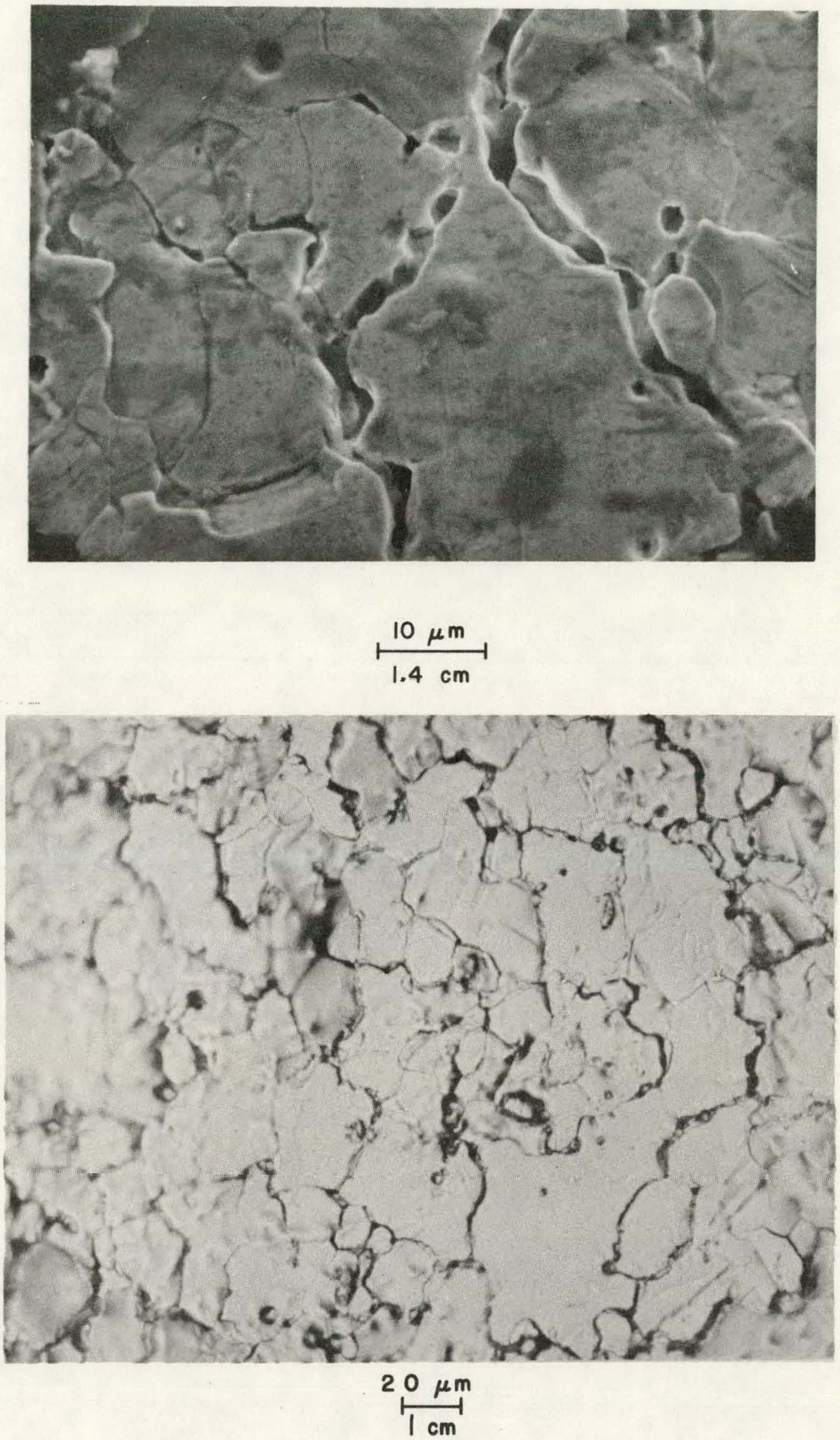

FIGURE 2 Pores and Cracks Along Grain Boundaries of ETP Copper Exposed to Hydrogen at 500°C
(From Reference 5)

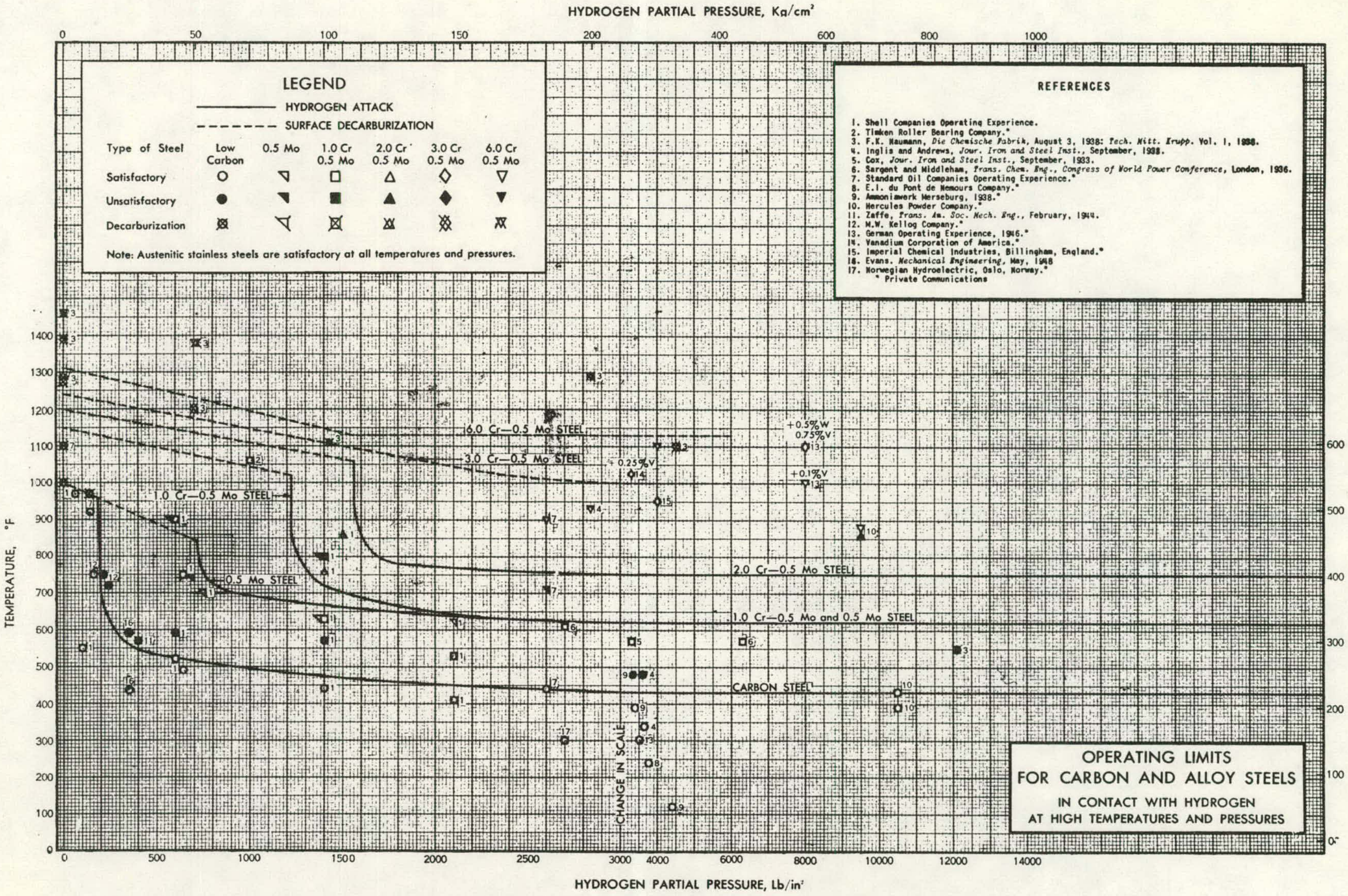

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FIG. 4 OPERATING LIMITS FOR CARBON AND ALLOY STEELS IN CONTACT WITH HYDROGEN AT HIGH TEMPERATURES AND PRESSURES

FIGURE 3 Nelson Diagrams

(From Reference 27)

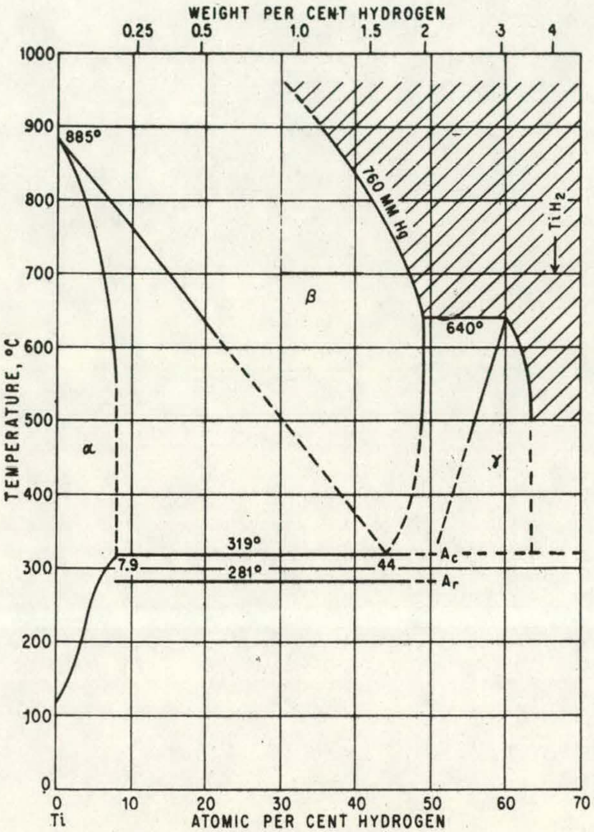

FIGURE 4 Titanium-Hydrogen Phase Diagram for Hydrogen at One Atmosphere
(From Reference 29)

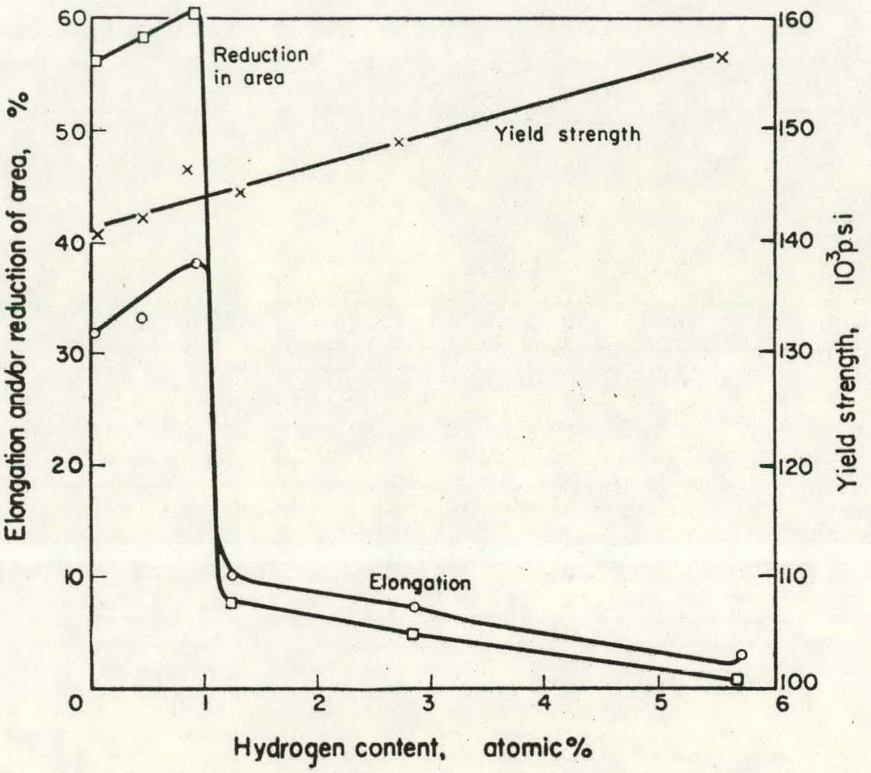

FIGURE 5 Tensile Properties of Ti-8Mn as a Function of Hydrogen Content
(From Reference 30)

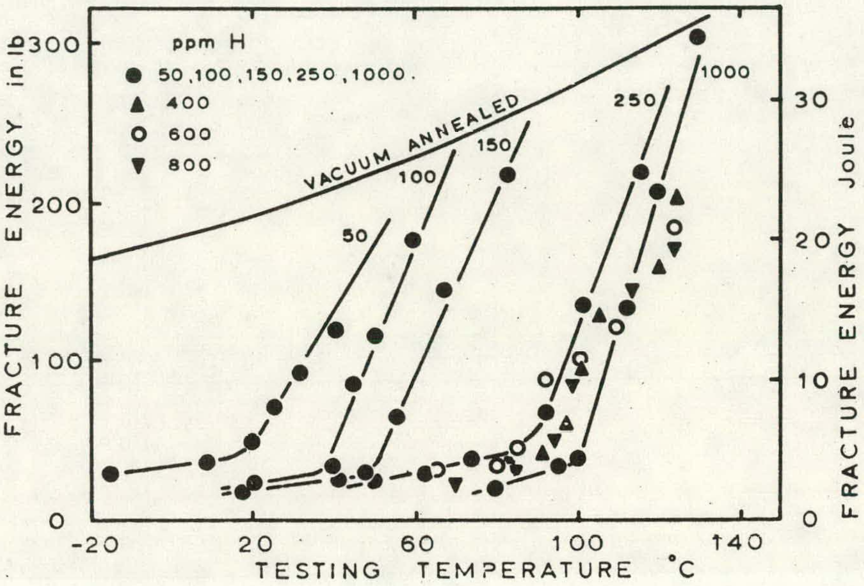

FIGURE 6 Ductile to Brittle Transitions in Hydrided Zirconium
(From Reference 35)

FIGURE 7 Cracking Along Metal-Hydride Interface. Cracks developed during tensile tests of hydrided Zircaloy-2.
(From Reference 39)

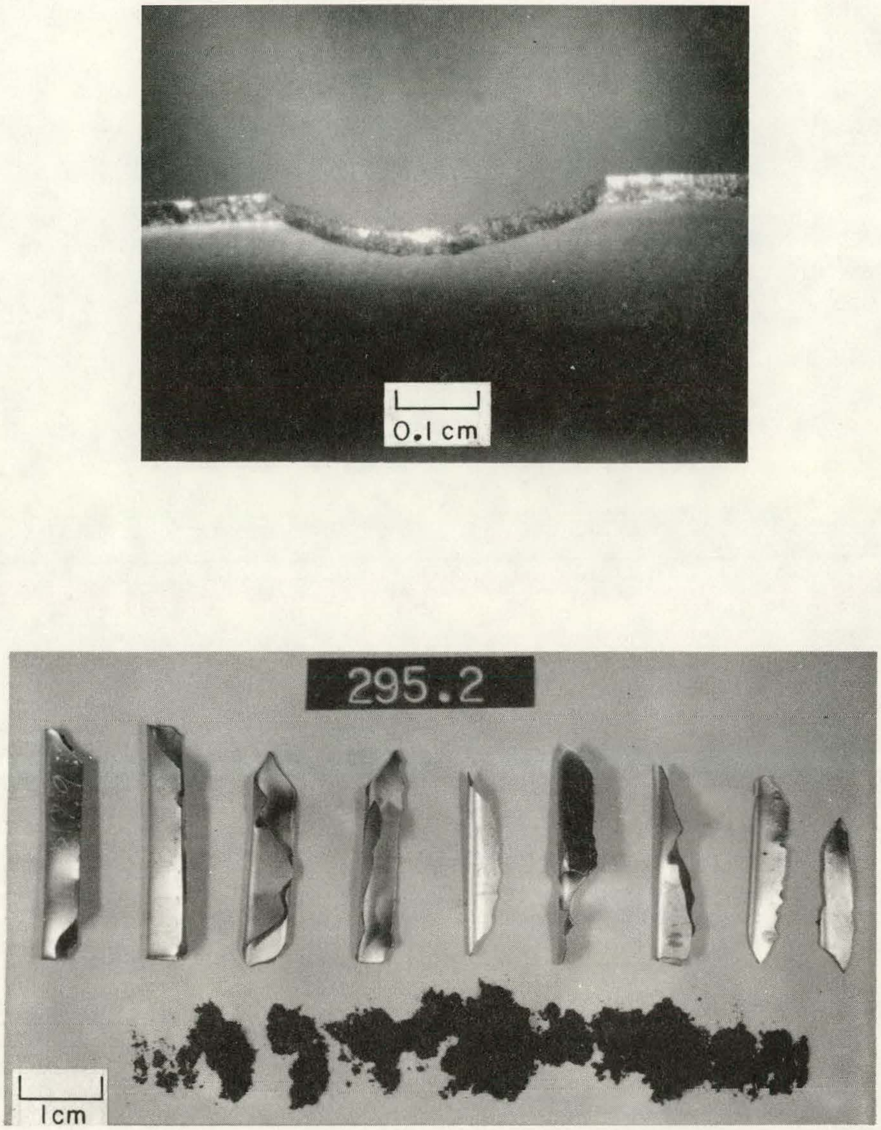

FIGURE 8 Disintegration of Ti-5Al-2½Sn to Hydrogen at 71°C
(From Reference 42)

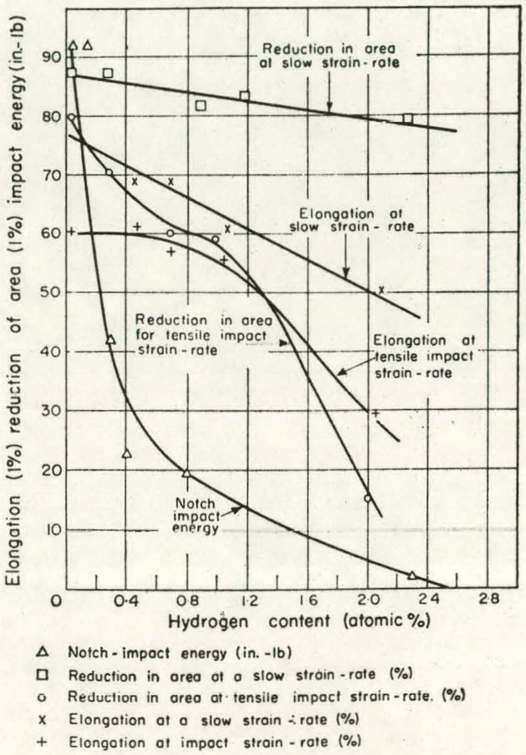

FIGURE 9 Effect of Testing Techniques in Revealing Susceptibility of Hydrided to Hydrogen Embrittlement
(From Reference 43)

FIGURE 10 Hydride Platchet Near the Edge of a Weld in Titanium Alloy *(From Reference 44)*

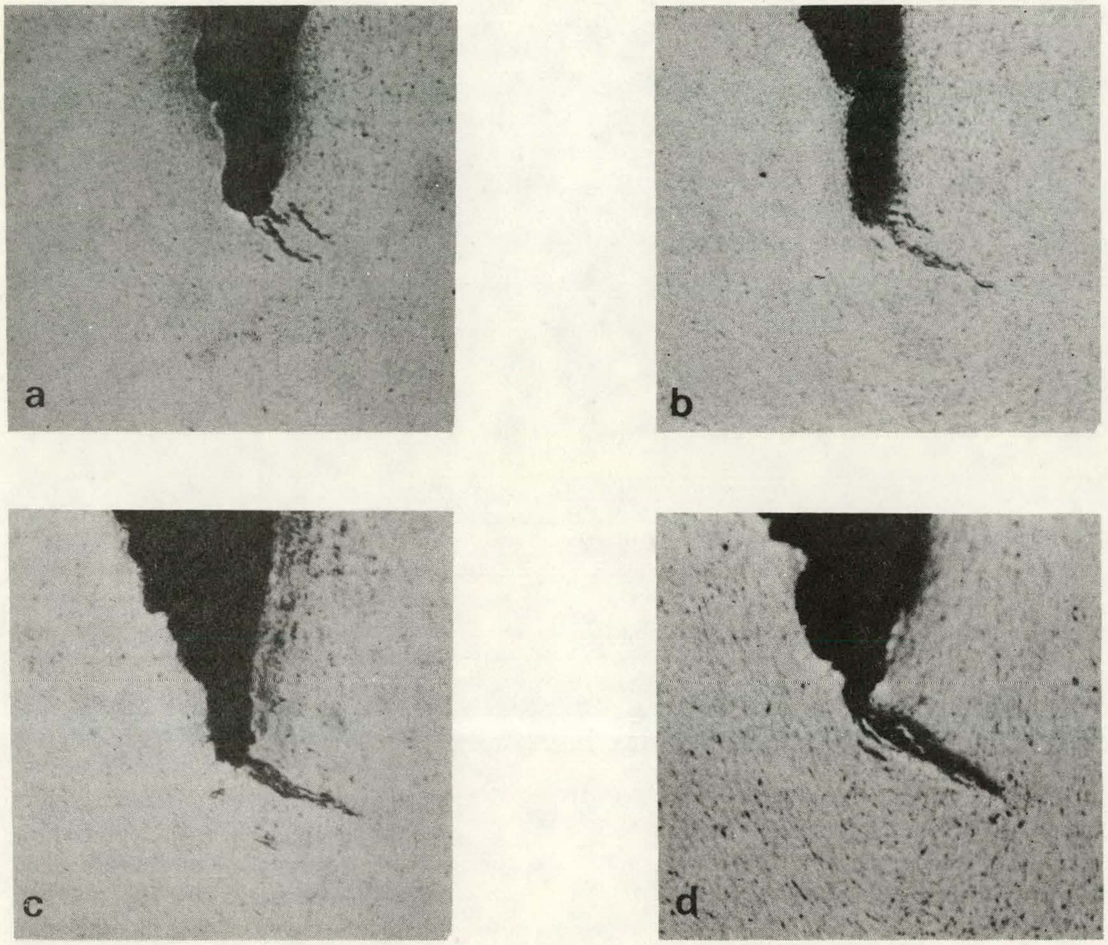

FIGURE 11 Progressive Growth of Hydride Precipitate at the Tip of Weld Defect in a Zirconium Alloy. The times between welding and specimen examination are (a) 2 weeks, (b) 4 weeks, (c) 8 weeks, (d) 24 weeks. *(From Reference 45)*

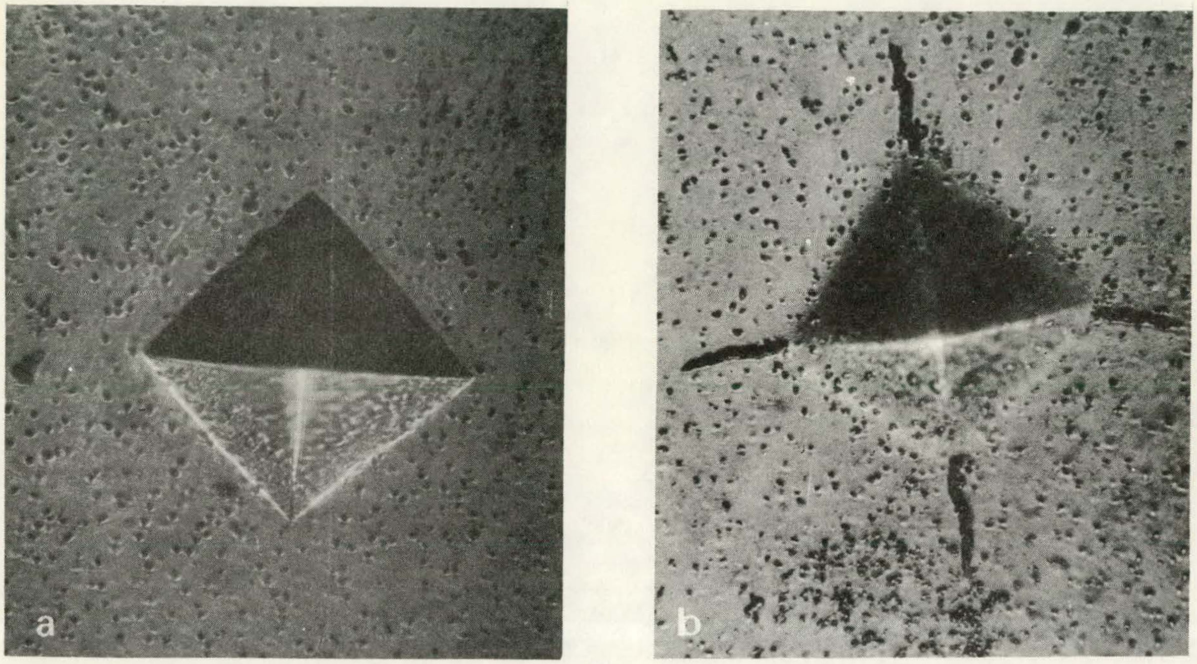

FIGURE 12 Hydride Growth at a Vickers Hardness Impression in Zr-2.5 wt % Nb Alloy. (a) A few minutes after making the hardness impression. (b) Six months after making the hardness impression. *(From Reference 45)*