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Hydrogen Embrittlement Tests of Cryogenic Metals*

by

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Introduction

In the field of cryogenics it is well known that many metals become dangerously brittle at low temperature. If the cryogen used is liquid hydrogen, the metal may suffer an additional embrittlement due to its possible exposure to hydrogen gas at room temperature and high pressure.

Although it is generally understood that metals can be embrittled by cathodic hydrogen during electrolysis and by gaseous hydrogen at high temperature, it is only within the past ten years that any attention has been paid to the embrittling effect of high-pressure gaseous hydrogen at ambient temperature.

Various alloys with acceptable low-temperature properties are currently being considered for cryogenic applications which also require exposure to high-pressure hydrogen gas at ambient temperature. In particular a question arose concerning the susceptibility of certain aluminum alloys to hydrogen embrittlement. The only relevant test found in the literature was for commercial grade 2-S aluminum, which was reported to be unaffected.¹⁾ It is the purpose of the present study to determine whether or not aluminum alloys are embrittled by hydrogen. Invar and a promising titanium alloy²⁾ are also tested.

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Experimental

Nine materials were examined for the possible embrittling effect of hydrogen. These included the six aluminum alloys, 1100, 2024, 5052, 6061, 7039, 7079; Invar-36; titanium-6Al-4V; and mild carbon steel AISI-C1019. Although not considered a good low-temperature steel, AISI-C1019 was included for comparison since its embrittlement by hydrogen was roughly known. At least six specimens $2.165 \times 0.394 \times 0.063$ in. were prepared from each material - three to be exposed to hydrogen and three to serve as controls.

In a previous paper³⁾ it was shown that specimens of carbon steel subjected to the action of cathodic hydrogen for 30 min at a current density of 1 amp/sq in. exhibit embrittlement which is similar to that for specimens exposed for 20 hr to room-temperature hydrogen at 40,000 lb/sq in. While it remains to be proven that the detailed mechanism by which hydrogen enters metals is the same for cathodic and high-pressure exposures, it is probably true that the embrittling effect of the absorbed hydrogen is much the same. As pointed out by Perlmutter and Dodge,¹⁾ surface coatings of metal, adsorbed gas, or oxide film can temporarily keep high-pressure hydrogen gas from entering the metal, but eventually penetration will result. One expects, therefore, that in the case of cathodic exposure, metal surfaces will tend to be cleaned by the nascent hydrogen evolved, whereupon diffusion into the metal can take place more rapidly. Because of the saving in time and effort, the present study employed only cathodic impregnation, with the exception that three specimens

of Invar were also exposed to 1700 ± 100 -lb/sq in. hydrogen gas at ambient temperature for 24 hours.

The electrolytic bath consisted of a 4% H_2SO_4 aqueous solution "poisoned" with CS_2 and P. Three de-greased specimens at a time were suspended as cathodes in the bath equidistant from a platinum gauze anode. Direct current was supplied by a small battery charger. A variable resistance was adjusted periodically to maintain a current density of 1 amp/sq in. during the 30-min exposure at room temperature.

It was previously shown^{1,3)} that a simple bend test gives a rather sensitive measure of the embrittlement suffered by metals exposed to hydrogen. The strip specimens were bent first 90 deg in one direction over a 1/2-in. roller by a second roller, then similarly 180 deg in alternate directions. The first bend was counted 1/2, all 180-deg bends were counted one, and a fractional count for the last bend was determined from the angle at which fracture occurred.

Discussion of Results

Averages of the number of 180-deg bends to break multiple samples of each material are summarized in Table 1. For the entries in Cols. 3 and 4, the irreproducibility averages about 6% although in one case it is as large as 20%.

From the large variation in the number of bends necessary to break the control specimens, it can be seen that the materials tested covered a wide spectrum of initial ductility. The titanium alloy broke after only one bend while 22 bends were required to break aluminum-1100. The 21 bends needed to break AISI-C1019 when compared

with previous work³⁾ on C1020 and C1022, all very similar alloys, indicates that the material was in the annealed state.

If one takes the ratio of exposed to control bends in Table 1 as a measure of change in ductility and considers a 10% change from unity as being significant, it appears that only the mild carbon steel AISI-C1019 was embrittled. With a ratio of 0.52 the embrittlement of this steel is comparable to that found previously³⁾ for annealed C1020 and C1022. Mild steel in the annealed state still remains quite ductile after hydrogen exposure, as evidenced by the 11 bends required to break C1019. It was found,³⁾ however, that with prior cold-working to a tensile strength of 100,000 lb/sq in., specimens of C1020 and C1022 were so embrittled by cathodic hydrogen that they broke after only 1/3 bend, giving an exposed/control ratio of ~ 0.05 .

As shown in Table 1 all of the aluminum alloys and Invar resist hydrogen embrittlement under the test conditions of electrolysis. Likewise the samples of Invar exposed to tank-pressure room-temperature gaseous hydrogen were not affected. Titanium-6Al-4V shows no measurable embrittlement but the bend test loses much of its sensitivity when applied to such a brittle material. It was earlier reported¹⁾ that neither commercial titanium nor titanium-7Mn was significantly affected by exposure to 52,000-lb/sq in. gaseous hydrogen for periods up to 60 days, although in the same test titanium steel was severely embrittled.

With the exception of AISI-C1019, it is feasible to use any of the materials in Table 1 for liquid hydrogen service if one bears in mind

that several of them, notably titanium-6Al-4V, aluminum-2024, and aluminum-7079, can be initially extremely brittle. Even so, exposure to cathodic hydrogen apparently does not confer additional embrittlement.

References

- 1) Perlmutter, D. D. and B. F. Dodge, Ind. Eng. Chem. 48, No. 5, 885 (1956).
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Table 1. Results of Bend Test

Material	Condition	Number of 180-deg bends to break ^a		Ratio Exposed/control
		Control	H ₂ Exposed	
AISI-C1019	Annealed	21.0 ± 0	11.0 ± .7	0.52 ± .04
Aluminum-1100	H-14	21.8 ± 1.7	22.0 ± 0	1.01 ± .08
Aluminum-2024	T-4	2.0 ± 0	1.8 ± .2	0.90 ± .10
Aluminum-5052	H-34	10.3 ± .4	11.0 ± 0	1.07 ± .04
Aluminum-6061	T-6	6.3 ± .4	6.3 ± .4	1.00 ± .13
Aluminum-7039	T-6	4.7 ± .4	4.3 ± .9	0.91 ± .28
Aluminum-7079	F(as forged)	2.5 ± .4	2.3 ± .3	0.92 ± .28
Invar-36 (free machining)	Cold drawn	9.3 ± .4	8.4 ± .4	0.90 ± .08
Invar-36 (free machining)	Cold drawn	9.3 ± .4	8.7 ± .4 ^b	0.94 ± .08
Titanium-6Al-4V	(unspecified)	1.0 ± 0	1.0 ± 0	1.00 ± 0

^a Average of at least three specimens.

^b Exposed to 1700 ± 100 lb/sq in. H₂ gas for 24 hours at ambient temperature.