

# Testing method and techniques for damage control and protection of industrial fasteners and weldments from failure due to Hydrogen Embrittlement

### Swasti Shree Jain<sup>1</sup>, Dr. Manish Vishwakarma<sup>2</sup>

<sup>1</sup>PG Scholar, Mechanical Engineering.Department, MANIT Bhopal, Madhya Pradesh, India <sup>2</sup>Assistant Prof. Mechanical Engineering, Department, MANIT BhopaL, Madhya Pradesh, India

### ABSTRACT

Hydrogen embrittlement is a popular occurrence in which a metal is undermined by the inclusion of hydrogen in or beneath its surface. Hydrogen embrittlement (HE) of steels is of grave concern in many industries e.g. power, oil, aerospace, automobile, transport & other key applications, where breakdown can have disastrous consequences. This paper focuses on the testing methods of industrial fasteners whose working conditions are teemed with hydrogen in molecular as well as atomic form. This paper also emphasizes on the damage control and protection from failure due to HE. The prevention of HE is a chief concern for designers, manufacturers and application engineers. This is principally true with respect to the selection and application of materials, manufacturing process, application environment &service conditions. In path of describing the techniques for protection from HE, the various significant aspects of hydrogen embrittlement e.g. failure characteristics, mechanism, identification, testing etc. have been discussed with a view to provide a whole insight of the subject. This paper presents a re-examination of recent research emphasising to protect the equipments/ components with special reference to fasteners from HE failure leading to augmentation in service life. The literature shows that regardless of much work done on hydrogen embrittlement, the researchers agree that major part is still hazy and substantial scope of discussion exists regarding the mechanisms.

Keywords : Hydrogen Embrittlement, Mechanism, Astm Testing Methods, Embrittlement, Susceptibility.

### I. INTRODUCTION

### A. Concept of Hydrogen Embrittlement

Hydrogen Embrittlement is the process by which metals such as steel become brittle and fracture due to the introduction and subsequent diffusion of hydrogen into the metal. This is often a result of accidental introduction of hydrogen during forming and finishing operations [1]. This phenomenon was first described in 1987 by B. Phull [2]. Hydrogen Embrittlement results in diminished toughness or ductility of a metal due to atomic hydrogen. During hydrogen embrittlement, hydrogen is introduced to the surface of a metal and individual hydrogen atoms diffuse through the metal. Because the solubility of hydrogen augments at higher temperatures, raising the temperature can increase the diffusion of hydrogen [3]. When assisted by a concentration gradient where there is significantly more

hydrogen outside the metal than inside, hydrogen diffusion can occur even at lower temperatures.

These individual hydrogen atoms within the metal gradually recombine to form hydrogen molecules, creating pressure from within the metal. This pressure can increase to levels where the metal has reduced ductility, toughness, and tensile strength, up to the point where it cracks open (hydrogen-induced cracking, or HIC).

### **B.** Typeface of Hydrogen Embrittlement

Basically there are two forms of HE; (a) Internal Hydrogen Embrittlement (IHE). IHE is the hydrogen embrittlement which is caused due to the residual hydrogen originated from processing and manufacturing methods, prevention of which is discussed later in this paper. Some processes resulting into IHE are electroplating, acid cleaning etc. [4]. The other form of HE that the researchers deal with is- (b) External Hydrogen Embrittlement (EHE). EHE relates to the induction of hydrogen from external sources like hydrogen affluent environment. SCC (Stress corrosion cracking) falls under the class of EHE [5].

### C. Mechanisms for HE

Hydrogen embrittlement is a very intricate process with many concealed mechanisms. Often, failure results from a amalgamation of numerous factors, making the ascertaining of driving mechanism very hard. Till now, three main embrittlement mechanisms have been proposed: hydrogen-enhanced decohesion (HEDE), hydrogen-enhanced localized plasticity (HELP), hydride-induced embrittlement (HIE), Adsorption induced dislocation emission (AIDE), Stress sulphide corrosion (SCC) [6].

In the following sections, each mechanism has been discussed –

### 1) Hydride-induced embrittlement (HIE)

The stress-induced hydride formation and cleavage mechanism is one of the well-established hydrogen embrittlement mechanisms with extensive experimental and theoretical support. The nucleation and growth of an extensive hydride field ahead of a crack has been observed dynamically by Robertson et al. In a-Ti charged from the gas phase in situ in a controlled environment transmission electron microscope. In their observations the hydrides first nucleated in the stressfield of the crack and grew to large sizes not by the growth of individual hydrides but by the nucleation and growth of new hydrides in the stress field of the others. They showed that these small hydrides grew together to form the larger hydrides. This auto-catalytic process of hydride nucleation and growth together with brittle nature of them seems to be the main cause of embrittlement of typical hydride former element, i.e. the element of the group Vb; e.g. Vanadium, Niobium, Titanium and Zirconium.

### 2) Hydrogen-enhanced decohesion (HEDE)

The decohesion model is one of the oldest models used to represent the change of properties as a result of atom hydrogen. It was described first in 1941 by Zapffe and Sims. It is based on the increased solubility of hydrogen in a tensile strength field, for instance on the tip of a crack or in areas with internal tensile strength or in the tension field of edge dislocations .The increased solubility of hydrogen in this tension field results in a decrease in the atom binding forces of the metal lattice. The influence of stress results in a premature brittlematerial fracture the grain boundaries along (intergranular cleavage) or network levels (transgranular cleavage) owing to the decrease of the binding forces.

### 3) Hydrogen-enhanced localized plasticity (HELP)

The most recent process model by far is the so-called HELP (Hydrogen Enhanced Local Plasticity) process. A prerequisite for the HELP process is, as is the case with decohesion model, the accumulation of hydrogen in the field of stress, for instance, in the vicinity of the tips of cracks or in the stress areas of dislocations (carriers of plastic deformation in a metal grid). During the initiation of a dislocation movement by introducing external stresses, the existing active hydrogen considerably eases the dislocation movement through shielding the fields of stress of the dislocations against each other as well as against other grid defects. Therefore, a local dislocation movement will already occur at low levels of shearing stress, which is caused by a local drop of yield stress due to hydrogen.

A sliding localization occurs, leading to a micro crack caused by the formation of micro pores and shearing action. As soon as the crack leaves the area of reduced yield stress, it will not propagate any further.

### 4) Adsorption- Induced dislocation emission (AIDE)

This is the combination of HEDE and HELP mechanisms and is based on weakening of interatomic bonds due to H atoms as was the case with HEDE whereas crack growth is caused by localized slip as in HELP. According to AIDE the hydrogen present in environment gets adsorbed at the cracked tips thus weakening of bonds by inducing the dislocations from a crack tip, subsequently giving arise to crack growth due to slip and ultimately forming microvoids.

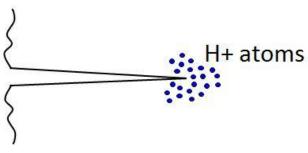


Figure 1: AIDE Mechanism

### 5) Stress Sulphide Corrosion (SCC)

SCC is the mechanism of the phenomenon in which the hydrogen atoms get trapped during the corrosion of steel by Hydrogen Sulphide.

This type of failure is prominently witnessed in petroleum and natural gas systems that are saturated with aqueous hydrogen sulphide in transport and processing. Researches and studies show that almost 30% of the damages in refining industries are associated with hydrogen damage.

The reaction between wet hydrogen sulphide and steel creates atomic hydrogen a part of which is absorbed into the steel. In the absence of stress, blistering occurs, while in the presence of stress sulphide corrosion takes place.

## II. Common Failure mishaps due to Hydrogen Embrittlement

Hydrogen embrittlement is an unpredictable phenomenon which comes across in almost every industrial branch of engineering like power-plant, chemical, gas processing, automobile, aerospace etc. Hydrogen embrittlement damages the components by reducing their ductility and strength, so it is difficult to predict the life of component.

Some of the critical components failure on account of HE are discussed here –

### A. Failures of high strength steel fasteners-

High strength mechanical steel fasteners are broadly characterized by tensile strengths in the range of 1,150 - 2,150 MPa (165 - 315 ksi), and are often used in critical applications such as in bridges, vehicle engines, aircraft, where a fastener failure can have disastrous consequences. [7].

When high strength steel is tensile stressed, as is the case with a high strength fastener that is under tensile load from tightening, the stress causes atomic hydrogen within the steel to diffuse (move) to the location of greatest stress (e.g., at the first engaged thread or at the fillet radius under the head of a bolt). As increasingly higher concentrations of hydrogen collect at this location, steel that is normally ductile gradually becomes brittle. Eventually, the concentration of stress and hydrogen in one location causes a hydrogen induced (brittle) micro crack. The brittle micro crack continues to grow as hydrogen moves to follow the tip of the progressing crack, until the fastener is overloaded and finally ruptures. This hydrogen damage mechanism can cause the fastener.



Figure 2: Typical Fastener Failure by Hydrogen Embrittlement [8]

In 2013, six months prior to opening, the East Span of the Oakland Bay Bridge failed during testing. disastrous failures occurred in shear bolts in the span, after only two weeks of service, with the failure attributed to embrittlement, possibly from the environment.

# **B.** Hydrogen induced cold cracking (HICC) in a low in high strength steel weldments

In high strength steel weldments, hydrogen is introduced by the arc of welding and diffuses to the heat affected zone where susceptible microstructures such as martensite and bainite are present. This causes hydrogen embrittlement. At critical residual stress, the Hydrogen Induced Cold Cracking (HICC) occurs. Thus, HICC occurs due to three factors: i) a susceptible microstructure; ii) sufficiently high concentration of • diffusible hydrogen and; iii) a critical stress intensity. [9]

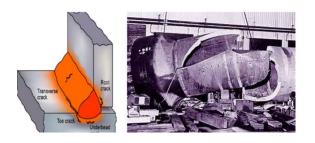


Figure 3 : Weldment failure due to HE

# C. Significant Past events of hydrogen embrittlement failure in industry

A first example is the failure of a storage tank for compressed hydrogen. The consequences of this can be confirmed from Fig. 3. This failure was caused by the growth of large fatigue cracks which was induced by hydrogen insurance. The total damage paid by insurance in this case was approximately US\$55 million. Hydrogen damage is more frequent than many people would suspect.

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In the minds of people, hydrogen is often synonymous with danger especially since the Hindenburg disaster on 6 May 1937. On that day, the Zeppelin inflated with 200,000 m3 of H2 ignited in less than a minute resulting in the death of 35 out of the 97 passengers who jumped out of the airship out of panic.

## III. General Characteristics of HE failure

- HE phenomenon occurs with high strength steel fasteners.
- Fasteners subjected to protective coatings e.g. Zinc electroplating are more sensitive for HE damage.
- Parts in contact with sulphuric acid and hydrochloric acid during manufacturing or service may subject to HE failure.

- If the failure is due to IHE it must have occurred just after some time of installation, usually one hour to one day.
- The fasteners hardened to at least Rockwell C38 may subject to HE failures. Unhardened fasteners are less prone to hydrogen embrittlement. [10]
- The appearance must be that of an "transgranular" failure. [10]

# IV. Factors That Influence Hydrogen Embrittlement on Parts

The severity and mode of the hydrogen damage depends on:

- Source of hydrogen—external (gaseous)/internal (dissolved).
- Exposure time.
- Temperature and pressure.
- Presence of solutions or solvents that may undergo some reaction with metals (e.g., acidic solutions).
- Type of alloy and its production method.
- Amount of discontinuities in the metal.
- Treatment of exposed surfaces (barrier layers, e.g., oxide layers as hydrogen permeation barrier on metals).
- Final treatment of the metal surface (e.g., galvanic nickel plating).
- Method of heat treatment. Level of residual and applied stresses.

### V. Hydrogen Embrittlement Test methods

- A. Disk Pressure test
- B. Constant load test
- C. Inclined Wedge test
- D. Four-point bend test

### A. Disk Pressure test

The method provides quantitative information on hydrogen embrittlement susceptibility of metallic materials. Materials to be investigated are manufactured into thin disks of specified size. Disk-shaped specimens are subjected to increasing gas pressure until they burst. Some of the samples are tested in helium (control) gas and some in hydrogen gas. The benefits of this test method include its simplicity and quantitative results. A limitation is that the specimen geometry is disk-shape only. In addition, the actual test may be fast, but set-up for the test is somewhat complex and time consuming. [11]

The test method requires a machined test cell, specimens, high pressure helium and hydrogen gases, valves, pressure gages, tubing, and a vacuum pump as shown in Fig 4(a) and (b).

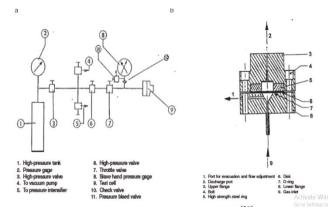
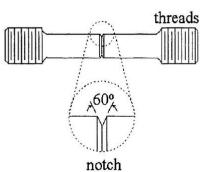


Figure 4. (a) Schematic of disk rupture test; (b) Disk pressure Test cell.[6].

#### **B.** Constant load test:

Hydrogen Embrittlement of Steels – Notched Specimen Test" describes a constant load test for assessing hydrogen embrittlement in steels residual from electrolytic or chemical surface treatments. Specimens are subjected to an axial load equal to 75% (+/-2%) of the tensile strength of a notched, uncoated specimen, for a period of 200 hours.

Notched test specimens and a device for delivering constant load are required for the test. ASTM F519, "Standard Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating Processes and Service Environments" employs a notched round bar tension test under constant load, Type la; see Figure 5. [12]



**Figure 5.** Type 1a specimen geometry ASTM STP 543,[4].

### C. Inclined Wedge test

The test method has two main functions: 1) it may be used in the statistical-basis determination of batch lot rejection or acceptance, and 2) it may be used to determine the effectiveness of processing steps (i.e., pre and post-baking treatments) in reducing mobile hydrogen in threaded metallic items. Testing is carried out after performing hydrogen embrittlement relief heat treatment. Selection of samples should be performed in accordance with the required quality assurance level. A minimum sample size of 30 pieces is necessary from each embrittlement relief treated batch that exceeds 500 pieces of plate as a single group. [13]

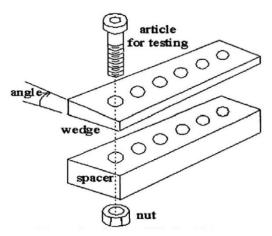


Figure 6. Inclined Wedge Fixture (ASTM B839)

#### **D.** Four-point bend test

ASTM F519 describes two types of four-point bending specimens and loading methods: Type lc notched round bar specimens are tested using a self-loading fixture; see Figure 7(a). Type le notched square bar specimens are tested using appropriate four-point bend adapters under displacement control; see Figure 7(b). An important

benefit of these self-loading fixtures is that the assemblies may be inserted into closed test vessels and exposed to environments without interruption. [12]

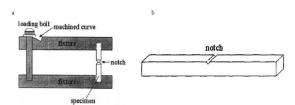


Figure 7.(a) Notched round bar bend by self-loading fixture (After ASTM F519); (b) Type 1e specimen geometry (ASTM F519) [5]

The results are interpreted as follows:

1) For test specimens that exceed 90% of their RSL fracture strength the plating bath is considered to be nonembrittling.

(2) For test specimens that exceed 75% of their RSL

fracture strength, the plating bath is considered to be of acceptable quality.

# V. Methods/ techniques for prevention of hydrogen embrittlement

Steps that can be taken to avoid hydrogen embrittlement include reducing hydrogen exposure and susceptibility, baking after plating (mandatory and as soon as practical) and using test methods to determine if a material is suspect. Other options that could help in avoiding hydrogen embrittlement include the use of lower strength steels (not always viable), the avoidance of acid cleaning, the utilization of low hydrogen plating techniques and the reduction of residual and applied stress [7].

**Reducing Corrosion Rate-**Hydrogen embrittlement occurs frequently during pickling operations where corrosion of the base metal produces vigorous hydrogen evolution. By careful inhibitor additions, base metal corrosion can largely be eliminated during pickling with a subsequent decrease in hydrogen pickup.

**Using Clean Steel-**Rimmed steels tend to have numerous voids, and the substitution of killed steel greatly increases the resistance to hydrogen interstitials for embrittlement because of the less number of voids in this material.[14] **Baking-** Hydrogen embrittlement is an almost reversible process, especially in steels. That is, if the hydrogen is removed, the mechanical properties of the treated material are only slightly different from those of hydrogen-free steel. A common way of removing hydrogen in steels is by baking at relatively low temperatures at 200-300 F [15].

**Practicing Proper Welding-**Low hydrogen welding rods should be specified for welding if hydrogen embrittlement is a problem. Also, it is important to maintain dry conditions during welding since water and water vapour are major sources of hydrogen.

**Substituting Alloys-**The materials most susceptible to hydrogen embrittlement are the very high-strength steels. Alloying with Ni or Mo reduces susceptibility. Because, Nickel-containing steels and Nickel-base alloys have very low hydrogen diffusion rates and best way to prevent from hydrogen embrittlement [16].

**Protective coatings-**A plethora of coatings exist that have been demonstrated to reduce either the outgassing of hydrogen in vacuum systems, or as diffusion barriers to the ingress of hydrogen. The most modern non structured coatings e.g. graphene, Tic, Tin, Al2O3 are still need to be proved for enhanced service life and barrier for hydrogen embrittlement.

### **VI. Conclusion**

We have discussed basic mechanism of hydrogen embrittlement along with its characteristics and factors affecting the development and behaviour of HE in specific service conditions. Also efforts are made to describe the various methods and techniques available to evaluate the effect of HE on high strength steels. Techniques for prevention of HE are also enlisted briefly. The discussion emphasise that research conducted are multidirectional and future efforts are to be made to develop economical and commercially means available to combat the hydrogen embrittlement.

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