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Review Article

Hydrogen embrittlement in different materials: A review

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ABSTRACT

Hydrogen embrittlement (HE) is a widely known phenomenon in high strength materials. HE is responsible for subcritical crack growth in material, fracture initiation and catastrophic failure with subsequent loss in mechanical properties such as ductility, toughness and strength. This hydrogen is induced in the material during electrochemical reaction and high-pressure gaseous hydrogen environment. LIST, SSRT and TDS techniques are performed to know the effect in mechanical properties and amount of hydrogen available in the material. For microstructure examination SEM, FESEM and TEM are performed to know the effect of hydrogen in the internal crystal structure. Also, various mechanisms which are responsible for crack growth and final fracture are discussed. This paper deals with HE definition, mechanisms which causes HE, subcritical crack growth, the concentration of hydrogen measurement and prevention activities are discussed which act as a barrier for hydrogen diffusion.

Contents

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Introduction

Some specific hydrogen induced damage to metals and alloys are hydrogen induced blistering, hydrogen embrittlement (HE), cracking due to hydride formation, hydrogen attack and cracking due to precipitation of internal hydrogen [1]. Hydrogen in high strength material such as steels has a most common effect known as hydrogen embrittlement [2–4]. It is defined as the process by which strength of a material can reduced significantly by introduction of hydrogen atom when working on hydrogen environment. Simultaneously ductility of a material is reduced and make it brittle. Austenitic stainless steel has widely accepted for production of hydrogen energy-related equipment because they have less susceptible to hydrogen embrittlement but after some time that embrittlement problem still occurs. This embrittlement is increased by slow stain rate and elevated temperature. Hydrogen transportation, storage system and components of fuel cell vehicle (FCV) are directly exposed to high pressure hydrogen environment. So, the embrittlement problem arises on that system. However, the main mechanism by which this degradation process happened is still not clear and some debate is still going on this [3]. The main mechanism which was highly responsible for hydrogen embrittlement are hydrogen enhanced decohesion mechanism (HEDE), adsorption induced dislocation emission (AIDE), hydrogen enhanced localized plasticity (HELP). The detail of such mechanisms is broadly available in a different paper [5–7].

The mechanical properties such as tensile strength and fatigue strength is reduced due to the effect of hydrogen embrittlement. Slow strain rate tests were also performed to evaluate the mechanical properties of the specimen under tensile stress with hydrogen charging [13]. The interaction hydrogen and slip band play an important role in hydrogen embrittlement [8]. Y Mine et al. were found due to this interaction hydrogen induces a micro crack along slip bands and enhances the fatigue crack growth [9]. There are the several parameters that affect the mechanical behaviour of steel under the presence of hydrogen such as chemical composition, condition of hydrogen charging (Temperature, source of hydrogen, surface condition, time for hydrogen charging), microstructure of steel and testing condition (temperature, specimen preparation, dimension, deformation rate) [12].

There are the several methods by which hydrogen can be entered in the material such as cathodic charging, electroplating, during welding etc [134]. From the literature, it was found that if we are performing the cathodic charging then the current density plays an important role in hydrogen absorption in steel. It was also found that for hydrogen induced phenomena in steel materials the current density which we are using must be in between 0.02 mA/cm^2 to 40 mA/cm^2 [10,11]. Higher the current density, more changes of hydrogen to get diffused into the material.

In case of welding, hydrogen assisted cracking (HAC) occur at a heat affected zone due to diffusible hydrogen content. It was advisable that electrodes are preheated before welding process to reduce the HAC of weld metal. Sometimes post weld heating or treatment of welds should be done after the welding to eliminate the risk of HAC. It is also found that the low welding current and negative polarity are responsible for the minimum level of diffusible hydrogen. Various test methods are available for determining the diffusible hydrogen contents such as glycerin technique, vacuum hot extraction method, mercury method and gas chromatography process [1]. Mercury technique is most simple and suitable one when compared to others techniques to measure the diffusible hydrogen quantity when it available in low quantity.

This paper reviews the hydrogen embrittlement in different materials such as high strength steels, high Mn steels, some advanced high-strength steels (dual-phase (DP), quenching and partitioning (Q&P), and twinning-induced plasticity (TWIP)), iron aluminides (TiAl, NiAl, FeAl) and aluminium alloy [15–18,116,118–120,124]. Also, some case studies were performed. Linearly increasing stress test (LIST) and thermal desorption spectroscopy (TPD) are the technique to evaluate the HE in a material (14,122). To know the effect of embrittlement on mechanical properties of materials, the slow strain rate test (SSRT) has been performed and found that fracture stress was reduced due to hydrogen introduction in notched and smooth specimen [23]. Also, the microstructure analysis of fractured specimen has been done by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). To reduce this HE in different materials, some prevention techniques must be followed such as selection of material which is least susceptible for HE, coating of different materials which reduces the susceptibility of HE such as graphene and niobium, alloying of titanium and aluminium and providing carbon and nitrogen diffusion layers above the materials [19–21].

Hydrogen embrittlement in materials

Generally, high strength materials are very much susceptible to hydrogen embrittlement when they work on hydrogen environment. Materials which are susceptible to HE is high strength steels, high Mn-steel, Aluminium alloys, Titanium, Magnesium and Magnesium alloys etc [16–18,116,118,119].

Steels whose strength are more than 1000 MPa is susceptible for HE [14]. These high strength steels are used in many places such as aerospace application, nuclear industries, high pressure hydrogen storage tanks, transportation industries, automotive applications etc. After some time, the development in high strength steel has occurred and a new class of steels such as High Mn steel, martensitic advanced high strength steel (MS-AHSS) is being used in many applications. This high Mn steel has various advantages such as excellent strength, ductility. But the embrittlement problem occurs on this steel and the HE susceptibility of high Mn steel is due to its chemical composition [15]. MS-AHSS is a new class of steel and strongest in all AHSS. It used in car manufacturing due to its high strength to price ratio [14]. But its ductility is slightly low as compared to other AHSS materials. Most of the studies of MS-AHSS has been done under control laboratory atmosphere. One question remains unclear that how this laboratory result should be implemented in actual working or service condition. This will need to be examined. Aluminium alloys are also affected due to HE with simultaneous corrosion cracking [18].

Factors which are responsible for susceptibility of materials for HE is (see Fig. 1) [14,25]

1. Strength of material & Residual stress in material.
2. Pressure, temperature & exposure time.
3. Applied strain rate & Surface condition of a material.
4. Concentration or amount of hydrogen & Amount of hydrogen trap.
5. Metallic coatings & specific precipitates.
6. Microstructure of a material.
7. Solution that make a reaction with metals (acidic solution).
8. Heat treatment of a material.

Characteristic of hydrogen embrittlement

For the degradation of mechanical properties caused by hydrogen embrittlement, three conditions must be fulfilled.

a. A material that is susceptible for hydrogen embrittlement if UTS>1000 MPa.
b. Hydrogen either pre-existing in the alloy or in the environment.
c. Tensile stresses in service condition and surface hardness must be greater than HRC 37.

The main characteristic of hydrogen embrittlement is strain rate sensitivity and susceptibility of delayed failure. HE is enhanced by slow stain rate. If we are applying the higher strain rate then the material is less susceptible for HE. Similarly, in HE parts the susceptibility of delayed failure has increased. It is also known that the hydrogen transportation in steel and alloys are possible because of diffusion of hydrogen and dislocation movement [23].

Hydrogen embrittlement is very well-known phenomena in high strength material. The effect of embrittlement is that the material failed catastrophically below its desirable strength or stress. So the study of fracture mechanics is required for knowledge of how the crack can grow in the material due to the effect of hydrogen or corrosion reaction because sometimes corrosion reaction can produce hydrogen atom. Due to the effect of hydrogen, material became brittle and crack growth happened due to dislocation emission or microvoid coalescence (MVC). From the study, it was found that sometimes combination of both dislocation emission and MVC may be responsible for crack growth and ultimately fracture happened in material and it will fail below its permissible limit.

The study of fracture mechanics is very well desirable for knowing the behaviour of crack growth and crack growth rate in a material due to the effect of hydrogen and simultaneously study of various crack growth mechanism is required. Hydrogen assisted cracking (HAC) describes the subcritical crack growth in material.

Hydrogen embrittlement can be classified in two different categories

(i) Subcritical crack growth above a threshold stress which is below the yield stress,
(ii) Fracture initiation at a stress between the yield stress and the UTS, in which the fracture can either initiate by microvoid coalescence (as for fracture in air) or by a more brittle fracture mode initiating at the surface.

Initially either defect is available in material or due to dislocation emission, a defect is generated. This defect is responsible for a crack generation. If the operating condition is varying such as loading condition and environmental condition (hydrogen environment) then these cracks can grow and when this crack become critical i.e. when the crack has grown in sufficient length (critical crack length) the propagation become unstable and material failure has occurs.

(i) Subcritical crack growth

If the hydrogen concentration is very high at the crack tip then absorption of hydrogen atom take place and HE began in material and the crack has grown. Basically, hydrogen reduced the fracture resistance of a particular material and supports the subcritical crack growth [88]. Crack growth occurred due to some chemical corrosion happened at crack tip. This crack growth is called as subcritical crack growth. This phenomenon is similar to stress corrosion cracking (SCC) [81,129]. If the rate of passive film formation is more than the dissolution process at crack tip then the crack growth happened slowly. This Crack growth is slow and continues (example in the case of fatigue) until the critical crack length is reached and when this condition has reached the stress intensity factor become critical i.e. critical stress intensity factor. Subcritical crack growth is only possible if the actual stress intensity factor (KI) is greater than the threshold stress intensity factor (KIH) [89]. The value of stress intensity factor is decreased as the crack propagates [95].

The value of critical crack length (ac) is given by Griffith which is

$$ac = \frac{2E\gamma}{\pi\sigma max^2} \tag{1}$$

where E is defined as the modulus of elasticity of the material and γ is the material surface energy. σmax is maximum stress occurring in material.

Griffith theory deals with the threshold for unsteady crack growth either it is ductile or brittle. It gives us criteria for crack growth whether it is possible or not.

There is a limiting stress above which subcritical crack growth is possible is called threshold stress. This threshold stress value is different for all material. Generally, this threshold stress is lower the yield stress of the material [33,82,83]. Its value for Mg–Al alloys which is stressed in distilled water is generally half of the yield stress [84–86]. Sometimes for higher stress rate, its value is one-third of fracture stress. The value of threshold stress is decreased if applied stress rate is decreased [87].

Loading rate has most pronounced effect in hydrogen diffusion. At high load hydrogen influence is minimum and at low or light load its effect is maximum because sufficient time has provided for the hydrogen atom to diffuse inside the material and subcritical crack can grow due to this effect. So it is concluded that a low loading rate is associated with subcritical crack growth [81,137].

(ii) Fracture initiation at a stress between the yield stress and the UTS

If a crack has grown in sufficient length and critical crack length has achieved which is provided by Griffith in equation (1) then fracture occurs below the ultimate tensile strength of the material. The material will fail in brittle fashion if hydrogen percentage or concentration is very high and it affects the mechanical properties. Similarly, ductile failure occurs if due to the effect of hydrogen no material property dilapidation takes place [81,90–94]. Sometime fracture initiation occurs due to micro-void coalescence and sometimes more brittle manner of fracture involves; that is intergranular or transgranular fracture occurs [89, 95].

Basic formula for fracture mechanics is $K = Y.\sigma\sqrt{\pi a}$ (2)

where K is the stress intensity factor, Y is the geometry factor, σ is the stress and a is the crack length. When crack become critical i.e. when the crack has grown to sufficient length and the propagation is now going to become unstable then the stress intensity factor becomes critical and called as critical stress intensity factor (Kc). Most common form of the Kc is called fracture toughness (KIC) and this fracture toughness is a very specific value of the stress intensity factor [96]. Stress intensity factor (K) is very effective for defining the stress value and its distribution around the flow.

For HE failure of a particular material, stress and hydrogen concentration are required. At high stress, a lot of microcracks have generated over the specimen and after some time these microcracks grew and converted into voids and finally, failure occurs due to coalescence of these voids [97]. By fractographic examination of the final fracture surface, it was seen that fracture comprises of mixed mode failure. At someplace there are a number of voids which combine to give micro-void dimple fracture and several areas where brittle (fish eye) features are present. These two micro-mechanisms are matched up with each other for final fracture. These fish eyes are surrounded by MVC. Hydrogen assisted microfracture occurs due to these two fracture mode. Decohesive hydrogen fracture (DHF) and dislocation emission are also responsible for fracture of material [81].

Mechanism

There are variety of mechanisms available which were responsible for HE of a material such as hydride formation, hydrogen enhanced decohesion mechanism (HEDE), hydrogen enhanced local plasticity model (HELP), adsorption-induced dislocation emission (AIDE) [98,99,117,125–128,135,136]. Sometime combination of these mechanisms is responsible for degradation and embrittlement of material. Still the actual mechanism which causes embrittlement occurs in a material is unclear and research will be going on to identify the main embrittlement causes of a material. Major mechanisms are discussed in detail below.

Hydrogen enhanced decohesion mechanism (HEDE)

This mechanism was first introduced in the year of 1959 by Troiano and a simplest one in all mechanism. It is based on the decreasing the cohesive strength of a material at the region of the crack tip by induction of a hydrogen atom. Interatomic bond strength between the atom has reduced due to hydrogen 1s electron comes in 3d cell of iron atom. Decohesion take place when the critical crack tip opening displacement (CTOP) is reached [5,7,27,99,125–128]. When hydrogen atoms are available all around the material and some type of stresses are acting on it then hydrogen atom is diffused inside a material and decreasing the interatomic strength or cohesive strength of a material at crack tip and cleavage like fracture occurs. Because of decreasing the cohesive strength of a material, the surface energy is reduced so that fracture stress is also reduced and fracture occurs below its permissible value. Measuring the cohesive force is the only limitation of this model [26].

Hydrogen enhanced local plasticity model (HELP)

This mechanism was first presented in the year of 1972 and widely accepted mechanism. In this model, the hydrogen accumulated near the crack tip decreases the resistance for dislocation motion so that mobility of dislocation increases and dislocations behave as carriers of plastic deformation in a metal lattice [6,26]. Local dislocation movement is possible at the low level of stress because of local drop in yield stress due to hydrogen. This means that fracture surface shows the local plastic deformation in embrittled material and slip bands at the crack tip [7]. However, it is also found that the there is no direct link between planned or proposed HELP model and actual embrittlement mechanism [7]. The various fracture modes, for example, intergranular, transgranular and quasi-cleavage might be seen depending on the hydrogen concentration, microstructure and stress intensity of crack tip. By fractography examination it was concluded that it has more local plastic deformation with reduction in macroscopic ductility. Activation energy for dislocation motion is reduced with the effect of hydrogen atom and activation area is also reduced with same effect [14,125,127,130,131,133,139]. This mechanism has been seen the various material such as a material having FCC, BCC, HCP structure, pure and alloys material [28].

Adsorption-induced dislocation emission (AIDE)

Basically, this mechanism is a combination of both HEDE and HELP. In this mechanism, solute hydrogen atoms are adsorbed at the surface in the region of stress concentration for example crack tips. Adsorption of hydrogen at crack tip results in weakening of interatomic bond or cohesive strength of material by HEDE mechanism and facilitating dislocation injection from a crack tip and then crack growth by slip and formation of microvoids by HELP mechanism [26,27]. In this mechanism nucleation and growth of crack has occurred due to decohesion and dislocation emission at the crack tip. Crack growth and simultaneously fracture occurred due to the combined effect of slip at the crack tip with micro-voids coalescence (MVC). High amount of adsorbed hydrogen on surface have been detected in Fe, Ni and Ti as proof to support AIDE mechanism [29,125,127,132,139].

Hydrogen Enhanced Macroscopic Ductility (HEMP)

It is also called hydrogen enhanced macroscopic plasticity model. Hydrogen has influenced the mechanical property of steel if it available in large volume (concentration) and whole specimen volume is interacted with the hydrogen then due to hydrogen diffusion and solid solution softening by hydrogen atom, yield strength of material was reduced. It was also evident that because of yielding, plasticization in whole specimen length (volume) had occurred so macroscopic enrichment of plasticity. This yield strength reduction due to effect of hydrogen is ascribed as Hydrogen Enhanced Macroscopic Ductility (HEMP). Dislocation motion on the area of high hydrogen percentage enables the macroscopic movement of hydrogen at yield stress [104,105,138].

Hydrogen changed micro-fracture mode (HAM)

Hydrogen changed the micro fracture mode of material so fracture mode was changed from ductile to brittle. Ductility of material was reduced due to hydrogen charging and mode of fracture at ultimate tensile strength has changed from cup and cone to brittle shear fracture mode. This is also due to high concentration of hydrogen at dislocation assists the shear fracture mode. This changing of microfracture mode due to effect of hydrogen is called as Hydrogen Assisted Micro-fracture (HAM) [81,104,105,138].

Decohesive hydrogen fracture (DHF)

It is basically a brittle fracture which is initiated with the effect of hydrogen or decohesion assists by hydrogen and causes final fracture of material or specimen. This fracture was initiated with the ductile fracture.

Mixed fracture (MF)

Sometime during the final fracture surface examination (by SEM or TEM) it was seen that the final fractography comprises of both brittle (fisheye) and ductile (MVC) features. So this it is concluded that the final fracture of material will take place through the combined effect of these two microfracture mechanisms. Fish eyes are originated at central area, generally at inclusion. The growth and fracture of these fish eyes take place in the radial direction until they come in contact (come close) to with ductile region or micro-void coalescence. These fish eyes are enclosed by MVC fracture. This type of fracture is called the mixed fracture mechanism where both fracture mode (hydrogen microfracture mechanism for fish-eyes and MVC) are competed with each other [81,104,139].

Hydrogen assisted micro void coalescence (HDMC)

Micro-void coalescence (MVC) is basically a ductile fracture mechanism. This MVC crack propagation happened in various stages such as void nucleation, void growth, void coalescence and crack extension and breaking of remains existing ligament by shear. Due to the effect of hydrogen, material possesses dislocation motion and localized plastic deformation take place. The crack growth happened in a zig-zag pattern by joining of voids present in crack propagation direction. MVC dimple produced due to the effect of hydrogen in sample but they possess poor ductility. Final fracture occurs due to shear stress so parabolic shallow shear dimple presented. Some brittle intergranular fracture is seen at the edge of the specimen due to the effect of hydrogen. This is called hydrogen assisted micro-void coalescence (HDMC). So it has seen that the hydrogen has an effect on ductile MVC process. Sometime fisheyes are also found in fracture propagation region which possess MVC shear dimple and these dimples are enclosed by MVC dimples [106,107,139].

Mechanisms that explain the advance of subcritical cracks by hydrogen

These are very essential and widely accepted mechanism for HE in steel and particular alloy. As seen from the literature that sometimes one or combination of these mechanisms are responsible for crack propagation and HE effect. So, by the effect of these mechanisms, subcritical crack propagation happened in material due to the effect of hydrogen. Macro and microscale fractography are very much desirable for complete knowledge of crack initiation and mechanism responsible for crack propagation and failure [102].

HEDE mechanism is based on decreasing the cohesive strength of material due to the effect of hydrogen at crack tip when the material is subjected to a particular value of stress. Hydrogen atom accumulated at the crack tip, decreasing the cohesive strength and subcritical crack growth occurs when tensile stress exceeds the interatomic strength of material at crack tip opening. After reducing the interatomic strength of material, hydrogen atom takes a new crack front position and further cracking propagated until the critical crack length get achieved [99,100]. So a large amount of stress at the crack tip with high hydrogen accumulation and hydrogen trapping causes crack growth.

HELP is very well accepted phenomenon for subcritical crack growth due to dislocation motion at crack tip because of accumulation of hydrogen atom. Due to this hydrogen atom, local plastic distortion happened (plastic flow affected) and this plastic flow is brittle in nature. Here dislocation happened so easily because of hydrogen atom accumulated at dislocation cores and reduced the elastic interaction or interface energy between moving dislocation. So stress for dislocation motion is decreased, enhance plasticity and crack growth happened very easily at lower applied stress. This propagation occurs along the grain boundary and its matrix [3,101]. Crack is generated and propagated as a result of dislocation motion along the grain boundaries and sometimes crack generated in the oxide layer at the grain boundary.

Crack growth facilitated by AIDE mechanism is based on the adsorption of hydrogen at the crack tip. This hydrogen weakens the metal atom bond strength and simultaneously facilitating the emission of dislocation from crack front. In this mechanism crack is nucleated and growth happened due to the effect of hydrogen. At the time of loading plastic deformation happened at the crack tip and micro-void formation occurs. The combined effect of formation and linking of these voids with slip bands of dislocation emission maintain a sharp crack tip and crack growth happened [27,98].

Various types of fracture mode intergranular, transgranular, micro-void coalescence or dimple rupture depend on amount of hydrogen concentration, microstructure of material due to effect of hydrogen and stress intensity factor at crack tip [103,139]. Transgranular fracture has seen in many places because crack propagation take place at slip step due to dislocation emission.

Hydrogen charging characteristic

Hydrogen charging plays an important role during the experimental investigation of the amount of hydrogen available in material. Generally, hydrogen charging of a sample (material) has done by electrochemical (Cathodic Charging) or by high-pressure hydrogen gas charging. These charging techniques cause hydrogen diffused inside the material degrade the material properties and causes hydrogen embrittlement take place in material [108,134,138]. Sometimes these hydrogen charging introduce or generate hydrogen trap in material and in those traps sides, hydrogen atoms accumulated. These traps are either reversible or irreversible. At the time of loading condition, reversible traps are supplied hydrogen to the highly stressed region in the lattice which causes HE type catastrophic failure occurred [100,108–112,126]. Hydrogen trapping contribute toward the fact from the study, it has been found that high negative applied potential causes more hydrogen atom to evolve at the surface so hydrogen diffusion takes place. Also, at high negative applied potential, high peak is generated and this peak comes at a lower temperature. In case of high gaseous hydrogen pressure the same thing happened as hydrogen pressure increases, the intensity of peak and hydrogen concentration also increases and peak moved to lower temperature. Thermal desorption spectroscopy (TDS) was used for evaluating the hydrogen content inside the material during hydrogen charging or in gaseous condition [121].

Perez Escobar et al. were also concluded that in polished specimen more hydrogen adsorbed as compared to simple ground specimen [113]. Venezuela et al. were found that in Na2SO4 solution maximum peak height was optioned as compared to NAOH solution [108]. Hydrogen fugacity of the electrochemical charging is equal to room temperature gaseous hydrogen charging hydrogen fugacity as long as the hydrogen concentration generated by cathodic charging is equivalent to the room temperature gaseous hydrogen charging [105,115,138].

Sieverts' Law proposed that concentration (quantity) of dissolved hydrogen in steel is proportional to partial pressure which is frequently expressed as fugacity [81,105,138].

$$\mathrm{CH} = \mathrm{S(fH)1/2} \tag{3}$$

where CH is concentration of hydrogen, s is the solubility constant which depend on the type of steel and temperature and f H2 is the hydrogen fugacity.

It was also found the relationship between the charging potential and hydrogen fugacity. As charging potential increases the hydrogen fugacity also increases. So because of that, we can predict the hydrogen acceptance during the process and if due to hydrogen concentration mechanical properties get affected then there will be the possibility of HE [99,122,123].

Equivalent charging pressure at the time electrochemical or cathodic charging is more than the gaseous high-pressure hydrogen charging. Microstructure also plays an important role in the hydrogen trap as compared to the composition of the material. So, the influence of microstructure is more in steel [114].

Techniques and tools to measure HE

Linearly increasing stress test (LIST)

This is a mechanical testing method for determining the HE in materials. In the earlier stage, two techniques are widely used for knowing the effect HE in materials one is constant load test and another one is constant extension test. In both test firstly, the threshold limit of stress is determined. It is defined as a stress below which no failure has occurred [31]. One difficulty has observed while applying these testing was that component does not fail in the specific or certain amount of time and it will take a long time for fracture or failure.

A new technique called constant extension rate test (CERT) was introduced. This testing was also called slow strain rate testing (SSRT) [32]. In this testing, a constant increasing elongation has applied with time until the specimen will fail or fracture occurs. One disadvantage of this testing was that after reaching the threshold limit (threshold stress) sample extend longer and it will take more time to fail. This is cumbersome technique.

To overcome the above difficulty, a new novel technique called linearly increasing stress testing was introduced. This testing technique is somehow similar to CERT but has advantages over it. In this testing, a sample was loaded and the applied stress was gradually increased until the failure occurs. By the movement of weight, a load is applied and rate of motion of it is controlled by a motor. Fig. 2 represents the schematic of LIST technique. CERT and LIST both are different technique because the CERT is basically a displacement control and LIST is load control [33]. From the literature, it was also found that CERT and LIST both give the same value of threshold stress but in LIST after reaching the threshold stress the specimen will fail and a test was completed [117].

TDS

Temperature desorption spectroscopy (TDS) is also called as temperature programmed desorption (TPD) is a most acceptable technique in the study of HE and hydrogen induced failure [23,34,35]. From the literature, it is found that there is a number of techniques are available to know and measure the amount of diffusible hydrogen in steel and the function of hydrogen in failure. The mobility of hydrogen also plays an important role in HE [36]. TDS has an ability to qualify and quantifies the diffusible hydrogen.

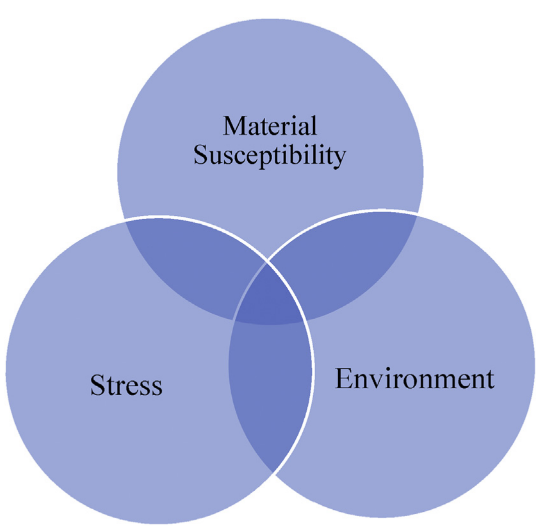

Fig. 1 – Factors responsible for hydrogen embrittlement [25].

Amount of desorbed hydrogen can be measured by TDS technique by controlled and limited heating. Traps are found in steel and these traps are responsible for hydrogen accumulation. When heat is supply to steel then hydrogen absorb the thermal energy and released when a critical level of absorbed energy is reached which is equal to desorption activation energy. So, desorption temperature is that temperature on which hydrogen atoms are released. The quantity of desorbed hydrogen is measured by quadrupole mass spectroscopy [37,108].

Fig. 3 shows the schematic of TDS. It has a unique quality that it has high sensitivity and can measure the small amount of desorbed hydrogen more accurately than other techniques (0.1 mg/kg) [14,38]. If TDS technique has used with other tests then it can give high HE detection and a better understanding of HE mechanism.

Hydrogen permeation test

This permeation testing is also used to measure the diffusible hydrogen in steel and the simplest technique for measurement. If diffusible hydrogen quantity is known then HE susceptibility of steel can be determined and accessed. For successful implementation of this permeation test in steel, it has used with the combination of other testing technique [42].

This permeation test is basically a double cell set up in which one chamber is entry cell also called a charging cell and the another is oxidation cell (exit cell). These two chambers are separated by a steel membrane [40,41]. The electrochemical process has used to hydrogen charging. This hydrogen is firstly introduced in charging the cell and then goes to oxidation cell with the help of membrane.

Microstructural analysis

Microstructural analysis plays an important role in the evaluation of HE susceptibility of a material and alloy. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are widely used analytical technique for knowing the internal microstructure of a material and effect of hydrogen in internal microstructure and characterization of material. SEM is most simple in use, a versatile and preferred technique for it. Also, preparation of the sample is easier as compared to others technique [43]. SEM also provide a three-dimensional image. This is a very essential thing when the study of fracture structures is required such as microvoids, dimples and fisheyes [44]. The only drawback of this process is its lesser magnification and low resolution as compared to other microscope.

TEM is most powerful and preferred technique. TEM works on high magnification and resolution to overcome the difficulty of SEM. TEM is capable of working on a million times of magnification and nanometer resolution [45]. Morphology and topographical information can be obtained with the use of TEM. Crystallographic information also got from this microscope. However, the one difficulty has arrived when dealing with TEM is the preparation of sample was required. For TEM imaging the sample must be electron transparent and if TEM is applied for microstructure analysis then the thickness of sample must be less than 100 nm.

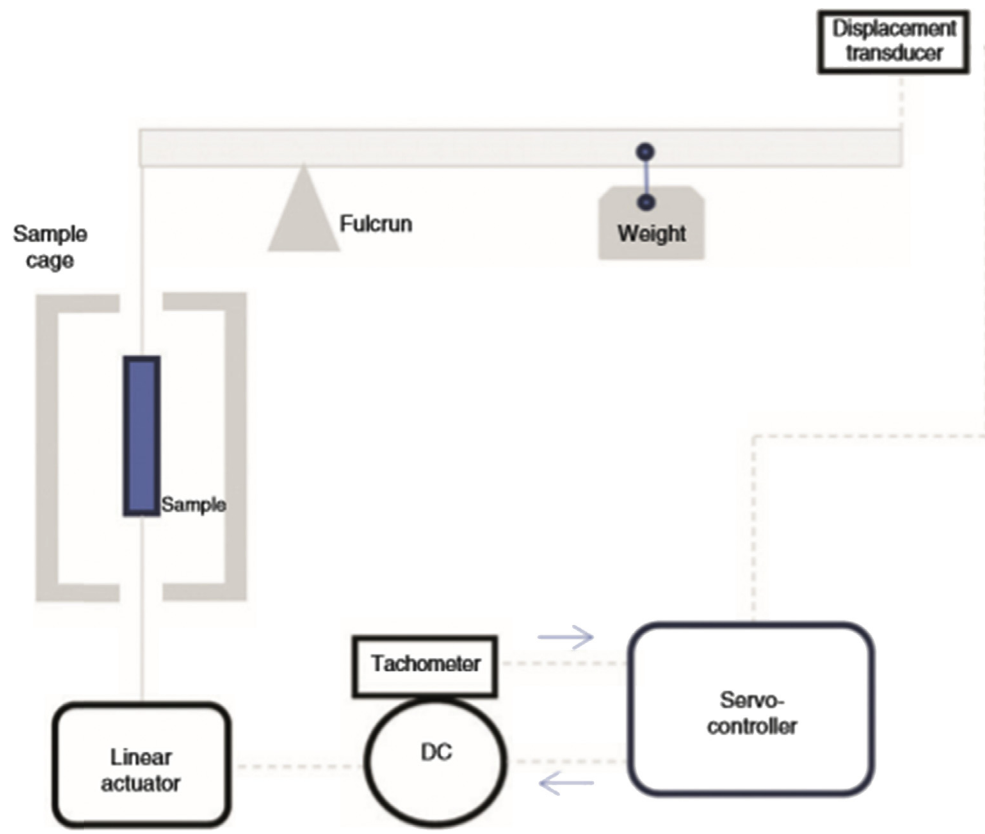

Fig. 2 – Schematic of LIST technique [14,30].

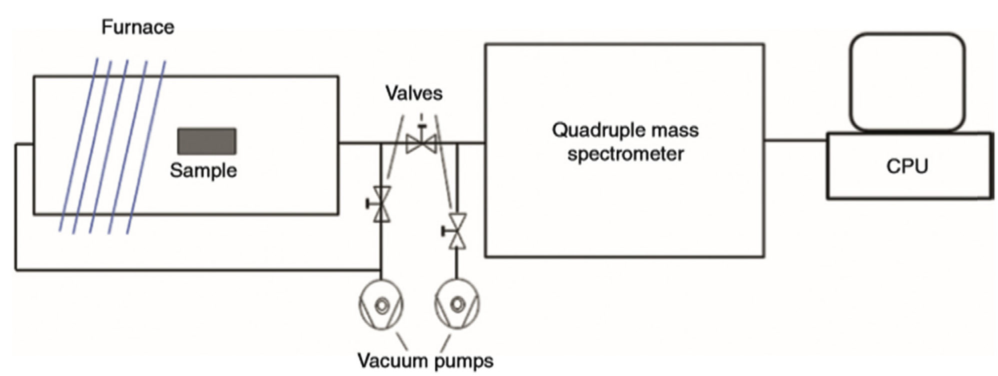

Fig. 3 – Schematic of TDS testing apparatus [14,37].

Hydrogen microprint technique (HMT)

This technique has used to know the path of diffusible hydrogen in metal. If these paths are known then specific microstructure of paths can be identified and effect of hydrogen must be known. This HMT technique is very simple and unique and available with better accuracy and high resolution [46,47]. To know the hydrogen distribution on stress field for example in notched steel and deformed steel, HMT technique was applied. HMT technique can be applied to the different material such as in low carbon steel, high strength steel and austenitic stainless steel [48–50].

During the process, the surface of the hydrogen-charged material is covered with the thin layer of AgBr gel. When hydrogen is coming out from the metal, it reacts with a silver salt. After the reaction, metallic form of silver ion has created and leaves a track where hydrogen contact happened. Silver particles are present on this location and the excess unreacted gel is take off from that place. SEM examination of the sample is carried out and during the examination exit of hydrogen occurs where the silver is present [51].

HE prevention

For prevention of high strength materials from HE, the actual source of hydrogen and mechanism which is responsible for it must be known. Selection of a suitable material plays an

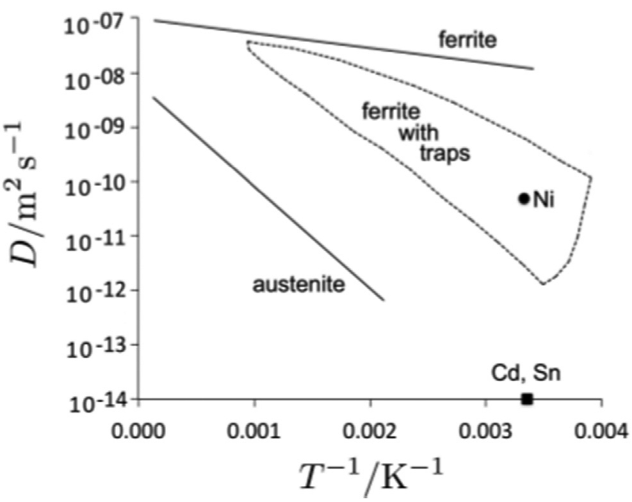

Fig. 4 – Hydrogen diffusion coefficient in ferrite and austenite. Also showing Cd and Sn lower hydrogen diffusion coefficient [52,61].

important role when it works on hydrogen environment. The material should be low strength and high resistance alloy for HE. Design of material must be appropriate. Notches, sharp and regular variation must be avoided and residual stresses are removed before the processing [53,54]. The baking operation has performed in the material to detach hydrogen which is absorbed and can cause damage or failure occurs. Baking is basically a heat treatment process and baking temperature will depend on the material on which this operation has performed [55]. Pickling operation has performed to remove some scale and oxide products from material with the use of acidic solution. This acid is responsible for hydrogen diffusion so to reduce that some mechanical means such as sand, grit blasting and vapor blasting will be performed.

HE prevention can also be done by adding some metal alloys to the base material and by applying protective coating above it. Some coating techniques are vacuum deposited coating, organic coating and some mechanical plating. Use of effective inhibitors also plays an important role. Titanium content can reduce the HE susceptibility of a hot stamped boron steel by forming titanium carbide in the material if titanium is available in a large quantity [21]. Some author suggests that the aluminium alloying also help to reduce the HE effect. Niobium and graphene coatings can also prevent material from HE [19,39]. Kim and Kim were found that the reduced graphene oxide layer provides sufficient diffusion barrier for hydrogen and stable after hydrogen charging. It was achieved due to diffusion length enhanced and C-h bond formed during hydrogen charging atmosphere [13,19]. Cadmium is used as a protective barrier against hydrogen diffusion in steel. The diffusion coefficient of hydrogen in cadmium and tin are lower than the ferrite (see Fig. 4).

Nickel when plated on steel act as a diffusion barrier for hydrogen. Various types of coatings such as TiC, TiN, TiO2, alumina, BN, Cr2O3 and WC have used a protective barrier against the permeation of hydrogen [52]. J. Cwiek said that Pt, Cu, Cd, Ag, Al and Au coatings can reduce the hydrogen diffusion inside the steel. He also said that oxides and nitrides surface layer to act as a barrier and lower the hydrogen diffusion. From the literature, it is also concluded that the corrosion product act as a barrier for hydrogen entry on the surface of material [53]. Actual working or functioning of the coating depends on the structural reliability and defects in coating. Service condition also plays an important role. If localized stress occurs in the coating then it may be possible that they wear out and hydrogen can diffuse on the base metal [56]. The diffusion layer of nitrogen and carbon are very effective to reduce the crack propagation rate although the cannot purely eliminate hydrogen environmental embrittlement. If proper adhesion, a thickness of the coating and defect-free coating are provided then HE possibilities are reduced [20].

Some elements cause hydrogen can enter on the structure of material if they work on hydrogen environment. These elements are Te, Sn, S, Hg, Se, Pb, As and Bi. They help hydrogen for entry and if they already available in material then they separate the grain boundary so strictly avoided [53]. It is found that there is a critical level of diffusible hydrogen concentration denoted by H_c below which no failure occurs. This value can have determined by some experiment. If diffusible hydrogen concentration is less than the H_c than no delayed fracture or failure can occur during the service or working condition of metal and alloy [52,78–80]. So HE causes a delayed failure in steel and alloys.

HE prevention by cadmium and nickel plating

Steels whose strength high are electroplated with zinc and cadmium because they provide corrosion protection from the environment. But this plating process also responsible for origin and introduction of hydrogen in steel. So to reduce this the heat treatment of that plated steel has done at a temperature of 190–230 °C and time duration for that is 10–24 h. Because of this, the diffusible hydrogen comes out from the material. One assumption has made during the process is that heat treatment does not change the mechanical properties of steel and alloys [52,57,58].

Hillier work on alloying of zinc with nickel and found that penetration of hydrogen in steel reduced by application of this. It was found that nickel first deposited on material forms a layer of it over the material and act as a barrier for hydrogen [58]. The diffusivity coefficient of hydrogen in nickel is very low and it is 5×10^{-11} m^2/s [59]. Sometimes hydrogen can go through the coating and it depends on the diffusivity of hydrogen in coating. Also, in some cases adsorbed hydrogen combine at a place to form a molecule and before entering inside the coating they getaway forming bubbles. Cadmium is responsible for recombination of hydrogen in a coating and reducing the entry of hydrogen coating [52,60].

Cadmium and titanium plating

This plating process was firstly introduced in 1960 [62]. Initially, cadmium cyanide solution was used with the titanium compound which is soluble in cyanide. The titanium contains in the deposit was 0.1–0.5% if the operation has done accurately. This method has used for coating threaded rods, gear actuation cylinder and connected shafts working on high stress environment [63]. The noncyanide solution was also obtained in which titanium contain was 0.1–0.7%. Titanium compound is steady and constant on noncyanide solution. It is concluded that noncyanide solution works better than the cyanide solution and this process has been used for putting a protective coating on high-quality instrumental steel, high strength structure steel and spring wire [64].

Other methods for preventing HE

Ion plating practically known as physical vapor deposition eliminates the hydrogen embrittlement problem. Because of this process has performed in a vacuum so the probability of embrittlement is reduced. Ion plated Al coating has been used in aircraft applications for more than 20 years. It is found that this ion aluminium coating process protects the surface better than other electroplating processes [65].

Some material such as Pt, Cu and electroless nickel is used as a protective layer in which a thin layer is introduced above the surface and this can reduce the penetration of hydrogen in steel and other alloys [66]. Au, Sn and some Sn–Pb alloys coatings are also very effective and act as a barrier for hydrogen permeation [67–70]. If Pt coating is used then protective layer thickness of 0.0015 μm is sufficient for reducing hydrogen penetration in iron. It was also found that for decreasing the hydrogen diffusion in iron, Cu is more effective than Ni [66]. Lead coatings are viable in counteracting hydrogen cracking on an assortment of steels working in a distinct environment [71–73].

Hydrogen environmental embrittlement prevention of electrodeposited nickel was done by coat the nickel with copper or gold. It was found that by application of both coatings either copper or gold, the ductility of the electrodeposited nickel is remaining same [68]. Alumina is creating a barrier for hydrogen diffusion. The plasma technique is used to deposit alumina over the steel and a layer of crystalline α-alumina having a thickness of 1 μm is deposited [74]. When this coating is worked at a high temperature such as 800 °C, it found that there is no effect of temperature on it and coating still working. During this process, enriched alumina surface layer has created over the steel. So, it can be concluded that this alumina has a tendency to reduce the hydrogen permeation in steel and reduced permeation rate having an order of 3–4 magnitude as compared to bare steel [52,75].

The amorphous surface layer has a unique quality in that it provides the sufficient diffusion barrier. A new phosphorus ion implantation technique works on this principle [76]. For hydrogen, it is very difficult to goes through the disordered structure and penetrates from it to reach the base material. Santos found that the hydrogen diffusion from this amorphous iron base alloy is slow and lesser than the ferrite steel [77].

Bhadeshia found that sometimes traps can reduce the susceptibility of hydrogen diffusion in steel. Due to this trap the amount of hydrogen on that region must be high means hydrogen concentration increased on that particular location but that hydrogen is not harmful [52].

Case study

Abhay et al. explained the effect of hydrogen embrittlement on cadmium plated fasteners. They found that fasteners are failed from the threaded region when torque is applied on it. The crack was propagated in the direction parallel to fasteners axis and perpendicular to fractured or damaged surface. When this failed region was examined then it found that the failed fasteners comprised of two regions, the first is outer black region and second is centre bright region. When the SEM examination of the fractured surface has been carried out then it was found that black region was spread all around the periphery has an intergranular structure while the bright region at the centre available with dimple structure. So, the structure might be changed from intergranular to dimple mode of fracture. Also, from microstructure examination, it was concluded that the initially de-bonding in material took place and this debonding took place at the interface of two regions. During crack propagation along grain boundary, burst opening was found in the outer region and this burst opening is due to accumulation of hydrogen at discontinuity and voids, create a high pressure so voids growth is possible and burst opening take place. Initially crack propagation is possible due to decohesion model and ductile tearing is found in an outer region of a fractured bolt. Dislocation movement is responsible for plastic flow at the crack tip. The possible source of hydrogen trapment during this study are furnace temperature at the time of spheroidization treatment, residual stress at the time of cadmium plating and baking operation does not performed sufficiently [24].

Djukic et al. worked on HE of boiler tubes of power plants. During the study, the number of damaged samples are cut from the boilers tube, failure analysis of damaged samples are done and then the mechanism which are responsible for HE failure on boiler tube are identified. Tensile, hardness and impact test are performed to know the behaviour of HE in boiler tubes. After that microstructure examination has carried out. From the investigation it was found that high temperature hydrogen attack (HTHA) is mainly responsible for boiler tube failure. HEDE and HELP both mechanism is responsible for HE of boiler tube in power plant. It depends on the local concentration of hydrogen in examined steel. Ductility and impact strength was reduced due to effect of hydrogen in steel and both mechanism have some contribution towards it. Due to HTHA mechanism, the window type fracture occurred on boiler tube surface. The HE phenomena was evaluated by HE index. Higher the HE index more chances of material to be susceptible for HE. A mixed mode fracture called quasi cleavage fracture has arrived during the examination of boiler tube due to combination of HEDE and HELP mechanism. Due to HEDE mechanism brittle fracture arrived and by HELP mechanism ductile mode microvoid coalescence found on the surface. It was concluded that the HTHA with combination of HEDE and HELP mechanism is responsible for HE in steel. They provide the correlation between the degradation in mechanical properties of steel and SEM fractography examination of damaged surface when combination of both HEDE and HELP HE mechanism simultaneously acting [22].

Conclusion

HE is now widely known phenomena is steel and other material. This paper deals with material which are susceptible for Hydrogen penetration or diffusion, causes and mechanism which are responsible for degradation in mechanical properties and hydrogen related failure such as HE. Simultaneously prevention activities to eliminate or reduce the hydrogen penetration in the material are discussed. These prevention methods can create a barrier for hydrogen diffusion and reduce the chances of HE. The conclusion of this work is written as follows;

1. Diffusible hydrogen is responsible for hydrogen related failure such as hydrogen embrittlement in materials.
2. If the diffusible hydrogen content is below than the critical level of hydrogen which is known as critical hydrogen concentration (H_c), then the chances of HE can be neglected. If diffusible hydrogen content is more than the H_c then HE happened in material and seriousness of HE is depend on the amount of hydrogen concentration.
3. To reduce this HE phenomena, selection of a material is very much important. Also baking operation performed to restore the material in original condition.
4. HE prevention can be done by adding some alloys such as titanium and aluminium with base material.
5. It is also concluded that some diffusion layer such as oxide, nitrogen and carbon can create a barrier for hydrogen penetration in steels so hydrogen cannot diffuse in material and susceptibility of HE is reduced.
6. Plating techniques such as zinc and nickel plating are very effective to create a layer over material and work as a barrier for hydrogen diffusion.
7. Coating techniques are reduced the susceptibility of hydrogen in material. Some coating such as graphene and niobium are very effective to prevent material from HE. If Pt coating is used then protective layer thickness of 0.0015 μm is sufficient for reducing hydrogen penetration in iron.
8. Traps and corrosion product also acts as barrier for hydrogen entry on surface of material. Amorphous structure material also reduces the hydrogen penetration in material.

The correct mechanism which is responsible for HE in the material is still unclear and doubtful. Sometime combination of mechanisms is responsible for the hydrogen-related failure. Also, the correlation between the laboratory result to the actual working condition is unclear because the laboratory experiment is done under a control condition and when material works on the real situation (hydrogen environment) then the actual condition is different from the others. So still some work must be done on it in future.

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