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Review Article

A focused review of the hydrogen storage tank embrittlement mechanism process

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HIGHLIGHTS

- Hydrogen storage tank is critical in renewable energy.
- Hydrogen tank performance can be enhanced by appropriate material selection.
- Microstructural modification reduces the hydrogen embrittlement.
- Embrittlement degradation mechanism affects hydrogen tank storage.

ARTICLE INFO

Article history:
Received 22 May 2022
Received in revised form
9 October 2022
Accepted 21 December 2022
Available online 7 January 2023

ABSTRACT

Hydrogen embrittlement is a widely known phenomenon in high-strength and storage materials. Hydrogen embrittlement is responsible for subcritical crack growth in material, fracture initiation, subsequent loss in mechanical properties, and catastrophic failure. Hydrogen is induced in the material during an electrochemical reaction between the hydrogen, storage materials, and high-pressure gaseous hydrogen environment. Various mechanisms which are responsible for crack development, growth, and fracture have been deliberated and reported. However, the fundamental mechanism of hydrogen embrittlement

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https://doi.org/10.1016/j.ijhydene.2022.12.252

Keywords:
Degradation
Hydrogen embrittlement
Storage tank
Hydrogen diffusion
Materials

remains unclear. Several techniques such as linearly increasing stress test techniques (LIST), constant extension rate test (CERT) and slow strain rate testing (SSRT), thermal desorption spectroscopy (TDS), permeation testing (PT), scanning electron microscopy (SEM), and transmission electron microscopy (TEM) have been utilized to determine the amount of hydrogen diffused and available in the hydrogen storage material. The review intends to categorize and provide a clear understanding of the degradation mechanism that occurs during hydrogen embrittlement. The improvement in mitigating the hydrogen embrittlement degradation as a function of modifying the structure and surfaces of the material is established. Prospects for addressing hydrogen embrittlement degradation through further experimental and numerical research are suggested. Lastly, this paper through recommendation endeavors to prevent hydrogen storage tank degradation and reduces high costs associated with the replacement of the component in renewable energy applications.

Contents

Introduction

Hydrogen has incredible qualities and has long been promoted as a fuel for a carbon-free future. Today, as global energy demands are increasing order, hydrogen is expected to play a critical role in future energy infrastructure. Hydrogen is also being used as an energy carrier in the hopes of weaning our current society away from carbon-emitting fossil fuels and mitigating their effects on the environment [1,2]. Hydrogen energy systems appear to be one of the most effective solutions and have the potential to play a significant role in improving the environment and ensuring sustainability. Hydrogen has also shown promise as a vehicle fuel, electricity storage via fuel cells, and a variety of other useful properties in the chemical and metallurgical industries [3,4]. The variety of potential supply sources is one of the key reasons why hydrogen is such an appealing energy carrier [5]. Large central plants, medium-scale semi-central plants, or small distributed units located at or very near the point of use, such as refueling stations or stationary power sites, can all make use of the produced hydrogen [6]. As a result, any method of storage and material of the hydrogen storage tank can significantly influence the cost of hydrogen fuel [7]. However, hydrogen can diffuse in many metals and compounds causing undesired outcomes [8,9]. Furthermore, its interaction with the crystal lattice of materials is a cause for concern in iron, steel, nickel, titanium, vanadium, zirconium, silicon, and other metals used in the design of hydrogen storage and other engineering applications [10]. Hydrogen embrittlement, Stress corrosion cracking, hydrogen-induced cracking, hydride cracking, and other negative effects caused by hydrogen in materials are commonly observed in materials leading to catastrophic fracture, malevolent repercussions, and frequently resulting in destructive cracks. Because catastrophic failure is unacceptable in engineering, hydrogen's negative consequences must be minimized to the greatest extent possible.

Hydrogen embrittlement is the most well-known effect of hydrogen in high-strength materials, such as steel [11]. It is referred to as the process by which the introduction of a hydrogen atom when working in a hydrogen environment can dramatically lower a material's strength. A substance simultaneously loses ductility and becomes brittle. Due to the influence of hydrogen embrittlement, mechanical qualities such as tensile strength and fatigue strength are decreased. Many metallic materials, such as steel, super alloys, and aluminum alloys, are affected by hydrogen embrittlement. The hydrogen-enhanced decohesion (HEDE) and adsorption-induced dislocation emission (AIDE) have been proposed as some of the mechanisms that cause hydrogen embrittlement by several authors [8,10,12]. However, the core mechanism responsible for hydrogen embrittlement is still unsatisfactory, and there is still considerable debate about it [11]. Johnson [13] focusing on the ramifications of iron and steel, launched an investigation into the effects of hydrogen on mechanical qualities around the end of the nineteenth century. The deleterious effects of hydrogen on several materials were demonstrated [13]. Because of the hydrogen embrittlement's complexity, it is important to understand how hydrogen interacts with another metal surface, enters the metal, travels through the crystal lattice, and interacts with crystal defects, precipitates, inclusions, interfaces, and more, as well as its effect on material properties modification. These defects, which include vacancies, dislocations, grain boundaries, solutes, precipitates, inclusions, and interfaces, act as traps for hydrogen atoms [14]. When the hydrogen escapes from reversible traps, it can wreak havoc on previously cracked materials resulting in material failure.

High-pressure hydrogen tanks are used in hydrogen transportation, storage, and fuel cell vehicles (FCVs). Due to the low density of hydrogen, the storage of hydrogen at reasonable energy densities poses a technical and economic challenge. The embrittlement issues are prevailing in the hydrogen storage structures and materials that interact with hydrogen [15]. To address the challenges of the hydrogen embrittlement on the storage system, surface and structural modifications of the storage material have been performed and several suggestions and results proposed [16,17]. Despite the successful results presented so far, degradation of the hydrogen storage tank is still being reported [18]. In combination with the load from the hydrogen storage system, the hydrogen will diffuse into the metal lattice, causing cracks that will eventually lead to fractures and the failure of the hydrogen storage tank.

To understand the hydrogen embrittlement degradation mechanism of the storage tank, it is worthwhile examining the structural and chemical composition of the hydrogen. Through the process, the interaction between hydrogen and storage materials can be appreciated and the degradation mechanism comprehended. Current research is predominantly focused on researching hydrogen embrittlement degradation mechanisms to address the long-term performance needs of hydrogen storage tanks [16]. There is likewise another need to evaluate the material development that can withstand the hydrogen embrittlement mechanisms and develop more durable and stable alloy materials for hydrogen storage applications. This study attempts to bridge the gap and provide a comprehensive review of the hydrogen embrittlement mechanism and the attempt made in addressing the challenges imposed by the degradation process. The knowledge gained from this review will help the material designers and the entire renewable energy society in selecting appropriate materials for hydrogen storage tanks and other applications.

Basic hydrogen description

Hydrogen is the most abundant element in the universe and more than 90% of all atoms are hydrogen. Hydrogen is the simplest atom consisting of one proton and one electron only. Apart from this ordinary isotope called protium, a small fraction of hydrogen atoms exist as deuterium and an even smaller fraction as unstable tritium [19]. If the spins of two hydrogen protons are parallel, the molecule is called ortho-hydrogen and if the spins are opposed, the molecule is called para-hydrogen. Ortho-hydrogen and para-hydrogen have slightly different properties [20]. At standard conditions, molecular hydrogen is a mixture of about 75 vol% ortho-hydrogen and 25 vol% para-hydrogen, which is called normal hydrogen. With a reduction in temperature, the content of para-hydrogen increases and reaches 100 vol% below 200 °C [20]. Para-hydrogen has a lower energy level than ortho-hydrogen and during the liquefaction of hydrogen, additional energy has to be dissipated to convert ortho-hydrogen to para-hydrogen. Due to its single valence electron, hydrogen is very reactive and usually combines to yield the molecule H_2 as shown in Fig. 1.

Hydrogen has an ambivalent behavior toward other elements, occurring as an anion (H^-) or cation (H^+) in ionic compounds, forming covalent bonds with carbon [21]. Hydrogen behaves as metal to form alloys or intermetallic compounds at ambient temperature. Fig. 2 shows the phase diagram of the hydrogen.

According to Fig. 2, at low temperatures, hydrogen is solid with a density of 70.6 kg m^3, and a gas at higher temperatures. Furthermore, hydrogen is a liquid in a small zone between the triple and critical points with a density of 70.8 kg/m^3 at 253 °C. Studies have shown that hydrogen is rarely found in its pure

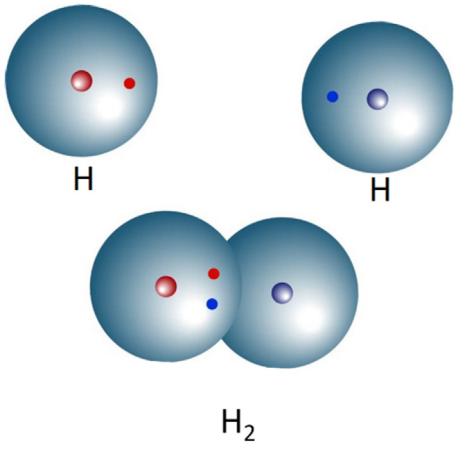

Fig. 1 – Schematic diagram of hydrogen and its molecule.

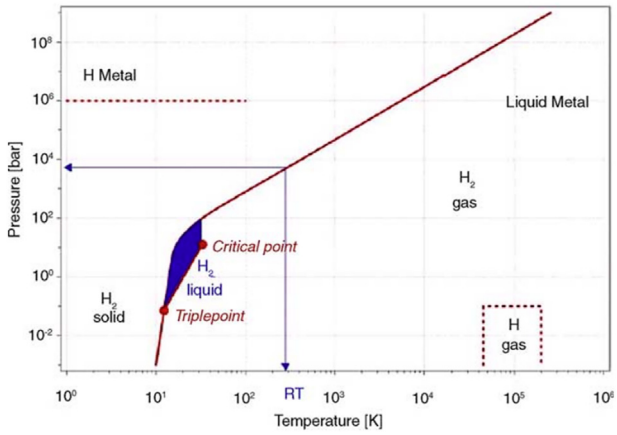

Fig. 2 – Primitive phase diagram for hydrogen [22].

form, but usually in a wide variety of inorganic and organic chemical compounds, the most common being water H_2O [23]. However, hydrogen forms chemical compounds with nearly all other elements [24].

Hydrogen storage and material requirement

For the storage of pure hydrogen, hydrogen has first to be separated from its compounds. Several technologies exist for the production of hydrogen from different sources [25–27]. Hydrogen is typically stored and transported in two forms: as compressed hydrogen gas or as a cryogenic liquid. The most common way to store hydrogen is in metal or composite cylinders/tanks of different sizes and capacities. However, due to the small size of its molecules, hydrogen is prone to leak easily through some materials, cracks, or poor joints of the storage tanks, as opposed to other common gases at equivalent pressures [24].

For compressed storage of hydrogen, the gas is usually compressed to pressures between 200 and 350 bar though, more recently, storage pressures of 700 bar and even higher have been under trial [28]. Such enormous pressures require consideration of questions regarding material choice, component dimensioning, and safety. Hydrogen tends to adsorb and dissociate at material surfaces; the atomic hydrogen then diffuses into the material and causes embrittlement and diffusion [16].

Hydrogen is generally non-corrosive and does not react with the materials used for storage containers, at a certain temperature and pressure conditions [29]. The major concerns related to hydrogen storage materials are the large amount of energy needed for the compression; the stress on the containers' materials caused by repeated cycling from low to high pressures; and the high weights and additional costs to design such vessels [28]. Other issues such as hydrogen permeation and embrittlement should be considered as well. Thus, the containers used to store hydrogen must be made of robust materials and must withstand high pressures without a loss of containment. The designed hydrogen storage vessels (as well as the materials they are made of) should comply with the requirements. Studies have shown that the pressure, at which hydrogen is stored, mainly affects the thickness of the storage container walls, the size/weight of the containers, the choice of materials, and the costs [30,31].

Usually, three types of materials are used for the design and manufacturing of hydrogen storage tanks: metals, polymers, and carbon fibers [32]. Metals should neither allow hydrogen permeation nor be subjected to hydrogen embrittlement, especially if they are to undergo extensive pressure/temperature cycling during their lifetime. The hydrogen tanks are designed for a maximum working pressure, with the minimum wall thickness dictated by the metal's tensile strength [33]. Apart from the container itself, valves for reducing the pressure, pipelines, and sensors to control pressure, temperature, and tightness are also affected by hydrogen [34]. Although the design of hydrogen tanks has been improved in recent years, particularly through the application of lightweight materials such as polymers and aluminum, the issues of large volumes and heavy weights remain. For example, the mass of hydrogen stored in a metal cylinder is only about 1% of its total mass. New lightweight composite cylinders have been developed that can withstand pressures up to 80 MPa so that hydrogen can reach a volumetric density of 36 kg/m^3, approximately half as much as in its liquid form at normal boiling point [33]. The ideal material for hydrogen storage should have a very high tensile strength, and a low density, and does not react with hydrogen or allow hydrogen to diffuse into it. As a matter of precaution, most pressure cylinders to date have used austenitic stainless steel (e.g. AISI 316 and 304, AISI 316L and 304L) above 300 °C to avoid carbon grain-boundary segregation [35,36].

Hydrogen storage vessels classification

Types of hydrogen storage vessels due to several unique hydrogen properties should be compatible with the materials the walls of the storage tanks are made of [37]. As a result, four types of vessels have been developed and used for hydrogen transportation and storage as shown in Fig. 3.

As shown in Fig. 3, type I tanks are seamless containers made of steel or aluminum and are very heavy, with thick walls. Type I tanks are designed for pressures not higher than 25 MPa and can be considered a relatively cheap storage option for some stationary applications [38]. The type I containers offer good properties concerning safety and strength, but at a high weight. The steel type 1 tanks are available with net volumes from 2.5 to 50 L. To reduce the weight, steel

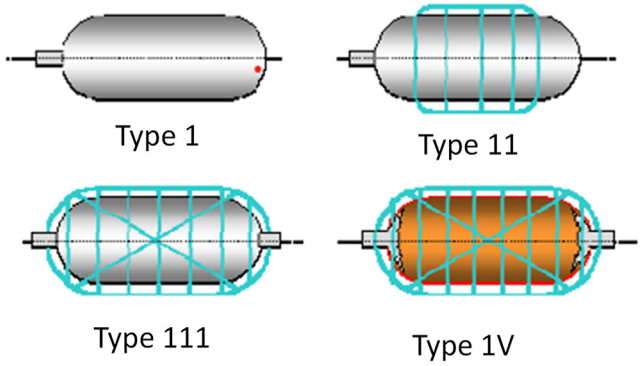

Fig. 3 – Cylinder types for hydrogen storage tanks [32].

containers have been replaced by composite containers and a thin inner liner of metal ensures gas tightness.

Type II tanks have seamless metallic tanks hoop-wrapped with fiber resin. They are also very heavy and can withstand pressures up to 45–80 MPa. These tanks can be used as high-pressure buffers at hydrogen refueling stations. Their cost is competitive due to a relatively low number of fibers used. However, both type I and type II vessels are not suitable for automotive applications due to their heavy weights and large sizes [39]. Types III and IV tanks are usually lighter and have thinner walls compared to types I and II containers. Type III tanks have seamless or welded aluminum liners fully wrapped with fiber resin composite and the materials used are less affected by hydrogen embrittlement.

Type IV tanks are made of non-metallic liners in a fiber/epoxy matrix as shown in Fig. 3. The fiber wrapping around the polymeric liner provides the required level of strength to contain pressurized hydrogen, while the liner mainly acts as a permeation barrier [39]. The disadvantage of type IV tanks is the possibility of hydrogen permeation through the polymeric liner. Although these cylinders are lighter than those containing all-metal liners they are more expensive compared to the other metal liners cylinders.

Interaction of hydrogen with storage materials

The interaction of hydrogen with metallic and polymeric materials which are primarily used for the storage tanks is important in hydrogen storage analysis. Due to the small size of its molecules and atoms, hydrogen can be easily absorbed by different materials including those used for hydrogen storage. This, in turn, leads to the degradation of the material's mechanical properties, which may result in unwanted hydrogen leaks and structural failures. The interaction of hydrogen with the materials is pertinent to all fuel cell vehicles (FCVs) and other renewable energy applications [10,40]. However, in addition to being compatible with hydrogen, the materials used for storage are often subjected to high pressures, low temperatures, and cyclic or static loading, thus they must be selected accordingly. Hydrogen has a low viscosity and small atoms that can also be absorbed into materials, so leaks and embrittlement of certain materials are possible, which can result in structural failure [41]. Mechanical degradation of structural materials under the influence of hydrogen is a serious problem and caused many accidents during production, storage, transportation, and use [10]. The correct selection of suitable materials for the components is crucial for the safety of hydrogen storage systems. This relates to piping, walls of storage vessels, filling connectors, valves, fittings, etc. [33].

Interaction of hydrogen with metallic and polymeric materials

The compatibility of hydrogen with metals is affected by chemical interactions and physical effects, which include: dry corrosion, wet corrosion, and hydrogen embrittlement [12]. Dry corrosion is a chemical reaction between a dry gas and a metal, which eventually results in the reduction of a cylinder wall thickness [42]. This type of corrosion is not very common, because its rate is very low at ambient temperature. However, at high temperatures hydrogen can react with some metals, forming hydrides [43]. In general, wet corrosion can occur in a hydrogen storage vessel following the water entry. At low temperatures, some metals can become more brittle due to a change from ductile to brittle behavior mode when the temperature is lower than a "nil-ductility" temperature, which is sometimes considerably higher than that of the cryogen [32]. Several accidents involving a cryogenic metallic storage tank have been caused by cold hydrogen embrittlement [44]. Hence, the selection of material for hydrogen storage needs to understand the interaction between the material and hydrogen.

Polymeric materials are increasingly being used for the liners and wrapping of hydrogen storage tanks [29]. These materials are characterized by their tensile modulus, tensile strength, and elongation [29]. Polymers are also present in some fuel cells as a material for membranes. However, two degradation phenomena are often associated with polymeric materials used in FCV applications. These two phenomena are common; a permeation of hydrogen through the materials and the degradation of the mechanical properties of the polymers are commonly observed [32]. Permeation of hydrogen through the polymeric liner can be one of the main issues for the hydrogen pressure vessels along with the hydrogen embrittlement [45].

There are other unwanted effects of hydrogen on polymeric materials [46,47]. Some of the phenomena are the swelling of polymers, which occurs due to a gas or a liquid absorption or adsorption [48,49]. These unwanted effects can lead to an unacceptable increase of components' dimensions, or a formation of cracks related to sudden outgassing when the partial pressure is decreased. However, the significant level of swelling can be obscured by the 'leaching out' of plasticizers and fillers frequently used in polymeric materials. Other important effects such as changes in a polymer's mechanical strength and hardness should also be considered when selecting material for hydrogen storage. Furthermore, relatively large amounts of hydrogen can dissolve in a polymeric material, thus a polymer exposure to hydrogen can cause not only swelling but also blistering [50]. It was reported that if gaseous hydrogen contains a certain type of impurities, which are not compatible with the polymeric materials, deterioration of mechanical properties and eventually a rupture of a component may occur [51]. The materials used for hydrogen storage must be light in weight but also should be able to withstand extremely high pressures whilst maintaining their integrity. Several studies relating to the interaction of hydrogen storage material with hydrogen and possible mechanisms have been investigated by different authors and reports presented as shown in Table 1.

Hydrogen embrittlement process

Embrittlement is a process, by which various metals, mainly high-strength steels, become brittle and crack after being exposed to hydrogen [12]. It is caused by the ingress of either molecular or atomic hydrogen into a metal lattice [64].

Table 1 – Hydrogen embrittlement mechanisms in some materials and alloys for hydrogen storage application.

Materials	Mechanisms	Ref.
Stainless steel	Hydrogen diffuses into the grain boundaries, combining with carbon to produce methane gas which enhances the pressure within the system and initiates crack.	[52]
Plain carbon steel	The entrance of hydrogen into material causes cathode hydrogen charging resulting in a decline in macro-mechanical properties. The transition from ductile to brittle failure transition occurs. Combined actions of hydrogen-enhanced localized plasticity and hydrogen-enhanced decohesion are enhanced by the diffusion of hydrogen into the steel material.	[53–55]
Aluminum and aluminum alloys	The diffusion of hydrogen to the casting defects and precipitates creates cracks due to the reduced solubility of hydrogen in the aluminum material at low temperatures. In aqueous conditions, stress corrosion cracking may be the prevailing mechanism when the high-strength aluminum alloy is electrochemically charged by hydrogen.	[56,57]
Copper and copper alloys	Formation of fissures and blisters due to diffusion of hydrogen into the oxygen-bearing copper and copper alloy annealed in a hydrogen environment. The fracture toughness and ductility of the copper material are decreased.	[58,59]
Nickel and nickel-based alloys	The adsorption of hydrogen at the crack tips of nickel and nickel-based material increases the rate of crack propagation in aqueous hydrogen	[60,61]
Titanium and titanium alloys	The mechanism occurs through the breakdown of titanium oxide protective film due to high cathodic charging current densities can initiate the penetration of atomic hydrogen into the material.	[62,63]

However, the exact mechanism of hydrogen embrittlement is not clear. The capacity of the material to deform or stretch under load is limited due to a metallurgical interaction between atomic hydrogen and the crystallographic structure. As a result, when stressed or loaded, it becomes "brittle" and the metal will fracture or break at a far lower strain or stress than expected. Hydrogen embrittlement is particularly dangerous because of its reduced breaking strength and the vulnerability of material to hydrogen embrittlement increases as its strength increases. Embrittlement caused by very minute amounts of hydrogen, which is undetectable by typical loss-of-ductility bend tests, has been a major source of concern for materials and system designers. This atomic level embrittlement occurs at hydrogen levels as low as 10 ppm [65]. Hydrogen atoms are reported to preferentially segregate at defect locations in the materials, according to the hydrogen pressure theory first presented by Zapfe et al. [9]. When hydrogen is concentrated or absorbed in particular locations of metallurgical instability, embrittlement occurs. The infiltrating hydrogen atoms tend to "sweep" across the atomic structure as a result of residual or imposed stress. When hydrogen atoms come in contact with the storage materials, the hydrogen atoms that have gathered locally are then united to form hydrogen molecules. As time passes, hydrogen atoms in the vicinity of these defects diffuse toward the defect sites of the material, resulting in a high hydrogen gas pressure. Hydrogen-induced cracking occurs when the local hydrogen gas pressure surpasses the material's critical strength. In the case of stress-induced hydrides for metals, the originally low hydrogen concentration is redistributed and the hydrides form when the local hydrogen concentration approaches the solubility of metals. While several studies have been performed regarding factors affecting hydrogen embrittlement, the course of this phenomenon is not yet clearly understood.

Although critical evidence on hydrogen storage using different materials and improvement in hydrogen storage has been reported, numerous inquiries regarding the key degradation mechanism of hydrogen storage vessels remain unanswered [66,67]. Mirabile Gattia [67] investigated the swelling process brought on by the cycling of pellets in a hydrogen storage tank. The objective was to determine the microstructural modification and degradation mechanism of the hydrogen tank to define strategies for material preparation capable of reducing the entity of the swelling process using various microstructural techniques. In another study, Halm et al. [66] studied the degradation of the outer layers of a type IV composite pressure vessel. They used an experimental setup and simulation techniques in the investigation. To simulate the combined effects of mechanical damage and temperature, a straightforward finite element model was developed. The composite tank's time to burst and the change from burst to leak were both found to be reliably predicted by this method. For the durability and sustainability of the hydrogen storage tank, the material has to be kept out of any form of degradation and operational damage. Although the exact mechanism(s) is still being researched, the reality is that components fail as a result of this phenomenon.

Hydrogen embrittlement mechanism and material failure

Fig. 4 shows the schematic diagram of the proposed hydrogen embrittlement mechanisms in a hydrogen storage tank. In accordance with Fig. 4, hydrogen penetrates the material in aqueous or gaseous state and spreads via various pathways, leading to material failure and degradation. Dissolved hydrogen may exist as atomic or molecular hydrogen, or in a mixed molecular form, once absorbed by a material [68]. Because these molecules are too big to permeate through the material, pressure builds at crystallographic defects (dislocations and vacancies) and discontinuities (voids, inclusion/matrix contacts), resulting in minute cracks. The combination of material strength, external forces, pressure, and

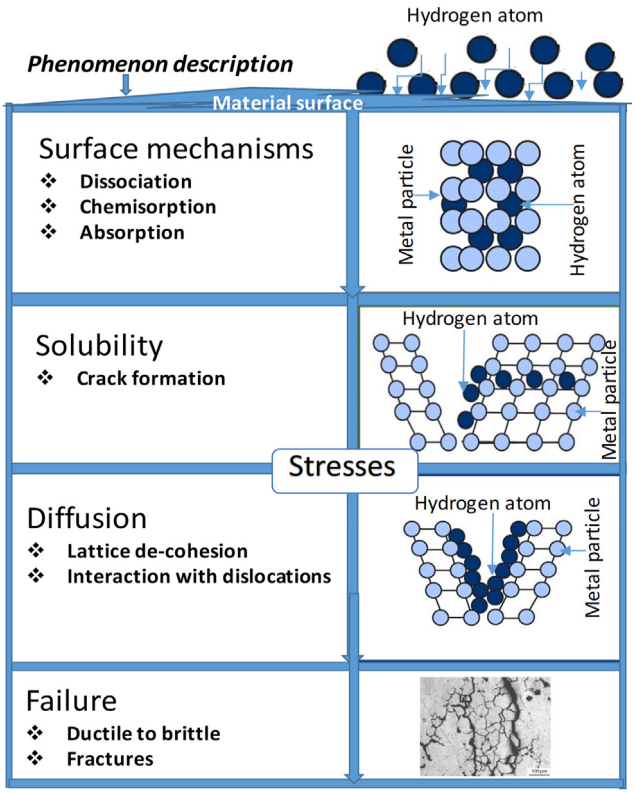

Fig. 4 – Schematic diagram of hydrogen embrittlement mechanism.

temperature determine whether this absorbed hydrogen causes cracking or not [18].

This type of embrittlement occurs when hydrogen is concentrated or absorbed in certain areas of metallurgical defects. As shown in Fig. 4, aqueous or gaseous hydrogen embrittlement mechanisms entail the entry of hydrogen into the material, which reduces its ductility and load-bearing capacity. Stress below the vulnerable material's yield stress leads to cracking and catastrophic brittle failures (see Fig. 4). There are several other mechanisms proposed to explain the hydrogen embrittlement mechanisms in hydrogen-material interactions by different authors [8,69]. Amongst them are hydride formation and fracture mechanism, HEDE and hydrogen enhanced local plasticity (HELP) shown in Fig. 5.

The HEDE mechanism proposed that introduction of hydrogen into the material reduces the cohesive strength of the material at the region of the crack tip. It was reported that the process occurs when the critical crack tip opening displacement is reached resulting in a cleavage like the type of fracture. On the other hand, the HELP mechanism theory reports that the accumulation of hydrogen near the crack tip decreases the resistance of the material for dislocation through a local drop in the yield stress of the material as a result of local dislocation movement at the low level of the material stress. Another mechanism called AIDE combines the theories of HELP and HEDE to demonstrate that the nucleation and growth of cracks occur as a result of decohesion and dislocation emission at the crack tip. The mechanism further explains that the growth in crack and fracture is enhanced by the combined action of slip at the crack tip with the presence of micro-voids coalescence. While Hydrogen changed micro-fracture mode (HAM) and other mechanisms are focused on the transition from the ductile to brittle micro fracture to the mode of material which reduces the ductility of the material, other mechanisms such as hydrogen enhanced macroscopic plasticity (HEMP) center around the macroscopic plasticity of material due to hydrogen influence that affects the mechanical property of the material when hydrogen occurs in large volume. Subsequently, through hydrogen diffusion, solid softening occurs and the yield strength of the material is reduced [71]. According to the literature, fracture propagation and the hydrogen embrittlement effect are occasionally caused by one or a combination of these mechanisms. As a result of these mechanisms, the influence of hydrogen caused subcritical fracture growth in the material.

For a thorough understanding of crack initiation and the mechanisms that cause crack propagation and failure in hydrogen embrittlement processes, macro and microscale fractures are very desirable [10]. When a material is subjected to a certain amount of stress, the effect of hydrogen at the fracture tip causes the material's cohesive strength to decrease resulting in subsequent failure. Furthermore, when tensile stress exceeds the material's interatomic strength at fracture tip opening, hydrogen atoms concentrate at the crack tip, reducing cohesive strength and causing subcritical crack propagation. After the material's interatomic strength is reduced, the hydrogen atom moves to a new crack front position, and cracking continues until the critical crack length is reached [10]. Fracture growth is caused by a huge amount of tension near the crack tip, as well as excessive hydrogen accumulation and hydrogen trapping. HELP is a well-known phenomenon for subcritical crack growth caused by dislocation motion at the fracture tip due to hydrogen atom buildup in the process. Local plastic distortion occurs as a result of the hydrogen atom, and the plastic flow becomes brittle. After the material's interatomic strength is reduced, the hydrogen atom moves to a new crack front position, and cracking continues until the critical crack length is reached and the material fails [10].

Techniques for measuring hydrogen embrittlement

Determination of hydrogen embrittlement in materials has been performed using several methods and some of these techniques will be described in the subsequent section. The methods are; the linearly increasing stress test (LIST), constant extension rate test (CERT), Slow strain rate testing (SSRT), temperature techniques desorption spectroscopy (TDS), permeation testing (PT), scanning electron microscopy (SEM), and the transmission electron microscopy (TEM).

Linearly increasing stress test techniques (LIST)

In linear increasing stress test measurement (LIST), two procedures are commonly employed in the early stages to determine the influence of hydrogen embrittlement in materials. While constant load and constant extension tests are

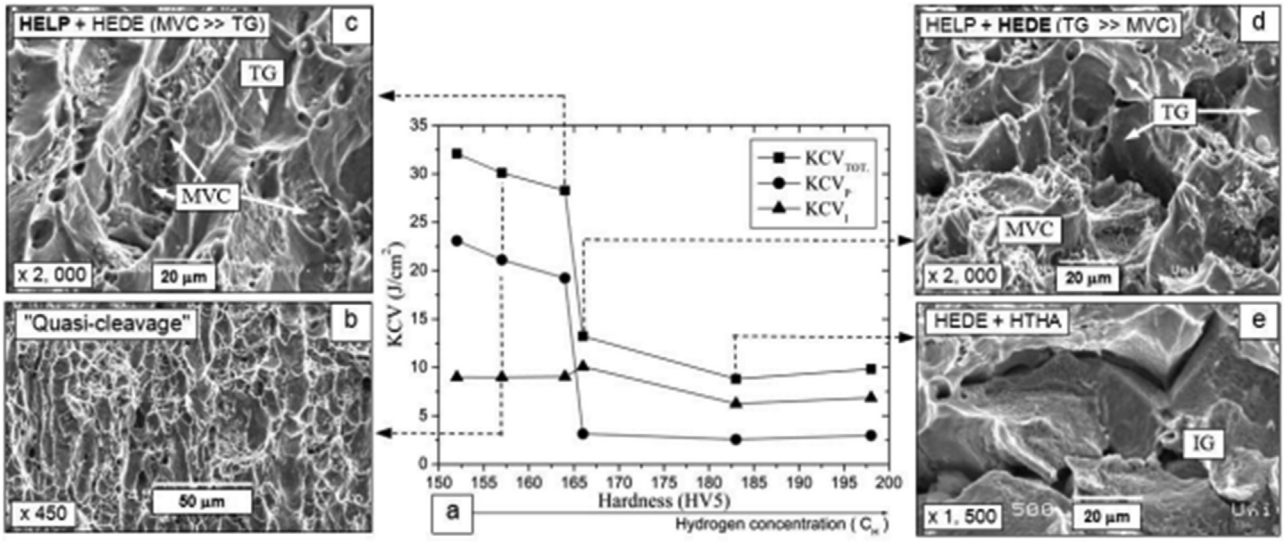

Fig. 5 – Different mechanisms of hydrogen embrittlement of carbon steel showing: (a) Changes in the impact strength (KCVTOT) of a Charpy specimen as a function of the specimen's hardness (hydrogen content), as well as its crack propagation component (KCVP) and crack initiation component (KCVI); (b–e) SEM fractography of the examined Charpy specimens' fracture surfaces [70].

applied, the stress threshold limit is first calculated in both tests and the stress below which no failure has occurred is determined [72].

However, the constant extension rate test (CERT) and Slow strain rate testing (SSRT) are other methods employed to address the deficiencies of the LIST method. In the SSRT, a constant progressive elongation is applied to the specimen with time till it fails or fractures. Nevertheless, the downside of this testing is that if the sample reaches the threshold limit (threshold stress), it will take longer to fail. Furthermore, the process is a time-consuming method, and a fresh revolutionary technique is required. The CERT test provides the capacity to relate SCC to strain rate in addition to providing information about the critical strain rate at or below which susceptibility to SCC occurs. For instance, in some alloy systems, reducing the strain rate first results in a decrease in the failure strain and time to failure, then a return of the ductile values at extremely slow strain rates. The specimens are tested in the CERT at 600 °C in a salt media with initial strain rates ranging from 4×10^{-8} to 2 × 10–5 s^{-1}. When tested in molten salt, the structure of the oxide film on the specimens is altered by the continual deformation that is applied. The LIST and CERT methods are depicted schematically in Fig. 6.

According to Fig. 6a, a high-precision displacement transducer with a 25 mm stroke is mounted at the end of the lever beam so that the transducer could track the angular deflection of the beam. Any movement of the lever beam generates a feedback signal for the servo controller to control the 1.27 N m continuous stall torque DC servomotor. The DC servomotor drives a linear actuator, which moves the load string in the opposite direction of the lever arm movement until the lever arm returns to its initial horizontal position via a pulley and timing belt connected to a worm gearbox. The motor's velocity is proportional to the deflection of the lever arm, while the acceleration is proportional to the speed of the lever arm. In the servomotor, a tach0 generator generates an analogue signal proportional to the motor's direction and velocity; this signal is used as feedback to the servo controller, allowing accurate control of the velocity and acceleration. One challenge encountered while doing these tests is that the component does not fail in a definite or predictable length of time, and fracture or failure takes a long time to occur.

Where an electromechanical sensor known as an LVDT (linear variable differential transformer) is used to turn mechanical motion or vibrations, particularly rectilinear motion, into a variable electrical current, voltage, or electric signals and vice versa.

Thermal desorption spectroscopy (TDS)

Thermal desorption spectroscopy is a popular method for studying hydrogen embrittlement and hydrogen-induced failure [74]. According to the literature, there are a variety of approaches for determining and measuring the amount of diffusible hydrogen in steel and the role of hydrogen in failure. The diffusible hydrogen can be qualified and quantified using controlled and regulated heating to determine the amount of desorbed hydrogen. When heat is applied to steel, hydrogen absorbs the thermal energy and releases it when a certain level of absorbed energy equals desorption activation energy is reached. Under carefully controlled temperature ramping circumstances, measurements of the hydrogen desorption flow are made using the thermal desorption method. The sample can be heated using a radiation furnace in the high-vacuum system from ambient temperature to 1120 K at a configurable heating rate. In order to avoid any interference, the thermocouple is put outside the analysis chamber. A quadrupole mass spectrometer is used to find the tested mass numbers' partial pressures. The vacuum system's internal pressure ranged from 10^{-8} to 10^{-2} Pa during the

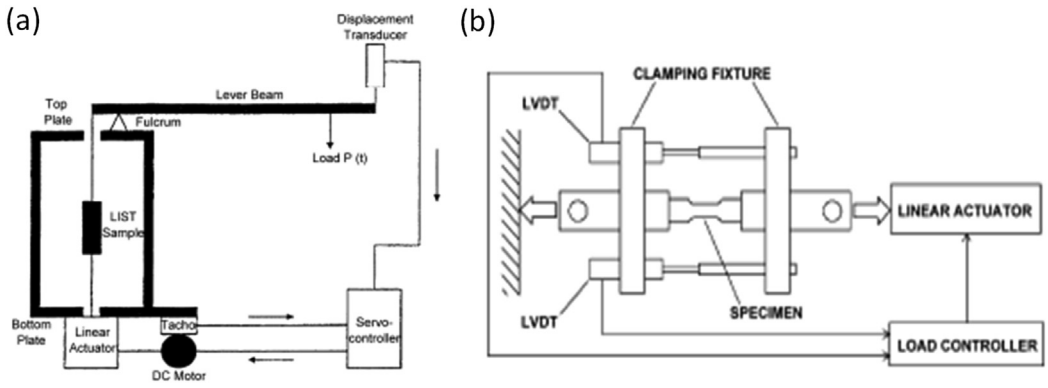

Fig. 6 – Image of the test set-up of (a) LIST and (b) CERT apparatus [73].

measurement. The samples are heated for 12 h at 1273 K in a high vacuum before hydrogenation. To thoroughly desorb any hydrogen that might have been stored within the sample, this was done. The samples are next cleaned with nitro-hydrochloric acid and acetone to get rid of the surface oxide coatings. The samples are further hydrogenated for around 20 h at 350 K in a hydrogen environment of 0.004 MPa before being cooled to room temperature. The Gadolinium (Gd) atoms serve as hydrogen atom trapping centers at this hydrogen pressure, which results in hydrogen concentrations that prevents the formation of a hydride phase. Therefore, by weighing the samples before and after, the hydrogen concentration of the samples can be discovered.

Quadrupole mass spectroscopy is used to determine the amount of desorbed hydrogen [75]. It is unique, in that it has high sensitivity and can reliably quantify small amounts of desorbed hydrogen compared to other techniques [76]. When combined with additional tests, the TDS approach can provide high hydrogen embrittlement detection and a better knowledge of the hydrogen embrittlement mechanism.

Hydrogen permeation test

The amount of diffusible hydrogen in the material can be determined using the permeation test which is the most basic method of measurement. When the amount of diffusible hydrogen is known, the HE susceptibility of steel can be calculated and accessed. This permeation test has been successfully implemented using a mix of different testing techniques [77]. This permeation test is essentially a two-chamber setup, with one serving as an entering cell (also known as a charging cell) and the other as an oxidation cell (exit cell). In the set-up, a steel membrane separates these two chambers, and hydrogen charging is employed in the electrochemical procedure. The cleanliness of the cell has a direct impact on the results' repeatability. All cell parts were meticulously cleaned in a sulfochromic solution before to each test. Both sides are kept in a nitrogen environment after the steel membrane is placed within the cell. The sodium hydroxide (NaOH) is often the solution used in the anodic side of the cell. Without any touch with the air, this solution is delivered to the cell. Anodic current is measured throughout time while a potential of 0.2 V is applied. The hydrogen is first used to charge the cell before being transferred to the oxidation cell via the membrane as shown in Fig. 7.

Microstructural analysis

In determining a material's and alloy's hydrogen embrittlement susceptibility, the microstructural investigation is critical. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) are common analytical techniques for determining the internal microstructure of a material, as well as the effect of hydrogen on the internal microstructure and material characterization. SEM is the most user-friendly, adaptable, and recommended technique.

The electron gun, which is located at the top of the column of the SEM, generates the electrons and accelerates them to energies ranging from 0.1 to 30 keV. A high-resolution image cannot be formed because of the diameter of the electron beam that the hairpin tungsten cannon produces. Therefore, the electron beam is focused, defined, and formed into a narrow-focused electron spot on the specimen using electromagnetic lenses and apertures. With this method, the electron source's size (around 50 mm for a tungsten filament) is reduced to the desired spot size (1–100 nm). It requires a high vacuum to prevent air from scattering electrons as they move. Real-time observation and image capturing of the specimen surface are made possible by the specimen stage, electron beam scanning coils, signal detection, and processing equipment. In addition, compared to other techniques, sample preparation is simpler and a three-dimensional image of the fractured surface and secondary cracks morphology is also produced by SEM as shown in Fig. 8.

This is crucial when studying fracture morphologies such as micro voids, dimples, and fisheyes [80]. Since a lower magnification and resolution can be more challenging in SEM than in other microscopes, the most effective and recommended approach is TEM. To circumvent the difficulties of SEM, TEM uses high magnification and resolution. More so, a million times magnification and nanoscale resolution are possible with TEM [45]. TEM can be used to collect morphology and topography information since the technique can provide crystallographic data. In the TEM, the interaction between high-energy electrons and the atoms in a solid is crucial to many physical processes. There are several potential

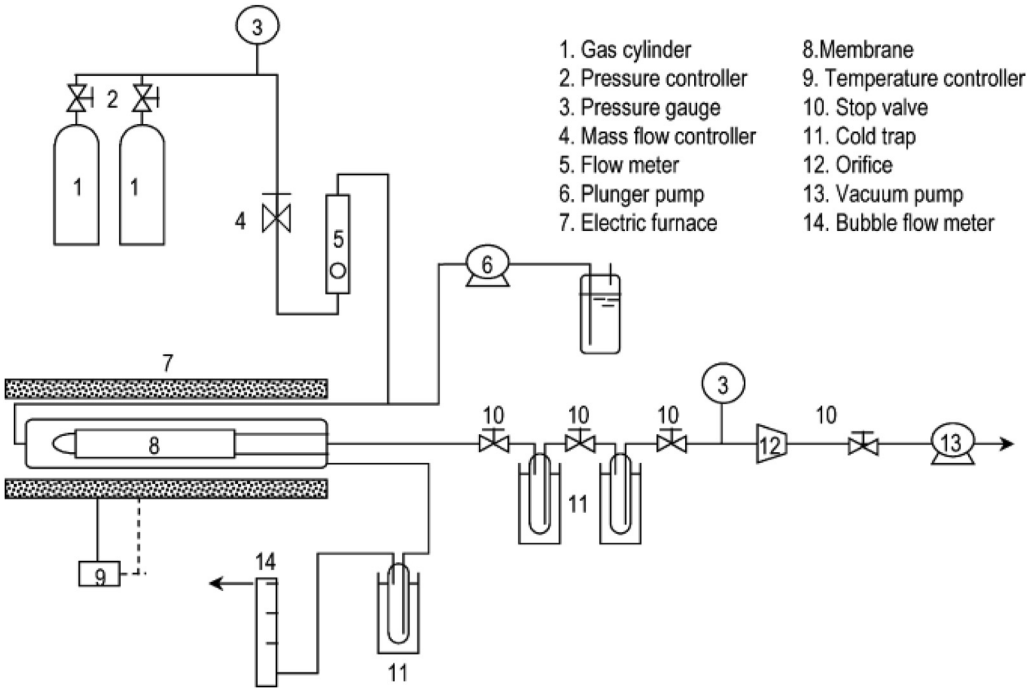

Fig. 7 – Schematic diagram of experimental apparatus [78].

interactions, and some of the more beneficial ones are those that result in quantifiable effects. The simulations that follow allow for individual interactions between high energy electrons typically 20 keV or higher and a single aluminum atom. This atom donates three electrons to a valence band or conduction band and is thought to be a component of a solid metallic specimen. Fan et al. [81] investigated the role of reversed austenite in hydrogen embrittlement fracture of a martensitic stainless steel using TEM for the microstructural analysis as shown in Fig. 9.

However, sample preparation can be one issue that has arisen while working with TEM since the sample must be electron transparent for TEM imaging, and the sample thickness must be less than 100 nm if TEM is used for microstructure investigation.

Improvement in the hydrogen embrittlement prevention

Degradation of the hydrogen storage material is a significant deterrent in the far-reaching development and commercialization of hydrogen tanks for creating a green society. Among various degradation processes, hydrogen embrittlement

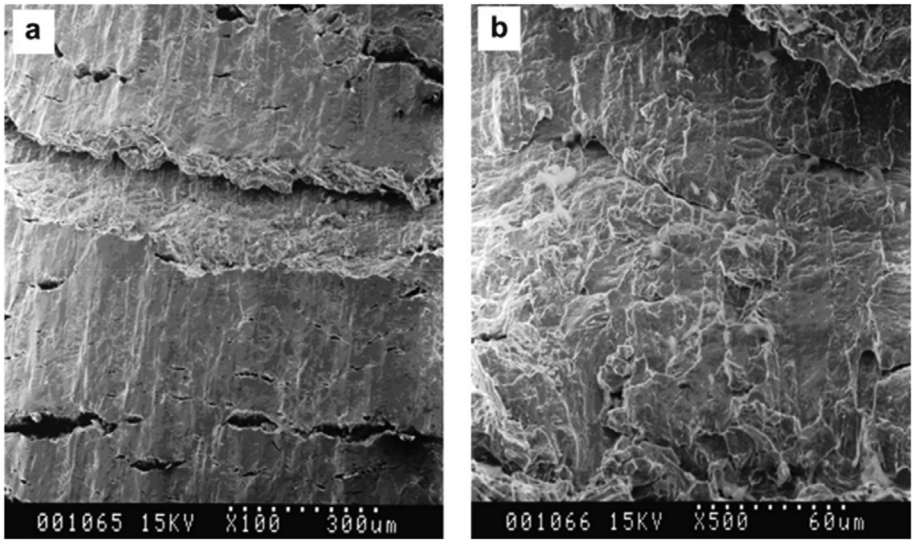

Fig. 8 – Shows a SEM image taken at a secondary crack after 48 h of cathodic charging in 0.1 M H2SO4 for a sample of 12% plastic strain; (a) the image shows the exterior surface and (b) a close-up of the secondary fracture [79].

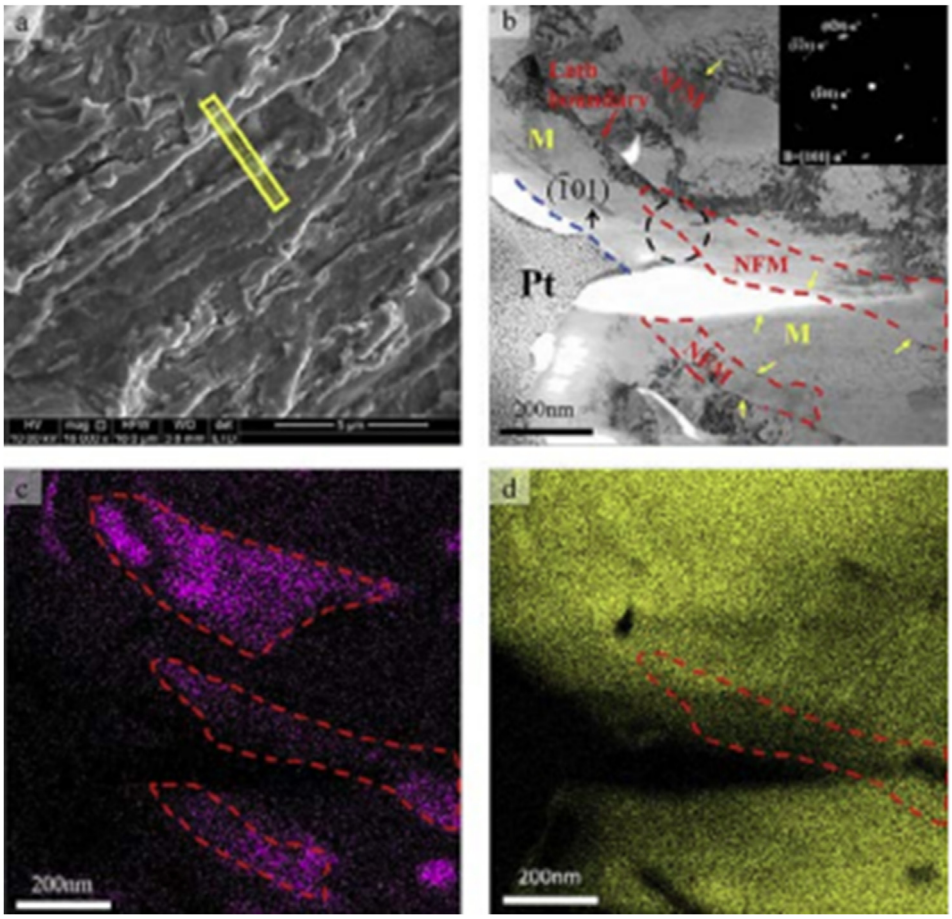

Fig. 9 – TEM images depicting the microstructure beneath the fracture surface and the behavior of crack propagation in DT samples subjected to dynamic hydrogen charging. Fig. 9a, FIB sample extraction position, 9b shows the bright-field picture and accompanying SAED pattern, 9c is the EDX elemental mapping of Ni in 9b, and 9d is the EDX elemental mapping of Fe in 9b are all examples of information that is given. Phase boundaries are indicated by yellow arrows [81]. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

through different mechanisms has been reported [8,11]. Hydrogen diffuses toward material and often accumulates in the stress concentration region. Studies have shown that fracture failure of materials occurs when the local hydrogen concentration approaches a critical value, which is currently unknown. Different techniques have been utilized to identify and understand the degradation mechanism of hydrogen storage material. For instance, mechanism HEDE mechanism, HELP mechanism, AIDE, and HEMP, etc. have been used to understand the hydrogen embrittlement mechanism that occurs in hydrogen storage tanks [8]. The general understanding is that the introduction of hydrogen into the material reduces the cohesive strength and yield strength of the material at the region of the crack tip. Subsequently, the nucleation and growth of cracks occur as a result of decohesion and dislocation emission at the crack tip when the critical crack tip opening displacement is reached resulting in a cleavage like the type of fracture. The failure mechanism can affect the integrity, performance, and efficiency of the storage tank.

Several attempts have been made by authors to mitigate the hydrogen embrittlement and improve the hydrogen storage facilities. Among the techniques, surface coating and material microstructural modifications have been at the forefront of this strategy. While the first approach involves the application of the surface coating and surface modification, the second method involves altering the material microstructure by adding or removing appropriate alloy components and optimizing the alloy microstructure. Often surface blackening which involves the production of an oxide layer on the metal surface with a thickness of 1–3 is useful for enhancing steels' corrosion resistance, surface coating, and modification of hydrogen storage material. Hydrogen entrance into the alloy or material is reduced when a metal surface is coated with a layer, providing the alloys with good hydrogen embrittlement resistance. The results of hydrogen permeation tests demonstrate that the hydrogen flow and hydrogen diffusion efficiency of blackening treatment samples are lower than those of non-treated samples [82]. More reports have been presented on the coverage of material surfaces with nickel (Ni), cadmium (Cd), aluminum (Al), and Al–Ni complex films resulting in successfully high hydrogen embrittlement resistance. Figueroa et al. [83], revealed that the AISI 4340 steel coated with a Zn–Al film demonstrated a greater hydrogen embrittlement resistance than the alloy

coated with a Cd film. Further analysis of the result and comparison with other coating materials showed that Ni–graphene complex film effectively lowered the hydrogen transport coefficient [84].

In the hydrogen embrittlement of steels, alloy elements and material microstructure modification play a significant impact. The amount of carbon (C), silicon (Si), phosphorus (P), and Sulphur (S) elements in the material can be reduced while the amount of Ni, Al, and Mo elements can be increased. It was discovered, for example, that the hydrogen embrittlement of Mn–B steel increases as the C element percentage rises [85]. For C concentrations larger than 0.3%, however, sensitivity to hydrogen embrittlement stays constant [52]. Aside from the goal of improving hydrogen storage tank material performance, further research and work are needed to better understand hydrogen storage degradation mechanisms. The application of appropriate materials microstructural modification and surface coating to increase and improve the performance of hydrogen storage materials is also suggested in this review. The creation of novel material through a microstructural and surface modification that can increase the performance of hydrogen storage tanks and enable more applications in the renewable energy system can be realized through these research and development methodologies.

Prospects and concluding remarks

Researchers have recently focused on hydrogen storage and its materials components [32,86]. The studies have increased the awareness of different hydrogen storage tanks and applications. However, degradation of the hydrogen-containing tanks has been a great concern to the green energy community. A review of the degradation mechanism of hydrogen storage tank materials is offered within this framework to provide a better understanding of the hydrogen embrittlement mechanism in storage tanks. Surface and materials modifications for the efficient operation of hydrogen storage containers are one of significant advancements made. The surface covering with elements like Ni, Cd, Al, and others has also proven to be effective in preventing hydrogen embrittlement. The accomplishments have been improving furthermore with additional comprehension of the material selection and surface modification roles in increasing the hydrogen embrittlement resistance as well as investing in innovative materials and alloys for hydrogen storage tanks. Indeed, such a lack of understanding of the hydrogen embrittlement degradation mechanism makes the explanation of the macroscopic level disintegration of the material in a hydrogen storage tank a hard task. Experimental and numerical models have been used to understand the degradation phenomenon in hydrogen storage materials [39,87]. Besides the aim to improve the performance of the hydrogen storage tanks, studies, and work are required to further understand the storage material degradation mechanisms. It is also suggested in this review to apply the developed enhanced surface and material modification strategies in improving the performance of the hydrogen storage tank materials. More new hydrogen storage materials that can enhance the efficiency of hydrogen storage vessels and offer more applications can be produced through these research and development methods.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgment

This study is supported via funding from Prince Sattam bin Abdulaziz University project number (PSAU/2023/R/1444).

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