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Investigation for the Response of High Strength Steel 42CrMo4 to in-situ Hydrogen Loading through Tensile Testing

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ABSTRACT

Hydrogen embrittlement remains a significant challenge in industries requiring high-strength materials operating in hydrogen-rich environments. This study examined the Investigation for the Response of High Strength Steel 42CrMo4 to in-situ hydrogen loading through tensile testing, aiming to unravel the intricate relationship between electrochemical conditions, hydrogen exposure, and the mechanical properties of the alloy steel. Slow strain rate tests were employed to comprehensively explore the material's response under varying conditions. The uncharged specimen in test-1 exhibited remarkable mechanical properties, including high tensile strength, appreciable hardness, and substantial elongation. The microstructural analysis confirmed the presence of a martensitic structure, reflecting inherent strength. However, as the current density increased in tests-2 and 3, hydrogen-induced embrittlement manifested. These tests revealed diminished mechanical integrity, evidenced by decreased ultimate tensile strength, reduced hardness, and limited elongation. The fracture modes observed, characterized by brittle fractures and distinctive fracture surfaces, underscored the susceptibility of 42CrMo4 to hydrogen embrittlement. In conclusion, this research advances the understanding of how current density interacts with hydrogen loading, influencing the mechanical properties of 42CrMo4 alloy steel. The findings contribute to theoretical knowledge, practical applications, and the pursuit of materials that thrive in demanding hydrogen-rich environments. As industries seek to fortify their materials against evolving challenges, the insights from this study provide a solid foundation for the development of hydrogen-resistant components, ensuring safer and more reliable structures in the face of adverse conditions.

Keywords: Hydrogen embrittlement, Current density, 42CrMo4 alloy steel, Brittle fracture, Fracture surface, mechanical property, Hydrogen-rich environment.

RÉSUMÉ

La fragilisation par l'hydrogène reste un défi important dans les industries nécessitant des matériaux à haute résistance fonctionnant dans des environnements riches en hydrogène. Cette étude a examiné "l'étude de la réponse de l'acier à haute résistance 42CrMo4 à la charge d'hydrogène par des essais de fatigue", visant à démêler la relation complexe entre les conditions électrochimiques, l'exposition à l'hydrogène et les propriétés mécaniques de l'acier allié. Des tests de vitesse de déformation lente ont été utilisés pour explorer de manière exhaustive la réponse du matériau dans des conditions variables. L'échantillon non chargé dans le test 1 présentait des propriétés mécaniques remarquables, notamment une résistance à la traction élevée, une dureté appréciable et un allongement substantiel. L'analyse microstructurale a confirmé la présence d'une structure martensitique, reflétant la résistance inhérente. Cependant, à mesure que la densité de courant augmentait dans les tests 2 et 3, une fragilisation induite par l'hydrogène s'est manifestée. Ces tests ont révélé une intégrité mécanique diminuée, mise en évidence par une résistance ultime à la traction réduite, une dureté réduite et un allongement limité. Les modes de fracture observés, caractérisés par des fractures fragiles et des surfaces de fracture distinctives, ont souligné la sensibilité du 42CrMo4 à la fragilisation par l'hydrogène. En conclusion, cette recherche fait progresser la compréhension de la façon dont la densité de courant interagit avec la charge d'hydrogène, influençant les propriétés mécaniques de l'acier allié 42CrMo4. Les résultats contribuent aux connaissances théoriques, aux applications pratiques et à la recherche de matériaux qui prospèrent dans des environnements exigeants riches en hydrogène. Alors que les industries cherchent à renforcer leurs matériaux contre l'évolution des défis, les informations de cette étude fournissent une base solide pour le développement de composants résistants à l'hydrogène, garantissant des structures plus sûres et plus fiables face à des conditions défavorables.

Mots-clés: Fragilisation par l'hydrogène, Densité de courant, Acier allié 42CrMo4, Rupture fragile, Surfaces de rupture, propriétés mécaniques, Milieux riches en hydrogène.

ACRONYMS AND ABBREVIATIONS

CT: compact tensile specimens

da/dN: Fatigue crack growth rates

ECCI: Electron Channeling Contrast Imaging

HE: hydrogen embrittlement

HEE: Hydrogen Environment Embrittlement

HEDE: hydrogen-enhanced decohesion

HELP: Hydrogen-Enhanced Localized Plasticity

HRE: hydrogen reaction embrittlement

IHE: internal hydrogen embrittlement

In-situ: test performed in a high-pressure hydrogen environment

ΔK: stress intensity factor range

LOM: light optical microscope

NTT: normal tensile test

PWHT: post-welding heat treatments

RT: room temperature

SSRT: slow strain rate test

SEM: scanning electron microscopy

TDS: thermal desorption spectroscopy

wppm: weight parts per million

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GENERAL INTRODUCTION

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GENERAL INTRODUCTION

The use of hydrogen as an alternative energy source has gained significant attention due to its potential to reduce carbon emissions [1]. In this context, high demand for hydrogen in the future will mean that we want to design more efficient and economical steel materials for storage and delivery facilities to be able to deal with the huge amounts of hydrogen production and technology. However, to accomplish this task, the hydrogen storage and transport sector (tanks, valves, vessels, engines, turbines, and pipes) will play a crucial role in withstanding higher hydrogen pressures between 40 and 100 MPa [2]. Specific equipment and materials have been fabricated to engage metallic materials with high hydrogen embrittlement resistance, mainly aluminium alloy and austenitic stainless steel. These metals have lower strength and are more expensive than conventional steels, such as low-alloy steels and carbon [3]. The approach could be the use of medium- and high-strength tempered martensitic steels, which are economically viable and allow decreasing vessel and pipe thicknesses and hence a lower cost of the steel. The BCC structure of high-strength steel, 42CrMo4, easily absorbs hydrogen due to its small atomic size. It can gently diffuse into the crystal lattice structure of the steel, driven by concentration gradients and stress [4,5]. However, unequal lattice imperfections such as internal interfaces, dislocations, and vacancies are called hydrogen traps [6].

42CrMo4 is an alloy steel containing carbon, Cr-Mo, and manganese, used in quenched and tempered conditions for applications requiring strength and toughness. It is one of the most excellent candidates for the manufacturing of hydrogen storage tanks, pipes, vessels, and valves to cater for high fatigue strength and wear resistance under high pressure hydrogen gas [7,8]. Nevertheless, it is believed to be more sensitive to hydrogen embrittlement than other forms of alloy due to susceptibility increasing with the strength level of the steel [9,10]. As a result, 42CrMo4 steel is usually tempered at high temperatures with reduced strength and hardness to reduce hydrogen embrittlement and HE-related problems. 42CrMo4 steel is normally applicable in aerospace, oil and gas industries, automotive, agriculture, and defence. The fatigue test is one of the most significant elements in material science, where failure occurs due to a component subjected to cyclic loading at stress considerably lower than the yield stress of static loading. Fatigue can be subdivided into three stages: fatigue crack formation, growth, and propagation. The formation is on two types of surfaces or sub-surfaces: the surface formation crack happens due to different defects, roughness, and notches on the surface, which can be the result of thermal treatment [2]. Furthermore, sub-surface cracks are initiated when the lubricant used on the surface is finished and the area is subjected to high contact stress. Fatigue crack propagation under contact loading ultimately leads to pitting and, consequently, to the failure of the contact surfaces. High Strength Steel is a type of steel alloy that provides superior strength and hardness compared to other types of steel [12]. It has a higher yield strength and tensile strength, making it ideal for use in structures and components that are regularly exposed to heavy stress and strain. This special alloy is often used in the construction of bridges, buildings, and automobiles, as well as in the manufacturing of aircraft and ship parts. Through its superior strength, it allows these items to be lighter and more efficient compared to those constructed with other materials, increasing performance and durability [13,14].

This research aims to bridge the gap in our understanding by investigating the intricate relationship between current density and hydrogen embrittlement behaviour in 42CrMo4 alloy steel. By systematically varying the current density during hydrogen loading, this study seeks to uncover the specific mechanisms through which electrochemical conditions influence hydrogen uptake, diffusion, and subsequent material degradation.

The previous investigations usually focused on the mechanism of hydrogen loading through fatigue tests and the mechanical properties of special steel grades via different testing methods. Few studies compared the various testing methods to evaluate the HE of 42CrMo4. There is still a limited understanding of how to interpret the research results and apply them to industrial applications. However, hydrogen can also cause embrittlement of metals, which can compromise the safety and reliability of hydrogen infrastructure [15]. High-strength steel 42CrMo4 is commonly used in the construction of hydrogen pipelines, but its response to hydrogen loading under cyclic loading conditions is not well understood. Therefore, this thesis aims to investigate the response of high-strength steel 42CrMo4 to hydrogen loading through fatigue testing.

This research aims to investigate the influence of varying current densities on the mechanical properties and hydrogen embrittlement behaviour of 42CrMo4, a commonly used alloy steel. The interaction between current density and hydrogen loading in this material is not fully understood, and addressing this knowledge gap is crucial for enhancing the durability and safety of components subjected to both electrochemical processes and hydrogen-rich environments. By systematically studying the impact of different current densities on hydrogen uptake, diffusion, and distribution within the 42CrMo4 steel, this research seeks to provide insights that will contribute to the development of more reliable materials for applications in industries such as aerospace, energy, and transportation.

Research questions

1. How does varying current density during hydrogen loading (SSRT) affect the mechanical properties of 42CrMo4 alloy steel?
2. What is the relationship between current density and the amount of hydrogen absorbed by the 42CrMo4 steel?
3. How does the interaction between current density and hydrogen loading impact the fracture toughness and ductility of 42CrMo4 steel?

The primary objective of this research is to systematically study the effect of varying current densities on the mechanical properties of 42CrMo4 alloy steel under hydrogen loading conditions. By subjecting samples to controlled electrochemical environments with different current densities, the research aims to characterize how these variations impact the material's tensile strength, hardness, fracture toughness, and ductility. This investigation will provide crucial insights into the potential correlation between current density and the mechanical integrity of 42CrMo4, shedding light on the material's behaviour under real-world hydrogen-rich scenarios. Another key objective is to quantify the relationship between current density and the rate of hydrogen absorption and diffusion within the microstructure of 42CrMo4. Through advanced hydrogen analysis techniques such as permeation tests and thermal desorption spectroscopy, the research seeks to establish how different current densities influence the uptake and distribution of hydrogen atoms within the material. This quantitative understanding will help elucidate the interplay between electrochemical processes, hydrogen transport, and the resulting embrittlement phenomena. Furthermore, the research will endeavor aims to delve into the microstructural changes induced by varying current densities during hydrogen loading. Light optical microscopy and Scanning electron microscopy techniques will be employed to analyze the evolution of microstructures, the formation of hydrogen-induced defects, and the localization of hydrogen-related damage within the 42CrMo4 steel. The objective is to uncover the mechanisms responsible for hydrogen embrittlement and the role of current density in promoting the initiation and propagation of defects like cracks and voids. [16] studied the optimization of PWHT in a 1.25Cr-0.5Mo pressure vessel steel for high-temperature hydrogen service and suggested that a tempering stage in the range of 670–710°C for 8 hours represented the optimal solution for resistance to hydrogen embrittlement. Tensile properties have been studied by many researchers, showing the effect of hydrogen on medium- and high-strength ferritic steels, which validates that HE does not only depend on the property level of the steel but also on the microstructure itself [17]. Some factors have paved the way for hydrogen charge in the specimen gas electrochemical charge [18]; the structure and measurement of the equipment, for example, the availability of notches or cracks [19,20]. Furthermore, the parameters used for testing the applied strain rate [21,22]. All these aspects related to hydrogen embrittlement of 42CrMo4 steel quenched and tempered at different temperatures were already considered in a previous work [23]. Furthermore, quenched and tempered steels have been widely studied in recent years to understand the effect of hydrogen on fatigue crack growth [24,25,26] pointing out the influence of the applied Kc factor and test frequency on the hydrogen embrittlement phenomenon. Some works have shown the nature of 42CrMo4 under high-pressure gas [27] by cathodic charge [28] and the pre-charging phenomena of the specimens in high-temperature, high-pressure hydrogen gas [30]. The findings of this research hold substantial significance for both fundamental materials science and practical engineering applications. Understanding the interplay between current density and hydrogen embrittlement can offer insights into designing more robust materials that can withstand the challenges of hydrogen-rich environments. Ultimately, the knowledge gained from this study has the potential to enhance the durability, safety, and performance of critical components in industries where hydrogen-related embrittlement poses a concern.

CHAPTER I:

LITERATURE REVIEW AND THEORETICAL BACKGROUND

Chapter I: Literature Review and Theoretical Background

Introduction

The subchapter below explains the behavior of hydrogen diffusion in metals, there mechanisms, and different factors. This is important information that needs to be understood to fully comprehend the later chapters in the thesis. Fundamental theory is laid forward with general information about hydrogen embrittlement and its types; Furthermore, experimental work is performed to know the effect of hydrogen embrittlement on the mechanical properties of 42CrMo4 high-strength steel.

1.1. Hydrogen

Hydrogen is the first element in the periodic table with a single proton and electron having an atomic mass of 1.00794 g/mol. It is the most abundant and simple element in the universe. Hydrogen is said to be 90% of the visible universe. Moreover, due to its properties such as tasteless, odourless, colourless, highly flammable, and non-metallic. When combined by itself it produces hydrogen such as Deuterium and tritium isotope [31].

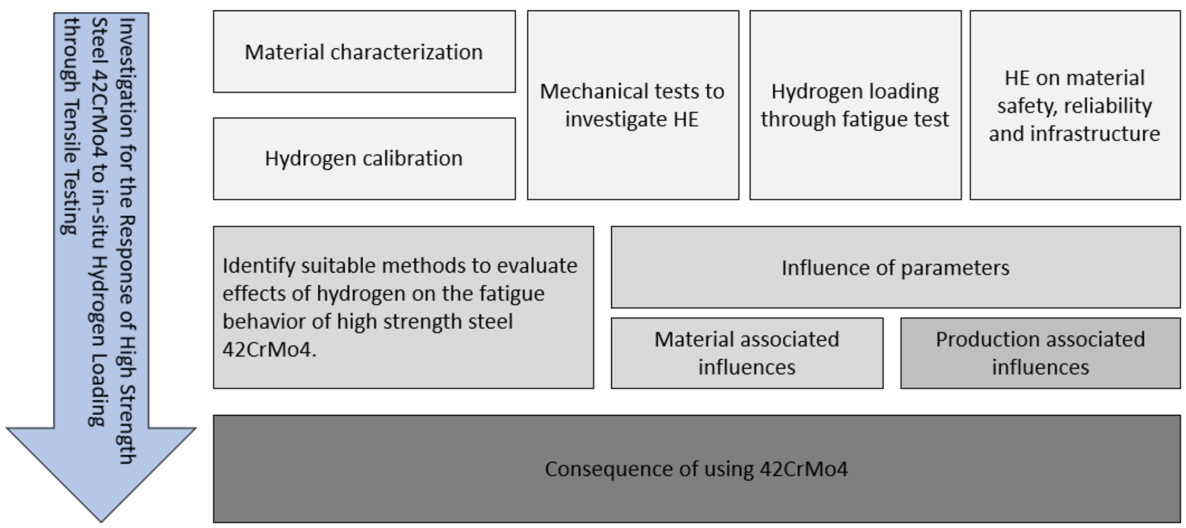

Figure 1: Structure of the thesis work, of the response of high strength steel 42CrMo4 to hydrogen loading through fatigue testing.

As illustrated in Figure 1, this thesis focuses on certain aspects, such as transport, energy, and even automobile industries. On the one hand, suitable methods to evaluate the HE is identified. On the other hand, the related influences of materials and production processes are investigated. Combining the test methods and related influences, the hydrogen embrittlement of 42CrMo4 in the steel industry can be better understood, leading to recommendations for the material selection and better manufacture of tanks, valves, vessels, and pipes.

1.2. Hydrogen embrittlement

The hydrogen embrittlement phenomenon is one of the causes of mechanical property failure (ductility, strength, and toughness) in most materials, leading to a high cost of loss. It is also called hydrogen attached, and it is a process where the metal becomes brittle or fractures due to the exposure or diffusion of hydrogen into the material. HE is divided into two forms: environmental hydrogen embrittlement, where failures occur due to the supply of hydrogen from the environment into the surface normally through the cathodic reaction of corrosion, and internal hydrogen embrittlement, where failures occur due to the supply of hydrogen during the heat treatment, electroplating, welding, melting, and solidification of the metal and alloys. Avoiding hydrogen mixing during manufacturing could help reduce internal hydrogen embrittlement. The fatigue crack growth in a material is affected by the following factors are: The R –value used in fatigue testing, b) The rate of diffusion of hydrogen in the material, c) The stress field present near the crack tip, d) The concentration of hydrogen in material e) The rate of diffusion of hydrogen in the material, and f) The frequency of cyclic loading applied [32].

1.3. Degradation of mechanical properties due to hydrogen

The presence of hydrogen in a material can reduce its mechanical properties, ductility, and load-carrying ability. It can lead to high fatal brittle failure due to applied stresses even below the yield strength as a result of the availability of hydrogen in the material [33]. Many researchs have shown that with increasing hydrogen content, strength and ductility decrease considerably [34]. Degradation is more apparent with increasing hydrogen content and with increasing strength of the steel [35]. Once hydrogen has entered a material, it can produce delayed failure (i.e., fracture resulting well after application of a load on the specimen). [35] describe the delayed fracture: Here hydrogen diffusion is an important step; hence, a certain amount of time is required during a test to develop a sufficient hydrogen concentration at a location to produce hydrogen-related damage. The method of testing for delayed fracture is simply to apply a static load below the ultimate tensile strength of a notch-tensile specimen until failure occurs. The resulting curve of applied stress vs time to failure Figure 2 provides information on the upper and lower critical stresses, incubation time, and fracture time. The upper critical stress corresponds to the rupture stress in an ordinary notch tensile test. The lower critical stress, or static fatigue limit, is the stress below which no delayed failure will occur [36]. The incubation time is required for crack initiation, and the fracture time is the total time until failure. Generally, the greater the hydrogen concentration, notch acuity, and strength level that the steel has, the lower the values of critical stresses, incubation time, and fracture time will be [37].

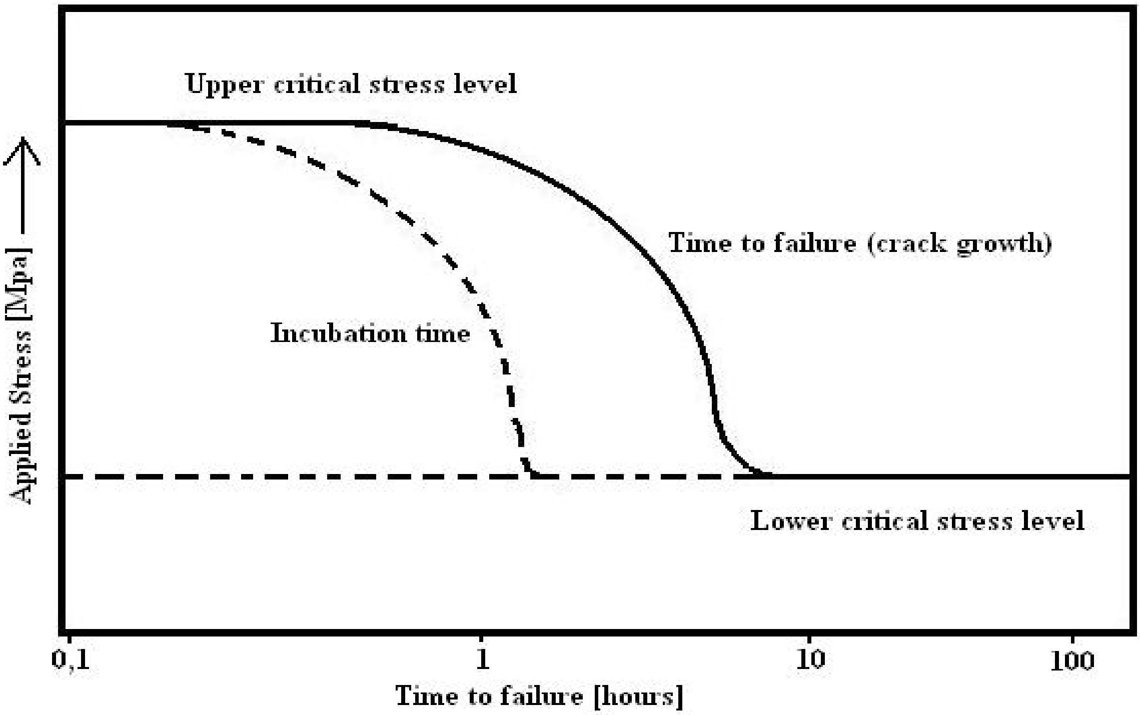

Figure 2: Schematic illustration of delayed failure for typical high strength steel alloy [37].

1.4.Different types of hydrogen embrittlement

Literature has demonstrated that there are three different types of hydrogen embrittlement (HE): hydrogen environment embrittlement (HEE), hydrogen reaction embrittlement (HRE), and internal hydrogen embrittlement (IHE) [38].

Table 1: Different type of hydrogen embrittlement [38].

Type of HE	IHE	HRE	HEE
Effect	Hydrogen absorbed at elevated temperature & long exposed times	Hydride formation. Methane and water vapour formation.	Surface cracking and accelerated cracking growth. Loss of ductility

Characteristic	Worse at room temperature	All alloys contain elements that react with H_2	Worse at room temperature
Examples	Almost all alloy, except Ag, Cu and Al alloy	Ti alloy and C steel	Ni base alloy

1.5.Hydrogen Embrittlement Mechanisms

The relationship between hydrogen and 42CrMo4 steel is crucial for understanding how their interaction initiates fatigue cracking and growth. These mechanisms will give us a comprehensive idea about the chapter toward developing a very good thesis and how it could help to carry out experimental work and analyze different results with their mechanical properties. There are different forms of hydrogen embrittlement mechanisms where the metals and steels interact with hydrogen internally these are:

1.5.1. Hydrogen-Enhanced Decohesion (HEDE):

A model that quantifies the behaviour of hydrogen solubility in a material, leading to low atomic binding energy in the metal’s lattice structure. This model based its mechanism on the hypothesis that hydrogen dissolved in materials such as metals and alloys could minimize the mechanical properties and the crystal structure. The HEDE mechanism depends on the type of fracture in the material, including grain size, grain boundaries, interface, intergranular, and cleavage planes. The main operation is shown in the diagram below. Figure 3 illustrates that as the applied stress is loaded in a gaseous hydrogen environment, the maximum stress is held in front of the crack tip, and this leads to hydrogen concentration exceeding the critical value. The formation of cracks nucleates and spreads along the plane [39]. [40] suggested that the interaction between electrons in dissolved hydrogen and iron causes the weakening of bond strength.

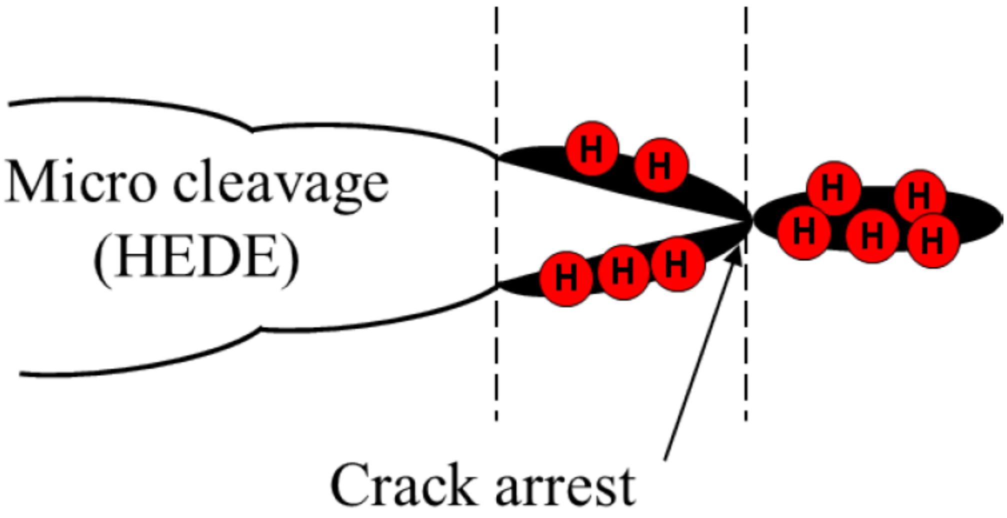

Figure 3: Schematics of HEDE mechanism in gaseous hydrogen [40].

1.5.2. Hydrogen-Enhanced Localized Plasticity (HELP)

Indicates that stress can cause dislocation of a material by shielding the emerging dislocation motions from each other [41]. The mode of mechanism occurred due to the presence of hydrogen and has impacted the nature of deformation, dislocation rate, and velocity, which have increased rapidly [42] According to this model, hydrogen lowers the dislocation interactions with elastic obstacles, which leads to a reduction in shear stress for dislocation motion; hence, localized deformation is promoted [43].

1.5.3. Adsorption-induced dislocation emission (AIDE)

Hydrogen embrittlement in this mechanism is a result of HEDE and HELP, which allow the solute hydrogen to be absorbed mainly at the surface of the material by stress concentration, causing a crack to initiate. Weakening is one of the mechanical properties observed because the interatomic bond of HEDE facilitates dislocation and the formation of cracks and micro voids by the HELP mechanism [44]. In this mechanism, nucleation and growth of the crack have occurred due to decohesion and dislocation emission at the crack. A high amount of adsorbed hydrogen on the surface has been detected in Fe, Ni, and Ti as proof to support the AIDE mechanism [45].

1.5.4. Mixed fracture (MF)

Microstructural examination (SEM) reveals two fundamental behaviours: ductility and brittleness. From this evaluation, it has been demonstrated that the fracture of the material will occur through the combination of these two phenomena in the microstructure mechanisms. Brittle, also called fish, originate from the central area, while ductility and micro-void coalescence (MVC) meet with fish through a radial direction until they come into contact [46].

1.5.5. Hydrogen changed micro-fracture mode (HAM)

A mechanism in which the mechanical properties of the steel changed from ductile to brittle [47]. This transformation is due to hydrogen charging, and the mode of fracture at ultimate tensile strength has changed from cup and cone to brittle shear fracture mode. According to [48], this could also be due to the high concentration of hydrogen at the edge dislocation, which helps the formation of shear fracture mode. The evolution is called hydrogen-assisted microfracture.

1.5.6. Hydrogen assisted micro void coalescence (HDMC)

As stated, mixture fracture (MVC) emerges from the ductile fracture process mechanism, where crack elongation occurs at different stages. These include void coalescence, void nucleation, void growth, crack extension, and the breaking of the existing remaining ligaments by shear. It has resulted in localized plastic deformation and possesses material dislocation motion. The nature of the propagation is in the form of a zigzag due to the holding of voids present in the crack propagation path. The presence of dimples in MVC decreased the ductility of the material due to the presence of hydrogen in the sample. The final fracture occurred due to shear stress with a parabolic shallow shear dimple [48-49].

1.6.Introduce hydrogen into steel and loading

Hydrogen loading is a term used to describe the process of using hydrogen to store and transfer energy. This process is becoming increasingly popular as a way to reduce emissions of harmful greenhouse gases and increase energy efficiency. Hydrogen loading involves capturing and storing hydrogen molecules in liquid or compressed gas form for later use. This stored energy can then be used in a variety of ways, such as powering fuel cells or running electric vehicles. As well as being relatively clean and efficient, hydrogen loading is also cost effective, which makes it an attractive option for many energy consumers. This work, we will take a closer look at the concept of hydrogen loading, how it works, and the potential benefits and risks associated with it. The introduction of hydrogen into materials has been observed by many writers; these include fabrication, shaping, melting, and casting [49]. The life span of the material could also experience hydrogen in the perimeter. Two main methods significantly induce hydrogen in metals and steels [50]. Arc-welding: if the electrodes contain moisture, water will be decomposed and hydrogen will be liberated in Cathodic protection process, e.g., with zinc This procedure is carried out in an electrochemical cell, where the metal is the cathode. The electrolyte contains hydrogen ions, which produce hydrogen molecules at the cathode when external current is applied [51]. Pickling in acids such as, hydrochloric or sulfuric acid these acids liberate a large amount of hydrogen when they come into contact with the material. Heating in an atmosphere containing water vapor or hydrogen Furthermore, hydrogen is very difficult to control or avoid because of its interaction with materials. Even under stress, the sample material is exposed to atmospheric gaseous hydrogen, which binds to the metal [52]. By controlled methods, hydrogen can be introduced into metal in many forms; among the various charging methods, two are broadly used 53]. [54] stated that the combination of hydrogen charge and stress applied to the material has a synergistic effect on the medium carbon and low alloy steel of 42CrMo4 by reducing 91% brittle fracture. There is less statistical difference between materials charged for different hours, and the longer the electrochemical charge of the material, the poorer the ductility. Generally, hydrogen-pre-charged specimens performed better on smooth tensile tests with quite low embrittlement indexes, while a notched tensile test shows a high increase. It was observed that these indexes always increase as the applied displacement rate decreases [55].

1.6.1. Cathodic method to introduce hydrogen

One of the common ways of charging hydrogen to the metal is in an aqueous medium called an electrolyte. Hydrogen molecules are concentrated on the surface of the sample specimen cathode through the application of external current, which flows throughout the circuit. The electrolytes used in this process are water, sodium hydroxide (NaOH), and sodium chloride (NaCl), but the most familiar electrolyte is H_2SO_4. Due to the large molecular size, it is tough to enter the samples. As_2O_3, which is mainly used for hydrogen recombination poison to prevent the formation of molecular gaseous hydrogen, is added to the electrolyte. Evaluations have shown that with increasing current density and charging time, the hydrogen concentration inside the sample will increase. This method is mostly used to study embrittlement of a stainless steels [55].

$Anode: Zn \rightarrow Zn^{2+} + 2e-$ equation (1)

$Cathode: O_2 + 2H_2O + 4e- \rightarrow 4OH-$ equation (2)

or

$Cathode: H_2O + e- \rightarrow OH- + H$ equation (3)

1.6.2. Gaseous method to introduce hydrogen

Hydrogen embrittlement in high strength steel pressurize vessel are studied using this method [56]. It operates in a condition where hydrogen is placed in a furnace at hydrogen rich gas atmosphere. The solubility depends on pressure (40-60) on hydrogen that will allow charging to occurred. The charging time is reduced with an increase in temperature [57].

1.7. Hydrogen diffusion and solubility

Numerous factors that influence the amount of dissolved hydrogen in a metal are diffusion, pressure, temperature, microstructure, and material composition. At room temperature hydrogen is basically at some few ppm [58]. When dissolved in a metal it occupied tetrahedra and octahedral interstices [59].

1.7.1. Diffusion

A vacancy space in a metal always allow hydrogen molecules to migrate from one interstitial to another, this diffusion phenomenon happened due to low in Gibbs free energy. During the heat treatment of metal diffusion rate increases as temperature material allows the flow of molecules.

Mathematically it can be described by Fick´s first and second laws the equation is also shown below for calculating the hydrogen penetration depth of hydrogen in metals [60].

$\mathrm{X} = \sqrt{2} * \mathrm{D} * \mathrm{t}$ Equation (4)

where X is the hydrogen penetration depth, D is diffusion coefficient (usually between 10^{-6} - 10^{7} cm^2/s for steel) and t is time in seconds.

$$D = D_0 e^{\frac{-Q}{RT}} \qquad \text{Equation (5)}$$

where T is temperature, Q is activation energy, D_0 is diffusion constant and R is universal gas constant 8.314 J K^{-1} mol^{-1}.

1.7.2. Hydrogen trapping

According to [61] local distortion or crystal impurities have a great influence on hydrogen solubility in metals, with cold working of steel hydrogen saturation increases and the lattices defects are introduced by cold working leading to hydrogen trapped. As a result, diffusion of hydrogen is retarded. However, there are different mechanisms by which hydrogen could be trapped in a material, such structural defects binding to impurities or microstructure entities in carbides alloy. The binding could be assigned to electrostatic forces (proton defect interaction), chemical potential gradients, physical trapping temperature gradients and stress fields. The trapping nature could be reversible dislocation, stacking fault or non-reversible (or irreversible) (grain boundaries, non-metallic inclusions, precipitates, or individual solute atoms). [62]. The trapping sites of hydrogen in 42CrMo4 refer to the locations within the material where hydrogen atoms can be trapped and held. These trapping sites play a crucial role in the diffusion and behavior of hydrogen in the material. Additionally, some common trapping sites includes: grain boundaries vacancies and interstitial sites. As shown in the sinusoidal curve in the figure 4, E_a stand for activation energy for hydrogen diffusion in perfect crystal lattice, while $E^{'}$ has high barrier energy and ΔE_x than the normal sites. For any diffusion to take place hydrogen requires much higher energy than $E^{'}+E_a$. The trapped hydrogen could leave at the condition when $E_a+E^{'}+\Delta E_x$ [62]. Equation (6)

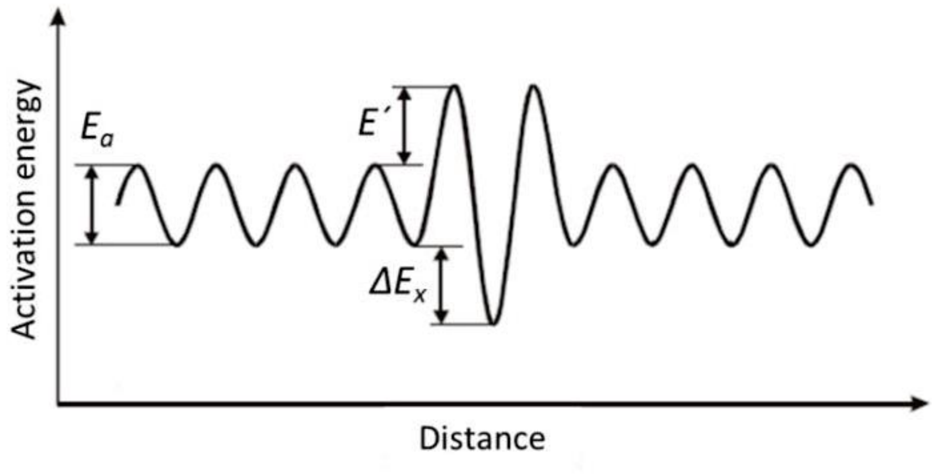

Figure 4: Energy interaction of atomic hydrogen and trapping sites [19].

1.7.3. Pressure influence

The Sievert's law equation defines the relationship between hydrogen solubility and pressure as shown in equation (6): [62].

$C = K * \sqrt{P}$ Equation (7)

Where the parameter C is the dissolved hydrogen concentration, P is the hydrogen gas pressure and K is a constant. The equation is valid at standard pressure of 120 atm and temperature higher than 200 °C. The reason being that above these values hydrogen will attach the steel to form a compound of methane which is difficult to diffuse out of the metal and instead will be collected in the voids, resulting in high pressure and initiating cracks in the steel [63].

1.7.4. Influence of Temperature

To study the influence of temperature on the high-strength steel, the equilibrium iron-hydrogen phase diagram was used. From the illustration given below, the hydrogen solubility is very low at room temperature (20 °C) for iron (-Fe) body centered cubic (BCC). The solubility increases gradually with an increase in temperature to almost 900 °C. At 910 °C, with an increase in solubility, the iron transforms to -iron face centered cubic (FCC structure). The solubility increases gradually until -iron transforms to -iron (BCC). After the transformation, the solubility decreases considerably. At the transition from solid iron to molten iron (at temperatures around 1535°C), the solubility increases once again [64].

The takeaway from the graph is that the liquid phase can solve more hydrogen than the solid phase; an example is molten iron or steel. It concluded that weld metal can absorb a large quantity of hydrogen, leading to hydrogen embrittlement or cracking when solidified.

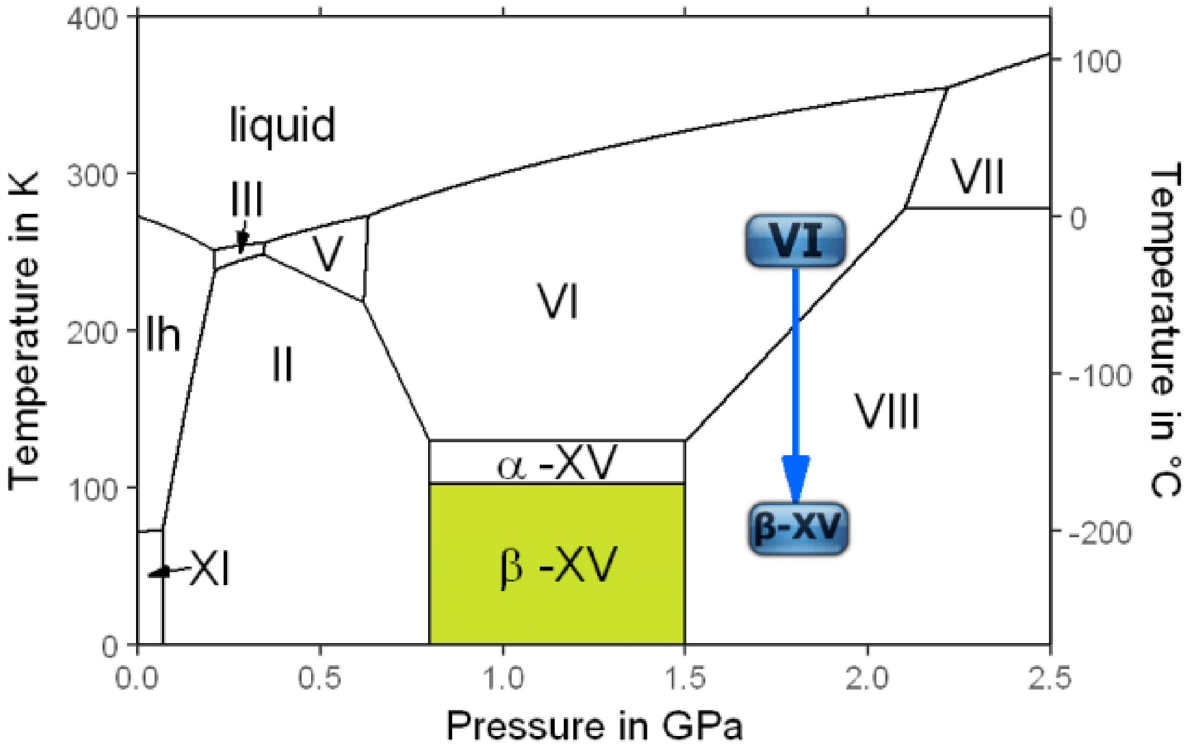

Figure 5: Equilibrium iron-hydrogen phase diagram [64].

The hydrogen solubility increases at 910°C and decreases at 1400°C.

1.7.5. Microstructural Influence

Understanding the microstructure of any material is a key operational factor in defining its mechanical behaviour. Microstructure allows us to know how slow, delayed, or fast hydrogen diffusivity is in a material by increasing the residence time of the molecular movement in the 42CrMo4 steel, leading to the risk of hydrogen embrittlement [65]. Hydrogen diffusivity in steel could be characterized by different technical methods, but the most reliable one is said to be the electrochemical hydrogen test. For instance, [66] studied hydrogen diffusion in a 2.25Cr1Mo steel submitted to a variety of heat treatments, reporting an inverse correlation between steel hardness and hydrogen diffusivity. In general, during definite heat treatment at very high temperatures, the density of hydrogen traps decreases due to dislocation of atomic molecules, thus increasing the diffusivity of hydrogen. also demonstrated that dislocations are the main factor in controlling the mobility of hydrogen in quenched and tempered martensitic steels [66]. I designed fatigue tests to evaluate the effect of hydrogen loading on the fatigue behaviour of high strength steel 42CrMo4. The tests will involve exposing high strength steel specimens to hydrogen under various conditions (e.g., pressure, temperature, exposure time) and subjecting the specimens to cyclic loading. The fatigue behaviour of the specimens will be evaluated using standard testing methods.

1.8.Prevention Techniques of Hydrogen Embrittlement

Hydrogen embrittlement is a phenomenon that can occur in metals when they become susceptible to cracking and failure due to the ingress of hydrogen atoms. Preventing hydrogen embrittlement is crucial in industries that deal with hydrogen or hydrogen-containing environments. Here are some prevention techniques [67]:

- Choose materials that are less prone to hydrogen embrittlement. For example, some high-strength steels and certain alloys are more resistant to hydrogen-induced cracking than others.
- Reduce or avoid high tensile stresses in the metal. High stresses can accelerate hydrogen embrittlement, so designing components with lower stress concentrations is important.
- Apply protective coatings to the metal surface to act as a barrier against hydrogen absorption. Coatings like cadmium plating, nickel plating, and chromate conversion coatings can provide some protection.
- Add a hydrogen permeation barrier between the metal and the hydrogen source. This barrier can prevent the hydrogen from diffusing into the metal and causing embrittlement.
- Control the temperature of the environment to minimize the effect of hydrogen embrittlement. Lower temperatures generally reduce hydrogen uptake and decrease the likelihood of embrittlement.
- Limit the concentration of hydrogen in the environment or the material. In some cases, purging the hydrogen environment or reducing the amount of hydrogen in the atmosphere can be effective.
- Use appropriate heat treatment processes that can minimize the risk of hydrogen embrittlement during manufacturing and processing.
- Keep metals away from potential hydrogen sources, such as acid pickling baths or certain chemicals that release hydrogen. Perform stress relief annealing on the metal

after welding or other processes that introduce high levels of residual stress. This can help mitigate hydrogen embrittlement. For components with protective coatings, subjecting them to a baking process after plating can help drive out absorbed hydrogen and reduce the risk of embrittlement. Ensure that the manufacturing and handling environments are clean and free from contaminants that could introduce hydrogen into the metal.

1.9. Effects of Hydrogen on the Mechanical Properties of Steel

Hydrogen embrittlement is a phenomenon that can significantly affect the mechanical properties of steel and other metals, leading to reduced strength and increased susceptibility to brittle fracture. When hydrogen atoms diffuse into the lattice structure of steel, they can interact with the material's microstructure and weaken its mechanical properties.

1.10. Reduce ductility and toughness

Hydrogen can induce steel to become less ductile and tougher. It can alter the grain boundaries and dislocations within the material, making it more prone to brittle fracture even at lower stress levels.

1.11. Increased Susceptibility to Stress Corrosion Cracking (SCC)

Hydrogen can enhance the susceptibility of steel to stress corrosion cracking, where cracks propagate under the combined influence of tensile stress and a corrosive environment

CHAPTER II:

MATERIAL AND METHODS

CHAPTER II: MATERIALS AND EXPERIMENTAL METHODS

CHAPTER II: Material and Experimental Methods
Introduction

This chapter includes the material, equipment, software and methods that are used to investigate hydrogen embrittlement in high strength material through fatigue tests. This section serves as a foundation for the entire investigation, providing readers with an understanding of the materials used in the study and the methods employed to conduct experiments, gather data, and analyze results. By detailing the materials' properties, characteristics, and sources, as well as the methodologies employed for testing and analysing, this section establishes the context and credibility of the research findings.

2. Heat treatment of 42CrMo4 steel and Properties

High-strength steel 42CrMo4 is a ferritic alloy that has been widely used in the energy industry due to its strength and resistance to wear and tear. It is widely used for components that are subject to cyclic loading, such as drive-shafts and cam-shafts. A 42CrMo4 steel was used in the present study. Its chemical composition in weight % is shown in Table 2.

Table 2*:* Chemical composition in weight*% of 42CrMo4 steel* [44].

Steel	Cr	Mo	C	Si	Mn	P	S
42CrMo4	0.98	0.22	0.42	0.18	0.62	0.008	0.002

The material dimension under the test specimens were made from 42CrMo4 in the form of solid bars with a circular cross-section (Length, and diameter) of (70 and 5.98 mm). They were machined to the required tolerance with the tolerance (Length +/-0.02mm, Diameter +/-0.02 mm). The two upper parts were ground to aid in holding during the experiment.

The diagram below in figure 6 demonstrates martensite steel. This microstructure is characterized by a needle-like or plate-like pattern and is known for its hardness and strength. Similar results were obtained from [67]. [68] showed that the initial microstructure of the 42CrMo4 before heat treatment was 70% perlite and 30% ferrite. Perlite is characterized by a dark contrast (composed of ferrite plus cementite) and ferrite is characterized by a light contrast. Martensite appears as a unique phase with a distinct crystal structure within the steel. When the carbon atoms in the austenite are rapidly cooled, they become trapped in a supersaturated state, preventing them from diffusing and forming a different microstructure. This results in the transformation of austenite into martensite, which gives the steel its hardness characteristic. The density of the needle-like structures is higher in grains with a smaller diameter. A special design machine was used to cut the shape of samples from workshop lab. It was then programmed to cut the reduce area both the head and the body size with length, diameter, and the specimen was put inside the machine to confirm that cut was correct or not. The whole sample cutting was performed after the heat treatment process using bandsaw. The size and shape of tensile tests specimens are as follows. This designed is made as per the ASTM standards. The final sample is demonstrated in the figure bellow.

Figure 6: Specimen design as per the ASTM standards.

2.1.Thermal desorption spectroscopy measurement

The hydrogen concentration was determined by Thermal Desorption Spectroscopy (TDS). The apparatus of TDS is shown schematically in Figure 7. The measurement took place after the tensile test of the four sample specimens were immediately done. The equipment was clean initially and all the setting parameters such as the pressure at the range (10-5 or 10- 8Pa) where done in furnace, the samples were placed in a glass pipe and the thermocouple was placed outside in another glass chamber in an equivalent location. The surrounding conditions of the sample and the thermocouple were quite similar under high vacuum [68]. In the heating process the temperature of the radiation furnace was measured by the thermocouple. The absorbed atomic hydrogen (H_{ab}) desorbs from the substrate with increased temperature and re-combination takes place at the sample surfaces. Thereby the amount of desorbed molecule hydrogen was measured by quadruple spectrometers and recorded by computer. In this study, the TDS apparatus was calibrated by standard TiH2. The heating rate was 0.3K/s**.**

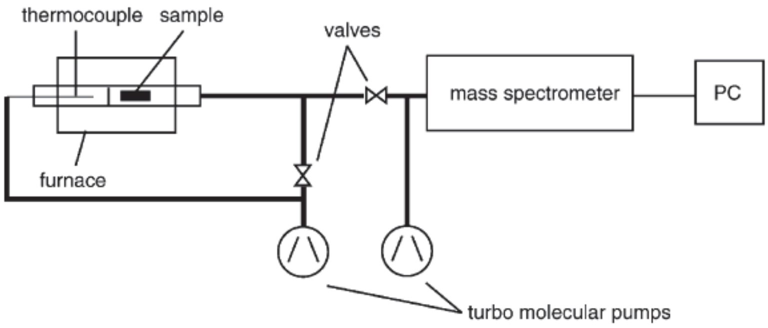

Figure 7: Schematic apparatus of thermal desorption spectroscopy

2.2. Tensile tests

A tensile test is a type of mechanical test used to measure the tensile strength, yield strength, fracture strength, percentage area reduction, and elongation of a material. It is performed by applying a force to a material sample until it breaks or deforms. The results of the test are used to determine the material's properties such as its strength, ductility, and toughness.

2.3. Tensile test at room temperature

Tensile tests on smooth round-bar specimens, whose dimensions and geometries are shown in Figure 8, were performed on (**Zwick/Z100**) tensile machine. Uncharged test was performed under a displacement rate of 2.16 mm/s.

The 42CrMo4 specimen was carefully mounted in the grips of the Zwick/Z100 testing machine. The grips were aligned to ensure that the specimen was centered and parallel to the loading axis. Once the specimen was securely mounted, the machine was started, and the TestXpert software was set up according to the predefined test profile. This included selecting the appropriate test type (tensile test), choosing the correct control mode strain rate at $5x10^{-6}$ s^{-1} and $5x10^{-3}$ s^{-1}, and specifying the required test parameters such as test speed, and gauge length. The machine started applying a gradually increasing force to the specimen. The applied force was measured using a load cell, which was connected to the machine and displayed on the TestXpert software. Simultaneously, the machine also recorded the elongation of the specimen using an extensometer, which was attached to the gauge section of the specimen. The extensometer measured the change in length of the specimen as the force was applied. Throughout the test, the TestXpert software continuously recorded and displayed the force and elongation data in real-time. This allowed for monitoring the behavior of the specimen and detecting any anomalies or deviations from the expected response.

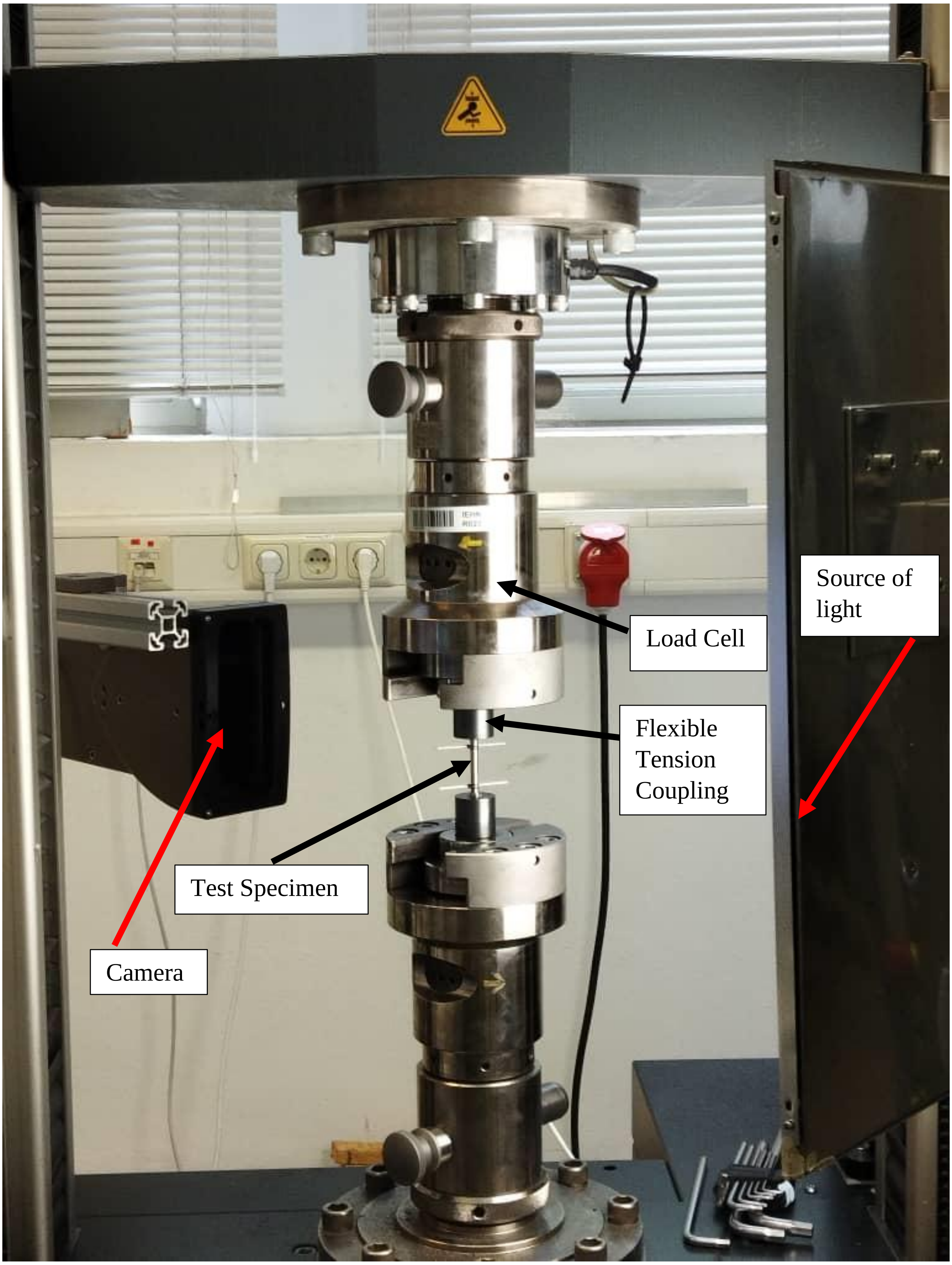

Figure 8: Uncharged tensile test machine (Zwick/Z100)

2.4.Tensile tests under in-situ hydrogen charging condition

The experimental set-up of the **Fritz-Fackert KG** test is displayed in Fig. 6. The machine is directly connected to the PC, which has LABVIEW software, where all the parameters of the test were inserted, such as the initial temperature of 21 degrees, the diameter of 5.97 mm, the current density of 0.01 mA/cm^2, and the round, circular, cylindrical shape of the sample. A cylindrical container was attached along with the specimen, which enabled it to hold the electrolyte of H_2SO_4, which then provided hydrogen diffusion into the specimen. The initial force was set at 15 KN, and after arranging all the required parameters, the machine was set to conduct a tensile test under in-situ charging. After the material has been broken it was carefully removed and washed with distilled water and kontaktspray 70 was on it to avoid corrosion and maintain it natural body. Another critical point is that imaging with high voltage under in-situ conditions may be dangerous because hydrogen is a flammable element. The voltage and current were set at high levels, and the experiment lasted nearly 5 hours.

Fatigue crack growth will be observed and compare the results with those obtained on un-charged specimens. Crack length was measured by optical microscope after the tests. The initial and final crack lengths were measured on the fracture surface of the broken specimen, and the measured ΔK values were corrected. The diagram bellow shows the indicated part of the tensile test machine.

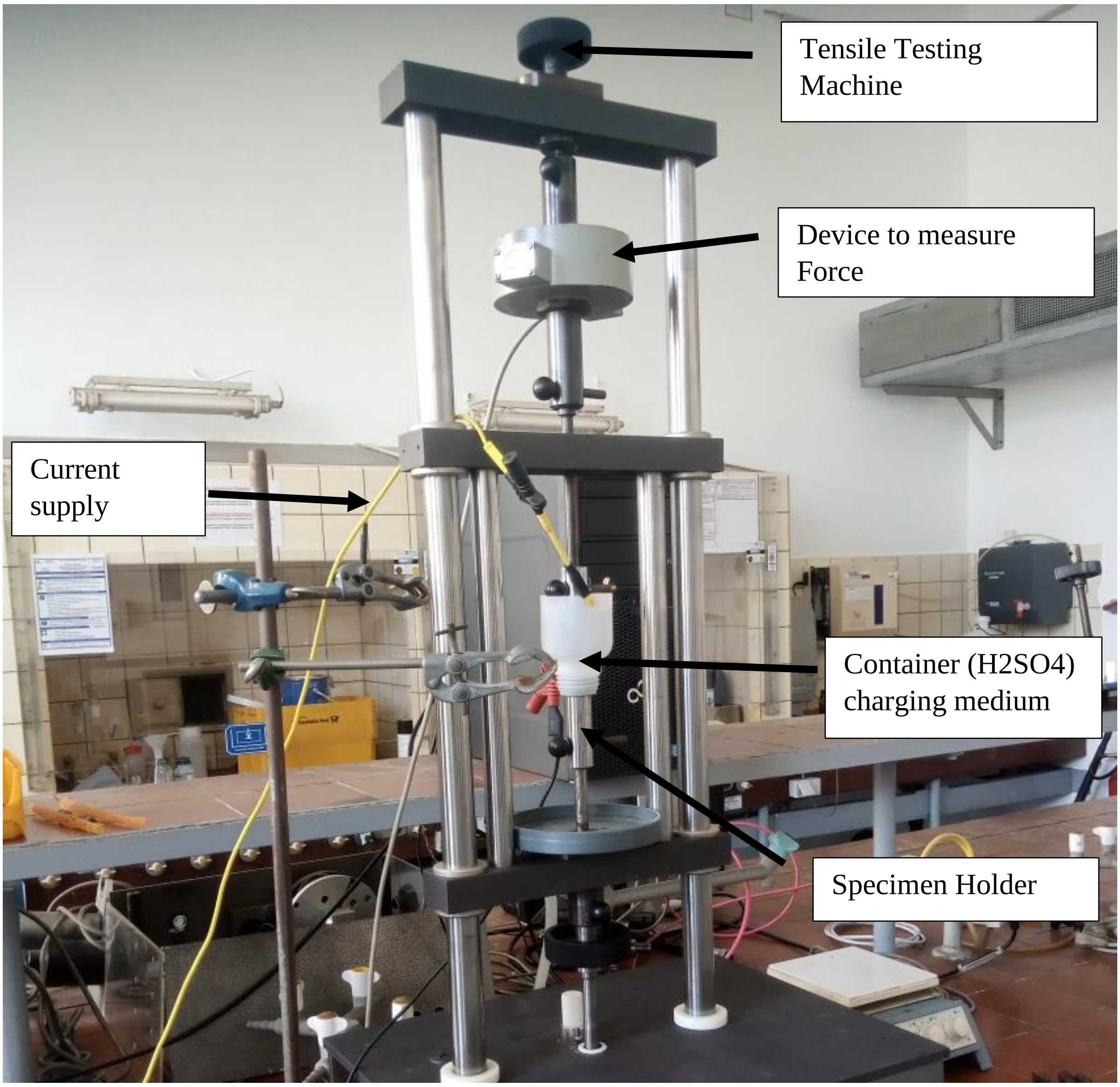

Figure 9: Experimental setup for SSRT with in situ hydrogen charging.

2.5.Hardness

Hardness testing was conducted to assess the mechanical properties of the in-situ charged and uncharged specimens. The average hardness values where computed according to AISI 420 martensitic steel of 42CrMo4. The tensile strength (MPa) can in turn be evaluated from Vickers hardness (VHN) data using the following expression [69].

TS (MPa) = 3.2 x HV

2.6.Statistical analysis of the results

CHAPTER II: MATERIALS AND EXPERIMENTAL METHODS

The statistical analysis of this master thesis has been conducted in Origin and Excel software. Initially, all the available data folders were checked using Shapiro-Wilk test at level of 0.05 with two similar values where tested using a hypothesis test for mean. For accurate statically normality all the results data where presented as the mean value (n=3).

2.7.Loss of mechanical properties

The loss of mechanical properties was computed from the reference uncharged slow strain rate test data, which served as the baseline for our analysis. As compared to the two other parameters, which were charged at current densities of 0.01 mA/cm^2 and 1 mA/cm^2, respectively. Such losses were established by the given expression below [68].

$$L\ (\%) = (1 - X_H/X) \times 100 \qquad \text{Equation (6)}$$

The two variables X_H and X stand for charged and uncharged, which correspond to the 42CrMo4 property. In this thesis, we focus more on ultimate tensile strength (UTS) and yield strength (YS).

2.8.Conclusion

The experimental methodology employed in this study involved the utilization of 42CrMo4 steel, meticulously designed and prepared within the laboratory setting. The material underwent a carefully controlled heat treatment process to transform it into a martensitic steel phase. Subsequently, a series of tensile tests were conducted in both hydrogen-rich and non-hydrogen environments, in the presence of H_2SO_4. The data acquired from these tests were employed to calculate various mechanical properties utilizing established equations, in conjunction with hardness measurements. Furthermore, we conducted thermal desorption analyses to assess and quantify the hydrogen concentration within the material. Finally, the experimental procedures encompassed material preparation, tensile testing under different environmental conditions, mechanical property calculations, and hydrogen concentration assessment through thermal desorption analysis.

CHAPTER III:
RESULTS AND DISCUSSION

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Introduction

This chapter will present the results of the Investigation for the Response of High Strength Steel 42CrMo4 to in-situ hydrogen loading through tensile testing, which includes the microstructures, and graphs using Origin software.

3.1.Microstructural view

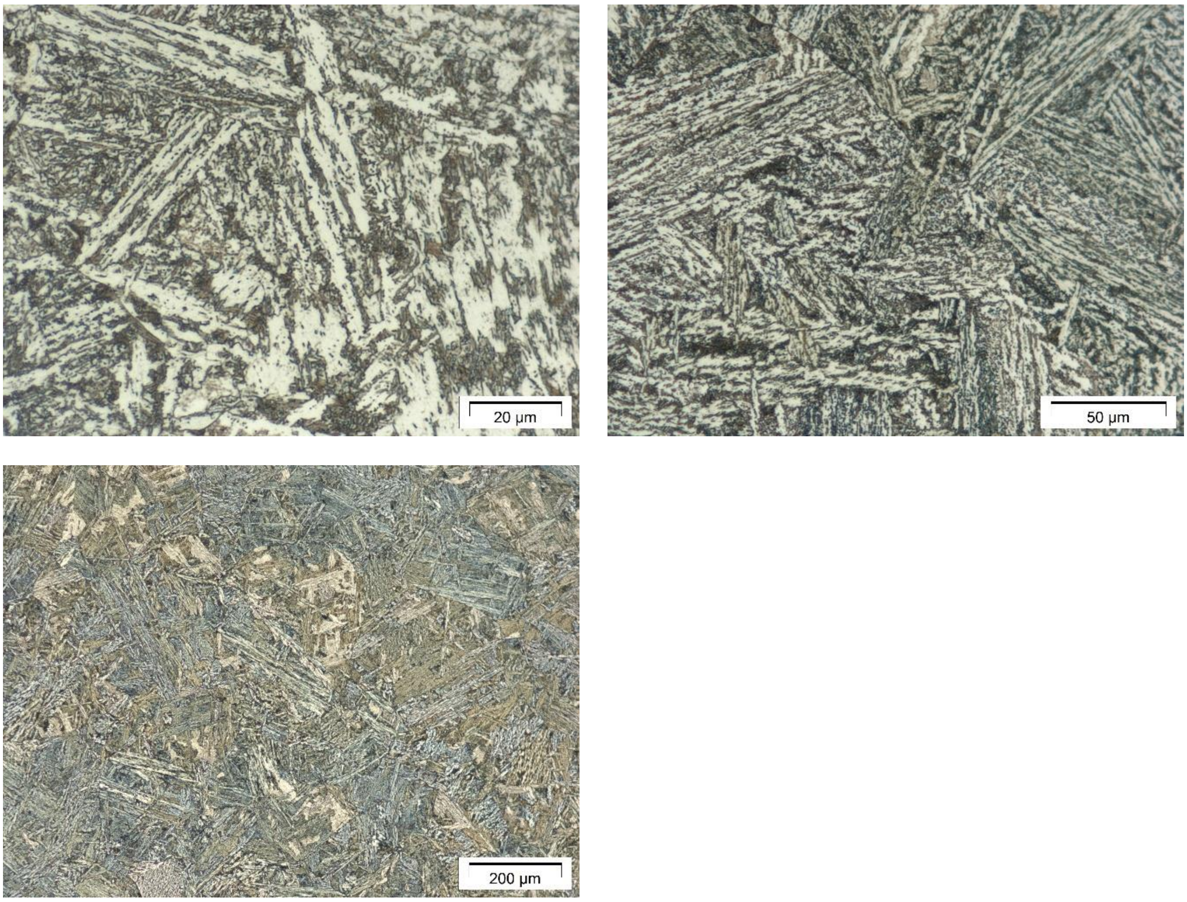


Figure 10: (LOM) Martensitic microstructure observed in 42CrMo4 before an experiment.

The heat treatment of austenite followed by rapid cooling, also known as quenching, is employed to transform the steel into martensite. Martensite is a metastable phase of steel that exhibits high hardness and brittleness. The microstructure of martensite given above is characterized by a needle-like or plate-like pattern, which was observed under a light optical microscope (LOM). This pattern is formed due to the transformation of the crystal structure during the quenching process. 42CrMo4 steel is specifically chosen for engineering applications due to its favorable combination of properties. The presence of chromium and molybdenum in this alloy contribute to its high strength, toughness, and wear resistance. These properties make it suitable for applications where high mechanical loads, impact resistance, and resistance to wear are required [43]. The specimen showed good hardness at 297.46 VH after the test.

3.2. Hydrogen diffusion and trapping

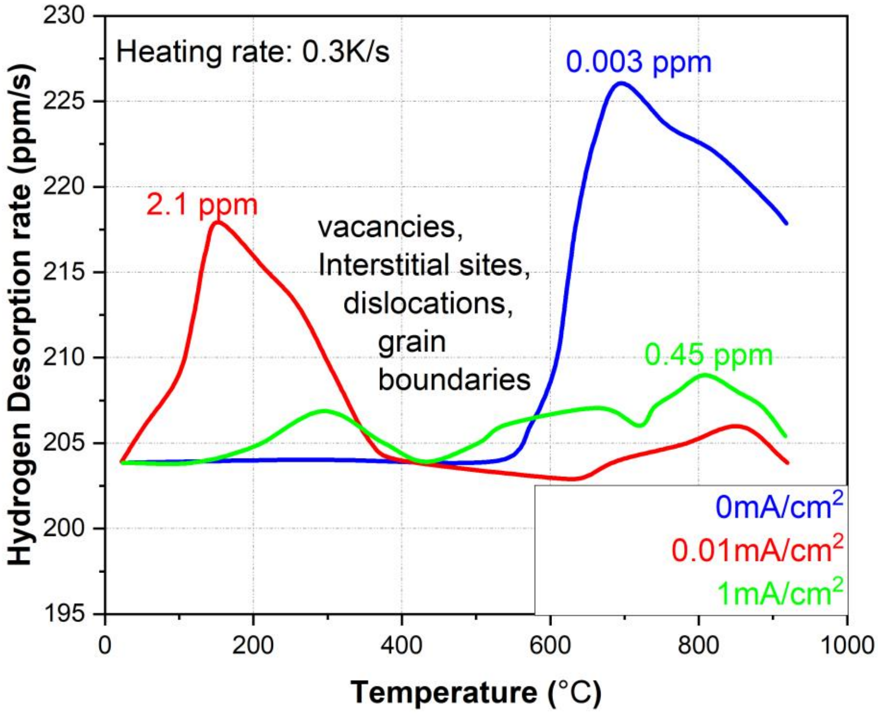


Figure 11: Hydrogen desorption curves of SSRT test with current density of 0, 0.01 and 1mA/cm^2.

Figure 11 illustrates the hydrogen desorption curves at the constant heating rate of 0.3K/s starting at RT to maximum temperature of 900˚C. The first peak at each curve highlighted with a low temperature is associated with hydrogen desorption from lattice defects where the hydrogen trapping energy is relatively low. These defects include vacancies, interstitial sites, dislocations, and twin and grain boundaries. The second-highest temperature peak in the red legend is associated with high hydrogen concentration, such as segregation interfaces and inclusions. The amounts of hydrogen under each desorption peak were demonstrated in the figure using Origin software. The amounts of diffusive hydrogen (H_{Diff}) were determined as 2.1 and 0.45 ppm in the 0.01 mA/cm^2 and 1 mA/cm^2 charged, respectively. In comparison to the diffusive hydrogen, the amounts of trapped hydrogen were not calculated in this work. The trapped hydrogen is assumed to be associated with inclusions and 42CrMo4 segregations, which were frequently observed in the investigated material and discussed in a previous work [69]. The amount of hydrogen diffused also known as hydrogen content is shown in figure 11. below with-it corresponding peak and temperature at 285, 720, and 830˚C

3.3. Mechanical properties

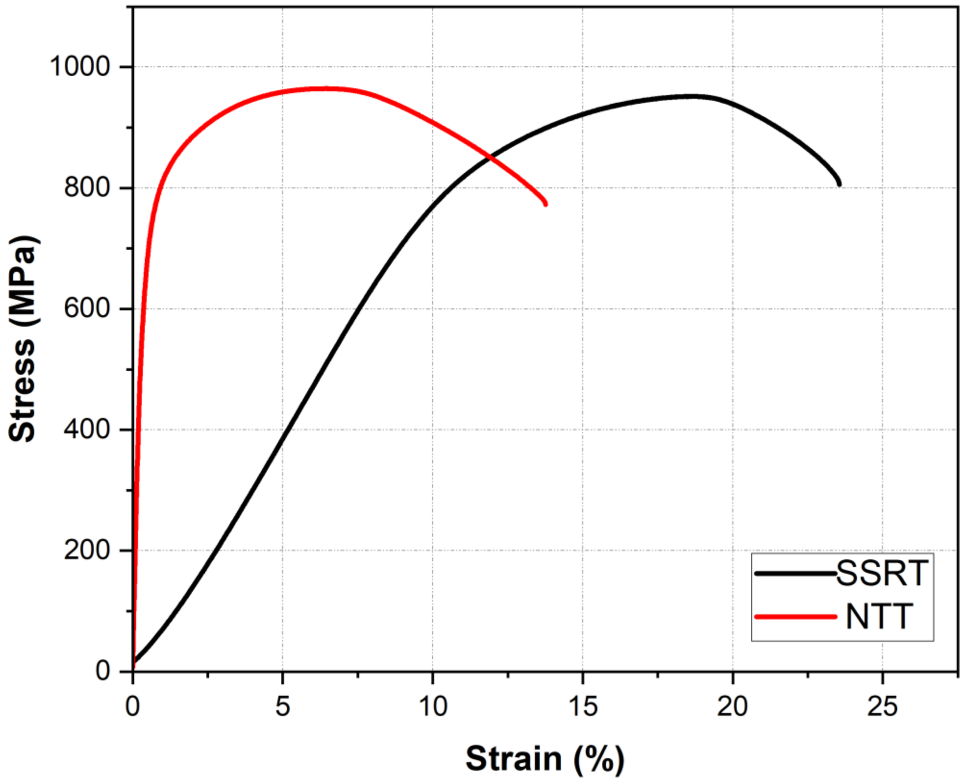


Figure 12: Engineering stress–strain curves of 42CrMo4 steel from SSRT and NTT

Tensile tests were performed on a smooth, rounded cylindrical shape without hydrogen charge on the specimens of each grade of 42CrMo4 steel. Figure 12 shows the stress-strain curves obtained with grades martensitic (A + Q). There is a significant difference between the two observed tests carried out at the same strain rate of $5x10^{-6}$ s^{-1} before the fracture strain. The slow strain rate has higher toughness, elongation, and energy absorbed than a normal tensile test. Both of them experience fracture at the region of plastic deformation.

3.4. Hydrogen effect on tensile properties and fracture mode

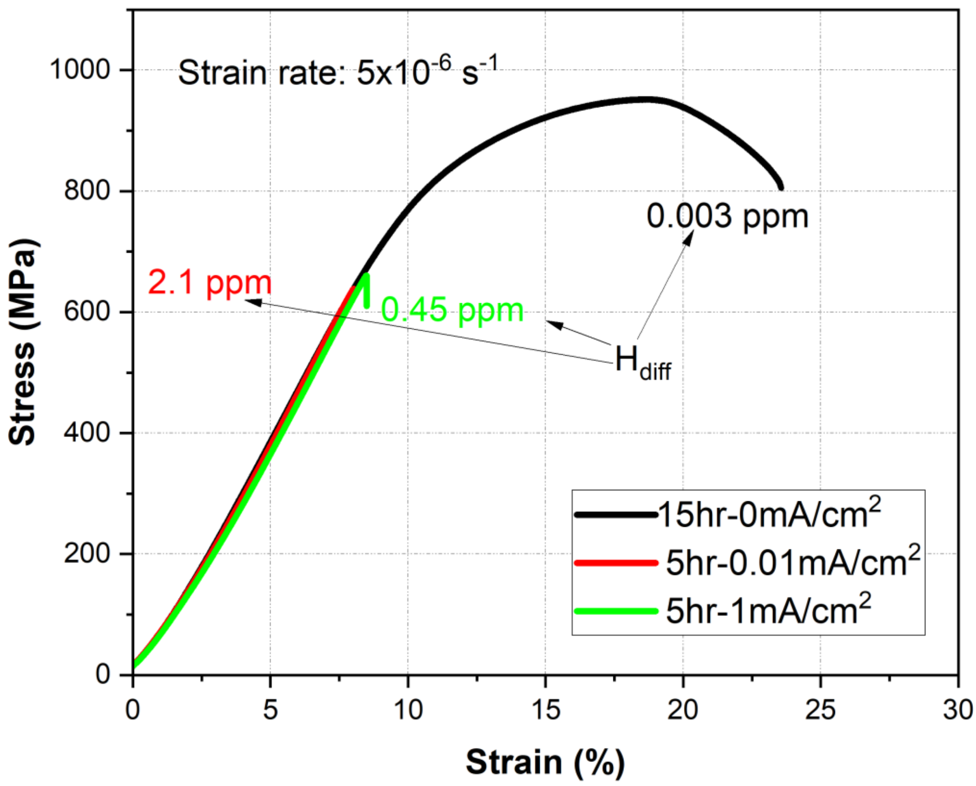


Figure 12: Engineering stress–strain curves in 42CrMo4 steel from slow strain rate test

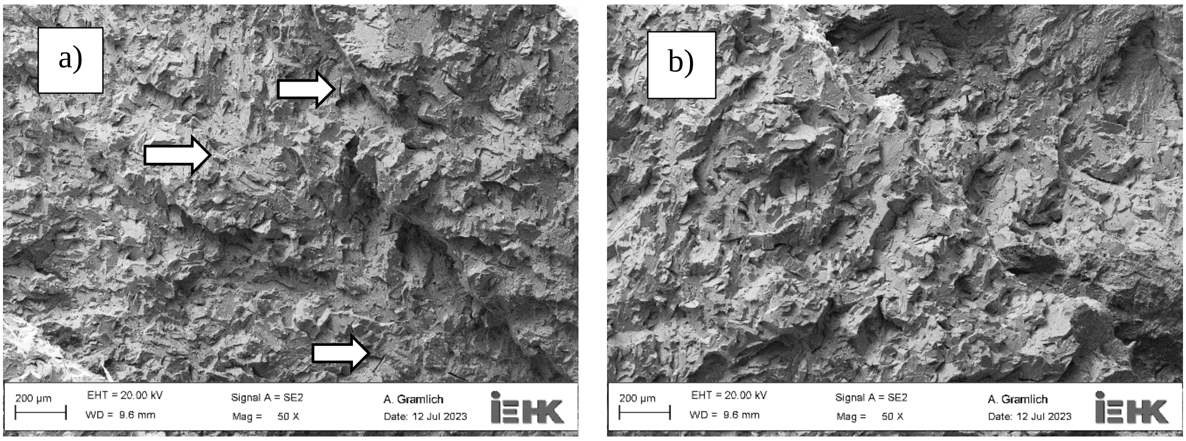
The Figure 12 shows the SSRT engineering stress-strain curves of the 42CrMo4 steel with different amounts of pre-charged and non-charged. SSRT is a test method used to evaluate a material's susceptibility to stress corrosion cracking in certain environments. A significant reduction of the engineering fracture strains was observed in hydrogen-pre-charged specimens compared to the uncharged ones. These results indicate the mechanical properties of the material when it is not subjected to any external current (0 mA/cm^2). The UTS represents the maximum stress the material can withstand before it fails in tension, and it is 951.86 MPa for the uncharged sample, while the in-situ charged samples are 638.63 MPa and 660.63 MPa, with a strain rate of 5x10^{-6} s^{-1}. The %EL indicates how much the material can deform before breaking; in this case, it can elongate by approximately 23.55% before failure for a normal tensile test. The result also denoted that the NTT has higher toughness, elongation, and energy absorbed than the SSRT, with ductile fracture on the surface of the work sample, while the charged sample manifested brittle fracture. The investigated material showed high susceptibility to hydrogen embrittlement.

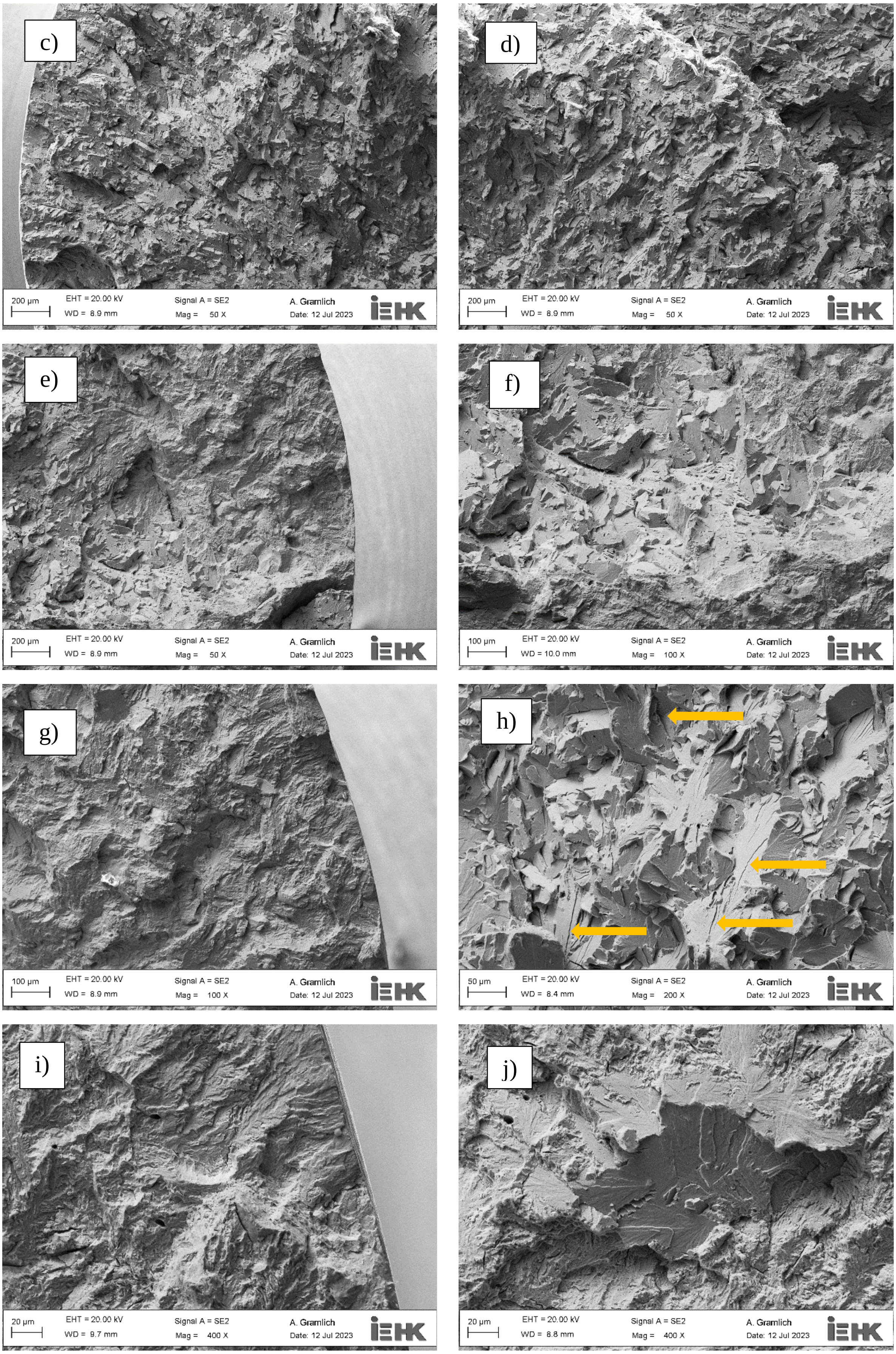
Table 3: Mechanical properties of martensitic samples, uncharged hydrogen at different strain rates, respectively.

Test $5x10^{-6}s^{-1}$	Environment	Fracture Toughness (KJ/m^3)	YS (MPa)	UTS (MPa)	Hardness (VH)	EL (%)
NTT	Air	…..	692.1547	964.5423	301.42	13.48697
SSRT	Air	160479.7	688.43	951.86	297.46	23.55
SSRT	H_2SO_4 + $0.01mA/cm^2$	24,695.56	402.34	638.63	199.57	8.05
SSRT	H_2SO_4 + $1mA/cm^2$	26,746.79	420.65	660.63	206.45	8.49

The toughness was calculated using the origin software by integrating the area under the curve for uncharged specimen the area falls under plastic region while the in-situ charged samples the area falls under elastic region.

3.5. Surface fracture of 42CrMo4 under Scanning Electron Microscope (SEM)




c)
200 µm
EHT = 20.00 kV
WD = 8.9 mm
Signal A = SE2
Mag = 50 X
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Date: 12 Jul 2023
d)
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WD = 8.9 mm
Signal A = SE2
Mag = 50 X
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Date: 12 Jul 2023
e)
200 µm
EHT = 20.00 kV
WD = 8.9 mm
Signal A = SE2
Mag = 50 X
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Date: 12 Jul 2023
f)
100 µm
EHT = 20.00 kV
WD = 10.0 mm
Signal A = SE2
Mag = 100 X
A. Gramlich
Date: 12 Jul 2023
g)
100 µm
EHT = 20.00 kV
WD = 8.9 mm
Signal A = SE2
Mag = 100 X
A. Gramlich
Date: 12 Jul 2023
h)
50 µm
EHT = 20.00 kV
WD = 8.4 mm
Signal A = SE2
Mag = 200 X
A. Gramlich
Date: 12 Jul 2023
i)
20 µm
EHT = 20.00 kV
WD = 9.7 mm
Signal A = SE2
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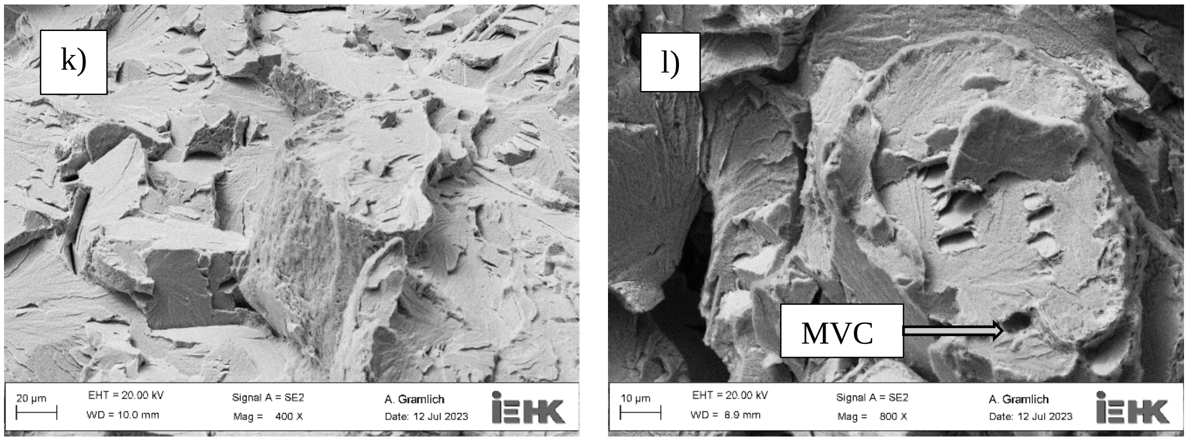

Figure 13: Fracture surface of 42CrMo4 steel with SEM and tensile properties at 0.01mA/cm^2.

The fracture surfaces of the fractured tensile specimen are demonstrated in the above figure at a condition of 0.01 mA/cm^2 under a slow strain rate test. The fracture surface was observed through Scanning Electron Microstructure (SEM) with different magnifications to thoroughly observe the nature of the crack from initiation to propagation in all orientations. Figures (a, b, c, d, g, I, j, and k) highlight the effect of hydrogen embrittlement on the work piece; a mixture of brittle fracture mode with intragranular cleavage and shear fracture with large separations can be observed. The microvoid coalescen (MVC) can be seen in figure (I), which demonstrated that as the material undergoes tensile deformation, microvoids can form and grow. These microvoids may coalesce and contribute to the overall fracture process. A mixture of intragranular cleavage and shear fracture with large separations can be observed. label inside the position of brittle. A change in fracture micromechanics is also observed for the hydrogen in-situ charging sample at 0.01 mA/cm^2. However, as shown in figure (13h), the fracture surfaces of the martensitic samples are characterized by a mixture of two mechanisms: intergranular fracture and decohesion [69]. The intergranular fracture normally occurs when the slip systems intersect prior austenite grain boundaries is a condition where crack occurred at the along the grain boundaries. Decohesion always depends on local hydrogen concentration and stress in the interface with dislocation slip and therefore hydrogen transport. The yellow arrows indicate secondary cracking is observed along the austenite grain boundaries of the fracture surface. It is also attributed to a high localized hydrogen concentration at these interfaces located ahead of the primary crack, where hydrostatic tensile stress is at its maximum. The white arrows in figure a demonstrated transgranular fracture. Some researchers have demonstrated that secondary cracking in martensitic steel could be triggered by hydrogen-induced deformation twins impinging on grain boundaries [70]. Overall, the fracture surfaces of the fractured tensile specimen under hydrogen embrittlement conditions show a mixture of intragranular cleavage, shear fracture, and decohesion. These different fracture mechanisms are influenced by factors such as hydrogen concentration, hydrogen diffusive, stress levels, and the presence of martensitic microstructures. The presence of microvoid coalescence and secondary cracking highlight the complex nature of hydrogen embrittlement and its effects on the fracture behavior of the material.

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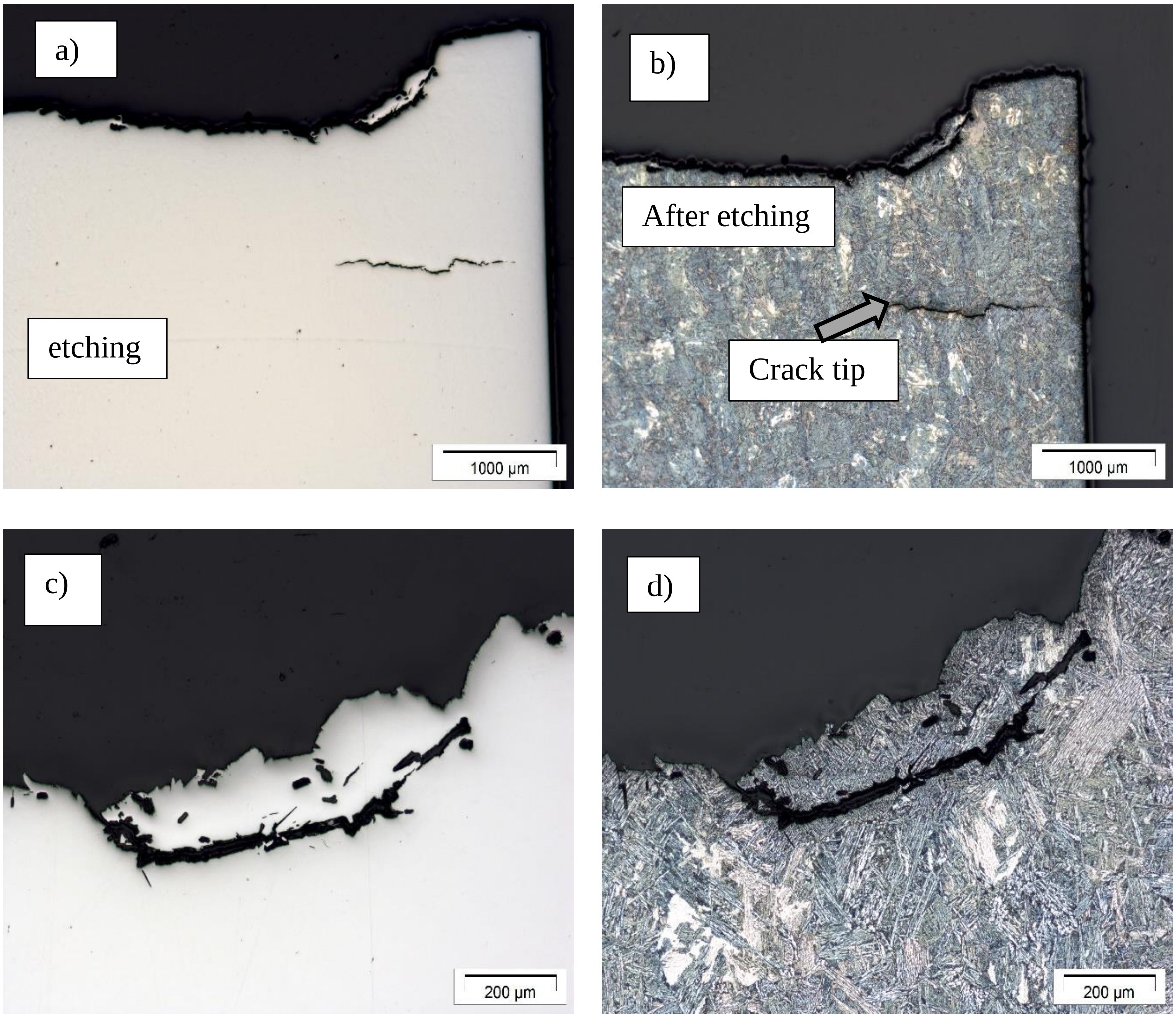

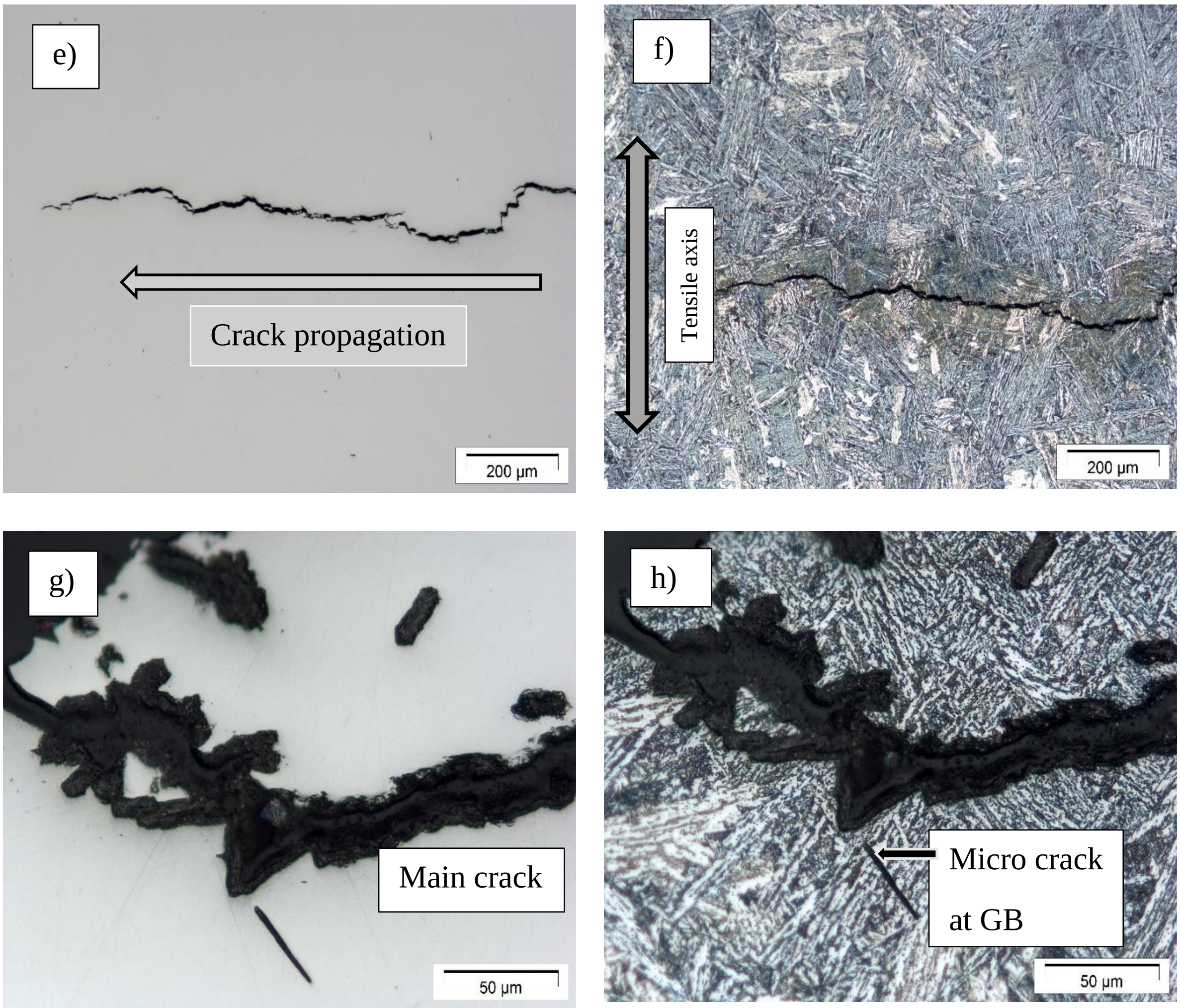

Figure 14: Microstructure of 42CrMo4 after SSRT under light optical microstructure (LOM) at different magnifications.

Figure 14. shows LOM images around the main crack. The main crack on that figure (14e and 14g) was initiated at the corners of the side edges of the specimen and propagated to the other side of the specimen. The direction of the macroscopic crack propagation was approximately perpendicular to the tensile axis as shown in figure (14f). Additionally, we followed the main crack path in our microstructure analysis to understand its propagation mechanism. The micro-cracks were also observed to investigate further crack initiation sites and to better understand the fine details of the crack propagation

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3.6. Comparison of the results of SSRT

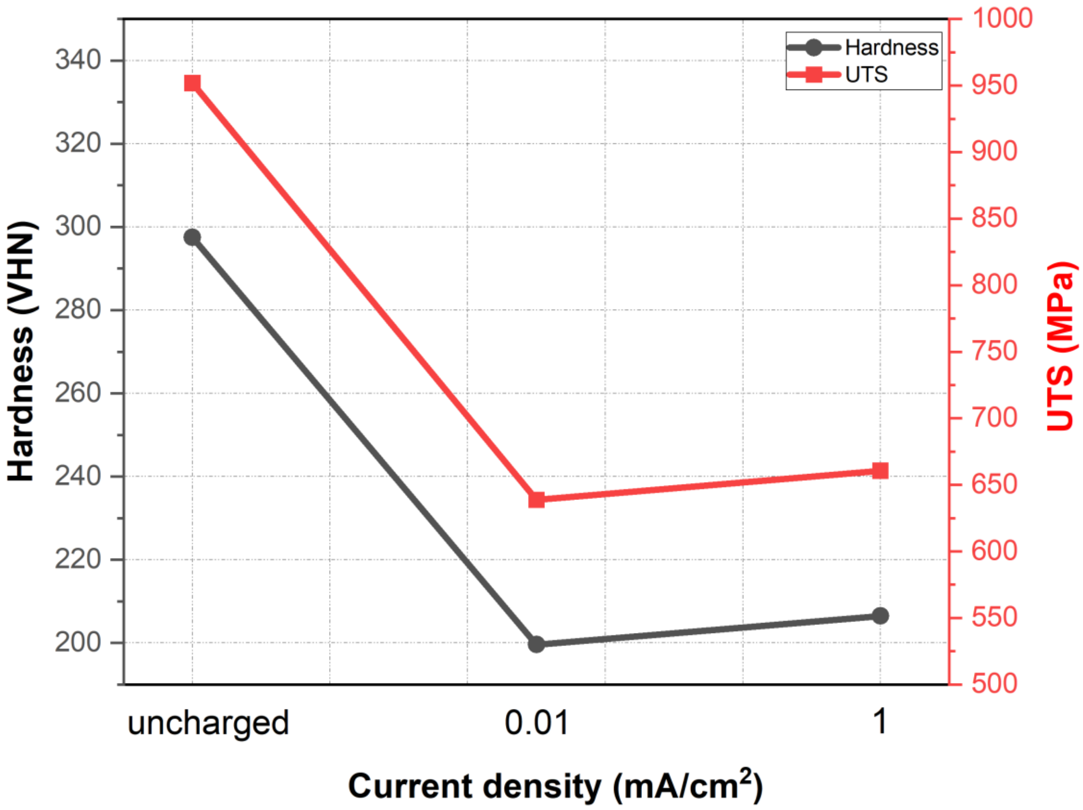

Figure 15: Current Density-Stress-Hardness

The test result above showed the behavior of an uncharged sample under stress rupture conditions. The ultimate tensile strength of 951.86 MPa indicates the maximum stress the sample can withstand before failure. In test-2, the sample was charged and subjected to stress rupture testing in an acidic environment of H_2SO_4. The ultimate tensile strength (UTS) of 638.63 MPa is lower than that of the uncharged sample, indicating that the sample's strength is reduced when exposed to the corrosive environment under slow strain rate test at current density of 0.01mA/cm^2. The final test-3 sample was charged and subjected to a higher current density 1mA/ cm^2 in the H_2SO_4 environment. The ultimate tensile strength of 660.63 MPa is slightly higher than in test-2, indicating that the higher current density might not significantly affect the material's strength. Additionally, while high current densities may enhance hydrogen diffusion and uptake in some cases, they can also lead to detrimental effects such as increased susceptibility to hydrogen-induced cracking or embrittlement. Overall, as the current density increases, the ultimate tensile strength decreases to some extent, but the average of the trends observed seems convincing. [70] also agreed that an increase in current density decreases with hydrogen-charged ultimate tensile strength. Test-2 results revealed that at 0.01 mA/cm^2, the sample has the lowest hardness value in an in-situ charged specimen of the steel, which shows some dimples as shown in figure 15, and stated in [71]. The study concluded by [70] mentioned that the internal hydrogen increases the mobility of dislocation at the point of lower stress, enabling plasticity localization in the region of lower diameter. This phenomenon increases over and over, giving rise to the formation of small voids into larger ones, as shown in figure 18 (I).

It was observed that in figure 15 as the ultimate tensile strength decreases, the Vickers hardness also tends to decrease. This indicates that there is a negative correlation between ultimate tensile strength and hardness. In other words, as the material becomes less resistant to deformation under tension with lower Ultimate tensile strength, it also becomes less resistant to penetration with lower hardness. The hydrogen concentration plays a role in reducing the mechanical properties of the material. In test-2 and test-3, where the samples were charged with hydrogen during the test, we can see that the ultimate tensile strength decreased compared to the uncharged sample in test-1. This is likely due to hydrogen embrittlement, where hydrogen atoms can diffuse into the material's lattice and cause localized weakening, leading to lower ultimate tensile strength and hardness. Furthermore, test-2 and test-3, we have seen that increasing the charging current from $0.01mA/cm^2$ to $1mA/cm^2$ resulted in a slight increase in ultimate tensile strength and hardness. This suggests that at higher charging currents, the hydrogen embrittlement effect may become less severe, leading to a relatively higher ultimate tensile strength and hardness compared to lower charging currents.

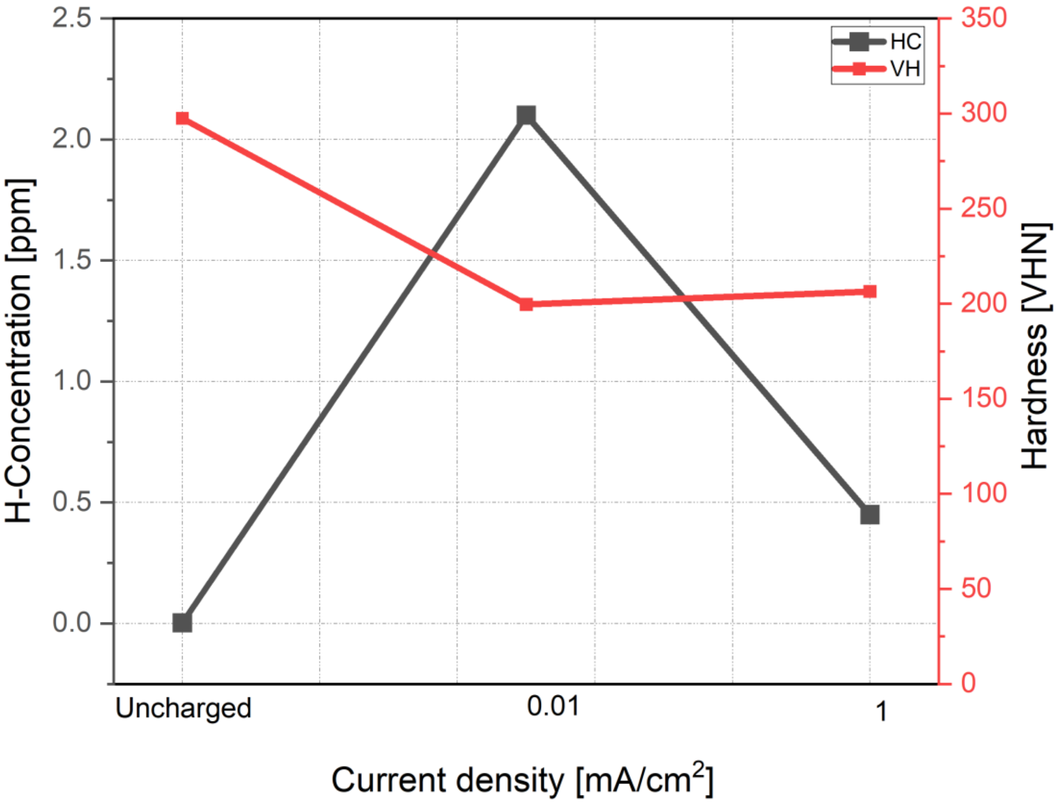

Figure 16: Comparative plot of hydrogen concentration, Vickers hardness, and current density.

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As the current density increases, the hardness of the surface layer also increases. This is because higher heat treatment temperature result in more intense electrochemical reactions at the surface, leading to a higher concentration of hardened phases like martensite or other hard precipitates. Consequently, the hardness of the treated surface increases. The time of exposure to the electric current during the treatment also influences the hardness. Longer treatment times allow for deeper diffusion of carbon (in 42CrMo4) into the surface, leading to increased hardness in a thicker layer. The chemical composition of the material, including its carbon content, alloying elements, and microstructure, plays a vital role in determining hardenability. For 42CrMo4, its alloying elements contribute to its hardenability, and the presence of chromium and molybdenum promotes the formation of hard phases during Electrochemical Hardening. As seen in test-1 and 3, the hydrogen concentrations are relatively low 0.003 ppm and 0.45 ppm, respectively. Test-2, on the other hand, shows a significantly higher hydrogen concentration of 2.1 ppm, indicating substantial hydrogen charging in the material. In tests-1 and 3, the hardness values are relatively higher 297.45VH and 206.45VH, respectively, as compared to those of test-2 which is 199.57VH. The lower hardness observed in this test might be attributed to potential hydrogen embrittlement effects, as a higher hydrogen concentration is associated with reduced mechanical strength and hardness [71]. The results suggested that in-situ charge test sample exhibited a significant increase in hydrogen concentration and a decrease in material hardness, which may be indicative of hydrogen embrittlement effects. Similar phenomenon of the embrittlement has been reported for several steels' grades with varying strengths, hardness, and microstructures [72].

Our study revealed that due to increase in current density from 0 to 0.01mA/cm^2 there was a rapid increased of hydrogen diffusivity from 0.003 ppm to 2.1 ppm which is due to the significant increase in hydrogen concentration that is attributed to the process of hydrogen charging, and specimen is exposed to hydrogen-containing environments, such as in the presence of acids like H_2SO_4. The hydrogen atoms from the acid solution diffused into the material's microstructure and accumulate. Moreover, the applied current during the slow strain rate test enhanced the hydrogen diffusion process, leading to a higher concentration of hydrogen within the material. The high concentration of hydrogen within the material can lead to hydrogen embrittlement, a phenomenon where the absorbed hydrogen atoms create internal pressure, leading to micro-cracking and reducing the material's ductility and mechanical properties and lead to sudden and catastrophic failures under stress. But as the current density increases more to 1mA/cm^2 there was not that significant increment as compared to the reference once.

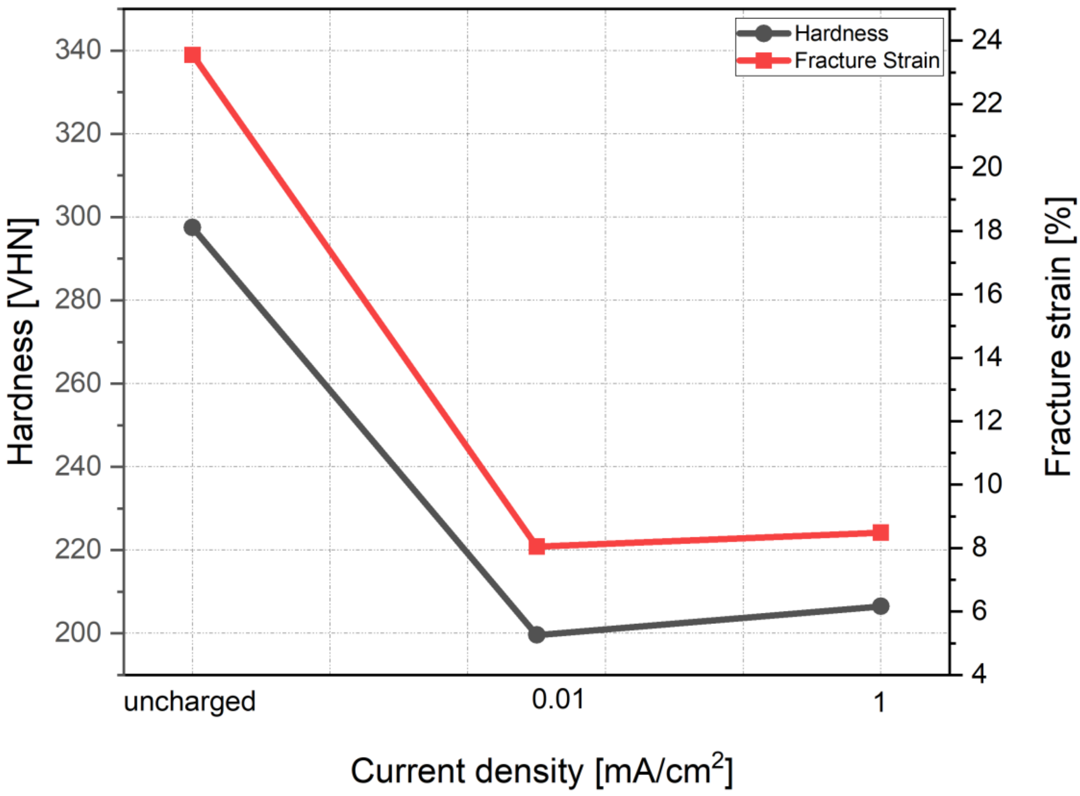


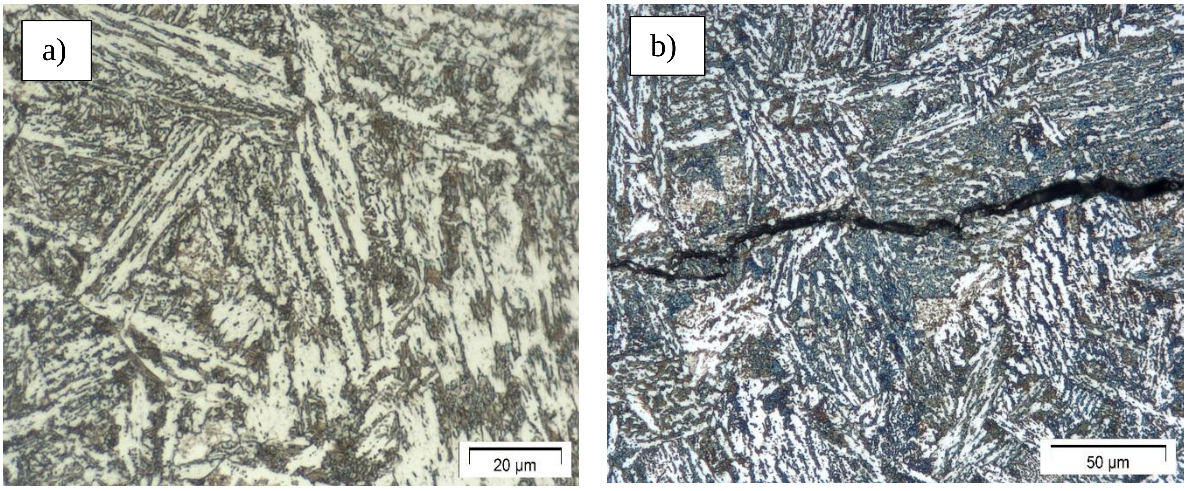
Figure 17: Susceptibility (fracture strain/Hardness) vs Current density

As the fracture strain decreases, the Vickers hardness tends to decrease as well. This indicates that there is a negative correlation between fracture strain and hardness. In other words, as the material becomes less ductile, it also becomes less resistant to penetration. hydrogen charging at different current densities seems to affect both the fracture strain and hardness of the samples. In test-2 and test-3, where the samples were charged with hydrogen during the test, we can see that the fracture strain decreased compared to the uncharged sample in test-1. This suggests that hydrogen charging contributes to a reduction in the ductility of the material, making it less capable of undergoing plastic deformation before fracture. [72] also found similar results which reveal a drastic loss of fracture strain for all hydrogen-charged specimens when compared with the uncharged reference conditions. Moreover, test-2 and test-3, we saw that increasing the charging current from 0.01mA/cm^2 to 1mA/cm^2 resulted in a slight increase in fracture strain. Other studies such [73] suggested that the enhanced evolution of hydrogen at high current densities is responsible for the formation of larger crystals and the unusual low tensile fracture strain. From the figure 17. However, test-2 and test-3 show that raising the current density to 0.01 mA/cm^2 resulted in a 33% reduction in hardness loss. Further increasing the current density led to a slight improvement in hardness, with a 3% increase. This indicates that at higher charging currents, the negative effect of hydrogen charging on ductility and hardness may become less pronounced, leading to relatively higher percentage elongation and hardness compared to lower charging currents. This material conceded more hydrogen concentration than the rest of the test samples and proved to have less fracture strain, hardness, and toughness at 24, 695.56 kJ/m^3.

3.7. Synergistic effect of current density and hydrogen concentration

As explained above the increase of current density to 0.01mA/cm^2 has led to dramatic increase in hydrogen concentration from 0.003 ppm to 2.1 ppm. This impact has showcased the dropped of mechanical properties such as yield strength and ultimate tensile strength with losses of 42% and 33% respectively. This hydrogen concentration is in the same range as those reported by Conde et al. [74].

3.8. Effect of hydrogen concentration on microstructure



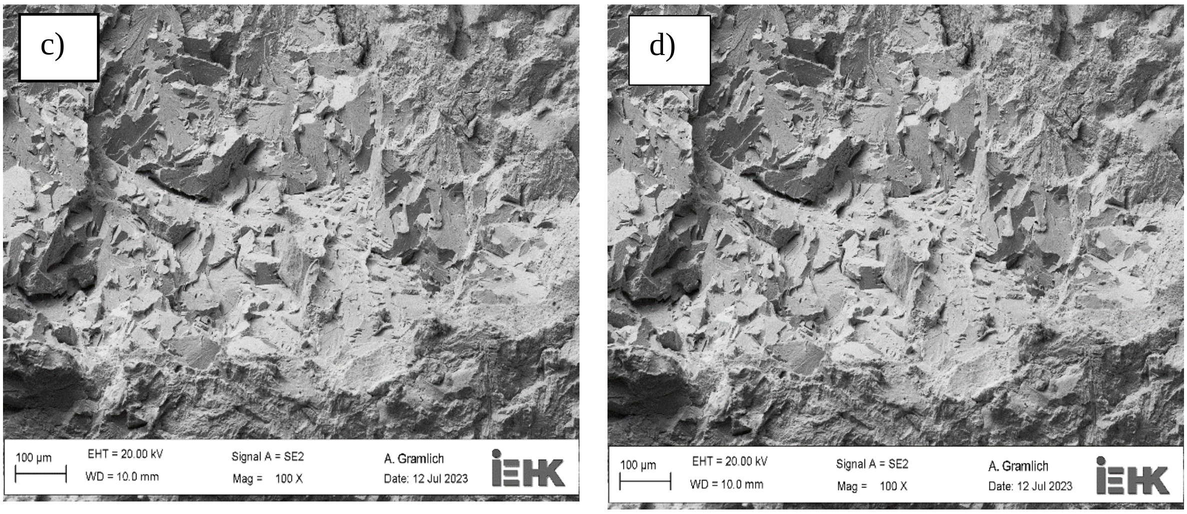


Figure 18: Comparative microstructure of uncharged, charged and fracture surface at 0.01 mA/cm^2

The effect of hydrogen concentration was observed in microstructural changes and fracture behaviour. It is seen that charged sample at 0.01 mA/cm2 does not showed significant changes in terms of its microstructure it remains martensitic characterized by a needle-like or plate-like pattern observed under a Light Optical Microscope (LOM) in figure (18b) even though there was high uptake of hydrogen concentration to 2.1 ppm as compared to uncharged sample. In test-2, the mixture of brittle fracture mode, intragranular cleavage, and shear fracture with large separations on the fracture surface indicates that hydrogen embrittlement has played a significant role, also being noted in figure (18c and d). The brittle fracture initiation at the corners of the side edges, along with the perpendicular propagation to the tensile axis observed under LOM, is indicative of brittle fracture due to hydrogen embrittlement. As seen in figure (18b). The consistency in microstructure between the two tests suggests that the microstructural changes might not be the primary cause of the observed differences in fracture behavior. Both tests exhibit a similar martensitic microstructure, but the key difference lies in the hydrogen concentration and resulting fracture behaviour. The higher hydrogen concentration in test-2, along with the observed brittle fracture characteristics, suggests that hydrogen embrittlement has occurred. The fracture behavior and hydrogen-induced embrittlement effects seem to be consistent with the observed microstructural characteristics in both tests.

3.9. Conclusion

This master's thesis studies the investigation of hydrogen embrittlement on high-strength steel, 42CrMo4. To investigate the influences of mechanical properties on hydrogen embrittlement susceptibility, the experiment was basically based on current density, and it was subdivided into three uncharged specimens and two charged samples at a current density of 0.01 and 1 mA/cm2, both of which were conducted through in-situ hydrogen charging. The tensile results and the computed parameters are stated in Table 6. The deformation behavior of the steel was also affected by hydrogen charging. The uncharged specimens exhibited primarily brittle fracture, with some areas of localized plastic deformation. In contrast, the charged specimens showed evidence of hydrogen-induced cracking and transgranular fracture. The results of this study confirm that high-strength steel, such as 42CrMo4, is susceptible to hydrogen embrittlement. The susceptibility increases with increasing current density, indicating the importance of controlling the charging conditions to mitigate the risk of hydrogen embrittlement.

The tensile test conducted under normal air conditions at room temperature showed that the fracture occurred at the point with the smallest diameter. It was also observed that during the slow strain rate test, necking started at this point and continued until the specimen broke. The point with the smallest diameter not only allowed necking, but also allowed the diffusion of hydrogen molecules into the specimen due to high stress in the region. This phenomenon was found to increase under external loads, which were governed by hydrostatic stress [74]. The premature failure of the workpiece was attributed to the high accumulation of hydrogen in this region [75].

GENERAL CONCLUSION AND OUTLOOK

GENERAL CONCLUSION AND PERSPECTIVE

This comprehensive study focused on the Investigation of the Response of 42CrMo4 to hydrogen loading through tensile testing, a profound understanding of the intricate relationship between electrochemical processes, hydrogen exposure, and the mechanical properties of the alloy steel has been achieved. The series of tests conducted under slow strain rate conditions provided a wealth of valuable data. Test-1, involving an uncharged specimen, exhibited the material's inherent mechanical prowess, showcasing high tensile strength, appreciable hardness, and significant elongation. The martensitic microstructure observed underscored the alloy's intrinsic strength and resilience. As the current density increased in test-2 and 3, hydrogen-induced embrittlement became apparent. The altered mechanical properties, characterized by lowered UTS, reduced hardness, and limited elongation, accompanied by brittle fracture modes and unique fracture surface features, underscored the susceptibility of 42CrMo4 to hydrogen embrittlement. Even at lower hydrogen concentrations, the material's integrity was compromised, highlighting the importance of current density in influencing hydrogen embrittlement behaviour.

The knowledge gained from this study directly informs material design and engineering strategies, offering guidelines to create components that can withstand the challenges posed by hydrogen-rich environments. From aerospace to energy sectors, this research provides tools to enhance the durability and reliability of critical structures. the research has successfully addressed the research problem by systematically investigating the impact of current density on 42CrMo4 alloy steel through hydrogen loading. The findings provide a holistic understanding of the intricate interplay between electrochemical conditions, hydrogen exposure, and mechanical properties. As industries strive for improved materials in the face of evolving challenges, the insights obtained from this study form a solid foundation upon which to build more robust, hydrogen-resistant materials and components. The implications span from theoretical advances in materials science to practical applications, ensuring safer and more reliable structures in an ever-demanding world.

GENERAL CONCLUSION AND PERSPECTIVE

RECOMMENDATIONS

This study revealed that with increase in current density the steel absorbed more hydrogen concentration leading to significant effect on material mechanical properties. Thus, based on the findings and the conclusions presented, the following recommendations are hereby suggested:

1. **Expanded Sample Size and Diversity:** Future studies could involve a larger and more diverse set of samples, encompassing variations in material conditions, microstructures, and testing environments. This could provide a more comprehensive understanding of the material's behavior and the impact of current density.
2. **Real-World Loading Conditions:** Considering the limitations of slow strain rate tests, researchers could explore the effects of hydrogen loading and current density under different loading conditions, such as cyclic loading or dynamic loading, to simulate real-world scenarios more accurately.
3. **Comprehensive Electrochemical Control:** To ensure accurate current density control, researchers could employ advanced electrochemical techniques and equipment to precisely regulate the current during hydrogen loading experiments.
4. **Influence of Alloying Elements:** Investigating the role of specific alloying elements present in 42CrMo4 on hydrogen embrittlement behavior could provide insights into their interaction with hydrogen and their impact on embrittlement susceptibility.
5. **Microstructural Analysis:** Advanced microstructural analysis techniques, such as electron backscatter diffraction (EBSD), could be utilized to correlate microstructural variations with embrittlement behavior and provide a deeper understanding of the mechanisms involved.

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