



## Review

# Effects of different microstructural parameters on the corrosion and cracking resistance of pipeline steels: A review

Meekness Nnoka<sup>\*</sup>, Tonye Alaso Jack, Jerzy Szipunar

Department of Mechanical Engineering, University of Saskatchewan, 57 Campus Drive, Saskatoon S7N 5A9, Saskatchewan, Canada

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## ABSTRACT

Several environmental challenges such as corrosion, low temperature, hydrogen-induced cracking (HIC), stress corrosion cracking (SCC), sulfide stress corrosion cracking (SSCC), and various other failure mechanisms contribute to the deterioration of the mechanical properties of pipeline steels, ultimately resulting in failure. In this review, diverse hydrogen attack sources, their possible failure mechanisms, and various strategies for their mitigation in different environments are explored. Optimizing the microstructure of pipeline steels can greatly improve their resistance to corrosion and cracking. This involves tailoring several microstructural parameters like the phase composition, dislocation density, crystallographic texture, grain size, grain boundary, and inclusions/precipitates, amongst others to the needs of the steel's service environment. The evolving research landscape concerning the role of these microstructural parameters in corrosion, HIC, SCC, and other failure mechanisms was discussed in this study. It was established that crystallographic texture and grain boundary characteristics have roles to play in improving the SCC resistance of pipeline steels. However, the degree to which crystallographic texture, amidst other microstructural parameters, affects cracking is not yet established. For instance, the direct influence of crystallographic texture in the arrest of propagating cracks is still unclear and debated, while low-angle grain boundaries and CSL boundaries have been seen to arrest cracks in steels. It was also established that dislocation density, amongst other microstructural parameters, has a more profound effect on the HIC resistance of pipeline steels. Furthermore, this review examines the effect of hydrogen in pipeline welds. It investigates strategies to adapt existing pipelines' microstructure to meet the demands of operations in arctic environments. It was established that grain size, amongst other microstructural parameters, has a more profound effect on the mechanical properties of pipelines designated for cold applications. Finally, this review explores recent advancements in the transportation of gaseous hydrogen using existing pipeline steels (natural gas infrastructure). Ultimately, this study reinforces the importance of microstructure optimization in pipelines designated for different service environments, detailing the contribution of individual microstructural parameters to their overall performance and failure susceptibility in these environments.

## 1. Introduction

High-strength pipeline steels encounter harsh operational conditions such as corrosive, low temperature, and hydrogen

<sup>\*</sup> Corresponding author.

E-mail address: [ysr538@usask.ca](mailto:ysr538@usask.ca) (M. Nnoka).

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environments, which accelerate structural failures and compromise their integrity. The hydrogen environment, whether aqueous or gaseous, is of grave concern, and the manifestation of cracks in pipelines can be attributed to the uptake of hydrogen into the steel structure. Over the years, research has shown that these vulnerabilities can be mitigated through meticulous control of the microalloying elements and processing parameters during thermomechanical processing (TMCP) while providing a good balance of strength and toughness for the steel [1]. However, it's important to note that elevating pipeline strength can increase its likelihood of failure, which is attributed to significant changes in the microstructural characteristics of the steel [2]. This empirical strength and failure relationship highlights the necessity of attaining a delicate compromise between properties, such as strength, weldability, toughness, corrosion, and cracking susceptibility. Achieving the proper balance requires careful analysis of the specific environmental conditions and intended applications of the pipelines, as well as the relevance of microstructure in determining the susceptibility of pipelines to failure [3–8]. Microstructure plays a pivotal role in the distribution of hydrogen within the crystal lattice, thereby dictating the vulnerability to hydrogen-induced degradation of pipelines. It encompasses a range of parameters such as phase composition, grain size, grain boundary characteristics, dislocation density, and crystallographic texture, all of which have been reported to exert a substantial impact on the corrosion and hydrogen-induced cracking resistance of pipelines [9]. For instance, it has been reported that boundaries between grains having  $\{110\}$  and  $\{111\}$  planes oriented parallel to the pipeline steel surface are more resistant to hydrogen-related degradation [10]. It has been also reported that a large amount of coincident site lattice (CSL) boundaries and low-angle grain boundaries (LAGBs) can improve SCC resistance in pipeline steels [11]. In this review, the role of these microstructural parameters in corrosion, HIC, SCC, and other failure mechanisms has been made known. These microstructural parameters can be optimized to improve pipeline steel performance in different service environments. The microstructural characteristics obtained after thermomechanical processing and microalloying hold the potential to significantly reduce the degradation of steel in different service environments. However, despite extensive work on the subject, HIC and SCC remain the most important failure mechanisms that must be mitigated. Understanding the deteriorating effect of hydrogen and developing pipeline steels having optimum resistance to hydrogen embrittlement (HE) is pivotal and remains the driving force behind this review. Due to the catastrophic failures that often occur because of HE, our focus is mainly on pipeline steel. However, other types of steel are occasionally mentioned. While extensive research has been conducted on the role of microstructure in hydrogen diffusion, the detrimental consequences of hydrogen in pipeline steels, the various hydrogen degradation mechanisms, and contemporary techniques for characterizing microstructures, controversies, and debates persist within the field [12–14]. Apart from microstructural phases, and control, sulfur, and hydrogen sulfide ( $H_2S$ ) are significant impurities in pipeline steels because of their potential to cause corrosion and embrittlement, which can compromise the integrity of the pipeline. Elevated amounts of sulfur can foster the formation of manganese sulfide (MnS) inclusions, which have been reported to cause HIC in pipeline steels [15–18]. While the shape of MnS matters, it has been reported that elongated inclusions have a lesser effect on pipeline steels [19]. There are several ways of controlling the morphology of inclusions. For instance, the use of fine oxide inclusions as heterogeneous nucleation sites by adding titanium, magnesium, and zirconium. Also, the use of calcium or rare earth treatment has been reported. However, there are shortcomings of this method because controlling the calcium sulfide (CaS) content in steel is difficult as it requires a very sensitive range of oxygen content for Calcium treatment.  $H_2S$  on the other hand fosters the ingress of hydrogen atoms into the pipeline structure, by acting as a recombination poison [20]. This review also discusses past and recent techniques used for controlling the morphology of inclusions. Another focus of this study is on the application of TMCP as a means of tailoring the microstructural parameters of pipeline steels to impact crack resistance, while pointing out the use of inhibitors to prevent pipeline failure due to corrosion. Apart from cracking being induced environmentally, the mismatch in deformation between the ferrite and cementite during cold forming can facilitate cracking which seriously deteriorates the fatigue life of spheroidized ferrite-cementite steel (SFC) steels. Wang et al. were able to utilize experiments and multiscale simulations to investigate the damage mechanisms and the dependence of microstructure features under uniaxial tension in SFC steel [21]. The authors found three damage mechanisms in SFC steel under uniaxial tension: cementite cracking, ferrite/cementite interface debonding, and ferrite cracking, after the in situ tensile test, and the matrix cracking was attributed to dislocation pile up or the crossing of multiple slip bands.

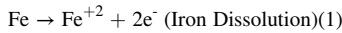
Furthermore, heightened operational demands for high-strength pipeline steel have driven increased research focus toward understanding the inherent failures observed in high-grade pipeline steel welded joints. These failures have significant implications for the microstructure, structural safety, and overall integrity of the welded joints [22,23]. For instance, Chen et al. revealed the effect of welding residual stress (WRS) on the fatigue life of rib-to-deck double-sided welded joints in orthotropic steel decks. The authors observed that WRS has a significant effect on fatigue life, and the fatigue life of the weld toe without considering WRS is about twice that of considering WRS [24]. Moving forward, it is also known that to improve the microstructure and properties of duplex stainless steel welded joints, it is very important that an appropriate  $N_2$  content was added in shielding gas during the DSS CMT-P welding process. Zhang et al. experimentally recommended  $Ar + 4\% N_2$  as the shielding gas to join DSS by using the CMT-P welding technique [25]. It is also imperative to understand the effect of hydrogen in pipeline welds, amidst other possible causes of cracking. As such, this review discusses the effect of hydrogen on the cracking resistance of pipeline welds. Our focus is on pipeline steels; however, other steels were also mentioned in this study. In the end, it outlines recent advances for transporting gaseous hydrogen using pipeline steels not originally designed for such purposes, while shedding light on the recent adaptation of current pipelines to operate effectively in demanding arctic environments. Overall, this study reinforces the importance of microstructure optimization in pipelines designated for different service environments, detailing the contribution of individual microstructural parameters to their overall performance and failure susceptibility in these environments.

## 2. Pipeline corrosion – Overview

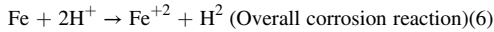
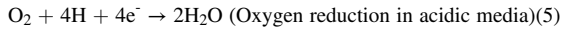
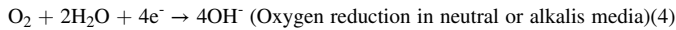
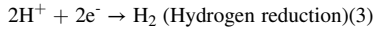
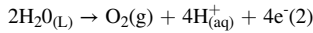
Pipeline corrosion is the backbone of the most environmentally induced failure mechanisms encountered in pipeline steels. It

remains a broad topic to discuss and can more easily be understood by discussing it in parts. Corrosion involves material deterioration due to environmental factors. It is an electrochemical process that involves electron ejection due to metal dissolution at the anodic site, and electron transfer to the cathodic site, resulting in the formation of ions and/or hydrogen evolution [26]. Understanding the reduction–oxidation (redox) reactions that make corrosion possible is most fundamental, and they are given as follows:

#### Anodic Reaction



#### Cathodic Reactions.

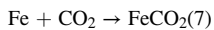


The above corrosion reactions occur in the anodic and cathodic regions of the pipeline. These anode and cathode sites are usually on the surfaces of the pipelines, and in combination with electrolytes, they make corrosion possible.

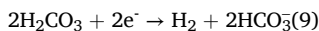
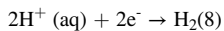
The role of the electrolyte is to complete the corrosion circuit, facilitating the flow of ions. In pipeline steels, corrosion usually occurs in the presence of carbon dioxide (CO<sub>2</sub>) or hydrogen sulfide (H<sub>2</sub>S). All the above corrosion agents are mostly present in oil and gas, and sometimes, with the combination of other corrosive agents like water, sand, microbes, and other organic compounds.

### 2.1. CO<sub>2</sub> corrosion (Sweet Corrosion)

This type of corrosion is also called sweet corrosion because it happens in the presence of CO<sub>2</sub>. Sweet corrosion happens by an acidic reaction where water (H<sub>2</sub>O) reacts with CO<sub>2</sub>. It is called sweet because it is usually less aggressive than sour corrosion (corrosion in the presence of a high amount of H<sub>2</sub>S). In this process, the corrosion agent is H<sup>+</sup> from H<sub>2</sub>CO<sub>3</sub>. CO<sub>2</sub> can be present in pipeline steels due to certain processes like steel recovery. As iron (Fe) reacts with CO<sub>2</sub>, sweet corrosion begins. The reaction is seen below:



H<sub>2</sub>O reacts with the absorbed surface complex, forming Fe<sup>2+</sup> (aq) and H<sub>2</sub>CO<sub>3</sub>, producing the cathodic reactant H<sup>+</sup> during dissociation. To date, the cathodic reaction mechanism for sweet corrosion is a complex reaction. However, it can be approximated as:

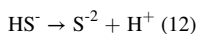
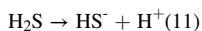


Several interpretations have been made of the sweet corrosion process however, the cathodic reaction mechanism of sweet corrosion is not understood [27].

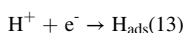
It was inferred that when the pH value is lower than 4, hydrogen reduction becomes the dominant mode for corrosion, while when the pH value varies between 4 and 7, the adsorbed H<sub>2</sub>CO<sub>3</sub> reduction becomes the dominant mode for corrosion [28]. For many oil and gas pipelines, sweet corrosion is not a threat, this is because of the presence of hydrogen sulfide (H<sub>2</sub>S). Sweet corrosion occurs in the absence of hydrogen sulfide (H<sub>2</sub>S).

### 2.2. Sour corrosion (H<sub>2</sub>S)

Unlike sweet corrosion, sour corrosion occurs in the presence of hydrogen sulfide (H<sub>2</sub>S). This is a major concern in the oil and gas industries, as H<sub>2</sub>S is present in the transported substances. In sour corrosion, H<sub>2</sub>S is decomposed into H<sup>+</sup> and HS<sup>-</sup> ions. HS<sup>-</sup> ion acts as a hydrogen recombination poison, limiting the formation of hydrogen molecules. The following reactions occur during sour corrosion:



Hydrogen atoms in the form of protons attract electrons from iron (Fe) and form hydrogen atoms:



The hydrogen atoms which do not recombine to form a hydrogen molecule, because of the recombination poison (HS<sup>-</sup>) are absorbed

**Table 1**  
Different microstructures of the X80 pipeline carbon steel [56].

Heat Treatment	Microstructure
For the as-received specimen	Ferrite and pearlite bands, having dispersed precipitates.
Quenched and tempered specimen	Incomplete recrystallized grains with incipient acicular ferrite and isolated pearlite grains.
Watered-sprayed specimen	Partial transformation of perlite grains with few precipitates.
Water-quenched specimen	Martensite with high segregation at martensitic lath boundaries and dispersion of precipitates.

into the pipeline's lattice.

Upon entering the steel, a lot of regions act as accumulation zones as the hydrogen atoms diffuse through the lattice [29]. They might be trapped at grain boundaries, dislocations, precipitates, inclusions, etc. The hydrogen atoms tend to recombine around defects or strong trapping sites to cause the degradation of mechanical properties, and eventually failure [30]. Also, the sulfide corrosion products passivate the steel at low temperatures, as far as chloride ions and oxygen are absent. When those substances are present, they may form a galvanic couple with the pipeline steel to aggravate the localized corrosion problems [31].

### 2.3. Under-deposit corrosion (UDC)

UDC due to the presence of deposits has become a major threat to the integrity of pipelines in oil and gas fields. UDC refers to the localized corrosion that occurs beneath or around deposits present on a metal surface. These deposits include silica sand, calcite, clays, alumina, corrosion products, organics, etc. [32]. UDC tends to cause the perforation of pipelines and they occur unexpectedly, resulting in unacceptable failures [33]. One of the ways of mitigating UDC in pipelines is by scale controlling, which involves preventing depositing (scale inhibition) and removing the deposits/scales that have already existed on pipeline surfaces [34]. Mitigating UDC by adding specially developed corrosion inhibitors is still promising, however, some inhibitors promote UDC in pipeline steels, therefore specialized inhibitors are recommended.

### 2.4. Microbiologically influenced corrosion (MIC)

MIC involves the pollution of pipeline steel surfaces by microorganisms and the subsequent creation of corrosive oxygen concentration cells [9]. It has been reported that MIC accounts for more than 20 % of pipeline infrastructure failures [35]. Many studies have been made regarding the corrosive actions of sulfate-reducing bacteria (SRB) and nitrate-reducing bacteria (NRB) on metal surfaces, in fostering MIC. SRBs, as anaerobic bacteria, reduce sulfur/sulfur ( $\text{SO}_4^{2-}$ ,  $\text{SO}_3^{2-}$ ,  $\text{S}_2\text{O}_3^{2-}$ , and sulfur) compounds to sulfide ( $\text{H}_2\text{S}$ ) from available carbon sources [36]. When they adhere to pipeline steel surfaces, they mature into a multitude of SRBs due to means of extracellular polymeric substances (EPS) secreted by their biofilms [37]. This fosters the production of  $\text{H}_2\text{S}$ , leading to electrochemical reactions that lead to localized corrosion modes [36].

## 3. Improving the corrosion resistance of pipeline steel

The use of cathodic protection (CP) has been a technique that favors the corrosion resistance of pipeline steels [38]. CP is a system that uses direct electrical current to mitigate the normal external corrosion that occurs on a metal pipeline due to soil and moisture conditions. CP is used where all or part of a pipeline is buried underground or submerged in water. Another method is the use of pipeline coatings and linings, as they protect the bare steel from coming in direct contact with corrosive conditions [39]. Corrosion inhibitors are also used to improve the corrosion resistance of pipeline steels. They can be added to the pipeline substance running through the pipeline to decrease the rate of attack of internal corrosion on the steel. As CP cannot protect against internal corrosion, inhibitors are of particular use in "wet" gas pipelines. Inhibitors like Pectin/nano  $\text{CeO}_2$  (mixture-based corrosion inhibitor) and Hydroxyquinoline-KI (mixture-based corrosion inhibitor) have been proven to reduce corrosion in pipeline steels, respectively [40,41]. The production of corrosion inhibitors poses high cost and time constraints. However, in a contemporary context, He et al. utilized machine learning, revealing the controlled synthesis of carbon dots-based corrosion inhibitors [42]. The optimization process increases time management while reducing cost and contributing to the sustainability and cleaner production of inhibitors.

### 3.1. Stress corrosion cracking (SCC)

Stress corrosion cracks are formed from the combined effect of sustained tensile stress and corrosion. The surface corrosion type can be either sweet or sour, while the tensile stress can be residual or externally applied. Pipelines are prone to SCC because of their corrosive environment and tensile stress which facilitates cracking. As such, SCC is often called environmentally assisted cracking (EAC) and has always been of major interest to gas pipeline manufacturers [20,43–45]. The SCC resistance of pipelines depends on their microstructure [46–50]. Different microstructural parameters influence the stress corrosion cracking susceptibility of pipeline steels [9]. Abubakar et al. reviewed several factors affecting SCC initiation and propagation in carbon steels [51]. Several types of microstructural phase constituents are observed in pipeline steels such as austenite, pearlite, ferrite, bainite, and martensite. The cooling rate applied during processing and the nucleate contents determine the different forms of ferrite grains that will be nucleated such as acicular ferrite, polygonal ferrite, allotriomorphic ferrite, globular ferrite, and idiomorphic ferrite [52]. Among these

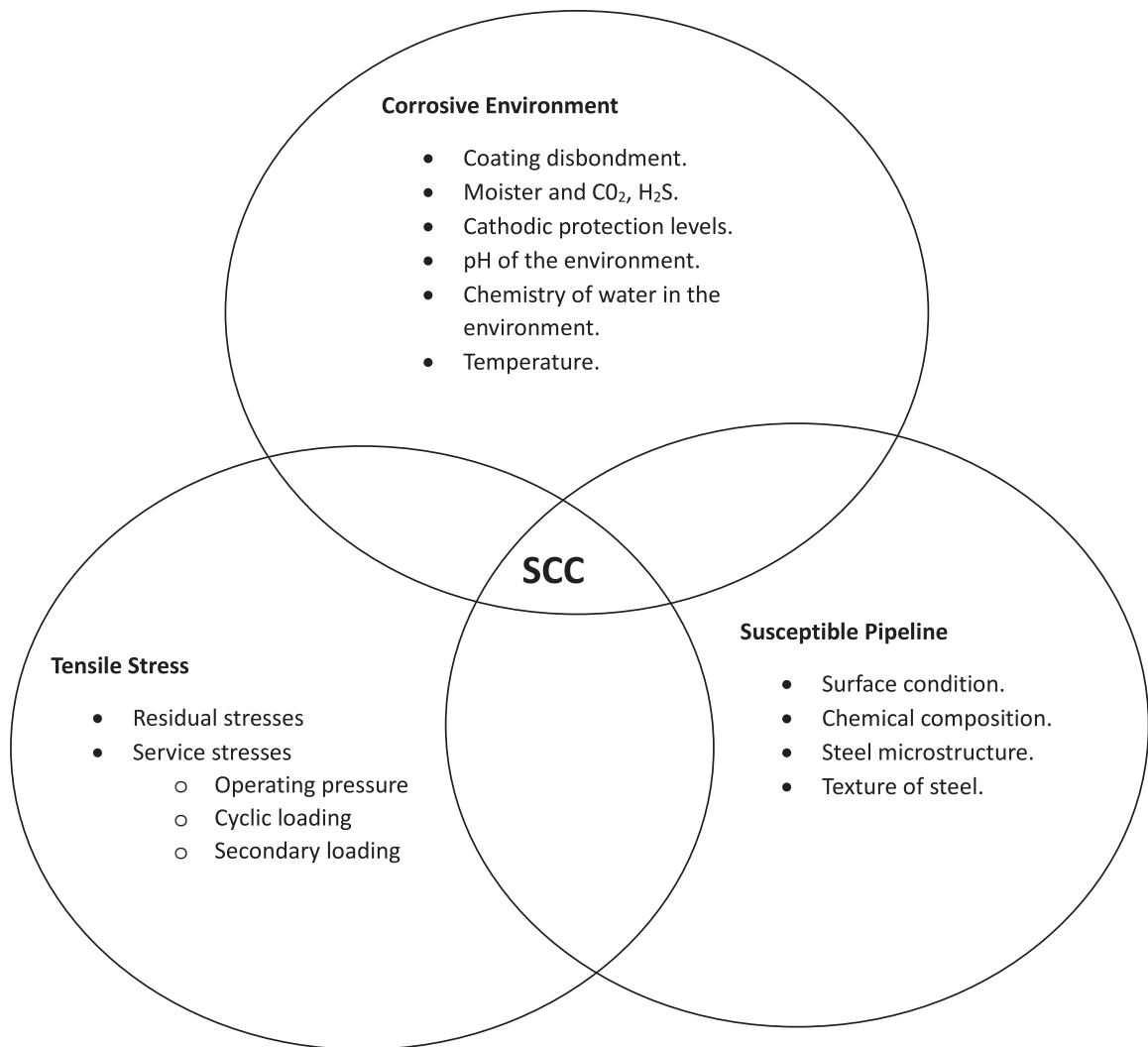
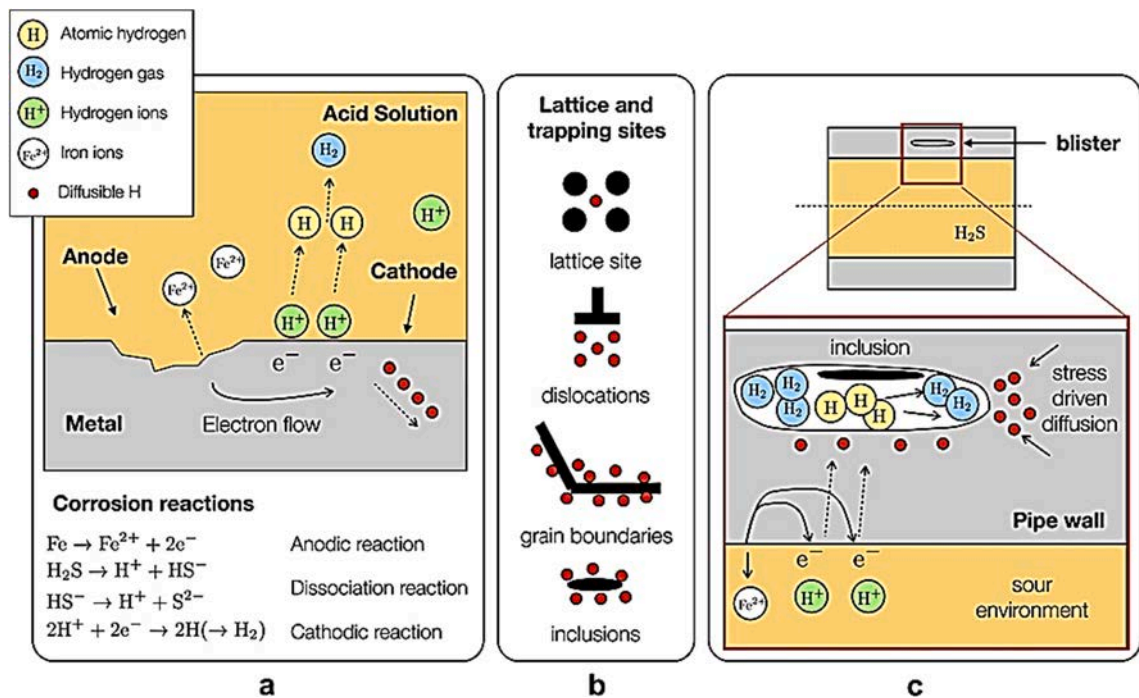


Fig. 1. Factors affecting SCC crack initiation and propagation in pipeline steel [27].

microstructures, acicular ferrite, which is a thin needle-like form of ferrite grain, possessing high dislocation density due to its fine-grained nature, has been understood to possess better SCC resistance [53,54]. On the other hand, pipeline steels with under-tempered martensitic or bainitic microstructures or those without post-weld heat treatment in the heat-affected zones (HAZ) are understood to have less SCC resistance [55].

Liu et al. investigated the SCC behavior of API 5L X70 pipeline steel with different applied heat treatments and microstructures as shown in Table 1 [56]. They conducted the test using the SSRT method with a strain rate of  $5 \times 10^{-7}$  in/s in an acidic soil extract (pH of 4.41) collected from 1.5 m underground. Cathodic potentials of  $-650$  mV saturated calomel electrode (SCE),  $-850$  mV (SCE), and  $-1200$  mV (SCE) below the corrosion potential were used in these tests. Interestingly, the results showed a dual-mode SCC mechanism, having a combined effect of anodic dissolution and hydrogen embrittlement. The water-quenched specimen having bainite grains had a higher susceptibility to SCC in the acidic soil extracts, while the as-received sample with ferrite matrix grains had a lower susceptibility to SCC. It can be deduced that the primary SCC mechanism changed with varying applied potentials. For instance, when a lower negative applied potential, the SCC mechanism was highly due to anodic dissolution. But when the applied potential was highly negative, hydrogen became the major SCC mechanism causing the cracking process, and *trans*-granular cracks were observed. With more negative potentials, the SCC mechanism became completely based on a hydrogen-induced mechanism, with a riverbed-shaped brittle character of the fractured surface.

This was also in line with Fu et al. study on the SCC behavior of X70 carbon steel at different cathodic potentials below  $E_{\text{corr}}$  [57]. They observed that at an applied potential of  $-450$  mV (SCE), the mechanism of SCC was anodic dissolution, while at a reduced potential of  $-850$  mV (SCE), the anodic dissolution of the X70 steel was inhibited. Now, when a more negative potential of  $-1200$  mV (SCE) was applied, the specimens showed a higher SCC susceptibility, and the major SCC mechanism was completely hydrogen embrittlement.



**Fig. 2.** (a) Mechanisms of hydrogen absorption in a steel plate exposed to H<sub>2</sub>S environment; (b) interstitial lattice sites and trapping sites in steel; (c) hydrogen pressure build-up inside a defect located inside the wall of a steel pipe [66].

In addition to microstructure, chemical composition, residual stress, crystallographic texture, water chemistry in the field, applied stress, pH of the environment, and AC density also affect the SCC crack initiation and propagation [27,58,59]. For example, Zhang et al. investigated the SCC susceptibility of API 5L X70 carbon steel in various CO<sub>2</sub> concentrations, using the SSRT technique at a strain rate of  $1 \times 10^{-6}$  in/s. Increasing CO<sub>2</sub> concentrations of 0 %, 5 %, 10 %, 15 %, 20 %, and 100 % balanced with nitrogen gas in soil extracts solution were used for the tests. The test results indicated a rapid fall in area reduction of specimens with increasing CO<sub>2</sub> concentration. The susceptibility of the specimens to SCC increased with increasing CO<sub>2</sub> partial pressure until a steady state value was reached at about 20 % CO<sub>2</sub> concentration.

There is also a chance that SCC may occur in the absence of hydrogen in pipelines. However, in such a scenario, passive film dissolution is usually responsible for crack propagation [9]. Fig. 1 shows the various factors affecting the SCC crack propagation in pipeline steels.

#### 4. Improving the SCC resistance of pipeline steels

The role of microstructure plays a major role in SSC mitigation. For instance, engineering grain boundary and crystallographic texture have been proven to improve the SCC resistance of pipeline steels. Arafin and Szpunar investigated the role of texture on X65 pipeline steel and concluded that tailoring the texture and grain boundary character will help to produce new pipeline steels with improved intergranular SCC resistance. It has been reported that boundaries between grains having {1 1 0} and {1 1 1} planes oriented parallel to the pipeline steel surface are more resistant to SCC. It has also been reported that large amounts of CSL boundaries and LAGBs can improve SCC resistance in pipeline steels [11]. Pipeline steels having a lesser number of inclusions or with a uniform distribution of inclusions will also possess better SCC resistance. This is because, the oxide and silicon-enriched inclusions are considered as SCC micro-crack initiation sites, as concluded by [60] Wang et al. In terms of microstructural phase constituents, Bulger et al. studied the effect of microstructure in pipeline steels on SCC resistance in near-neutral pH. The authors found out that fine-grained bainite and ferrite microstructure showed better SCC resistance unlike the pipeline steels having ferrite and pearlite microstructure [61].

##### 4.1. Hydrogen-related failures in pipeline steel

Hydrogen has been linked to several failures of pipeline steels [62]. Apart from understanding the harmful effects of hydrogen in pipelines carrying petroleum resources, it is also important to understand how pipelines respond to transporting hydrogen gas in the future. Usually, pipelines fail due to atomic hydrogen accumulation in the interstitial sites. Fig. 2 shows the mechanisms of hydrogen absorption in pipeline steel exposed to an acidic H<sub>2</sub>S environment. In Fig. 2a, the evolution of hydrogen occurs due to the corrosive interaction of the acidic solution and the pipe's surface. Fig. 2b shows the various trapping sites that hold hydrogen after its ingress into

the pipe's structure. While Fig. 2c shows typical hydrogen damage resulting in blisters, and cracks, caused mainly by stress-driven diffusion. This means more hydrogen would migrate to local stress concentrators to initiate pipeline steel failure.

Atomic hydrogen is introduced into the pipeline's structure through different processes:

1. Molecular hydrogen dissociation and atomic hydrogen absorption from the pipeline's surroundings into the steel.
2. Production and fabrication processes (e.g., welding).
3. The presence of agents with a strong affinity for producing hydrogen, electrochemical processes like corrosion, and/or hydride precipitation in hydride-producing components.

The risk of a pipeline crack in sour environments is high, because of the hydrogen recombination poison effect of H<sub>2</sub>S [63]. After pipeline cracks, there is leakage or rupture, and eventually, an economic loss. It is therefore imperative for pipelines to be capable of resisting severe environmental conditions, especially under sour environments. The effects of hydrogen often result without cracking. This reveals that cracking precedes hydrogen degradation mechanisms and acts in synergy with other factors like stress or corrosion [64]. In addition, the decohesion of structure can also occur, reducing the strength of the metallic bonds and facilitating failure below yield strength without the presence of a crack. Nevertheless, the internal defects formed during and after steel processing determine the extent of hydrogen-related degradation [65].

Hydrogen-related failures are better understood by different types of processes.

#### 4.1.1. Types of hydrogen-related failures

Hydrogen-related failures in pipeline steels are of significant concern in the oil and gas industry, as they can lead to potentially catastrophic incidents. These failures primarily result from the ingress of hydrogen into the steel structure, causing various forms of embrittlement and cracking. Some common hydrogen-related failures in pipeline steels include:

#### 4.1.2. Hydrogen-induced cracking (HIC)

From recent literature, a lot of attention has been given to hydrogen-induced cracking [20,43,63,64,67,68] because of the deleterious effect of hydrogen on the ductility and strength of pipelines. In offshore environments, hydrogen can also be introduced into the pipeline steel structure through moisture from cathodic protection (CP) [69]. Cathodic protection (CP), which involves the use of electric current to prevent pipeline corrosion, might become disastrous when uncontrolled [69]. This happens as the cathodic protection wears off, exposing the pipeline to the twofold effect of the cathodic environment and subsequently HIC. Cracking becomes inevitable, because of the synergistic effect of hydrogen concentration and stress level on pipelines. Also, the effect of interstitial atoms (H, C, N, etc.) migration on the physicochemical and mechanical properties of pipelines has been understood as time-dependent [63]. This reveals the dependence of the atom's migration in the lattice on exposure time, ambient temperature, and atom size. Within the  $\alpha$ -Fe lattice, H is usually less soluble than N atoms, though its small atom has an affinity for movement [70]. The decrease in hardness and ductility because of aging is a known effect in steels, involving C and N. However, the presence of H could simultaneously soften as well as harden metals, altering the results of their yield and tensile strengths, and creating a long-lasting controversy [71,72]. These controversies make it difficult to properly understand the effects of H on mechanical properties, making this subject very ambiguous even in recent times.

As said earlier, many studies on the effect of hydrogen exist, however, a lot of controversies abound. These controversies stem from different experimental designs and used methodologies, sample sizes, microstructural differences, and probing. Overall, pipeline embrittlement is attributed to the effect of hydrogen-induced failure, because of its deleterious effect on ductility. The degrading strength of steel depends on the mobility and effectiveness of hydrogen trapping sites. The mobility and trapping sites of hydrogen have been discussed in some studies [73,74]. These studies have shown the relationship between the HIC resistance of pipelines and their microstructural phase composition (bainite, martensite, ferrite, pearlite, etc.) as well as other microstructural features (grain boundary, dislocations, crystallographic texture, microalloying, inclusions, etc.). These HIC relationships with microstructure will be discussed later in detail.

Interestingly, a pipeline can have cracking initiated by the dual effects of both HIC and SSC. Some researchers define such an effect as hydrogen-assisted stress corrosion cracking. For instance, it was reported that anodic dissolution is dominant to stress corrosion cracking in a near-neutral pH environment, while a mix of hydrogen-based mechanisms becomes prominent at high pH [75]. According to the literature, the pH of the environment also affects the crack propagation, after crack initiation. For instance, the crack propagation mode is often *trans*-granular in near-neutral pH conditions but tends to be intergranular in high pH [75,76]. On the other hand, pipeline steels that are protected cathodically with highly negative applied potential, and with proper control of the protection processes, have better chances of withstanding hydrogen degradation [69]. When pipelines are not cathodically protected, or when these cathodic protections are not controlled, pipeline coating is not immune to hydrogen blistering. The blistering process in coatings is referred to as cathodic disbondment, and it is exacerbated by interactions between pipeline coating and OH<sup>-</sup> ions produced during water reduction [77]. Pipeline coatings experience accelerated water permeation at the edges at high cathodic potentials, which proceeds into pockets of hydrogen blisters and sites for localized hydrogen attack on the steel [77]. Inferences were also made that the high resistivity of coatings has a knock-on effect on the cathodic shielding, by preventing the permeation of cathodic current towards pipeline steel, thereby increasing the chances of SCC [78]. To confirm this inference, the cathodic-shielding behavior of high-density polyethylene and fusion-bonded epoxy coatings on pipelines were compared. Reports showed that high-density polyethylene (HDPE) coatings shielded cathodic protection current from reaching the pipeline's surface [79]. It can be assumed that pipelines coated with HDPE will experience hydrogen degradation under-coating. However, it was observed that the fusion-bonded epoxy-coated pipeline allowed permeation of CP current, creating more room for cathodic protection.

#### 4.1.3. Improving the HIC resistance of pipeline steels

It is widely reported that higher-strength pipeline steels are more susceptible to HE compared to lower grades. However, recent studies have reported the embrittlement in iron of much lower strength [80]. There are lots of debates when it comes to the effect of microstructure on the cracking susceptibility of pipeline steels. Some authors recommend acicular ferrite and bainitic ferrite for better resistance to HIC than ferritic pearlite or martensitic microstructure, but evidence in the literature has remained either inconsistent or less proven [81–86]. These inconsistencies might be a result of the different forms of hydrogen damage besetting pipeline steels. However, it is imperative to mention that tailoring the crystallographic texture, dislocation density, and grain boundary properties can positively influence susceptibility to HIC in pipeline steel [87–91]. Several studies have shown that dominant {111}, {110}, and {332}, planes oriented parallel to the pipeline surface can better resist crack propagation, unlike {001} planes of the same orientation. However, cracks have been observed across {110} ||ND textured grains, which necessitates further research on the influence of crystallographic texture on the HIC resistance in pipelines.

#### 4.2. Sulfide stress corrosion cracking (SSCC)

Like SCC, SSCC is a pipeline failure mechanism that also involves the combined effect of stress and corrosion. It is a more devastating failure mechanism that leads to HE in wet H<sub>2</sub>S media containing steel oil and gas lines [20]. The key parameters of SSCC are susceptible microstructure, corrosive environment, and tensile stresses. The kind of microstructural composition of the pipeline either increases or reduces SSCC susceptibility. As microstructure tends to control the mobility of hydrogen within the steel, it has been greatly investigated [1,92,93]. The hydrogen atoms generated at the surface of the steel substrates during cathodic reactions diffuse within the steel material. The diffused hydrogen atoms reside in reversible (e.g., grain boundaries, dislocations) or irreversible (e.g., inclusions, precipitates) traps, though escape from the latter is possible at ambient conditions [73]. Liu et al. reported the impacts of grain boundaries and dislocations on the SSCC susceptibility in casing steel [94]. Their results show that dislocations influenced SSCC behavior much more than grain boundaries. This could be due to the localized plastic deformation associated with hydrogen-mobilized dislocations. However, looking at the effect of dislocation and grain boundary separately will throw more light on their influence on SSCC. SSCC is usually classified by relating the two types: *type 1* and *type 2* [95]. For *type 1*, the formation of a hydrogen-induced blister crack may take place along the direction of the applied tensile stress, stress-oriented hydrogen-induced cracking (SOHIC). The failure mechanism of *type 1* may be in two stages depending on the direction of crack propagation (parallel or perpendicularly) concerning the applied stress. For *type 2*, the crack is propagated from the typical location of hydrogen embrittlement. There is a distinct difference between HIC (no external loading required for failure) and hydrogen-induced blister crack (HIBC) (stress is required for failure), however, these processes have similarities too - They are both explained by the internal pressure theory [96], which proposes that the accumulation of hydrogen in voids or cracks creates high pressures of hydrogen gas within the metal thereby initiating cracks [97] and after crack nucleation, they both propagate along the rolling direction of the steel. There remains insufficient literature revealing the understanding between HIC and SSCC resistance in the SOHIC mode [98]. From the literature, Cayard et al. showed that most HIC-resistant steels may also be more prone to SOHIC than some conventional ones; this further adds more difficulty to the selection criteria for SOHIC-resistant steel grade [99]. Kim et al. [95] have also tried to show a correlation between HIC and Type I SSCC failure mechanism in high-strength pipeline steel. They compared the SSCC resistance of pipelines under applied tensile stress with the one charged with hydrogen. They concluded that HIC occurs as an initial crack of type I SSCC.

Generally, hydrogen embrittlement is pronounced after crack initiation in a susceptible material from a combination of defined amounts of hydrogen and critical stress levels. While this situation is accepted to some extent, the exact hydrogen embrittlement mechanism is still a subject of intense controversy [100]. Formerly, it was inferred that hydrogen embrittlement is a result of restricted or promoted movement of dislocation under the influence of hydrogen within interstitial spaces in steel. To support this reasoning, it was reported that hydrogen influenced the decrease in cohesion between cleavage planes and/or grain boundaries [101]. Interestingly, Campari et al. utilized the machine learning approach to predict the hydrogen embrittlement susceptibility of metallic materials [102]. In their study, experimental data of tensile tests carried out in a hydrogen environment was performed and analyzed through an advanced machine learning approach. According to the authors, the embrittlement index was estimated and used as the determinant parameter for predicting the likelihood of component failures, which has an accuracy rate of 88.6 %.

Many theories and models have been introduced to shed more light on hydrogen embrittlement in pipeline steels. The first theory is the hydrogen-enhanced decohesion (HEDE) model which infers that bonding strength is reduced by interstitial atomic hydrogen [103–107]. It was revealed that in the HEDE theory, the hydrogen-induced weakening of metal–metal bonds leads to decohesion instead of slip. The HEDE mechanism is characterized by smooth brittle fracture surfaces with limited plasticity. Katzarov et al. were able to report the HEDE mechanism in sub-grain boundaries and *trans*-granular fracture of micro-alloyed steel. The authors also investigated HEDE within bcc  $\alpha$ -Fe (111) crystal planes [108]. The second model proposes that the occurrence of localized plastic deformation is due to hydrogen-induced dislocation processes. This happens as hydrogen atoms cause the movement of dislocations, causing localized stress in the steel lattice. In case of defects, such stresses can easily initiate a crack. This is the Hydrogen-Affected Localized Plasticity (HALP) model, pioneered by Beachem [109]. In recent times, this model has been modified into two: hydrogen-enhanced localized plasticity (HELP) [110,111] and Adsorption induced dislocation emission (AIDE) [101,112]. When the applied hydrogen-induced stress is in a magnitude greater than the cohesive strength along interfaces, the presence of impurities reduces the gross cohesive strength resulting in decohesion, and finally, the initiation/propagation of interfacial cracks [108]. Birnbaum also reported potential regions with greater affinity for hydrogen-related decohesion: (a) at crack tips as adsorbed species, (b) at particle–matrix pivots ahead of cracks, especially where dislocation shielding effects lead to the highest tensile stress, and (c) at locations of utmost hydrostatic stress [113]. The hydrogen-enhanced localized plasticity (HELP) model has also grown in popularity. The



**Table 2**  
Pipeline steel grades and their strength requirements.

API 5L Grade <sup>a</sup>	Min YS (Mpa/ Ksi)	Max YS (Mpa/Ksi)	Min TS (Mpa/Ksi)	Max TS (Mpa/Ksi)	Min elongation (%) for 0.2 sq in sample
A25	172/20	—	310/45	—	—
A	207/30	—	331/48	—	—
B	241/35	448/65	413/60	758/110	22.5
X42	289/42	496/72	413/60	758/110	22.5
X46	317/46	524/76	434/63	758/110	21.5
X52	358/52	531/77	455/66	758/110	21.0
X56	386/56	544/79	489/71	758/110	19.5
X60	413/60	565/82	517/75	758/110	18.5
X65	448/65	600/87	530/77	758/110	18.0
X70	482/70	621/90	565/82	758/110	17.0
X80	551/80	690/100	620/90	827/120	15.5
X90	625/90	775/112	695/100	915/132	—
X100	690/100	840/121	760/110	990/143	—

<sup>a</sup> American Petroleum Institute (grading system where identification number for each steel corresponds to minimum yield strength measured in Ksi); YS – yield strength; TS – tensile strength.

**Table 3**  
Classification of the new generation of high-strength pipeline steels based on microstructural control.

API- 5XL Steel grades	Steel manufacturing process	Microstructure	Microstructural features
X70	TMCP + QT	Bainite-Martensite multiphase steel: B + M + F	Slender bainitic sheaves along with martensite laths [124].
X80	TMCP + AcC	Bainite dual-phase steel: B + F	Smaller prior austenite grain (PAG) and fine ferrite grains [125].
		Bainite-Martensite multiphase steel: B + M + A	Martensite laths [124]. High volume fraction of filmy Austenite [126,127].
X100	TMCP + AcC + HOP	Bainite dual-phase steel: B-F	Polygonal and granular ferrite acts as the second phase in the bainite matrix [128].
X120	TMCP + AcC	Martensite dual-phase steel: M + F Tempered Lath Martensite: TLM	Fine ferrite acts as the second phase in the martensite matrix [129]. Martensite laths [130].

HELP model considers crack propagation through the coalescence of defects. Depending on specific temperatures and strain rates, the HELP model proposes that atomic hydrogen inside the steel lattice causes dislocation motion, leading to increasing deformation at localized regions [110]. When these hydrogen-engaged dislocations are close to defects (cracks), the resulting damage is no longer embrittlement but a localized plastic-type fracture [114]. Since both theories are not related to each other, it is best to understand that HEDE proposes that decohesion precedes cracking, while HELP proposes that enormous plasticity, which is induced by the enhanced velocity of dislocations precedes cracking. For HELP, when the elastic energy of steel is reduced due to hydrogen, there will be minimized obstructions and enhanced velocity of dislocation. As dislocations continue to interact, they pile up at microstructural defects, and this results in material failure [115].

Ultimately, a comprehensive understanding of these models indicates that the microstructural characteristics provide valuable insights into their intricate functions concerning the resistance of pipeline steels against cracking and corrosion.

#### 4.3. Improving SSCC resistance in pipeline steels

From a few studies, the elimination of residual stress can greatly improve the SSCC resistance of pipeline steels [116,117]. Zhao et al. proposed the use of nano-sized carbonitrides as a way of improving the SSCC resistance of acicular ferrite pipeline steel [118]. The reductions of inclusions in pipeline steel or attaining inclusions will also improve SSCC resistance. For welded joints, lowering the heat input can greatly improve SSCC resistance, especially in the heat-affected zone (HAZ) [118,119]. In addition, tailoring dislocation density, and crystallographic texture will favor SSCC resistance [117].

### 5. Microstructure overview

The role of microstructure in influencing the susceptibility of pipeline steels to hydrogen-related degradation mechanisms is crucial. However, a holistic comprehension of these functions hinges upon our grasp of the thorough range of microstructural analysis. The new generation of API-5XL steels is classified into four categories based on microstructural control, as shown in Table 4

In essence, microstructure constitutes an amalgamation of different features that exert a profound influence on the mechanical

**Table 4**

Shows how hydrogen at different pressures affect the mechanical properties of pipeline steels.

Pressure (psig)	Pressure (atm)	UTS (ksi)	Elongation (gage: 30 mm) %	Reduction of Area (%)
Ambient (Air)	1 (Air)	70.8	32	64
147 (H <sub>2</sub> )	10 (H <sub>2</sub> )		34.5	52
294 (H <sub>2</sub> )	20 (H <sub>2</sub> )		33	47
735 (H <sub>2</sub> )	50 (H <sub>2</sub> )		30	50
1470 (H <sub>2</sub> )	100 (H <sub>2</sub> )		30	36.5
2205 (H <sub>2</sub> )	150 (H <sub>2</sub> )		26	28
1470 (Argon)	100 (Argon)		36	62

properties of a material. It transcends mere phase composition, encompassing factors such as grain size, grain boundary attributes, dislocation density, crystallographic texture, and so on [9]. These diverse characteristics contribute to the understanding of the mechanical properties of pipelines (see Table 3). Table 2 shows pipeline steel grades and their strength requirements. To illustrate, consider the case of three API 5L X65 pipeline samples sharing identical microstructural phase compositions, characterized by pearlite and ferrite. These samples exhibited varying mechanical properties following quenching in different quenchants at different cooling rates. These differences in cooling rates introduced dissimilarities in crystallographic texture and altered the microstructure without necessarily affecting the phase composition [120]. Apart from microstructure, the choice of pipeline also depends on operational parameters like pressure, temperature, fluid velocity, and flow pattern [121]. A pipeline of higher tensile strength should be used to carry high-pressure substances to be able to withstand stress without deformation or failure. Additionally, pressure fluctuations, especially in systems with frequent changes, require pipelines capable of handling cyclic loading without fatigue. Elevated temperatures often cause a reduction in steel's strength and increased susceptibility to corrosion or embrittlement [122]. Pipeline steel with high-temperature resistance or insulation should be used for such conditions. On the other hand, pipeline steels with high fracture toughness and low ductile–brittle transition temperature (DBTT) should be used in arctic regions [123]. Lastly, high fluid velocities can cause corrosion within pipelines, especially at bends or areas of turbulence. The flow pattern, whether laminar or turbulent, also affects the forces exerted on the pipeline walls.

Our following sections will delve into an exploration of these diverse microstructural parameters and their respective contributions to the HIC and corrosion resistance of pipeline steels.

### 5.1. Microstructural phase composition

The microstructural phase composition of steel is normally used to define the microstructure of steel. However, we have explained how broad microstructure can be. Phase composition reveals the microconstituents of steel after processing. There are several microconstituents found in steels depending on their processing route, ferritic pearlite, polygonal ferrite, quasi-polygonal ferrites, Widmanstätten ferrite, acicular ferrite, bainitic ferrite, bainite, martensitic microstructural phases, etc. All these microstructural phase compositions can be formed by controlling critical TMCP parameters such as the cooling rate applied after hot rolling [1,93]. From various studies, different phase compositions have known advantages on the mechanical properties of steel, as regards the formation and propagation of cracks.

According to published reports, in ferrite grains, a decrease in slip length and a decrease in the deformation around the crack tip during crack propagation is observed [128]. In comparison, smaller prior austenite grains foster a decrease in dislocation pileups or increase the barriers against crack propagation [128]. Film-like austenite grains facilitate crack-tip blunting through the transformation of the retained austenite to the martensite. It is also known that a decrease in the propagation rate of the main crack due to the compressive residual stress formation and propagation of the secondary crack is associated with the transformation-induced plasticity effect (TRIP) [131]. In comparison, Martensite laths induce the uniform distribution of dislocations and the rearrangement of stress concentration along the high-angle grain boundaries and act as a blockage of cleavage crack propagation along martensite lath boundaries [126].

Polygonal ferrite, as a second phase in the bainite matrix, can significantly increase the yield strength and low-temperature toughness and optimize the fatigue limit of pipeline steels [131]. The bainitic phase with a smaller width of the bainite laths has a higher stress intensity factor than the threshold value ( $K_{th}$ ) for crack blunting [125,126]. In terms of toughness, acicular ferrite is known for its high toughness. From historical studies, the API 5L X70 plates have a ferrite-pearlite microstructure for the most part, and acicular ferrite (AF) microstructure was induced as the best microstructure in terms of toughness [132]. Also, the formation of quasi-polygonal ferrite (QPF), fine polygonal ferrite (FPF), globular ferrite (GF), and AF microstructural phases with fine grains and more dislocations, increases ductility, as well as contributes to a reduction in pearlite bands according to the conclusions of Amirjani et al. [132].

While tempered martensite is inferred to have better ductility, the bainite and martensite regions having low ductility have high SSCC susceptibility. This further reflects the difficulty in balancing the strength and toughness properties of pipeline steels, regarding HIC [133,134]. After quenching and tempering, the martensitic phases are more refined and uniformly distributed. Quenching/tempering is mostly used to produce SSCC-resistant martensitic steels.

Interestingly, accelerating the cooling rates of steel processing can result in desired micro constituents like acicular ferrite and polygonal ferrite, thus bringing a better balance in pipeline steel's strength and toughness [135]. Amirjani et al. reported that increasing the accelerated cooling rate and reducing the finishing rolling temperature was responsible for the ductile fracture of API 5L



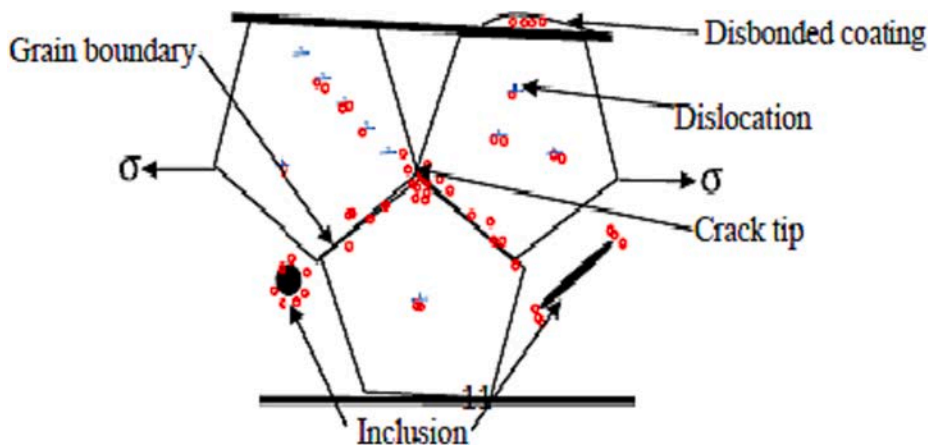


Fig. 4. Schematic representation of various trapping sites for hydrogen in pipeline steel [145].

Irreversible traps like martensitic interface and mixed dislocations were reported to possess binding energy in the range of 61.3–62.2 kJ/mol [146]. To prove the strong trapping proficiency of irreversible traps, the authors also recorded a higher hydrogen trap binding energy of up to 89.1–89.9 kJ/mol at grain boundaries with high misorientation and undissolved carbides. Their results spawned the conclusion that hydrogen atoms trapped irreversibly are excluded from those diffusing through the steel. On the other hand, the trapping sites where atomic hydrogen is weakly trapped are called reversible traps. These trapping sites require low potential energy barriers to permit atomic hydrogen escape. The strong presence of reversible traps has been noticed in cold rolled pipeline steels [143,147–150]. When enough plastic strain is in the pipeline steel because of cold rolling, a multiplication of dislocations and grain boundaries increases the number of reversible traps [147]. Till now, the detection of hydrogen atoms during their interaction with the steel structure remains complicated. To better understand the relationship between hydrogen embrittlement and trapping sites, information about the placement of hydrogen atoms concerning microstructural features at the nano/micro scale is required. However, because of the low atomic number of hydrogen, analyzing it at the nano/micro-scale has proven difficult by most available characterization techniques. For instance, lighter atoms like hydrogen pose scattering difficulties when X-rays and electrons are incident on them, unlike heavier atoms, making it difficult to analyze them through electron microscopy [151]. Even with thermal desorption spectroscopy (TDS) which provides macroscale information about hydrogen desorption, it remains difficult to evaluate hydrogen in traps, since this hydrogen originates from every possible trapping site, making it difficult to distinguish the different quota of contributions from the different types of hydrogen-containing defects [152]. However, neutron diffraction has achieved a significant milestone in the study of hydrogen interactions with metallic compounds [153–156]. While neutron diffraction might have shed light on hydrogen interaction with steel structure, its usage poses some challenges like cost and accessibility to users. Also, Mass spectroscopy techniques have been used to identify the location of hydrogen through the ratio of mass to charge state [151]. It was reported that the high spatial and mass resolution of atom probe tomography (APT) caused the easy mapping of hydrogen in nanoscale features in three dimensions [157–160]. Most times, APT is used to account for both precipitate analyses and the identification of microstructural defects such as grain boundaries and dislocations through associated elemental segregation [161–163]. According to reports, grain boundaries are the most efficient interface for trapping hydrogen in steels [164]. Yi-Sheng Chen et al. were able to observe the hydrogen-trapping behavior of grain boundaries, precipitates, and dislocations. They showed that the trapping affinity of many dislocations could override the trapping effects of grain boundaries in martensitic steel. However, on the annealed ferritic steel which had low dislocation density, more hydrogen-trapping affinity for the grain boundaries and carbide precipitates was observed [144]. Meanwhile, the types of hydrogen trapping sites and their role in hydrogen damage remain debatable within the scientific community. There are inferences that the high presence of irreversible hydrogen traps will increase the amount of hydrogen present within steel and will also reduce diffusible hydrogen within the lattice [165–168]. These inferences are because the irreversible traps starve the steel lattice of the much-needed hydrogen for crack initiation and propagation. Therefore, the strongly trapped hydrogen atoms are regarded as less likely to cause degradation. In other words, the combination of atomic hydrogen within irreversible traps is believed to produce hydrogen molecules and impede their diffusion through interstitial spacing towards crack-susceptible regions. It was also reported that widely distributed irreversible trap sites could potentially diminish hydrogen from reversible traps; hence lowering the chances of hydrogen embrittlement [169]. Jack et al. reported that more irreversible traps were responsible for the less severe cracks observed in the API 5L X65 pipeline after hydrogen charging when compared to the steel with more reversible traps. They also reported that hydrogen mobility through the steels was most prominent along the grain boundaries, indicating the significance of grain boundary character on HIC [142]. This was also in line with Diniz et al., as they mathematically simulated the effect of reversible hydrogen trapping effect on crack propagation in the API 5CT P110 steel. They concluded that crack initiation and propagation are both slower in pipelines with irreversible traps than in pipelines with reversible traps [170].

Peng et al. investigated the effect of MnS inclusions (irreversible trapping site) on the hydrogen trapping and HIC susceptibility of X70 pipeline steels. They found that most submicron scale inclusions in their steel were MnS, serving as irreversible traps for hydrogen. They also claimed that the amount of the trapped hydrogen can be significantly reduced by controlling the size of MnS inclusions below

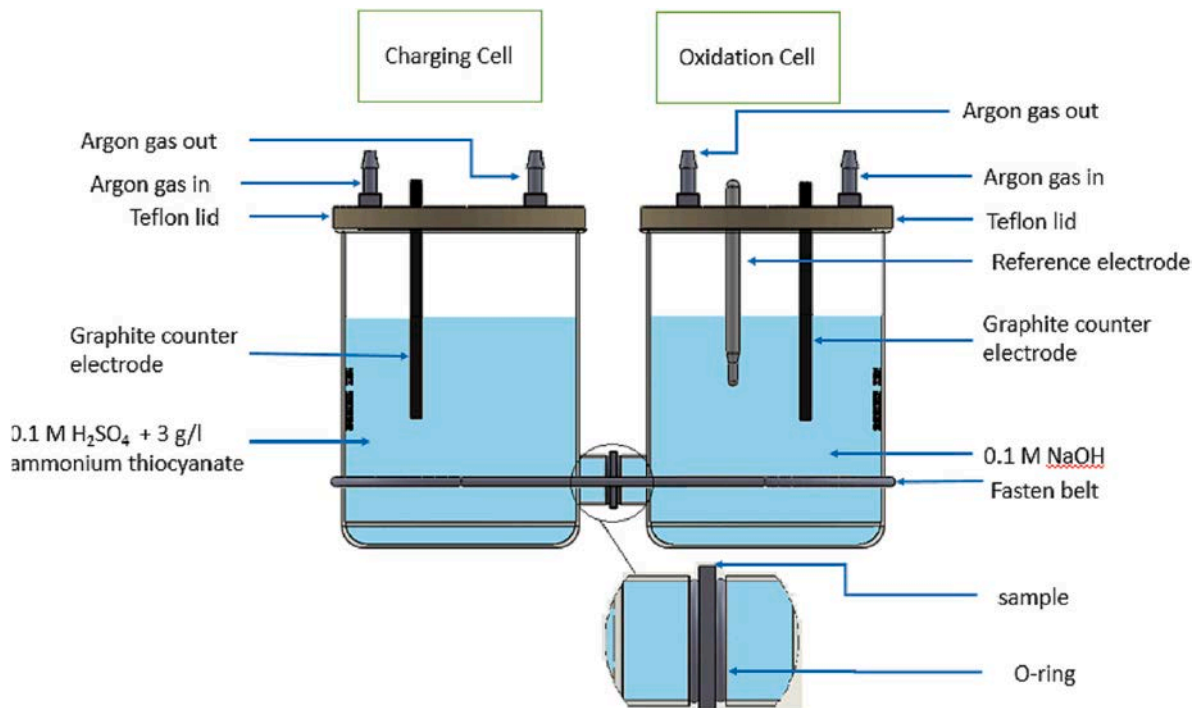


Fig. 5. Devanathan and Stachurski's setup for the hydrogen permeation experiment [173].

the submicron scale and distributing the inclusions uniformly in the steel, thereby reducing HIC susceptibility. This was also in line with Luu W et al. investigations on the effect of MnS on hydrogen transport in steels, where they concluded that MnS has more trapping affinity for hydrogen [15]. However, there are scanty studies regarding the multiplication of irreversible traps as a technique for minimizing reversible hydrogen trapping. This has to do with changes in other microstructural features, which play different roles in the HIC susceptibility of pipeline steels. Finally, the idea behind irreversible traps is that they mitigate the mobility of hydrogen in the steel matrix. It is believed that hydrogen in motion is a moving hazard to pipeline steels. However, Dadfarnia et al. concluded that although irreversible traps limit the amount of mobile hydrogen, we cannot infer that irreversible traps can be used to mitigate hydrogen embrittlement [171].

On the other hand, reversible traps, having low binding energy allow higher mobility of hydrogen, thereby increasing crack susceptibility [170,172]. With these parameters, the overall density of different trapping sites can be estimated. Fig. 5 shows the image of hydrogen permeation equipment used to determine the diffusivity of hydrogen, and for estimating the density of trapping sites in pipeline steels.

Interestingly, the impact of having a greater number of a particular trapping type in steel lattice has not been fully understood. Dadfarnia et al. used numerical simulation to predict that the greater density of one type of trap (whether weak or strong) can lower effective diffusion, but it does not necessarily affect the amount of hydrogen diffusing through normal interstitial lattice sites and any other type of trap [171]. However, this becomes a debate since it is somehow believed that the accumulation of hydrogen in traps could result in internal pressure, leading to crack initiation at high-stress concentration zones. Nevertheless, it was reported that increasing the density of irreversible traps can slow down the kinetics of hydrogen embrittlement [174,175].

### 5.3. Dislocation density

The dislocation density within the steel structure affects its mechanical properties, and therefore it should be considered when discussing microstructure. Generally, increasing dislocation density is a strengthening mechanism in materials. However, strengthening has a knock-on effect on the cracking susceptibility of pipelines. It has been reported severally that higher dislocation density reduces the HIC resistance of pipeline steels. Momotani et al. investigated the effect of dislocation density on martensitic steel [176]. In their research, it was concluded that high dislocation density facilitated hydrogen accumulation around prior austenite grain boundaries during deformation within the visible elastic strain regime. In the end, higher dislocation density led to the cracking of the steel. This can also be related to the HELP mechanism, where dislocations can move, interact, and pile up, thus causing bigger damage. Connolly also investigated the effect of dislocation on the HIC susceptibility of pipelines. From his interesting study, the steel sample with higher dislocation density had more cracks when compared with the sample with lower dislocation density [177]. When it comes to hydrogen trapping, dislocation density plays a bigger role in HIC susceptibility. Perng and Altstetter reported that the increased dislocation density allowed the trapping of more hydrogen, and it increased the effective hydrogen concentration [178]. The effect of

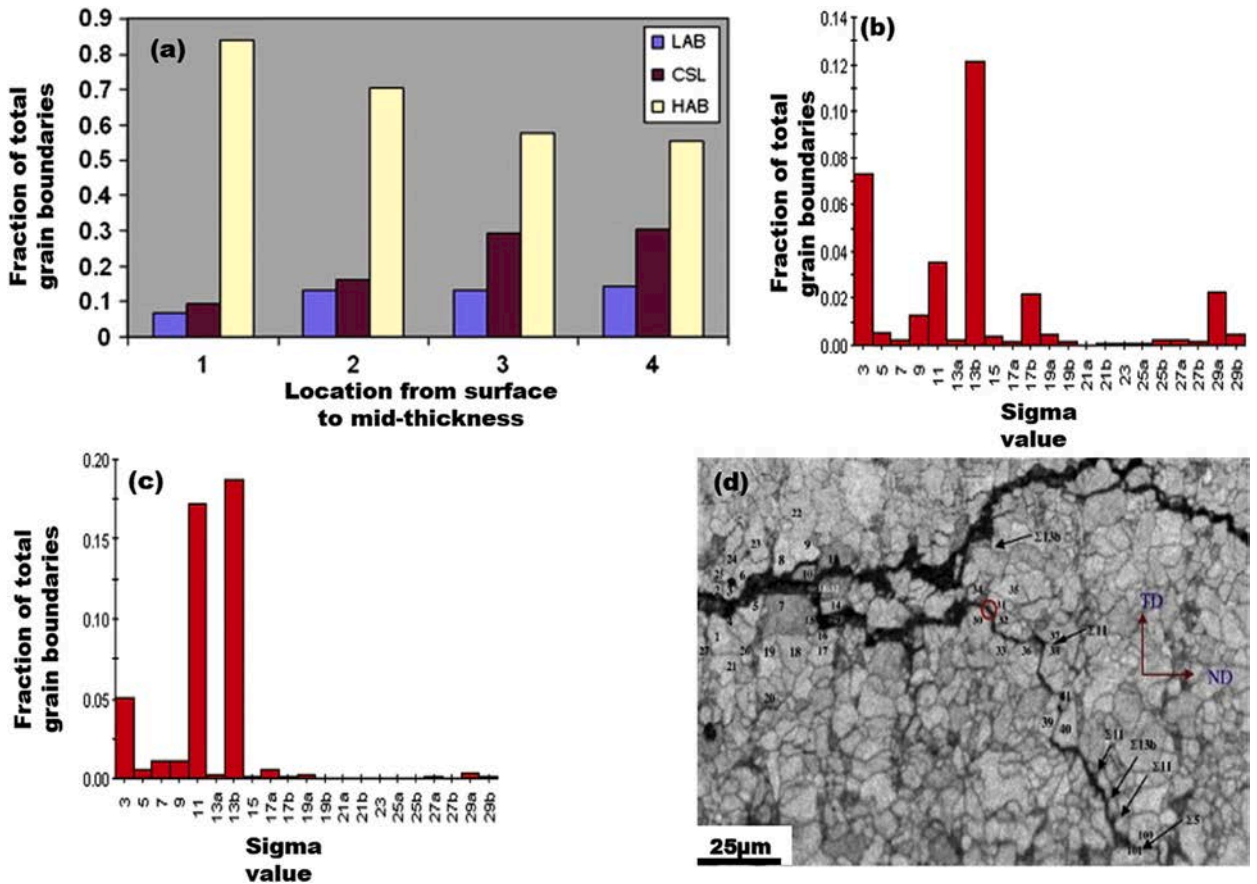


Fig. 6. (a) Distribution of grain boundaries from pipeline outer surface (location 1) through to mid-thickness (location 4), (b-c) shows the distribution of special CSL grain boundaries around crack-tips in X65 pipeline steel, and (d) EBSD IQ map showing crack propagation pattern along grain boundaries on ND-TD plane [11].

grain boundary and dislocation on the HIC resistance of pipelines has also been investigated. Grain boundaries are known to attract more hydrogen atoms than other trapping sites [164]. However, the conclusion of Yi-sheng et al. showed that dislocation density has a more pronounced effect on HIC than grain boundaries [144]. As such, dislocation density remains a vital parameter of note when optimizing pipelines for higher HIC resistance. On the contrary, few researchers have given different views about the effect of dislocation density on HIC resistance. Dislocations, to these researchers, are not trapping sites. Louthan and Derrick concluded that trapping sites were absent in austenitic steel that had dislocations induced through cold rolling [179]. However, dislocation density remains an important microstructural feature. Thomas and Szpunar showed that higher dislocation density due to cold working reduced the permeability and the effective diffusion coefficient of X70 steel relating to an increase in reversible trapping sites of hydrogen [150]. Generally, evaluating the effect of grain size without considering dislocation density is difficult. In such a scenario, pipelines are annealed to minimize the effect of dislocation on hydrogen transport within the steel lattice. Nevertheless, separating the mechanisms governing the transportation of hydrogen within the pipeline's structure, particularly in areas of pronounced transport propensity such as grain boundaries, and evaluating the significance of these regions, continue to be subjects of interest.

#### 5.4. Grain boundary engineering

The character and distribution of grain boundaries are important, especially to pipelines under sour environments. Because hydrogen atoms accumulate in grain boundaries, it is imperative to understand the effect of grain boundaries on the HIC susceptibility of pipelines and to properly engineer them to improve their performance in sour/acidic environments [164]. Grain boundaries possessing low energies are more resistant to HIC, and thus TMCP processes are used in engineering low-angle grain boundaries (LAGB) and special CSL boundaries in steels [180]. Generally, three different grain boundary distributions have been discussed in the literature, they include high-angle grain boundaries (HAGB), LAGB, and special CSL. It was reported that large amounts of HAGB were seen at the surface (RD) of X65 steel, while LAGB and CSL were dominant in the mid-thickness region [11]. Arafin and Szpunar investigated the role of grain boundary on pipelines' intergranular stress corrosion cracking resistance. They found out that cracks were arrested more often at LAGB and CSL ( $\Sigma 11$ ,  $\Sigma 13b$ , and, probably,  $\Sigma 5$ ). Also, cracking happened more often at grain boundaries having HAGB

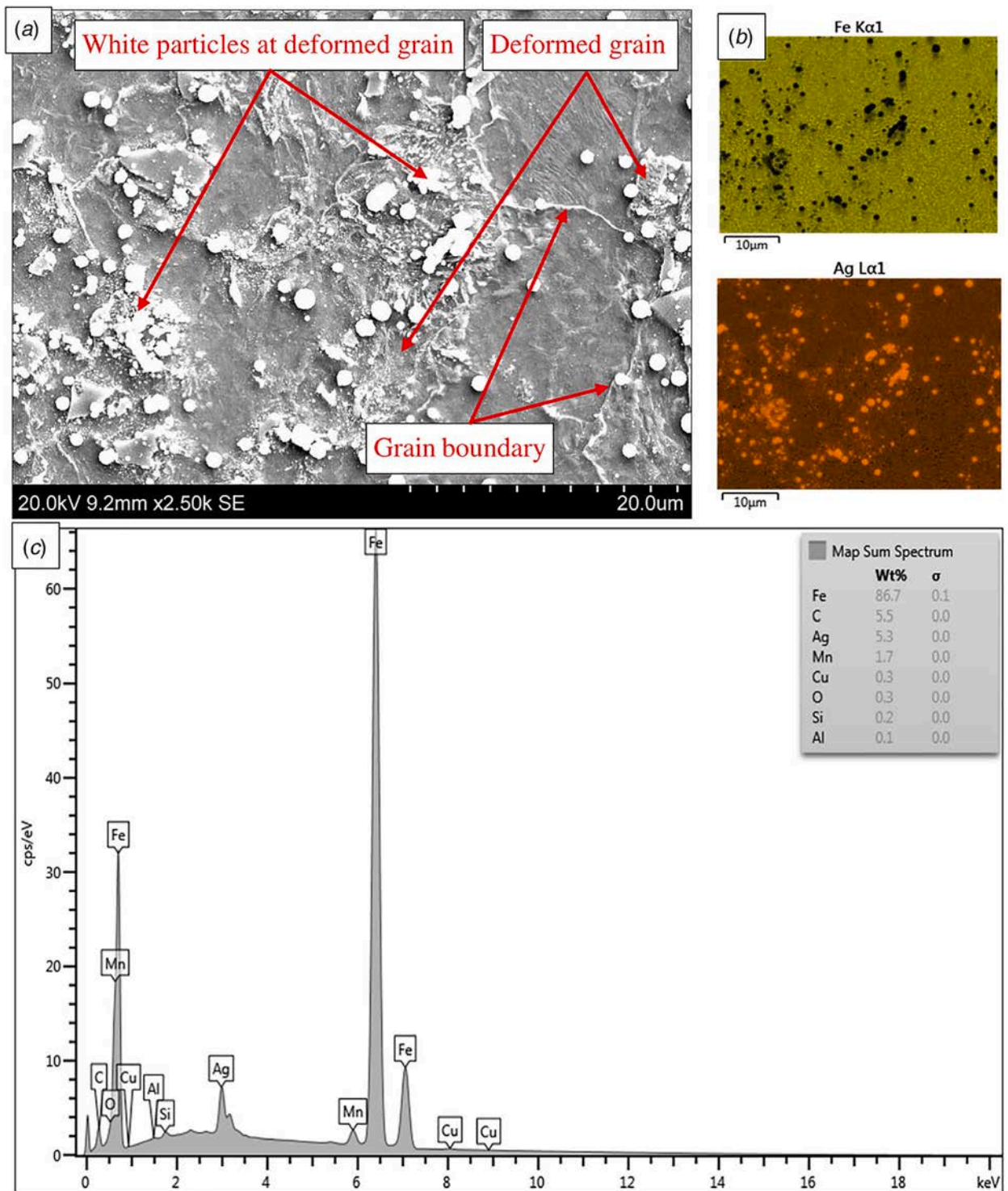


Fig. 7. Hydrogen diffusion in sample after HMT experiment (a) SEM image of deformed grains, (b) EDS maps of deformed grains, and (c) The EDS spectrum of deformed grains [150].

structure with an angle of misorientation greater than  $15^\circ$ . With the four different grain boundary types and the probability of cracking in a sour/acidic environment, it can be deduced that HAGB favors intergranular cracking. Interestingly, this was also confirmed as the cracking path followed the HAGB direction until it encountered a triple junction of HAGB, LAGB, and CSL sites as shown in Fig. 6(a–d). Thomas and Szpunar also investigated the effect of cold rolling on hydrogen diffusion in the X70 pipeline. They showed that grain

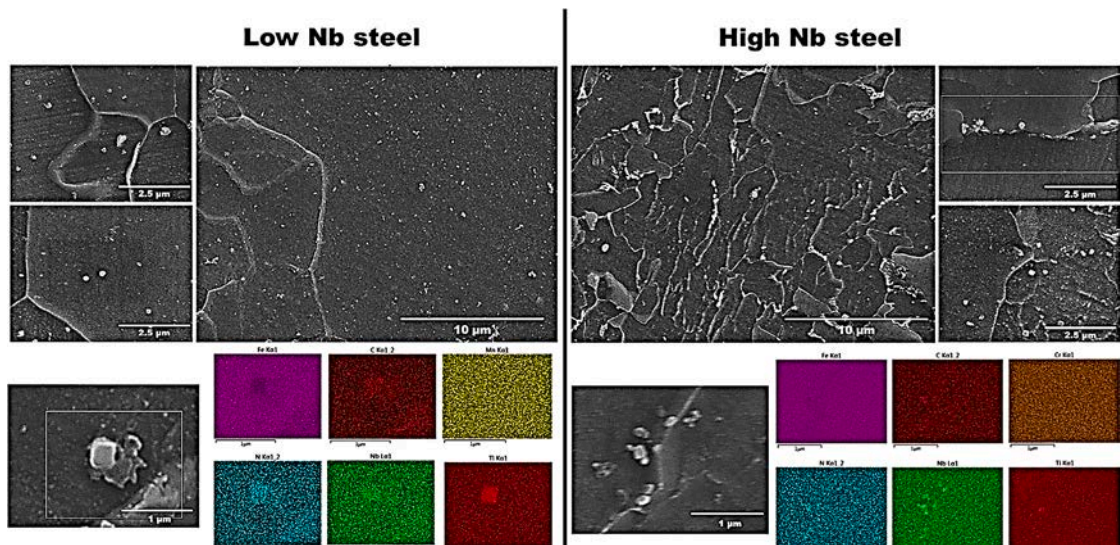


Fig. 8. Electron micrographs and energy dispersive X-ray spectroscopy of carbides observed on the top RD – TD planes of the low(left) and high (right) Nb steels [186].

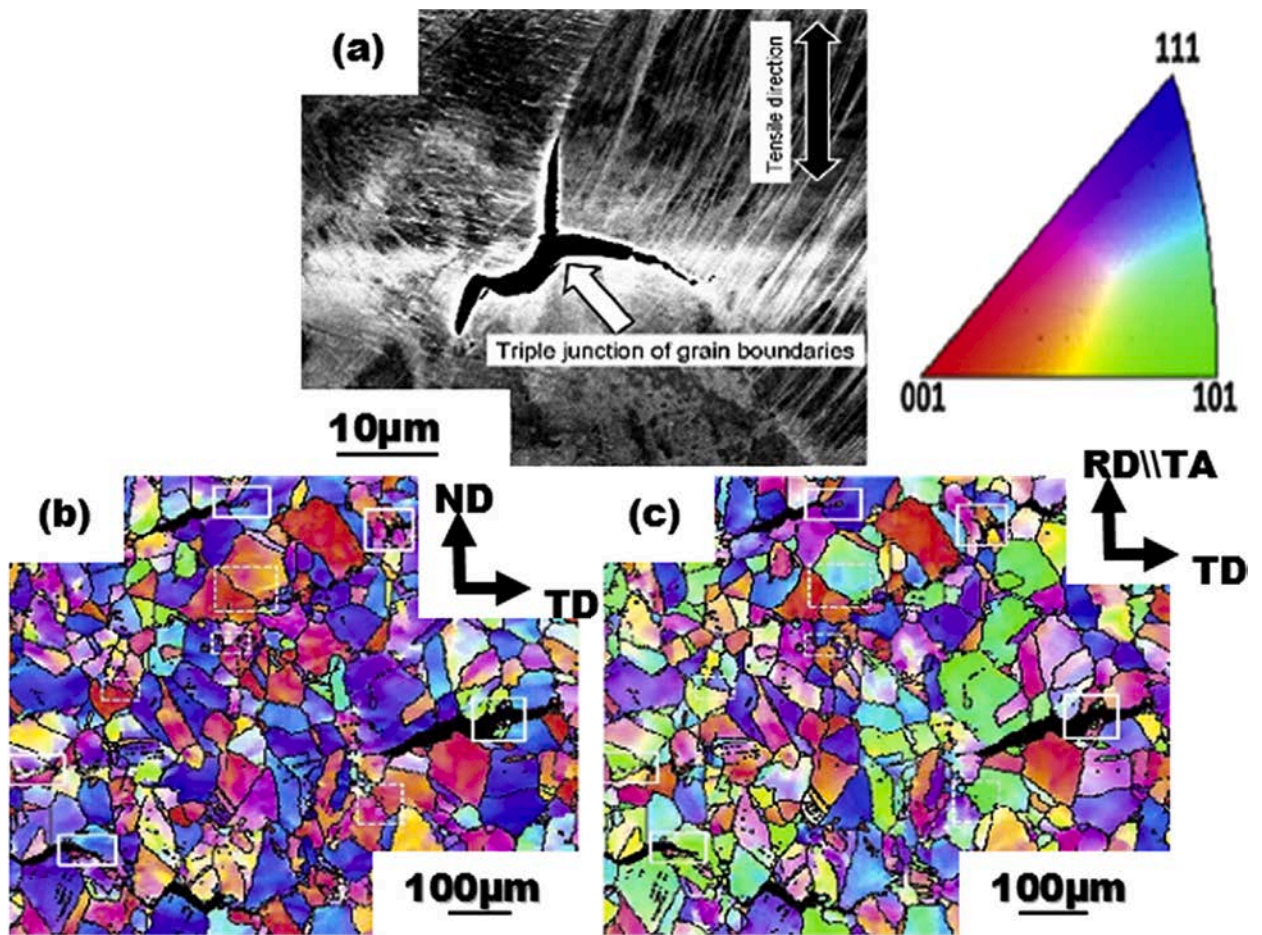
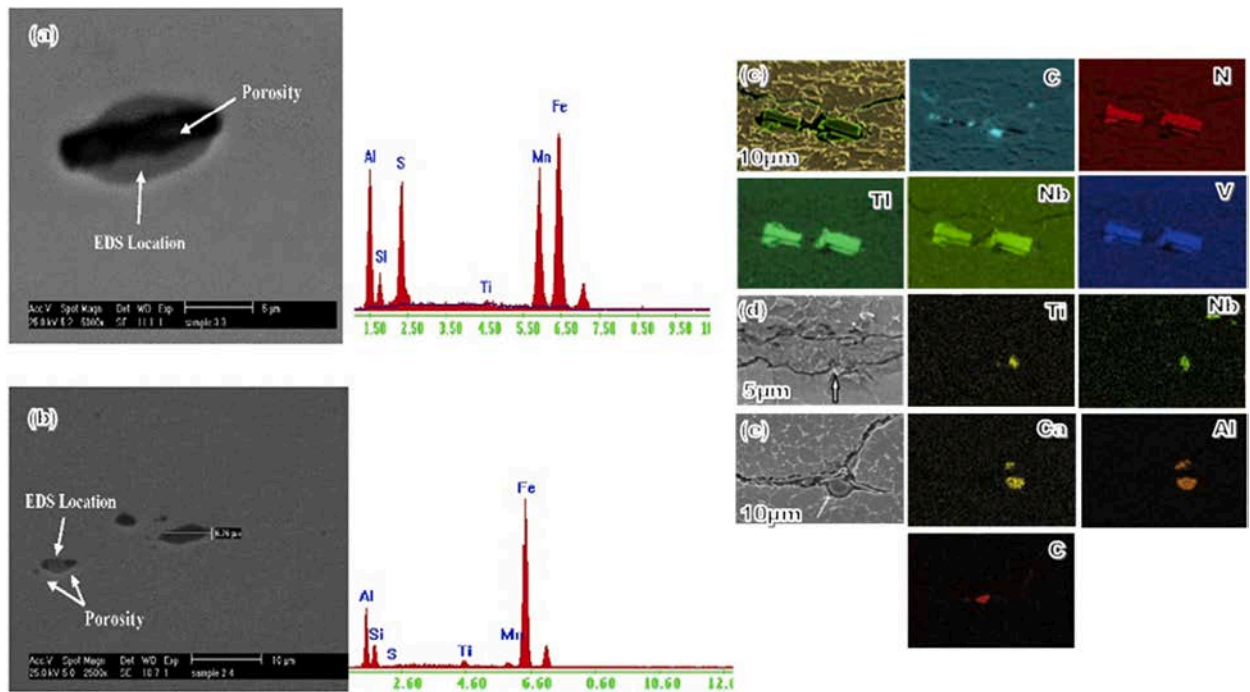


Fig. 9. HIC stricken TWIP steel (a) intergranular crack initiating at a triple junction (b) IPF map for ND-TD plane of the fractured tensile specimen (c) IPF map for RD-TD plane of the fractured tensile specimen (ND: normal direction, TD: transverse direction RD: rolling direction TA: tensile axis) [188].





**Fig. 10.** EDS point analysis on inclusions in pipeline steel (a, b) [200], inclusion maps of HIC nucleating at Ti-Nb-V enriched carbonitrides (c) [88] and Al-Ca-C enriched oxide inclusions in X70 pipeline steel (d, e) [182].

boundaries are reversible trapping sites through HMT experiments, visible in Fig. 7(a-d). The multiplication of grain boundaries due to 50 % cold rolling reduced the permeability and effective diffusion coefficient during the permeation test. This shows the overall effect of grain boundaries on hydrogen transportation within the steel lattice. However, when compared with the hydrogen diffusion rate on deformed grains, the grain boundary hydrogen-diffusion rate seems to be lower.

There is limited literature on proven methods for engineering pipeline steels towards having CSL and LAGB character, however, it has been reported that low stacking faults give rise to CSL boundaries. Also, twinning can give rise to these special grain boundaries. However, twinning is difficult to see in pipeline steels [181]. According to some researchers, warm (ferritic) rolling can induce a higher fraction of LAGB and CSL sites in pipeline steels [89]. Many publications have addressed the importance of low-energy grain boundaries in the reduction of HIC in pipelines [89,90,182,183]. It has been revealed that another characteristic of CSL boundaries and LAGB is that they act as reversible trapping sites, as they do not hold hydrogen for long. This is contrary to other inferences on grain boundaries. However, the theory behind it is that the low-energy grain boundaries do not allow hydrogen to build up to the critical levels of initiating cracks. Instead, they allow uniform distribution of atomic hydrogen within the steel lattice and subsequently allow desorption processes without tendencies of crack initiation [182]. Another interesting study showed that  $\Sigma 11$  type CSL boundaries are usually present around grains having orientations of  $\langle 110 \rangle$ , reducing their susceptibility to HIC [184]. Another area to consider is the relationship between grain boundary migration and pipeline steel. The pinning mechanism, known as the Zener drag/pinning effect is known to control grain boundary migration [185]. While there is no consensus on how halting grain boundary migration affects the corrosion resistance of pipeline steels, a study by Jack and Szpunar had an interesting insight. In their study, the addition of more niobium (Nb) resulted in the improved corrosion resistance of the steel due to certain favorable microstructural features, as against Nb deficient steel. Amidst many reasons for the improved corrosion resistance by the authors, the grain boundary pinning due to precipitates around them resulted in more refined grains, which was more beneficial for the steel in the selected acidic solution, hence improving its corrosion resistance [186] as shown in Fig. 8.

Also, in terms of corrosion resistance, Wright and Field proposed that the combined effect of grain boundary and crystallographic texture can effectively influence the corrosion resistance of polycrystalline materials [187]. Interestingly, with characterization techniques like transmission electron microscopy (TEM), electron backscatter diffraction (EBSD), and electron channeling contrast imaging (ECCI), the inner workings relating hydrogen interaction with dislocation pileups in grain boundaries have been probed. Koyama et al. made an interesting milestone in probing the effect of hydrogen-assisted degradation mechanism in twinning-induced plasticity (TWIP) steel. They concluded that initiated HIC is easily propagated at grain boundaries [188] as Fig. 9a shows crack initiating at a triple junction before.

propagating along grain boundaries. Fig. 9 (b-c) shows that most micro-cracks that were found along HAGB as indicated in the EBSD inverse pole figure (IPF) maps obtained for both ND-TD (Fig. 7b) and RD-TD (Fig. 7b) planes respectively.

With hydrogen trapping sites like grain boundaries gaining attention based on their affinity to hold hydrogen, it is imperative to understand other trapping zones and their role in the HIC sensitivity of pipeline steels.

### 5.5. Inclusions and precipitates

Unlike other microstructural features, there are few techniques available for investigating the role of inclusions and precipitations on HIC nucleation at the microscopic level. Nonetheless, many studies have been made on the effects of inclusions/precipitates on the HIC resistance of pipeline steels [189]. It is believed that inclusions and precipitates foster hydrogen attacks on pipeline steel, as they provide accumulation sites for hydrogen, which has a deleterious effect on the mechanical properties of pipeline steel [27,190,191]. It has also been implied that inclusions/precipitates are irreversible trapping sites, having higher binding energies to hold hydrogen for longer periods. Inclusions, which are usually harder than the steel matrix, are seen as crack initiation/propagation regions, as they can create an incoherent interface with the pipeline steel matrix, thus increasing hydrogen trapping and causing failure by cracking [191]. However, not all inclusions are incoherent sites. The coherent sites have less affinity for cracking initiation.

Inclusions that are either coherent or semi-coherent with the steel matrix have lower binding energy for hydrogen compared to those that are incoherent [146,192]. Wei et al. investigated the role of incoherent TiC precipitates in hydrogen trapping [193]. Incoherent TiC was revealed as a hydrogen trapping site based on temperature and volume of particles instead of the interfacial area with the steel matrix. According to the authors, the high affinity for hydrogen trapping within incoherent TiC particles was related to the high concentration of octahedral carbon vacancies in the semi-coherent particles with fewer carbon vacancies. In the trapping sites, an increase in the activation energy required for cracking with a reduction in coherency was noticed. Since in the service environment pipelines experience fluctuating temperatures, it is imperative to understand how the temperature affects the trapping nature of reversible traps or irreversible traps. From Fig. 10, the hydrogen accumulation mechanism around different inclusion types can be understood. An interesting region to note is voids. These voids become hydrogen-trapping sites and consequently become problematic. When hydrogen migrates into the interfacial voids, consequently, we might observe an increase in internal pressure and high-strain conditions to form cracks [194]. Usually, inclusions are either spherical or elongated, with both having their hydrogen-trapping uniqueness. Elongated inclusions are known to have hydrogen atoms accumulating more at the edges where there is a high-stress concentration. It has been observed that there are often colonies of several metallic inclusions along the HIC propagation path in pipeline steel [195]. Jack and Szpunar were also able to show how precipitates/particles that pinned grain boundaries induced some corrosion resistance on high niobium pipeline steel [186]. Given that cubic and rectangular-shaped particles were mostly found in the high Nb steel, a stronger and more effective pinning of grain boundary movement was reported. Ringer et al. also reported that cubic-shaped particles are far more effective than spherical and ellipsoidal particles in pinning grain boundaries, with a Zener pinning pressure almost two times that of the globular (spherical) particles [196]. The effect of the shape or morphology of inclusions and precipitates on the HIC resistance in pipeline steels has been controversial. There is a general belief that spherical-shaped inclusions don't affect the HIC resistance of pipeline steel, as much as other morphologies. Bonab et al. reported that spherical inclusions of oxides formed by aluminum (Al), calcium (Ca), manganese (Mn), and magnesium (Mg), which constituted 70 % of the total amount of inclusions did not cause HIC damage to X70 pipeline steel. Instead, the inclusions fostered a significant decrease in fracture toughness. While the authors believe that the significant reduction of HIC damage was a result of the spherical-shaped inclusions, which also supports the general belief, there still exists a controversy. Jin et al. reported that oxide inclusions of Al and Si induced HIC amidst other oxides of Ca-S-Al-O type and MnS found in X100 pipeline steel [194]. Fig. 10 a and b mainly show MnS and aluminum oxide types of inclusions surrounded by porous voids at the pipeline steel matrix/inclusion interface. It can be concluded from these results that cracks nucleated at the cavities formed because of poor adhesion of pipeline steel matrix and inclusion. In addition, Fig. 10e indicates HIC nucleating from oxide inclusions of Al and Ca, as well as carbonitride precipitates of Ti, Nb, and V (Fig. 10c and d). More findings also supported the findings that oxide inclusions of Ca and Al induce HIC in X70 pipeline steel [65,182]. Techniques for controlling the morphology of MnS inclusions have gained significant attention. For instance, the use of fine oxide inclusions as heterogeneous nucleation sites by adding titanium, magnesium, and zirconium [17,197]. Another method is modifying the morphology of the MnS inclusions by calcium or rare earth treatment [198,199]. Wang et al. proposed the addition of tellurium as a way of controlling the morphology of MnS inclusions [19].

While precipitates and inclusions are usually irreversible traps, several studies have shown that the precipitates are less harmful. Martensitic steels having carbide precipitates have higher strength, which often results in cracking and easy crack propagation. Qifeng et al. reported that carbide precipitates resulted in higher local normal stresses and lower plastic deformation, which increased the overall strength. The carbide precipitates impeded dislocation motion, resulting in high local stresses around the carbide precipitates, which led to large crack opening stresses on cleavage planes of  $\{100\}$  [201]. Inclusions, which are already inherent in the steel during the production phase, remain a bigger threat to the steel's HIC resistance. Peng et al. investigated the effect of non-metallic inclusions on the critical size for HIC initiation and their role in hydrogen trapping. Their study covered some MnS inclusions and some oxide inclusions. It was reported that MnS inclusions brought about a larger critical size tolerance of HIC nucleation (approximately 2.5  $\mu\text{m}$ ), but for some typical oxide inclusions, it ranged between 0.1 and 0.4  $\mu\text{m}$ . From their study, it can be deduced that finely dispersed non-metallic inclusions can trap more diffusible hydrogen and improve the HIC resistance of steel [202].

Overall, several inclusions have been identified in pipeline steels, which are enriched with silicon, aluminum oxide, magnesium, calcium oxide, carbide, iron, and manganese as shown in Figs. 11 and 12. The effect of inclusions on the HIC resistance of pipelines is dependent on their type, amount, size, and distribution. For instance, Kim et al. reported that the hydrogen-induced cracks primarily nucleated at inclusions enriched with Al and Ca oxides. Also, inclusions with a size over 20  $\mu\text{m}$  in length induced HIC damage in steels that contained a bainitic ferrite structure [203].

The number of inclusions also contributes to HIC in pipeline steels. Huang et al. investigated the effects of inclusions on the HIC susceptibility of X120 pipeline steel and concluded that the larger number of inclusions contributes to the HIC of the steel [204]. While it might be difficult to control the number of inclusions, the geometry of inclusion can be largely controlled. It was demonstrated that

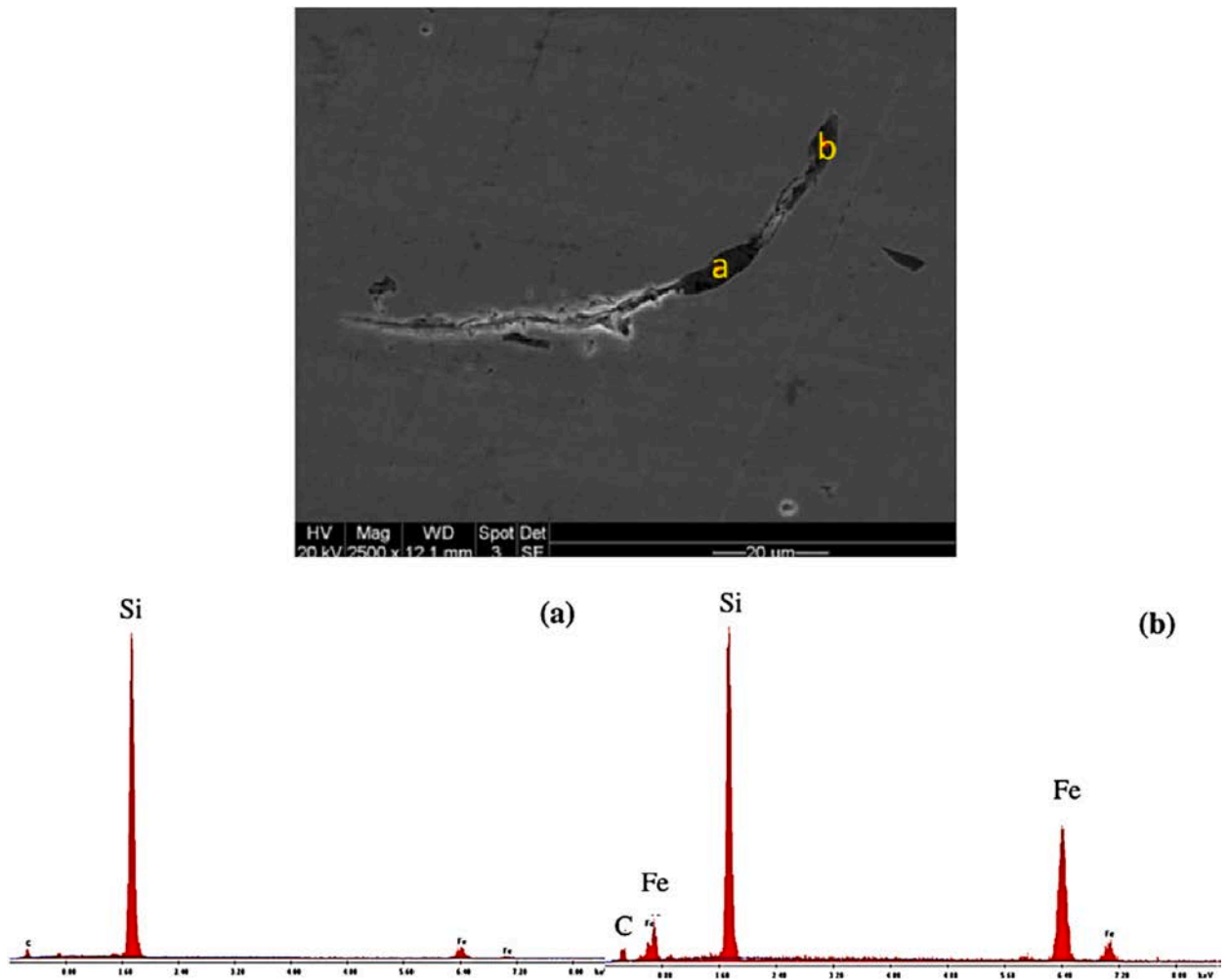


Fig. 11. SEM image of a crack initiating from a Si-enriched inclusion [204].

the resistance of steels to HIC can be improved by controlling the nature and geometric characteristics of the non-metallic inclusions [18]. Interestingly, Jiang et al. simulated the modification of inclusions by rare earth metals Ce and checked the SCC resistance, experimentally. The authors concluded that the risk of cracking was eliminated or alleviated, and the SCC resistance of the steel was improved [205]. Also, they reported that the composition of inclusions containing Ce will change during solidification, combining with S in molten steel to reduce its activity and further help to avoid the formation of CaS + Ca–Al–O duplex inclusions.

Interestingly, recent research indicates that the orientation of grains in pipeline steels also exerts an impact on their resistance to hydrogen-induced cracking (HIC). This preferred grain orientation is called crystallographic texture, and it has been a subject of interest to material scientists.

### 5.6. Crystallographic texture

Another important microstructural feature of pipeline steel is crystallographic texture. Texture represents the preferred orientation of grains and has been discussed concerning pipeline corrosion and HIC resistance. This is because TMCP induces deformation on the pipeline steel, causing grains to be oriented. The texture is often inhomogeneous through the thickness of the steel and these inhomogeneities have been reported to influence crack initiation and propagation in pipeline steel [206]. With these unavoidable texture inhomogeneities after hot rolling, it has been reported that boundaries between grains having {110} and {111} planes oriented parallel to the pipeline steel surface are more resistant to hydrogen-related degradation [142,207–209]. To ascertain the effect of crystallographic texture on the HIC resistance of pipeline steel, Lavigne et al. utilized three-dimensional crack images obtained from cracked X65 pipeline steel through X-ray microtomography. From Fig. 13, the cracks are observed to begin perpendicular to the pipe's outer surface but deviate out of its path on approaching the mid-thickness region.

To get a better understanding regarding the reason for the deviation, the authors applied neutron diffraction texture measurements, where they observed that the point at which the crack path deviated corresponds to the region where the crack encountered a strong

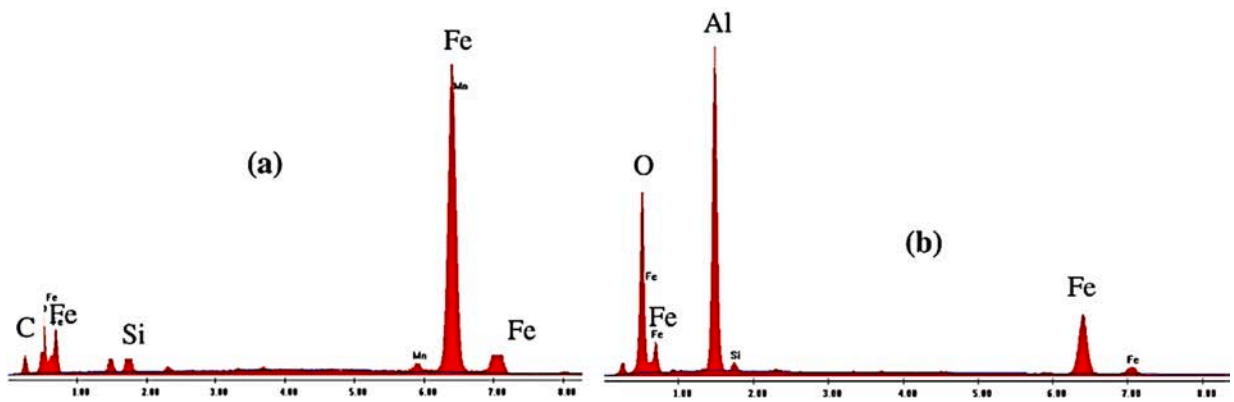
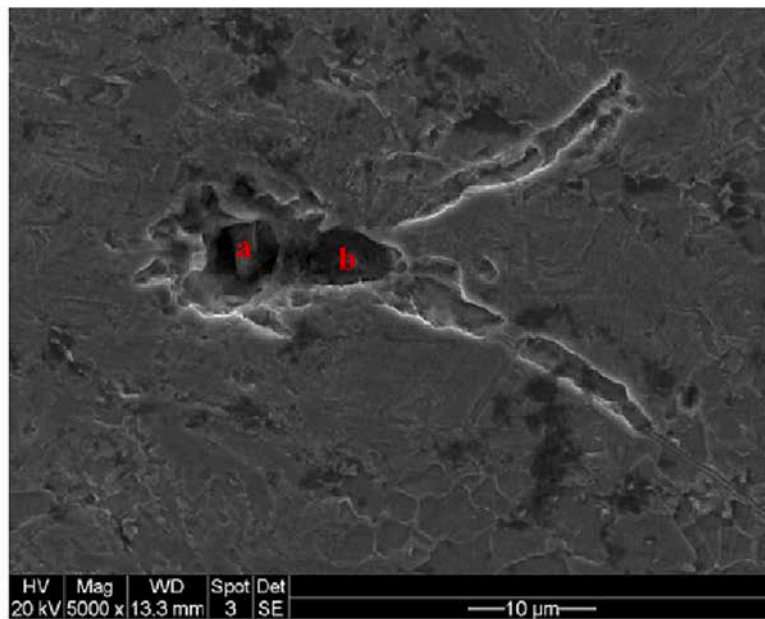


Fig. 12. SEM image of a crack initiating from an inclusion enriched in Si and ferric carbide [204].

presence of shear texture such as  $\langle 110 \rangle || TD$  with  $\{112\} \langle 111 \rangle$  orientation as maxima. However, the region where the crack propagated perpendicular to the outer surface before deviating was dominated by  $\langle 111 \rangle || ND$  (-fibre) and  $\langle 110 \rangle || R$  alpha fiber-oriented grains, with  $\{100\} \langle 110 \rangle$ ,  $\{111\} \langle 110 \rangle$  and  $\{111\} \langle 112 \rangle$  as maxima.

Interestingly, Jack and Szpunar had an observation regarding the role of texture on the corrosion resistance of high and low niobium steels. They found that the high Nb steel had conventional shear texture components, indicated by  $\{011\} \langle 100 \rangle$  and  $\{011\} \langle 211 \rangle$  along the  $\zeta$ -fiber in Euler space as shown in Fig. 14. This was also marked by stable  $\langle 110 \rangle || ND$ -oriented grains observed in the IPF and more  $\{110\}$  (green-colored) grains on the steel surface, as seen in the EBSD maps. The ODF of the low niobium indicated a strong presence of the rotated cube texture component,  $\{001\} \langle 110 \rangle$ , with more  $\{100\}$  (red-colored) grains observed in the EBSD maps. Also, from the IPF for the low Nb steel, the grains seem to be oriented more towards  $\langle 100 \rangle || ND$  than any of the other low-index directions. While these crystallographic texture dissimilarities were linked to internal strain differences between the two steel samples, it is worth noting that the high niobium steel showed better corrosion resistance than the low niobium steel because of their difference in texture [186].

Also, Arafin and Szpunar were able to establish that several patterns of intergranular crack propagation of the X65 pipeline were dependent on texture [11]. In their study, some cracks were mitigated at points having LAGB. This study confirms the general belief that HAGB promotes crack propagation, and it is imperative to understand that the cracking resistance of any pipeline steel is not always linked to one microstructural parameter, and most of the time several parameters are responsible for the cracking of pipelines, and this is why microstructure is greatly considered when studying hydrogen pipeline embrittlement. After the texture analysis of different cracks was obtained and the corresponding crack tip regions, it was established that texture was responsible for this result. From texture analysis, the cracks propagated dominantly through grains with  $\{100\} || RP$  (rolling plane). However, the crack tip region was dominated by grains with  $\{110\} || RP$  oriented grains and spawned the conclusion that intergranular cracks were arrested

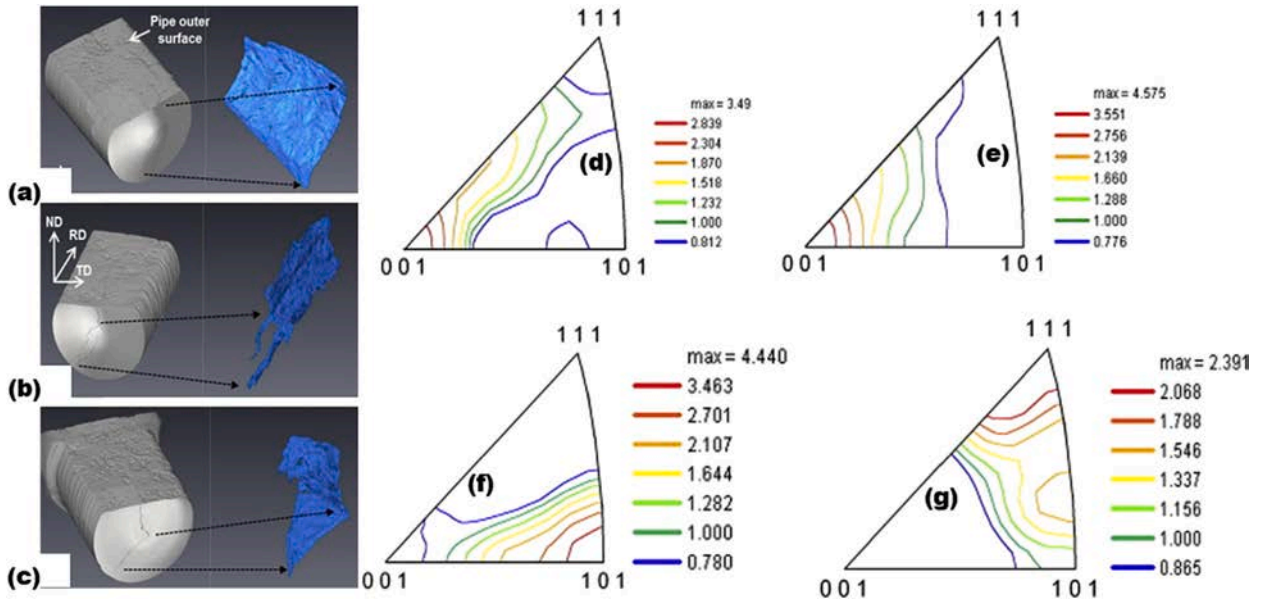


Fig. 13. Reconstructed three-dimensional X-ray microtomography images showing deflected cracks and volumetric segments of cracked area in (a-c) [10], IPF obtained from cracked regions (d, e), and IPF obtained ahead of crack tips (f, g) [11].

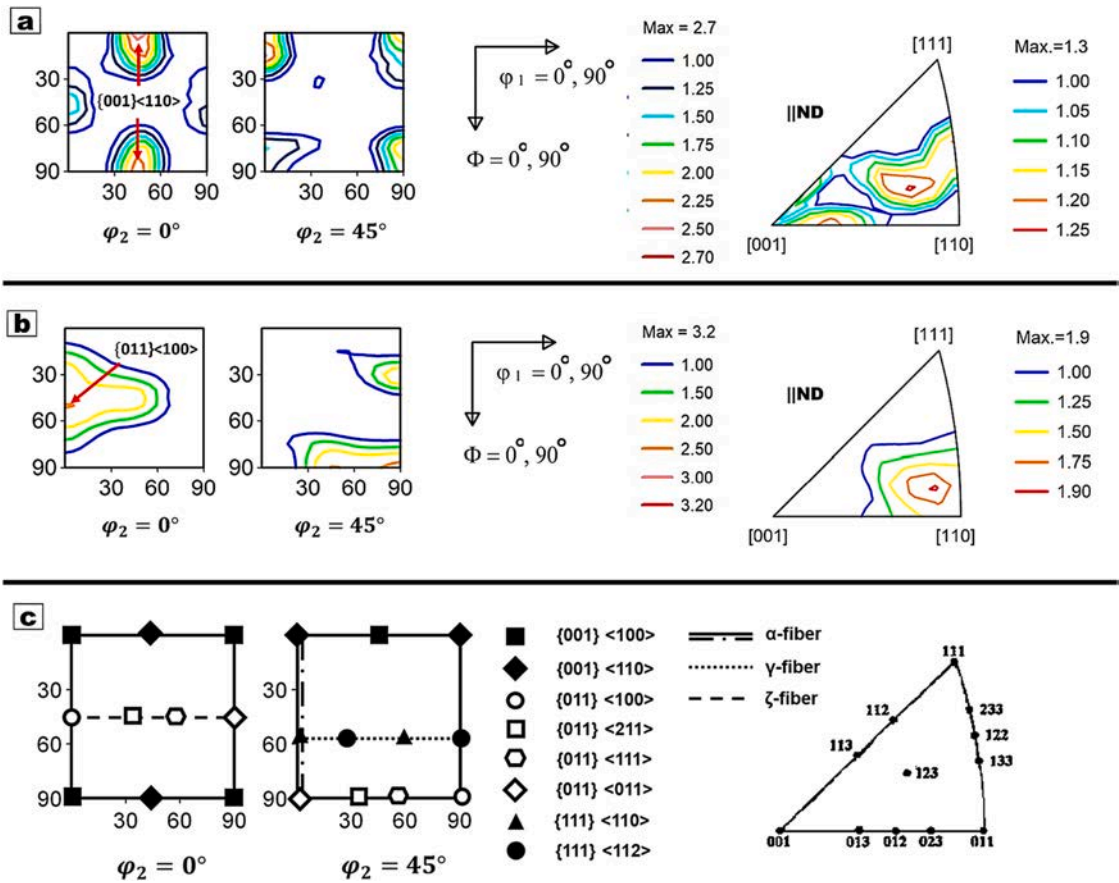
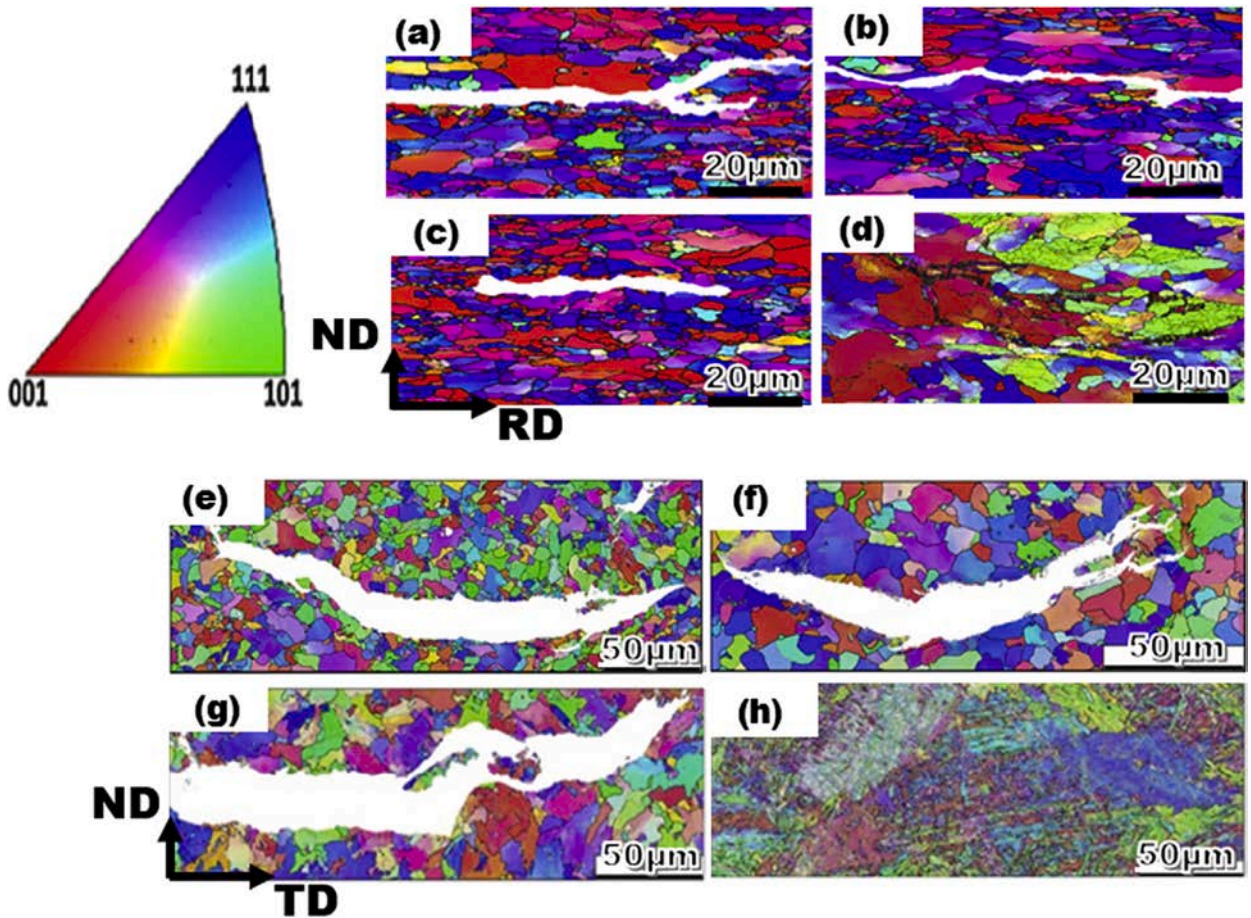


Fig. 14. XRD results showing the ODF and IPF of the top RD - TD planes of the (a) low and (b) high Nb steels, and (c) Some ideal texture components and fibers [186].



**Fig. 15.** EBSD orientation maps for HIC in X70 pipeline steel with different microstructures and texture (a to c) shows cracking at different spots containing small grains [10] (d) crack initiation and propagation through the boundary of  $\{100\}$  grain [87], (e) banded ferritic-pearlite (f) ferritic-granular bainite (g) equiaxed ferritic-pearlite (h) bainite-ferrite [182] (ND: normal direction, RD: rolling direction and TD: transverse direction of pipeline steel plate).

at grains with  $\{110\}||RP$  texture. While the authors studied intergranular cracks, it will be also good to look at other cracking types and their correlation with crystallographic texture.

Overall, emphasis has been placed on engineering pipeline steels having  $\{111\}$  planes parallel to their surfaces as a way of increasing their cracking resistance. However, there is no established consensus on how to engineer pipeline steels to achieve  $\{111\}$  planes. Interestingly, Omale et al reported that warm rolling with a finish rolling temperature of  $700^\circ\text{C}$  favored the development of the  $\gamma$ -fiber texture, due to complete recrystallization [210]. According to the authors, the  $\gamma$ -fiber texture is better formed at the mid-thickness compared to the surface. This was also in line with Jack et al. report, that the shear-type deformation at the surface layer due to the sticking effect of the roller and the steel surface can lead to the formation of shear textures, which often exhibit  $\{110\}$  orientations parallel to the normal direction,  $\langle 110 \rangle || ND$  [211]. The inner layers (mid-thickness) are mainly deformed by plane strain deformation, which gives rise to the formation of conventional textures ( $\gamma$ -fiber and  $\alpha$ -fiber) [212].

Masoumi et al. were able to establish that the development of dominant  $\{011\}$  grains parallel to the normal direction, and a small number of  $\langle 001 \rangle || ND$  grains obtained by isothermal rolling at  $850^\circ\text{C}$ , increased the hydrogen-induced crack resistance; while the hot rolled sample with sharp  $\langle 001 \rangle || ND$  textures was highly susceptible to cracking [90]. They revealed that the preferential HIC propagation occurs along regions dominated by small grains within the X70 pipeline steel microstructure, where the said regions were confirmed to contain more dislocations and grain boundaries, which are known as hydrogen trap sites and zones for HIC initiation and propagation in the steel structure as shown in Fig. 15 a-c. The authors established the reason to be the high stored energy associated with small grains which is known for crack promotion around such regions. However, we recalled a controversial inference in the study, where the authors suggested the possibility of cracks propagating along boundaries of  $\langle 111 \rangle || ND$  and  $\langle 011 \rangle || ND$  oriented grains contrary to the belief that these orientations are HIC resistant [11,87,187,207,213]. Nevertheless, it is also understood that the grain boundaries of crack-resistant grains can be susceptible to cracking depending on their elastic energies. A perfect example is shown in Fig. 15d where HIC cracks were induced at  $\langle 001 \rangle || ND$  oriented grain (red grain) but propagated through the boundaries of other grains with  $\langle 111 \rangle || ND$  and  $\langle 011 \rangle || ND$  orientation. The result was due to the grain boundaries through which the crack propagated, as

they were mainly HAGB. Despite the crack-resistant orientation of the so-called  $\{111\}$  grains, the crack continued its propagation because of the high stored energy stored in grain boundaries of small grains.

This is why some researchers have the opinion that texture has little or no effect on the HIC resistance of pipelines. Perhaps, since its effect is dependent on other microstructural features, it can be mitigated. However, it remains an interesting area of research and has attracted significant interest. Also, due to texture inhomogeneities, texture measurements will vary significantly depending on the processing history of the pipeline. Nevertheless, many studies have made use of the EBSD technique in correlating HIC and crystallographic texture even when a small sample area is used to account for the entire pipeline specimen [11,81,90,182]. We can draw inferences from different X70 pipeline steel samples with microstructures ranging from equiaxed ferrite-pearlite (Fig. 15g), banded ferrite-pearlite (Fig. 15e), ferritic-granular bainite (Fig. 15f), and bainitic-ferrite (Fig. 15h). It was established that *trans*-granular cracks propagated in all steels except the one with bainitic-ferrite microstructure. However, the most severe cracking occurred in the pipeline steel with banded ferritic-pearlite microstructure. While cracking resistance was noticed at CSL and LAGB, there was no dominance of a particular crystallographic orientation on the crack path. It was just composed of cracks that were preferentially propagating through the best-aligned slip planes, which correspond to  $\{110\}\{123\}$ ,  $\{112\}$  planes, as well as  $\{100\}$  cleavage planes. Consequently, it was concluded that cracking was controlled by a dominantly slip-based mechanism and less cleavage. This is not the first study revealing no crystallographic texture dominance in steel lattices. Venegas et al. have already established the absence of any dominant orientation along the HIC propagation path on X46 pipeline steel [183]. While their study was performed on specimens obtained from an already-formed pipeline, it is quite interesting to note that the cracks aligned towards the radial direction of the pipeline in a somewhat 'S' shape, indicating a crack connection along slip planes. Also, pipelines experience hoop stresses along their radial direction during service, which means that strain fields near cracks could potentially cause the convergence of smaller cracks into larger ones. One of the recent areas of research regarding the corrosion behavior of pipeline steels is the role of grain size and crystallographic texture on corrosion [209]. This has set up an array of interest regarding the role of grain size in HIC and corrosion.

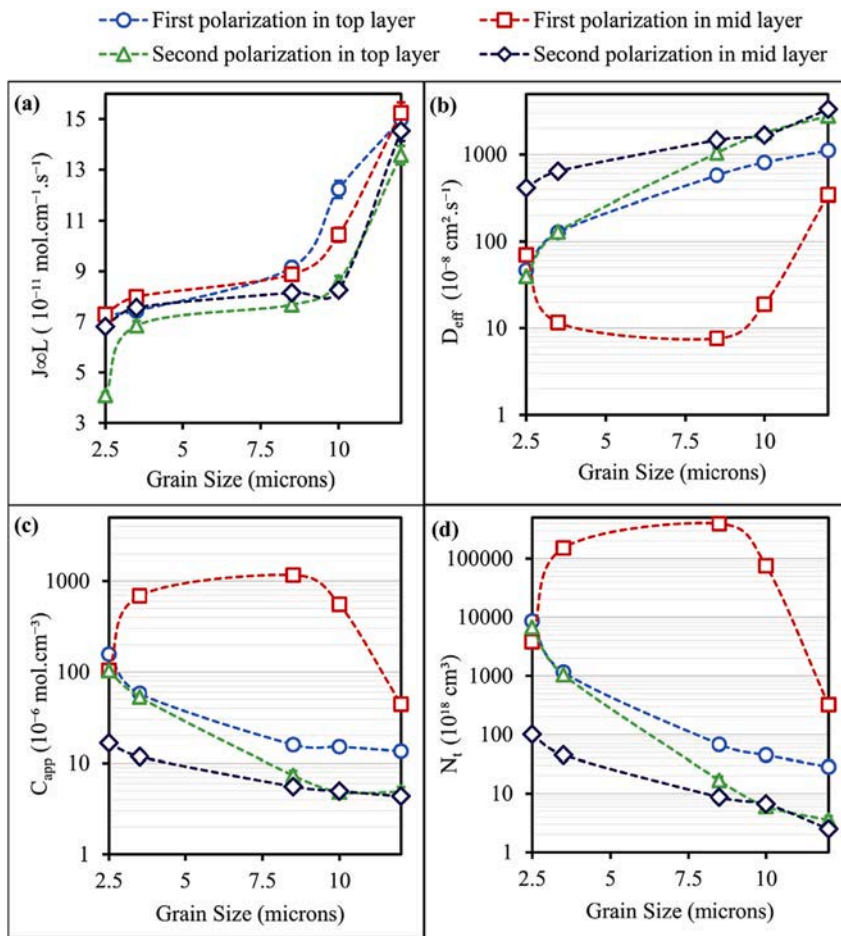
### 5.7. Grain size

Grain size is a microstructural parameter that hasn't been of great interest to researchers. Yet, few studies have shown its importance and roles in pipeline steel corrosion. Generally, ferrite grain size is an important factor for a pipeline's mechanical strength. That's why pipeline steel of properly refined grain sizes will show high mechanical strength [214–217]. However, due to higher energy and chemical activity on grain boundaries, as the density of these boundaries increases, there would be many electron activities and atom diffusion, subsequently resulting in failure [218,219]. While there is no established consensus regarding the role of grain sizes on hydrogen embrittlement, corrosion, and other problems encountered by pipeline steels, it is imperative to discuss a few studies regarding this subject matter. Another important idea is how grain sizes influence grain boundaries since bigger grain sizes will have a lower percentage of grain boundaries per area. Interestingly, grain boundaries have unique properties relative to the bulk material in terms of atomic coordination, reactivity, and diffusion rates [218]. On a plain view, it is expected that pipeline surfaces with relatively high grain boundary densities will show different electrochemical behavior in terms of corrosion than coarser-grained surfaces with lower grain boundary densities. While it has been established that yield strength is inversely proportional to grain size [215,220], there is no consensus on the relationship between grain size and corrosion rate, let alone, hydrogen-induced cracking (HIC). From a few studies, it is generally believed that the corrosion rate increases as grain sizes increase [221–224]. That is, the corrosion resistance of a material is directly proportional to its grain size.

Maryam et al. investigated the role of grain sizes on pipeline steel corrosion. The authors were able to study the corrosion resistance of different grain-sized pipeline steel samples. They concluded that two distinct stages for the dependency of corrosion current density ( $i_{\text{corr}}$ ) on grain size exist. The authors revealed that above a limiting average grain size of  $\sim 22 \mu\text{m}$ ,  $i_{\text{corr}}$  decreased slowly with increasing grain size. Below this limiting value,  $i_{\text{corr}}$  increased rapidly, which was related to the increased density of grain boundaries as interpreted by theoretical calculation of the number of grains per unit area. While the issue of limiting average grain size remains controversial, the authors were not able to reveal a reason for such a shift. Perhaps, since there were no strain analyses in the author's experiments, it will be very difficult to know the reasons behind their conclusions since internal strains can affect the corrosion resistance of pipeline steels. Also, since it is known that NaCl corrosion medium has low passivation affinity, unlike other mediums with hydrogen evolution potential, it might be the reason for their conclusions [209]. On the other hand, since different microstructural features affect corrosion, it will be very challenging to ascertain the effect of only grain size on corrosion.

Y Li et al. also investigated the role of grain size on the corrosion of surface nano-crystallized low-carbon steel. They revealed that the corrosion rate of the SNC low-carbon steel increased with the decreasing grain size [225]. The authors attributed the decrease in corrosion resistance to the increased number of active sites in SNC low-carbon steel. However, the corrosion rate is different in different corrosion media. Wang et al. were able to report the corrosion properties of fine and coarse-grained steel samples [209]. According to their study, grain refinement decreased corrosion resistance in the NaCl solution but improved corrosion resistance in the  $\text{NaHCO}_3$  solution. According to the authors, the reason is that coarse-grained steel is known to have anodic passivation, while grain-refined steel can induce self-passivation.

Interestingly, this was also in line with Jack and Szpunar's report on corrosion on similar steels that had ununiform grain sizes [186]. According to the authors, there was enough passivation in the region with greater density of grain boundaries, while little passivation in the region with bigger grain sizes (less grain boundaries). While the authors had the same conclusion as Wang et al., their reasons for their conclusion were quite different. They revealed that there is no hydrogen evolution during the corrosion process in the NaCl corrosion medium, unlike the  $\text{NaHCO}_3$  solution, therefore less passivation and more corrosion affinity were reported. As such, hydrogen emission in a corrosion process creates room for more passivation, and thus, better corrosion resistance. Another interesting



**Fig. 16.** Effect of grain growth on diffusion after dual-polarization (a) permeability (b) effective diffusion coefficient (c) apparent solubility and (d) trapping sites [226].

angle is how the diffusion of hydrogen in specimens of different grain sizes affects susceptibility to cracking. Thomas and Szpunar investigated the hydrogen diffusion and trapping in X70 pipeline steel. They concluded that the hydrogen diffusivity decreases with increasing grain size, and the density of trapping sites is reduced with decreasing grain size. Fig. 16 (a-d) shows the permeation parameters obtained for the annealed steel in the first and second polarization of the permeation experiments.

While this conclusion stresses the fact that finer grains have more trapping sites and experiences lesser hydrogen diffusion due to higher number of grain boundaries, it will be imperative to understand how grain size affects the mechanical properties of pipelines in relationship to diffusion and trapping density. While there is scanty literature on this, it remains worthwhile to carry more detailed investigation.

## 6. Effect of hydrogen on pipeline welds

The tailoring of the microstructure of pipeline steels in achieving desired microstructural parameters for better cracking and corrosion resistance is paramount, however, in an ideal situation, pipeline steels are welded together to transport oil and gas substances over longer distances. These microstructural variations across weld joints have been reported to cause cracking because of the formation of intermetallic compounds and high welding heat input [227]. With the presence of inclusions and retained atomic hydrogen, these cracks are easily initiated and propagated in pipeline welds [228]. The science behind the hydrogen effect in welds is that when hydrogen is introduced into the liquid weld pool, it becomes difficult for it to escape when the weld cools down, and this often initiates HIC in the heat-affected zone (HAZ) and the weld-metal (WM) due to stress build-up. When the applied heat is high, hydrogen solubility is increased, which in turn increases hydrogen retention potential after cooling down. Also, rapid cooling saturates the weld with hydrogen and enhances the tendency for the trapped hydrogen to migrate to other trap sites. This could potentially lead to cracking of the weldment, even at ambient temperature conditions. On the other hand, cooling welds at a slower rate causes low retention time for hydrogen in the weldment as it can escape before solidification. Welding often gives rise to three distinct zones such as unaffected base metal (BM), the heat affected zone (HAZ), and the weld metal (WM) or fusion zone (FZ). Out of these zones, the most



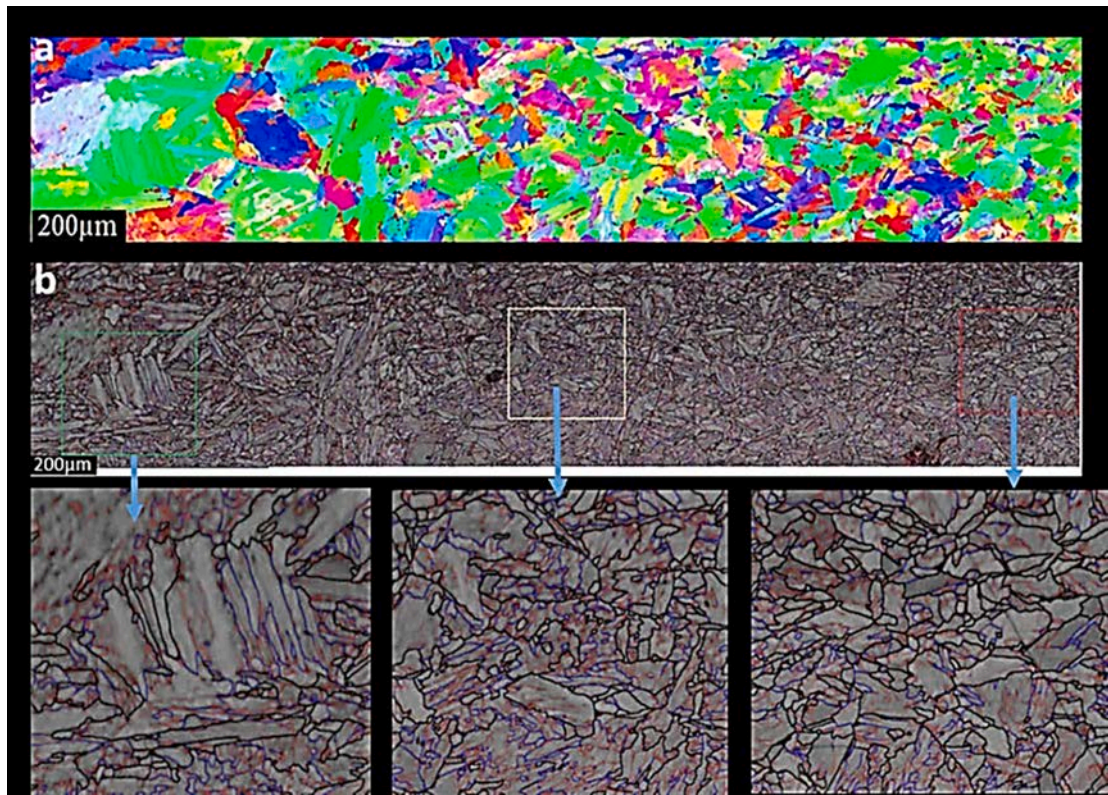


Fig. 17. (a) EBSD map of HAZ (b) image quality with imposed grain boundaries showing the subzones of HAZ (from CGHAZ to FGHAZ).

HIC-prone is the HAZ because of its higher hardenability in comparison to the base metal [229]. The HAZ, the zone between the BM and FZ, experiences peak temperature below the solidus temperature but is high enough to cause a change in the microstructure and mechanical properties of the area compared to the BM. The HAZ experiences different temperatures, moving from the FZ to the BM, this is what results in microstructural variation. This microstructural variation in the HAZ can be influenced by the heat input, the highest temperature reached, the holding time at the elevated temperature, and the cooling rate [230]. For instance, Zhu et al. investigated the effect of heat input on the interfacial characterization of the butter joint of hot-rolling CP-Ti/Q235 bimetallic sheets by Laser + CMT. They noticed that reducing the welding heat reduced the formation of intermetallic compounds, which improved the tensile strength of the weld when compared to higher welding heat [227]. Yuhua et al. also investigated the effect of welding crack in micro laser welded NiTiNb shape memory alloy and Ti6Al4V alloy dissimilar metals joints. The authors noticed a significant increase in weldment cracks after a higher laser power was used. The authors also revealed that a brittle Ti<sub>2</sub>Ni phase with more contents was present in the weld due to final solidification, which was the main cause of crack formation along with large stress concentration [231].

Due to temperature variations, four zones of the HAZ are known such as coarse-grained (CGHAZ), fine-grained (FGHAZ), inter-critical (ICHAZ), and tempered zones. It is worthwhile to know that the change in temperature leads to changes in grain sizes which in the end affects mechanical properties. For instance, Hashemi and Mohammadyani observed that the hardness of the welded joint in X65 pipeline steel varied as testing moved across the CGHAZ, FGHAZ, ICHAZ, and tempered zones [232]. Fig. 17 (a-b) shows the EBSD map and microstructure of the HAZ. From 17a, it can be deduced that there exists a variation of the grain boundary structures with distance from the CGHAZ to the FGHAZ.

In another study, Shaiful et al. investigated the impact of heat input on the hardness and toughness of austenitic 202-grade stainless steel weldments [233]. They found out that the joints made by using low heat input exhibited higher hardness and impact toughness values than those welded with medium and high heat input. It can be deduced that high heat input can lead to an increase in grain size, which has a knock-on effect on hardness and toughness value. The findings of Choubey et al. were also in line as they found that the tensile strength decreases with an increase in welding heat input [234].

There are many studies revolving around the influence of hydrogen on the integrity of pipeline welds. For instance, Yue investigated the HAZ hydrogen-induced cracking of high-strength naval steels using the Granjon implant test [229]. They found out that CGHAZ is the most HIC-prone HAZ because the CCT diagrams revealed both bainite and martensite form in the CGHAZ of HY-100 and HSLA-100 depending on the cooling rate, as shown in Fig. 18. While for BA-160, martensite was found to be the predominant phase in the CGHAZ. In addition, they advised pre-heating the pipeline before welding as a form of increasing HIC resistance of the HAZ, as pre-heat helps to reduce diffusible hydrogen during welding by slowing down the cooling rate. This was also in line with the study of [235] Wang et al. They also noticed brittle fractures in the CGHAZ for both the as-welded and PWHT X80 pipeline samples. Their reason for

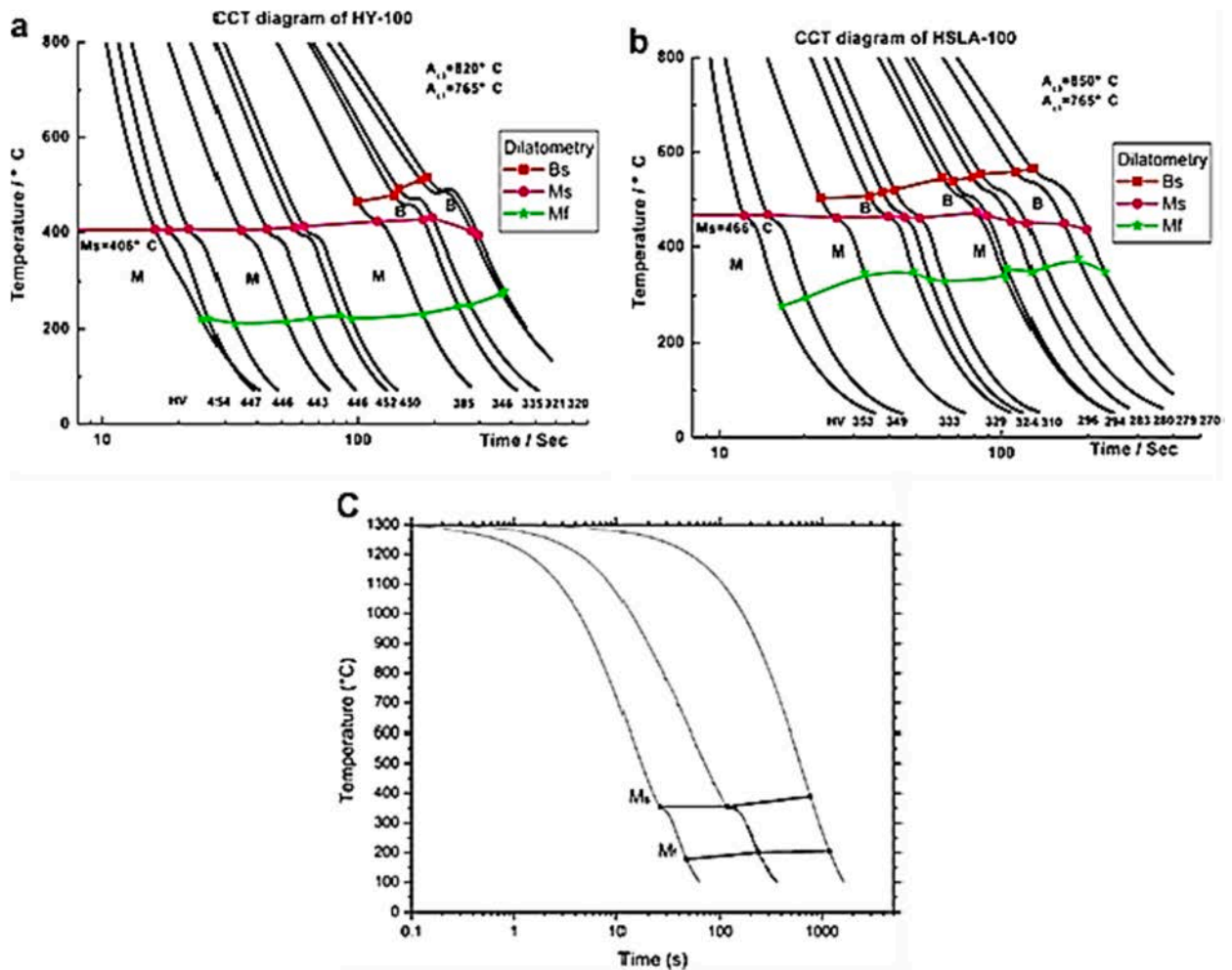


Fig. 18. Continuous cooling transformation diagrams for the CGHAZ of the three steels. a HY-100; b HSLA-100; c BA-160 [241].

the brittle fracture was because of the coarsening of the grain size on the fusion line, and the non-uniform distributions of Martensite/Austenite constituents. Fu et al. also investigated the HE behavior of SUS301L-MT stainless steel laser-arc hybrid welded joint localized zones. Utilizing an in situ electrochemical hydrogen charging during slow strain rate tensile (SSRT) tests, they noticed that the BM and HAZ zones exhibited higher plasticity loss than that of WM, but almost the same strength loss [236]. Luo et al. investigated the SCC behavior of X90 pipeline steel and its weld joint at different applied potentials in near-neutral solutions [236]. The authors noticed three mechanisms in the SCC behaviors of base metal and weld joint samples; anodic dissolution mechanism when the applied potential is open circuit potential (EOCP), anodic dissolution and hydrogen embrittlement mechanism when the applied potential is  $-850$  mV, and hydrogen embrittlement mechanism when the applied potential is  $-1000$  mV and  $-1200$  mV. Eliminating joint and residual stresses is another way of minimizing HIC in pipeline weldments [237]. This is because joint stresses in combination with the stress created from the presence of hydrogen, can lead to embrittlement. Interestingly, Zhu et al. studied the use of ameliorated longitudinal critically refracted—attenuation velocity method for welding residual stress measurement. The LCR-AV technique is based on the linear relationship between velocity, attenuation, and grain size, which is a valuable quantitative technology to estimate the residual stress of welded joints [238]. Also, the use of low hydrogen or baked electrodes, coupled with pre-heating and post-welding heat treatments is widely recommended [239,240].

## 7. Gaseous hydrogen and pipeline steels

Hydrogen transport using existing pipeline steels has recently stimulated a lot of research.

The awareness of the effect of greenhouse gases is setting hydrogen as the future energy vector [242]. While there is scanty literature on pipelines originally designed for hydrogen storage or transport, there is an increasing demand for the use of existing pipeline steels (not originally designed for hydrogen transport or storage) to transport hydrogen gas [243]. This has made this subject matter somewhat interesting to delve into. Hydrogen, which exists in 75 % of all matter [244] is colorless, odorless, and nontoxic. As a

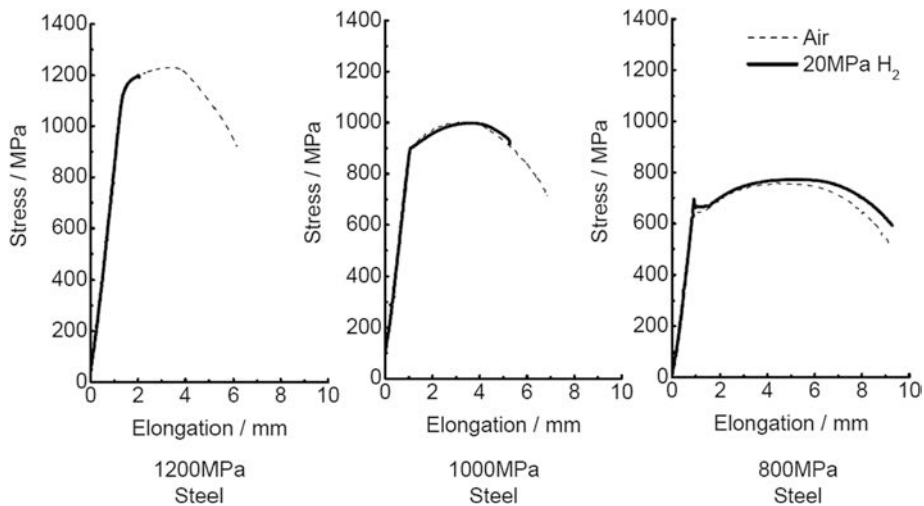


Fig. 19. Load-Displacement curve of pipeline steel sample [248].

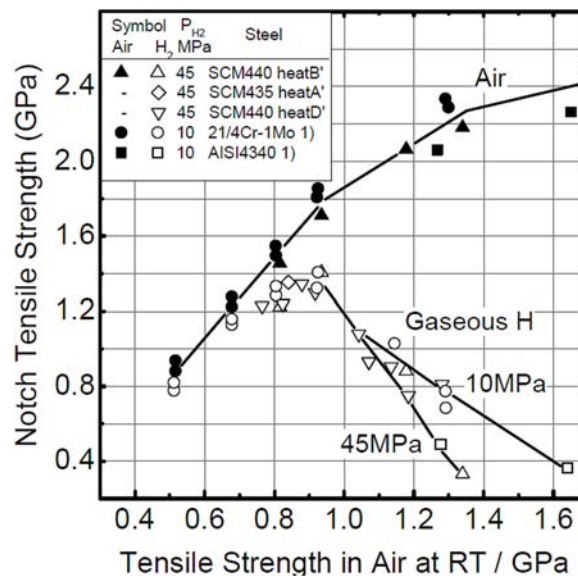
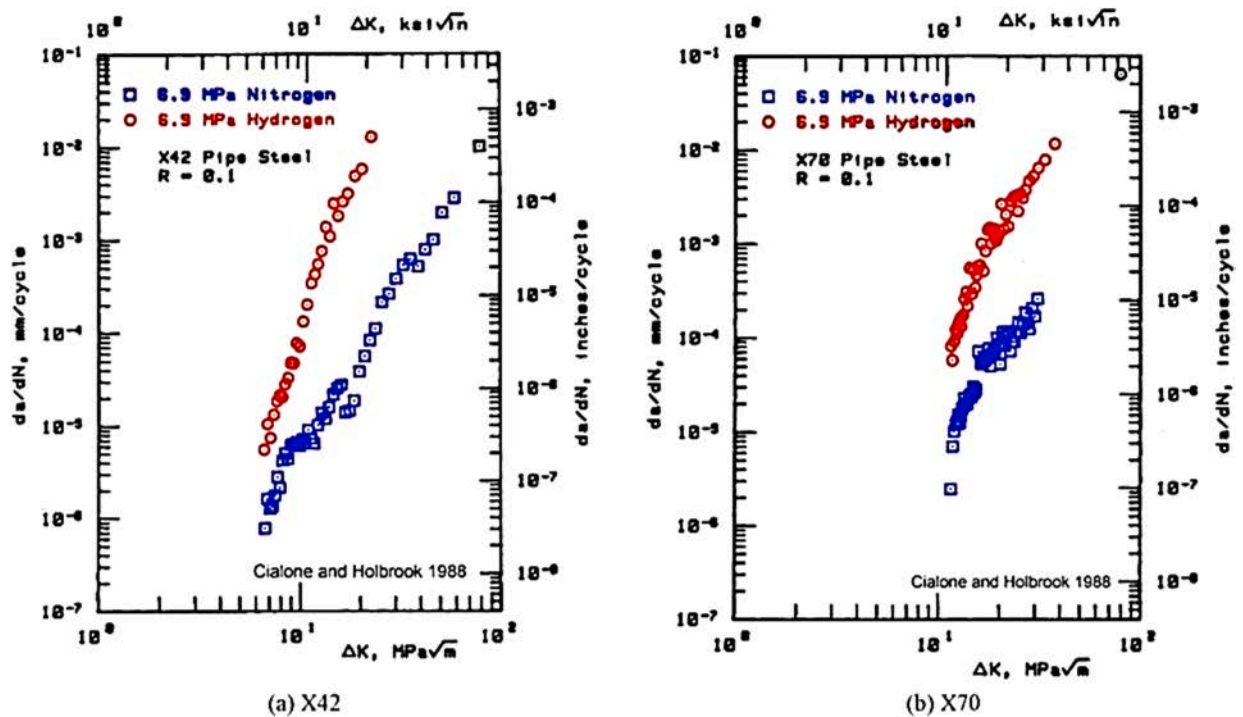


Fig. 20. Notch tensile strength as a function of the tensile strength of steel samples [248].

non-polluting substance, it can easily be released freely into the atmosphere [245]. However, it is very combustible and can be transported either as a liquid or as a gas [246]. While hydrogen can be produced in many ways, transportation and storage is more challenging than its production. Currently, hydrogen transportation and storage are operated under high-pressure (HP) conditions (i. e., 5–20 MPa for transportation, 35–100 MPa for storage) [247]. Just like other countries, Canada uses tube trailer trucks for transporting gaseous hydrogen, which is limited to the amount the truck can carry, causing high gas prices [242]. While Canada has developed a strategy for transporting hydrogen with trailer trucks, the envisioned sustainability during increased energy demand is not promising, increasing the need for using existing pipelines for transporting gaseous hydrogen. In the world today, only about 5,000 km of pipelines are used for hydrogen transport. However, increasing demand for energy has spiked the need for pipeline infrastructure expansion. This has led to the sorting of existing pipeline steels (not originally manufactured for hydrogen transportation.) for the transportation of hydrogen. To salvage the pressure on existing pipeline steels as a vessel for hydrogen gas transport, one needs to understand how hydrogen gas affects the mechanical properties of existing pipeline steels, by looking at the role of microstructure. Wada et al. were able to investigate the effect of hydrogen pressure on the mechanical properties of low alloy pipeline steels [248]. With a hydraulic servo-controlled testing machine, a tensile test was done on different steel samples under a gaseous hydrogen environment with a pressure of 20 MPa. From their mechanical testing, as the tensile strength of the pipeline increased, the elongation in gaseous hydrogen decreased. This means that a pipeline with high strength will fail faster in a gaseous hydrogen environment. It also shows that one of the main mechanical properties of pipeline steels vulnerable to hydrogen gas is ductility. These inferences are

**Table 5**  
Tensile ductility data for 0.22 % carbon steel (normalized at 900 °C) in hydrogen gas with various pressures [250].

Pressure (psig)	Pressure(atm)	UTS (ksi)	Elongation (gage:30 mm) %	Reduction of Area %
Ambient (Air)	1(Air)	70.8	32	64
147 (H <sub>2</sub> )	10 (H <sub>2</sub> )		34.5	52
294 (H <sub>2</sub> )	20 (H <sub>2</sub> )		33	47
735 (H <sub>2</sub> )	50 (H <sub>2</sub> )		30	50
1470 (H <sub>2</sub> )	100 (H <sub>2</sub> )		30	36.5
2205 (H <sub>2</sub> )	150 (H <sub>2</sub> )		26	28
1470 (Argon)	100 (Argon)		36	62



**Fig. 21.** Fatigue crack growth rates (da/dN) for (a) X42 and (b) X70 in 6.9 MPa (1000 psi) hydrogen and in 6.9 MPa (1000 psi) nitrogen at stress ratio R = 0.1.

evident as the steel sample with a tensile strength of 1200 MPa fractured under the hydrogen environment before reaching maximum tensile strength level, while the other sample with 1000 MPa strength fractured at the start of necking as shown in Fig. 19. The other sample of 800 MPa strength showed little or no hydrogen gas effect on tensile properties.

For the notched tensile test, the notch tensile strength(NTS) in gaseous hydrogen under the pressure of 10 MPa decreased above 930 MPa to 1000 MPa tensile level, while the NTS in gaseous hydrogen under pressure of 45 MPa had more significant decrease as shown in Fig. 20.

Hofmann and Rauls in 1961 carried out tensile test in hydrogen environment on 0.22 %carbon steel. These tensile tests obtained mechanical properties like yield stress, ultimate tensile strength, elongation, and reduction of area, of pipeline under hydrogen environment [249]. Table 5 shows the tensile ductility data for 0.22 % carbon steel (normalized at 900 °C) in hydrogen gas with various pressures.

Lam et al. also compiled a review showing the effect of hydrogen on the mechanical properties of pipeline steels, like tensile strength, fracture toughness, and fatigue strength [251]. Cialone and Holbrook investigated the tensile strength, subcritical crack growth, and fracture strength of API X42 and X70 pipeline steels under a hydrogen environment [252]. The fatigue test data of fatigue crack growth rate tests are shown in Fig. 21, where the fatigue crack growth rates in 6.9 MPa (1000 psig) hydrogen and 6.9 MPa (1000 psig) nitrogen are compared. A low-stress ratio (R = 0.1) was used in the fatigue testing. From Fig. 20, we can deduce that the da/dN appears to be higher in X42 steel than in X70 at the same ΔK level. In the case of X42, the fatigue crack growth rate can be 150 times greater than that in the nitrogen, under the same 6.9 MPa pressure. The tests were also carried out at higher stress ratios (R ranges from 0.1 to 0.8). These results for X42 are summarized in Fig. 22.

Zawierucha and Xu also investigated the fracture toughness of the as rolled and normalized API 5L Grade B pipeline steel [253]. Fig. 23 shows the corresponding fatigue crack growth rates with stress ratio R = 0.1 under 1.4 and 20.7 MPa hydrogen pressures. From

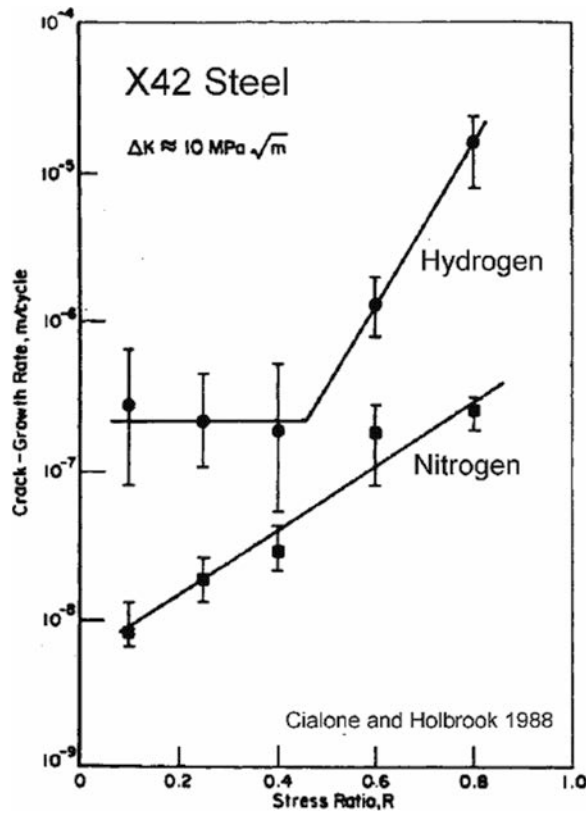


Fig. 22. Fatigue crack growth rates (da/dN) for X42 in hydrogen and in nitrogen at various stress ratios (R) [252].

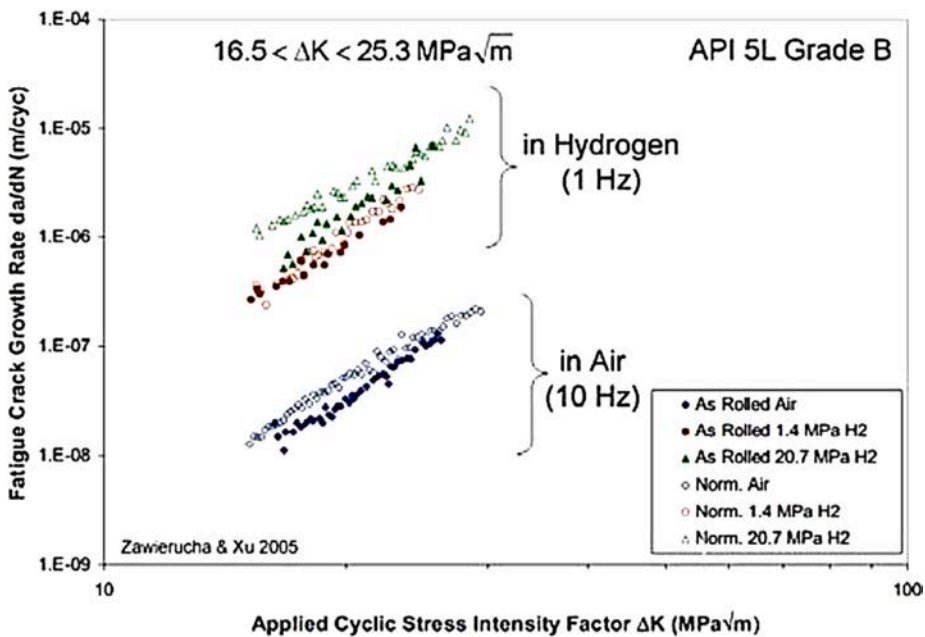


Fig. 23. Fatigue crack growth rates (da/dN) for as rolled and normalized API 5L Grade B steels in various pressures of hydrogen (1 Hz) and in air (10 Hz) [253].

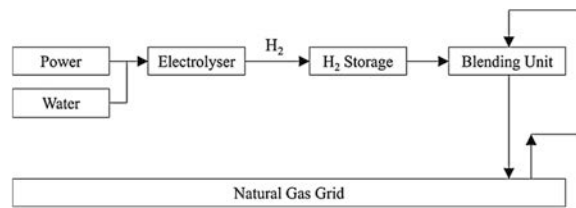


Fig. 24. Schematic showing a Typical System for Blending Hydrogen into Natural Gas Grids [256].

Table 6

Examples of Hydrogen Blending Systems [257].

Project	Country	Network	Electrolyser capacity	Hydrogen Blend (%)	Others
HyP SA	Australia	Distribution	1.2 MW	5 %	Uses green hydrogen
ATCO-CEIH	Australia	Distribution	0.15 MW	5–25 %	GC and PID control
HyDeploy	UK	Distribution	0.5 MW	20 %	Measures Wobbe index
Jupiter 1000	France	Transmission	1.0 MW	6 %	Injects hydrogen into transmission lines

Fig. 23, it can be deduced that the presence of hydrogen significantly increased the fatigue crack growth rate of the pipeline specimen by about 30 times that of the specimen in the air. In addition, over the tested  $\Delta K$  range (i.e.,  $16.5 < \Delta K < 25.3 \text{ MPa } \sqrt{\text{m}}$ ), the fatigue crack growth rate seemed insensitive to the pressure of hydrogen (i.e.,  $da/dN$  only increased about 1.5 times when the hydrogen pressure changed from 1.4 MPa to 20.7 MPa). Additional hydrogen pressures were applied in the fatigue crack growth tests. Fig. 23 shows the dependence of fatigue crack growth rate on the hydrogen pressure when  $\Delta K = 22 \text{ mMPa}$ .

The lack of dedicated pipeline steels for hydrogen transport or storage has encouraged the blending of hydrogen and natural gas together for transportation. To use existing pipelines for transporting hydrogen, blending hydrogen with natural gas becomes the best method in achieving that. However, hydrogen's molecule size and its different properties with natural gases can create a unique feature when blended [254,255]. This becomes a threat to pipelines, especially pipelines originally designed for just natural gas transmission. Transporting this blended mixture with optimum transmission conditions requires increasing the flow rate, unlike the rate used in transmitting only natural gas [256]. However, this may lead to an increase in pressure and a shift from the design specifications of the existing pipelines, exposing them to imminent failures. So, it is imperative to consider the design changes and trade-offs in transporting blended hydrogen and natural gas. For instance, homogeneity of the blended substances is necessary for uniform behavior along the inner linings of the pipeline [257]. Energy consumption is also an issue to investigate. It is usually necessary because of the different densities of the blended substances due to stratification [256]. To prevent stratification, blended gas is transported under turbulent flow conditions, according to an investigation by the HyDeploy project [258].

Fig. 24. shows the components of a typical system for blending hydrogen into natural gas grids. Currently, there are several blending systems in operation, but the safety process is to ensure a homogenous mixing of hydrogen (in specific percentages) and natural gas. Table 6 shows some hydrogen blending systems in operations, obtained from [258–262].

Blending hydrogen with natural gasses doesn't fully eliminate the hydrogen embrittlement effect from pipelines, especially when a high percentage of hydrogen is being blended. However, according to ASME B31.12, carbon steels, including ASTM A 106 Grade B, ASTM A53 Grade B, and API 5L Grades X42 and X52, are judged to be fit for the transportation of hydrogen with pressures up to 14 MPa [263]. Nevertheless, the rising need for hydrogen in the future will necessitate higher pressure requirements and raise the demands for existing pipeline steels for hydrogen transportation, which requires more research and work, regardless of the limited literature about the subject matter.

## 8. Processing of pipeline steels designated for cold regions

Another important aspect to look at is the processing of pipeline steels designated for cold regions. Arctic regions are known for their low-temperature setting, and the fluctuations of their temperature, ranging from  $+30^{\circ}\text{C}$  to  $-70^{\circ}\text{C}$  [264]. With the predicted decrease in petroleum from existing wells, the Arctic regions seem to be attracting interest from the oil and gas industries for petroleum exploration. While the data on petroleum in the uncharted parts of the world is still unclear, it is estimated that the undiscovered arctic petroleum accounts for 22 % of the unexplored fraction across the globe. That is, arctic petroleum represents 15 % of oil and 30 % of gas which is undiscovered worldwide [265]. While the Arctic region feels promising because of the abundance of petroleum resources, their low temperatures and climate fluctuations hinder exploration, because of the use of existing high-strength pipelines for resource transportation.

In summary, the microstructure of these existing pipeline steels needs to be tailored, as the mechanical properties of these existing pipelines need to be controlled to survive in Arctic environments. For existing pipeline steels, a swift temperature drop can ultimately alter the mechanical properties of the pipeline, due to a transition from a ductile to a brittle nature, called the ductile to brittle transition temperature (DBTT) [266–268]. As such the low-temperature toughness of pipelines becomes of interest, especially for pipelines designated for permafrost and arctic areas. For a pipeline to withstand low-temperature induced brittleness, it must have a

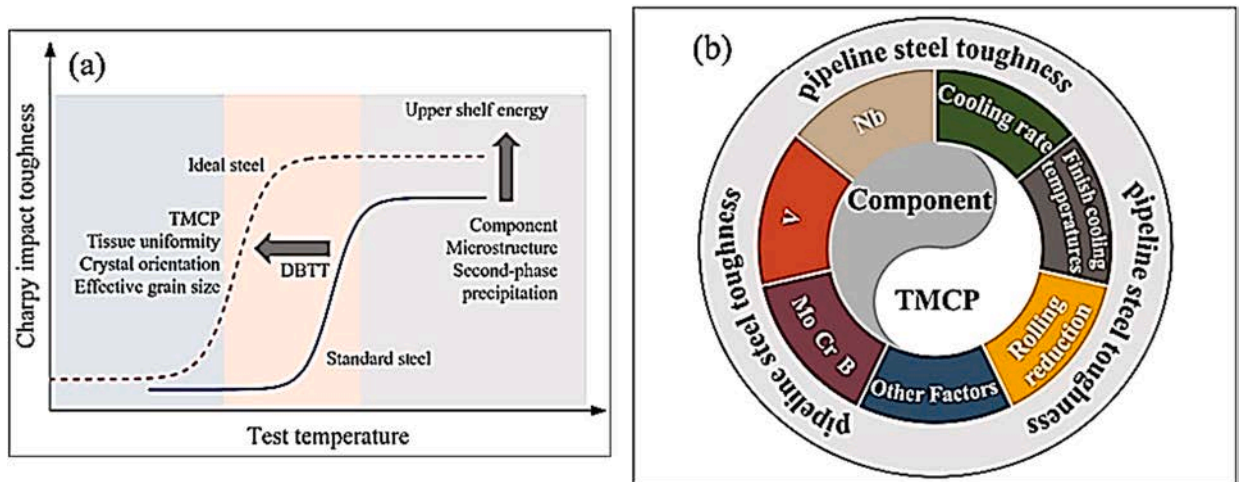


Fig. 25. Schematic diagram of low-temperature toughness optimization of pipeline steel. (a) Strategies for optimizing the low-temperature toughness properties of high-grade pipeline steel; (b) the typical factors affecting the low-temperature toughness properties of pipeline steel, including composition (Nb, V, Mo, Cr, B) and process parameters (finish cooling temperature, cooling rate, rolling reduction) [123].

low DBTT, responsible for maintaining its absorbed energy [123,269,270]. This DBTT depends on the chemical composition of the pipeline, microstructure, heat treatment, and so on [271]. Interestingly, grain size, amongst many other factors, greatly influences the DBTT of materials. Therefore, with adequate processing and microalloying, the low-temperature toughness of pipelines can be significantly improved. Fortunately, the method of making pipeline steels designated for cold environments is TMCP [84]. This involves the control of the processing of steels, right from the initial processing step to achieve better mechanical properties for the designated environment. From Fig. 25 a, the lower DBTT and higher upper shelf energy (USE) are desirable for the application of any specific pipeline steel.

Mechanical properties like toughness or ductility, at low temperatures, can be optimized by processing, which allows for the control of grain size, dislocation density (especially for pearlitic steels), phase composition, texture, and other microstructural characteristics, as shown in Fig. 22b [272]. Amidst the transportation and infrastructural difficulties in the Arctic regions, balancing pipeline strength and ductility remains a major concern. To balance strength and ductility, the processing of the pipeline needs to be controlled to enable:

1. Full dissolution of precipitates (carbonitrides and carbides).
2. Uniform refined ferrite grains through controlled dynamic recrystallization, precipitate hardening, and dissolution of carbides and carbonitrides [273].
3. Reduction in bainite lathes by controlling the finishing cooling temperature.
4. High presence of ferrite grains by creating more ferrite nucleation sites through controlled rolling reductions [274].
5. Optimum low-temperature toughness.

The following are the processing stages for producing pipelines designated for the arctic environment, with several recommendations from the literature.

### 8.1. Slab reheating temperature

This is the first important stage in the processing of steels for Arctic applications. It is important as it determines the austenite grain size (which gives an idea of the size of ferrite grains and their uniformity) and the quantity/quality of solid solution microalloying elements [275]. From Fig. 26a, it can be deduced that for the preferred precipitates to go into solution, a slab reheat temperature between 1130°C and 1180°C is recommended. From Fig. 26b, there is an insignificant increase in average austenite grain size between 1,130 and 1,180°C. Subsequently, a tangible austenite grain enlargement continues above 1,200°C. With this, it can be deduced that a slab reheating temperature between 1,140°C and 1,180°C is the recommended temperature for reheating X70 pipeline steel. It is, however, imperative to note that there is no optimum slab reheating temperature as it all depends on the composition of the steel pipe.

However, this is not in line with the findings of Ebrahimi et al. on Nb micro-alloyed steels. The authors revealed that the increase in the reheating temperature from 1180 to 1240°C minimized pearlite content and promoted acicular ferrite in the rolled steel. According to the authors, the increased ferrite presence in combination with carbonitride precipitates improved strength and toughness in the steels [276].

Di Schino and Rufini also investigated the effect of slab reheating temperature on the mechanical properties of steels [277]. They increased the slab reheating temperature from 1075°C to 1175°C and observed an increase in the steel's hardenability. The authors,

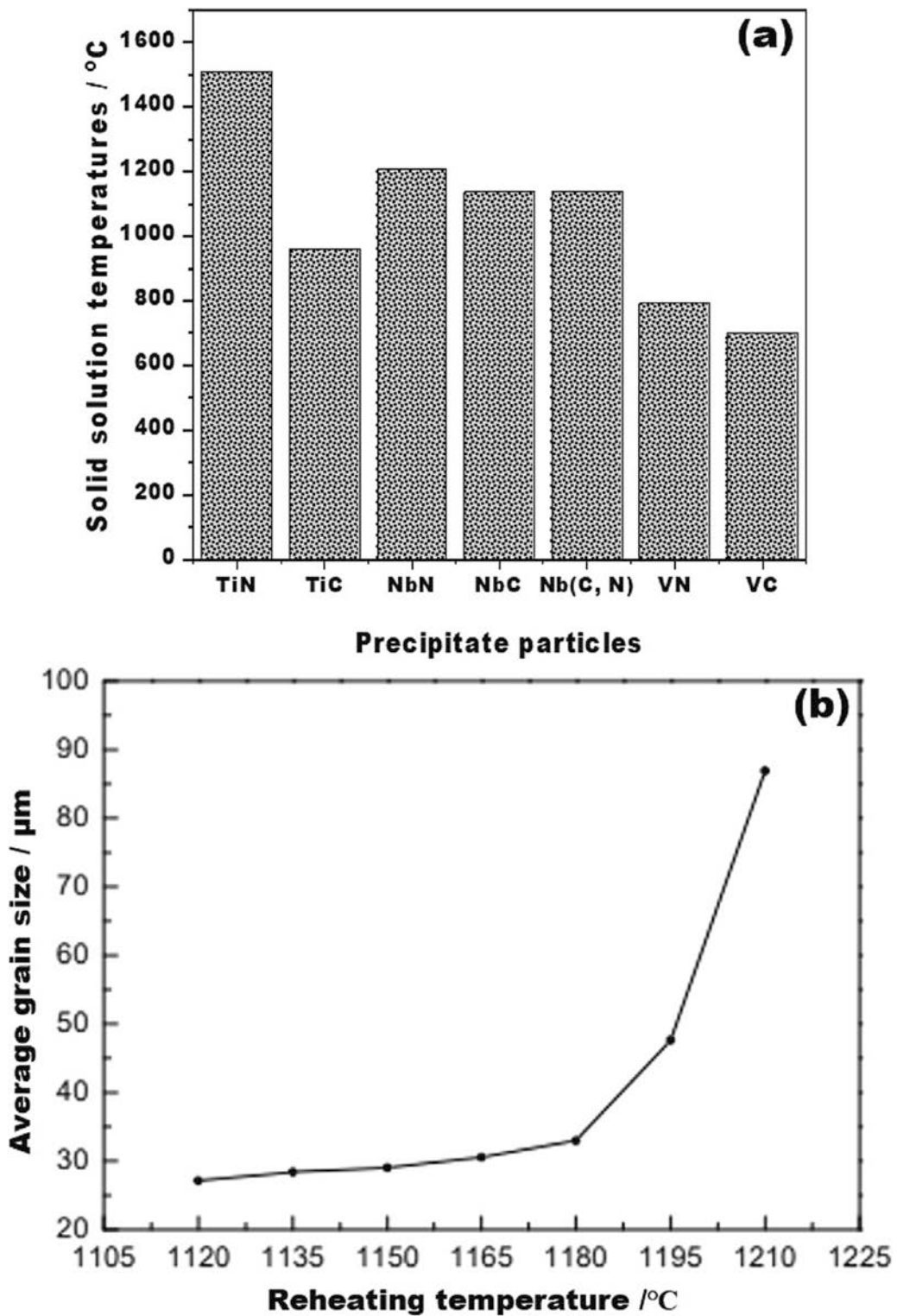


Fig. 26. (a) solid solution temperatures of typical carbonitride precipitate particles obtained from Nb, Ti, and V (b) influence of reheating temperature on average austenite grain size [275]).



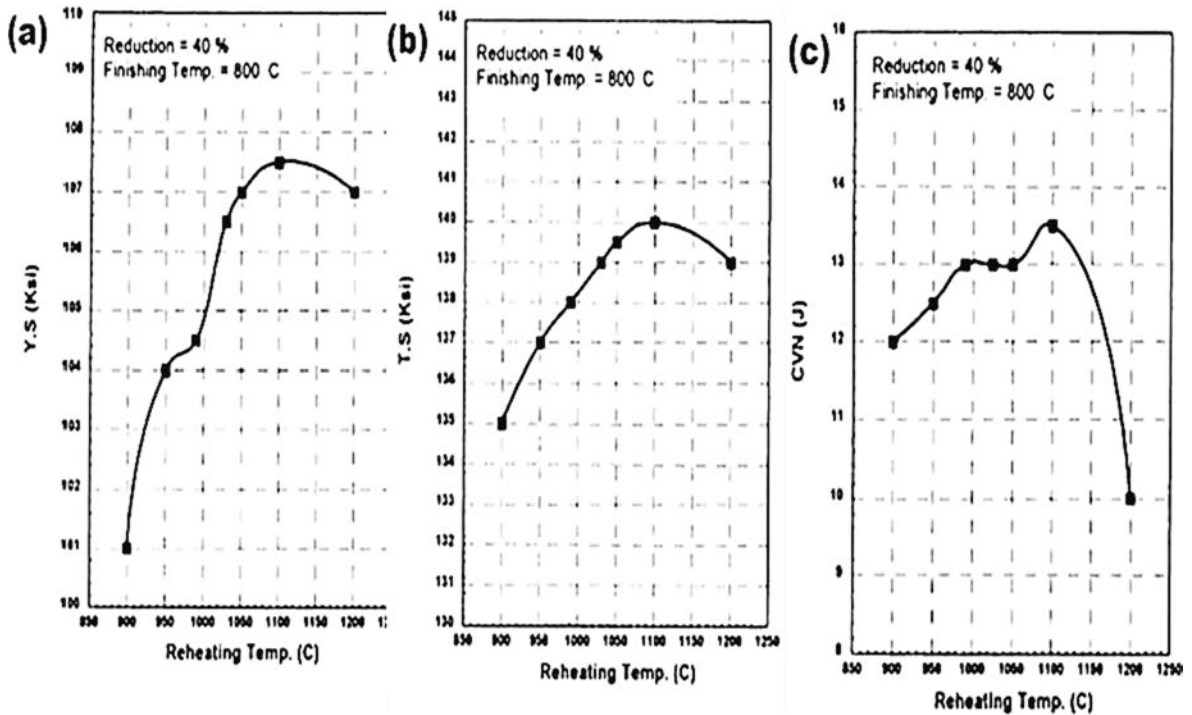


Fig. 27. Effect of reheating temperature on mechanical properties of AISI 4130 steel (a) yield strength vs. reheating temperature (b) tensile strength vs. reheating temperature (c) impact energy vs. reheating temperature [278].

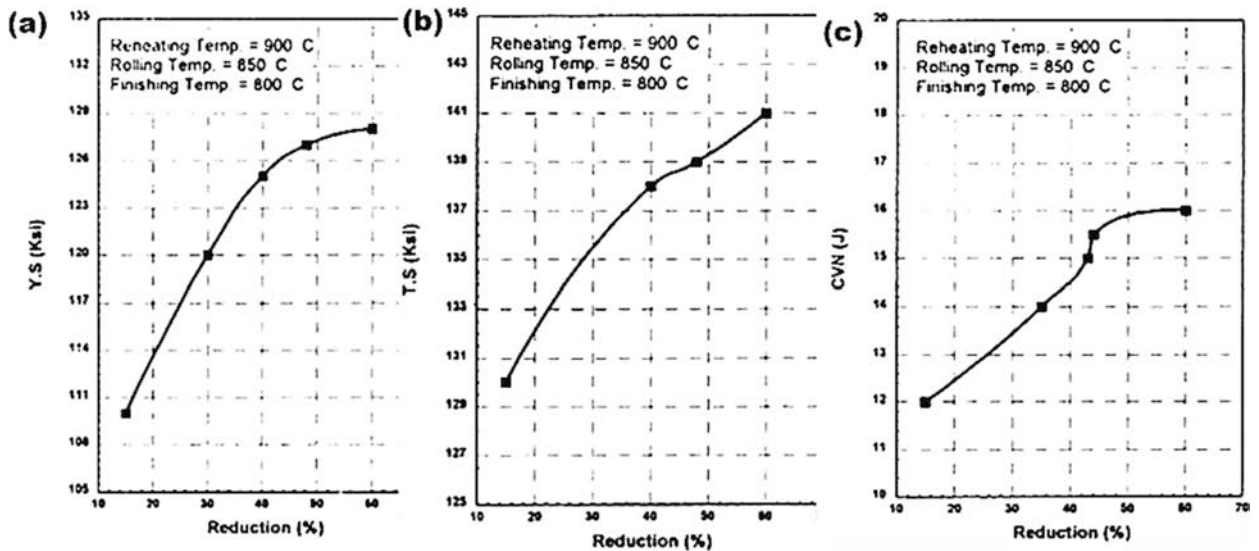


Fig. 28. Schematic representation of TMCP with the resulting EBSD micro- graphs obtained after applying different rolling reductions above and below  $T_{nr}$  [281].

however, attributed the increase to the size of austenite grains at relatively high reheating temperatures, which affects the extent of grain refinement in the final microstructure, which is also in line with the findings of Zhao et al. [275], as shown in Fig. 26b. The increased grain size contributed to the reduced toughness. Another interesting result was obtained by Jahazi and Egbali. They investigated how reheating temperature affects the mechanical properties of pipeline steels. In Fig. 27 (a-c), there is an illustration of the relationship between reheating temperature and the mechanical properties of pipeline steels. From the chart, it is visible that an increase in the reheating temperature results in an increase in strength and the absorbed energy, up to 1,100 °C. After that mark, at 1200 °C, there seems to be a decline in yield strength, impact energy, and tensile strength [278].

**Table 7**  
Effect of micro-alloys on the toughness of pipeline steels.

S/ N	ALLOYING ELEMENT	EFFECTS
1.	Niobium (Nb)	<ul style="list-style-type: none"> <li>• Delays austenite recrystallization.</li> <li>• Lowers the phase transformation temperature [285–287].</li> <li>• Enhances nucleation sites for phase transformation [288,289].</li> <li>• Refines austenite and deformed austenite grains.</li> <li>• Refines the final microstructure and enhances toughness and strength, as seen in Fig. 29.</li> </ul>
2.	Vanadium (V)	<ul style="list-style-type: none"> <li>• Compensates for insufficient Niobium content [268].</li> <li>• Better strengthening than Niobium but has poor grain refinement proficiency.</li> <li>• Retards the growth of austenite grains during heating to a lesser extent than Nb and Ti because of its high solubility.</li> </ul>
3.	Molybdenum (M)	<ul style="list-style-type: none"> <li>• Influences the precipitation activity of other microalloying elements like V, Nb, and Ti [290].</li> <li>• Impedes the static recrystallization of high-temperature austenite.</li> <li>• Increases the activation energy for carbon diffusion within austenite, thereby reducing the dispersion coefficient in carbon and inhibiting the formation of pro-eutectic ferrite.</li> <li>• Causes direct transformation of supercooled austenite to bainite without the concomitant formation of polygonal ferrite [291,292].</li> </ul>
4.	Chromium (Cr)	<ul style="list-style-type: none"> <li>• Acts as a ferrite stabilizer.</li> <li>• Improves strength through chromium carbides [268].</li> </ul>
5.	Titanium (Ti)	<ul style="list-style-type: none"> <li>• Microstructure refinement due to grain size control. More effective retarding action than Nb and V on the growth of the austenite grains with the temperature of heating for rolling [293].</li> <li>• Grain boundary strengthening.</li> <li>• Increased resistance to brittle fracture, and better toughness.</li> <li>• Less efficiency than Nb and V in precipitation strengthening, as titanium nitride TiN cannot be completely dissolved in the austenite in the heating for rolling at conventional temperatures [293,294].</li> </ul>

## 9. Hot rolling

The hot rolling process is necessary for dynamic recrystallization which brings refined or coarse grains. Controlling this process allows the creation of nucleation sites for refined ferrite grains. Sych et al. proposed a 950<sup>o</sup>C hot rolling temperature with a reduction of 70 percent to obtain fine-grained austenite [274]. The inability to rightfully control rolling deformation will lead to anisotropic microstructure in pipeline steel, making it unfit for arctic applications [264]. Some researchers have proposed reducing rolling temperature as a method of increasing toughness in pipeline steels. This was attributed to the minimization of recrystallization and austenite grain size enlargement [279,280]. Jahazi and Egbali also made an interesting conclusion related to the influence of rolling reductions on yield strength, tensile strength, and absorbed energy [278]. They found out that if the rolling reduction increased from 15 % to 60 %, strength also increased, however, the reduction recorded within 40 percent did not increase the absorbed energy. So, from Fig. 28 (a-c), it can be deduced that there is a balance in the rolling reduction between 45 and 50 % for pipeline steels designated for arctic regions, as from this range of reductions, we might see (a) pancaked austenite, (b) an increased dislocation density, and (c) an increase in nucleation sites for ferrite grains.

Apart from controlling these processing parameters to improve toughness of pipeline steels, the composition of micro-alloying elements also affects the temperature toughness of pipeline steels.

### 9.1. Effects of microalloying elements on the impact toughness of pipeline steels

The importance of microalloying elements goes beyond low DBTT to also affect the microstructure of pipeline steels. With a careful addition of micro-alloy elements with appropriate thermomechanical processing, a desired microstructure, such as AF [123,282] or fine polygonal ferrite [283,284] can be obtained. Table 7 shows the effects of some micro-alloying elements on the toughness of pipeline steels designated for low-temperature environments.

From Table 7, concerning V, Nb, and Ti, the retarding action on the growth of the austenite grains in hot rolling temperature is in this ascending order: V – Nb – Ti. But in the aspect of grain refinement: V – Ti – Nb.

## 10. Conclusion

Based on the above reviewed research articles on the effects of different microstructural parameters on the corrosion and cracking resistance of pipeline steels, the following conclusions can be made:

- Corrosion precedes HIC and remains the predominant environmental failure mechanism besetting pipelines under sour environments. The corrosion resistance of pipeline steels is mainly improved through cathodic protection, coating, and the use of corrosion inhibitors.
- Nb and Ti in micro-alloyed steels contribute significantly to retarding the austenite grain growth, thereby facilitating grain refinement, and improving mechanical properties. However, balancing toughness and strength property requirements largely depends on the judicious tailoring of several microstructural parameters through steel processing.

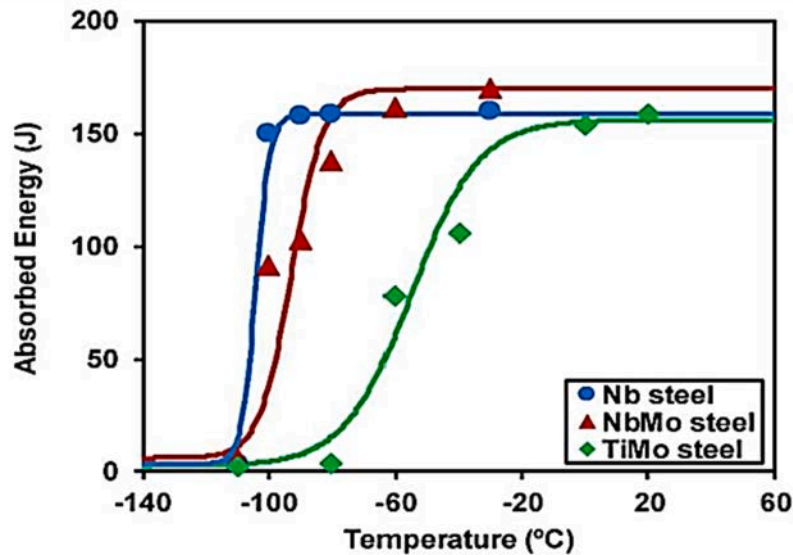


Fig 29. Different micro-alloyed steels (Nb, NbMo, TiMo) and their impact energy after coiling at 700°C [123].

- Dislocation density, amongst other microstructural parameters, has a more profound effect on the HIC resistance of pipeline steels. There is currently no established hierarchy revealing how different microstructural parameters influence the corrosion and cracking resistance of pipeline steels, however, dislocation density plays a prominent role in determining the susceptibility of these steels to failure. An in-depth understanding of the hierarchy of effects of different microstructural parameters will greatly help steel manufacturers modulate the mechanical properties of pipeline steels to harmonize them with the distinct demands imposed by diverse service environments.
- Grain size, amongst other microstructural parameters, has a more profound effect on the mechanical properties of pipelines designated for cold applications, while the relationship between grain size and corrosion susceptibility remains a multifaceted subject, having several influence factors.
- Crystallographic texture and grain boundary characteristics have roles to play in improving the SCC resistance of pipeline steels. However, the degree to which crystallographic texture, amidst other microstructural parameters, affects cracking is not yet established. For instance, the direct influence of crystallographic texture in the arrest of propagating cracks is still unclear and debated, while low-angle grain boundaries and CSL boundaries have been seen to arrest cracks in steels.
- Welding heat reduction, removal of joint and residual stresses, as well as pre-heating before welding, can greatly help reduce cracking in pipeline welds. Elimination of these residual stresses also has positive impacts on the SSCC resistance of steel.
- Mobile hydrogen is a major threat to the cracking resistance of pipeline steels.
- The lack of dedicated pipeline steels for hydrogen transport or storage has encouraged the blending of hydrogen and natural gas together for transportation.

### 10.1. Future recommendations

- In a contemporary context, the employment of machine learning techniques avails a promising avenue for optimizing steel microstructure as revealed in this review. The combination of empirical data encompassing historical and contemporary insights on hydrogen embrittlement, in conjunction with detailed knowledge of pipeline steel processing and thermodynamic modeling, forms the bedrock of this approach. Combining these facets with the insights provided by thermodynamic models could give rise to a simulated optimum microstructure. A pivotal aspect of this context involves the creation of simulated environments that mirror the distinctive operational environments of the pipeline steels. Through calibrated adjustments to the pipeline steel's composition, these simulated models can be subjected to simulated testing protocols to assess the impact of these environments on the mechanical properties of the pipeline steel. Such an approach represents an innovative and rigorous means to iteratively tailor pipeline steel microstructure, rendering it amenable to the multifaceted demands of real service conditions.

### CRediT authorship contribution statement

**Meekness Nnoka:** Writing – review & editing, Writing – original draft, Visualization, Methodology, Conceptualization. **Tonye Alaso Jack:** Writing – review & editing. **Jerzy Szpunar:** Writing – review & editing, Supervision.

## 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.

## Data availability

No data was used for the research described in the article.

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