



Influence of chemical attack on hydrogen embrittlement on bolt steels

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Bolt steel ;
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Abstract

This document delves into the impact of chemical attacks and hydrogen embrittlement on bolt steels, with a particular emphasis on galvanizing techniques utilized in sectors such as aeronautics and automotive manufacturing. The research highlights how hydrogen absorption during surface treatments can adversely affect the mechanical properties of steel, leading to structural failures. One of the primary methods discussed is hot-dip galvanizing, which provides corrosion protection by applying a zinc coating to steel. This process involves immersing the steel in molten zinc, a procedure that can inadvertently facilitate hydrogen absorption, resulting in potential embrittlement of the material. Hydrogen embrittlement (HE) is a critical concern as it occurs when hydrogen infiltrates the metal, causing it to become brittle and increasing the risk of fractures, particularly in bolts and fasteners that are subjected to mechanical stress. The study conducted an experimental investigation using 41Cr4 steel to analyze its behavior under various hydrogen loading conditions. The methods employed included heat treatment, microstructural analysis, and tensile testing to assess the steel's properties. The researchers utilized an embrittlement index to quantify the sensitivity of the steel to hydrogen embrittlement. The results indicated a significant reduction in ductility as the duration of hydrogen loading increased. In conclusion, the research emphasizes that the galvanizing process, especially during the acid pickling stage, presents considerable risks for hydrogen absorption. To mitigate these risks, the use of corrosion inhibitors is recommended. Furthermore, the study suggests that recovery of mechanical properties can be achieved through controlled degassing, underscoring the importance of managing hydrogen exposure during the processing of steel components.

1. Introduction

Hot-dip galvanized screws and bolts have good corrosion resistance, but this treatment requires surface preparation accompanied by significant hydrogen absorption. The optimization of their performance requires an understanding of all the factors that can influence their properties, especially the introduction of hydrogen, which leads to a decrease in mechanical properties and our it's at the origin of damage important, such as a sudden break or rupture delayed because of the appearance and propagation of cracks in the steel.

The corrosion is defined as the physicochemical interaction between a metal and its environment, resulting in changes in the properties of the metal, and is often a functional degradation of the metal itself [1]. The corrosion of a metal or an alloy can develop according to various processes that characterize each type of corrosion (chemical corrosion, electrochemical corrosion) [2,3]. Galvanizing is one of the most effective means of

protection of surfaces of steel structures against the development of corrosion process [4]. The tensile tests, the observations from the optical microscope allowed us to confirm the sensitivity of the steel to embrittlement by hydrogen.

2. Galvanizing of bolts

Galvanizing is one of the most effective means of protection of surfaces of steel structures against the development of corrosion process [5,6]. It is a technique unconditional basic and the most famous protection against corrosion for steel. The galvanizing process has low cost and the final product has a sufficient degree of protection against corrosion. The widespread use of the galvanization is due to its high productivity. Today, the hot-dip galvanizing is widely used in industry, in particular, in the production of fasteners of metal by the application of a zinc coating to

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the surface of metal structures, mainly in steel, from about 5 to 40 micrometers thick, by immersing the part in a bath of molten zinc heated to over 400°C. The protection by galvanizing is based on and determined by the difference of electrochemical potentials of Zn and Fe. The zinc protects steel in two ways. The first, the zinc coating forms a continuous layer on the surface of the steel, preventing any direct contact between the steel and the environment. The second is electrochemical or cathodic (called active, sacrificial or electrochemical) to the levels of damaged coatings and in the pores in which the zinc form a galvanic couple with the steel. The cathodic protection implies that zinc, when it is applied on the iron, form a galvanic couple in which the iron is a metal, a less active, and zinc is a metal more active. Thus, in a wet environment, the zinc coating acts as anode and undergoes the harmful oxidation reactions, thereby protecting the base metal of the bolt. The galvanized surface has a high degree of protection from the anode to the base metal. Such qualities have made the technology of galvanizing the most popular for the protection against corrosion of metal structures. Like any surface treatment, hot-dip galvanizing requires good surface preparation so that the reaction of iron and zinc is uniform when immersing parts in the galvanizing bath. It is after such careful surface preparation that the adhesion (adhesion force) of the zinc coating to the base metal is very high, which means that the galvanized metal will be protected from corrosion for many years. Zinc coatings are widely used in the areas where the reliability and the durability of the metal structures are required. Hot-dip galvanizing is suitable for the protection of the steel with low carbon content (carbon content not exceeding 0.30 wt.%) with a silicon content of up to 0.37wt.% and a relatively low level of alloy elements [2]. The critical step in hot-dip galvanizing is acid pickling, during which hydrogen is likely to penetrate the material. This phenomenon is at the origin of a major problem called hydrogen embrittlement (HE). The experimental strategy aims to highlight the influence of hydrogen on the mechanical strength of the steel studied from tensile tests on cylindrical specimens and screws, before and after hydrogen loading. The typical lifespan of galvanized products is about 50 to 70 years.

3. Experimental investigation

The analysis of literature survey the [7, 8, 9] has allowed to clarify the points to explore in more depth to better understand hydrogen embrittlement (HE) steel bolts. Thus, we established an experimental methodology based on mechanical tests to better understand the behavior of these steels in a hydrogenated environment. The chemical composition of 41Cr4 steel according to the standard (NF EN 10083-1) is shown in Table 1.

3.1. Heat treatment

The steel has undergone a heat treatment (quenching + tempering), to obtain a martensitic microstructure according to the following regime: heating to 870 °C / 40min; oil quenching, tempered at 540 °C / 90min, air cooling.

3.2. Microstructural analysis

Microstructural analysis was conducted to characterize the microstructure of the steel using a ZEISS optical microscope.

3.3. Hydrogen loading

The loading of hydrogen into the steel surface was carried out by loading cathode. Loading cathodic hydrogen is an electrochemical process where protons are reduced at the surface of the steel to form hydrogen atoms that penetrate into the metal, causing embrittlement. Loading cathode makes it possible to introduce high amounts of hydrogen, and that is the reason why we have chosen this technology [10,11]. Loading cathodic has been carried out in acid medium H_2SO_4 (0.1 N) with a current density of 30 mA.cm², and the charging time are : 1h – 2h – 3h - 4h – 5h. We used three test specimens for each case (thermal treatment, the presence or absence of hydrogen, duration of loading and degassing). Then, we studied the mechanical properties of 41Cr4 steel for the different loadings of hydrogen.

3.4. Tensile tests

Tensile tests were carried out on a TESTWELL machine. The steel is delivered in the form of cylindrical bars from which the tensile specimens are taken, as well as cold-formed screws as specimens. Tensile tests were carried out on cylindrical specimens with different hydrogen loadings. The tensile tests are carried out immediately after hydrogen loading.

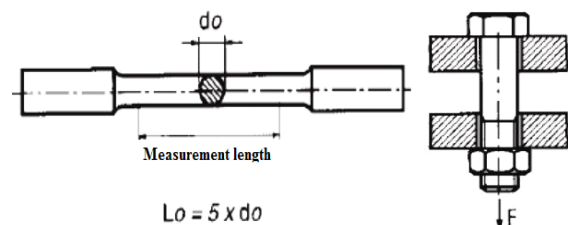


Figure 1. Tensile test specimens.

3.5. Vickers micro-hardness tests

Galvanizing and the Vickers hardness test are not directly related, but the Vickers hardness method is well suited for measuring the hardness of the thin iron–zinc alloy layers formed during galvanizing. The micro-hardness is a complementary part of the microstructural analysis that provides insight into the mechanical characteristics by measuring the hardness of the material.

These tests were carried out using an INNOVATEST 400 type rometer, analogous to a Vickers test, to measure the hardness of the different phases of the microstructure of the steel on a specimen after metallographic preparation by equation (1):

$$VH = 1,8544 \frac{P}{d^2} \quad (1)$$

P : indentation load (kgf),

d : arithmetic mean of the diagonals of the footprint (mm).



Table 1. Chemical composition of 41Cr4 steel (wt. %).

Elements	C	Si	Mn	Cr	Ni	P	S
41Cr4	0.38 - 0.45	≤ 0.40	0.60 - 0.90	0.90 - 1.20	≤ 0.12	≤ 0.035	≤ 0.035

3.6. Embrittlement index (HE sensitivity index) $E_i(\%)$

In general, the embrittlement index $E_i(\%)$ is a quantitative parameter evaluating the sensitivity to hydrogen embrittlement by measuring the reduction of a mechanical property linked to fracture in a material loaded with hydrogen, compared to the same material without hydrogen [12, 13]. The embrittlement index $E_i(\%)$ reflects the drop in ductility and the necking of steels induced by the presence of hydrogen within the material, by reflecting the loss of elongation at break of the steel in the unloaded and hydrogen-loaded state [12, 14].

Finally, in order to compare the behavior of the steel studied to embrittlement by hydrogen (HE), the embrittlement index $E_i(\%)$, was measured by formula (2), where A_0 and A_h are respectively the elongations at break before and after hydrogenation of the specimen:

$$E_i = \frac{A_0 - A_h}{A_0} \times 100 \quad (2)$$

Where: A_0 - elongation of the non-hydrogenated specimen and A_h - elongation of the hydrogenated specimen.

3.7. Influence of the inhibitor

In terms of anti-corrosion protection, the use of corrosion inhibitors is a very practical and effective method for reducing the adsorption of hydrogen and thus delaying the reaction of its evolution [14]. These are substances which, added in very small quantities to the corrosive reagent, make it possible to reduce or eliminate its aggressiveness towards the metal. In the case of acid pickling, these substances slow down the kinetics of metal dissolution while limiting surface roughness. These are generally organic products capable of adsorbing at the interface to control the electrochemical reactivity of the substrate [13]. In the case of galvanizing, these inhibitors also have the advantage of not causing any problem with the adhesion of zinc to the surface of the steel. Indeed, the inhibitor used during acid pickling is simply removed by rinsing and is no longer present on the surface during the immersion step in the liquid zinc bath.

Besides protecting against anodic attack of the steel surface and grain boundaries, their presence in acid pickling solution may also slow down the absorption and diffusion of hydrogen, but some are sometimes suspected of promoting hydrogen absorption [14].

3.8. Recovering of mechanical properties

After the study of the influence of the duration of hydrogen loading on the properties of the steel studied, we tried to consider the possibility of recovery of mechanical properties by degassing on a test specimen after loading in hydrogen for 1 hour. Afterwards, tensile tests were carried out on specimens loaded and degassed with hydrogen at a temperature of 200°C for 20 and 30 minutes.

4. Experimental results

The experimental results will be presented in two parts, taking into account the characterization of the steel for each structural transformation and the way of loading hydrogen in the 41Cr4 steel. Each type of result will be the subject of two paragraphs: the first will be used to show the results obtained, the diagrams used and the photos taken with the optical microscope, the second paragraph will be used to synthesize the results and to carry out the discussion using the other diagrams derived from the results obtained.

During the discussion of the results of low strain rate tensile tests, the embrittlement index (E_i) will be used, defined by at maximum strain.

4.1. The 41Cr4 steel in delivery state

4.1.1. Microstructure

In the annealed state, 41Cr4 steel has a ferritic-pearlitic structure (Figure 2). This steel, after controlled cold rolling, therefore exhibits, according to the longitudinal section (Figure 2-b), more elongated ferrite grains.

The grains thus remain deformed, work-hardened, with a controlled dislocation density. With the increase in magnification (Figure 3), a homogeneous matrix can be distinguished, this structure gives it optimal mechanical properties for good cold deformation capacity.

At delivery state, 41Cr4 steel has low mechanical properties: yield strength $R_e = 437 \text{ MPa}$, breaking strength $R_m = 544 \text{ MPa}$, with an elongation at break of approximately $\Delta l = 22\%$. This can be explained by work hardening following cold rolling. This makes it possible to obtain a high dislocation density when placed under stress and thus to reduce the elongation at break.

4.2. The 41Cr4 steel after heat treatment

4.2.1. Microstructure

In order to improve the characteristics of the steel, we carry out a heat treatment for hardening and tempering in the air. This treatment generates a structure essentially martensitic with a high hardness. The presence of alloying

elements such as chromium, molybdenum and vanadium allows the steel to preserve its excellent quenchability and air tempering (Figure 5).

This steel after heat treatment (quenching and tempering) has both better mechanical strength (1040 MPa) and good ductility (32.42%).

4.2.2. Study of resistance to HE

To perform the tests evaluating resistance to hydrogen embrittlement (HE), the test specimens were prepared from M12X60 screws, having undergone the same heat treatment.

After studying and determining the micro-structural and mechanical characteristics of 41Cr4 steel, we focused on its mechanical behavior for different levels of hydrogen loading. The conditions of the electrolytic charging, i.e. the determination of the parameters (time and current

intensity) were carried out according to the method applied in the work [9]. The optimal loading conditions are those that allow for significant embrittlement (modification of the tensile curve) while remaining simple to implement. We therefore vary the load time of between 2 hours and 5 hours, and the intensity of the charging current set to 60 mA.cm².

The hydrogen loading must ensure total penetration of the hydrogen into the specimen, with good reproducibility of the results.

4.2.3. Embrittlement index (Ei, %)

After cathodic loading of the test specimens (H₂SO₄ medium) for different durations from 2h to 5h, tensile tests are carried out immediately after loading with hydrogen.

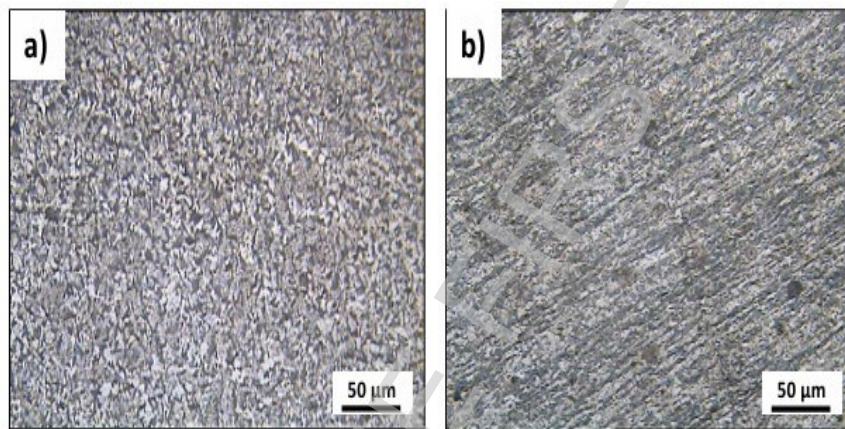


Figure 2. Microstructure of 41Cr4 steel in the delivery state: (a) transversal view (section perpendicular to the surface); (b) longitudinal view (section parallel to the surface).

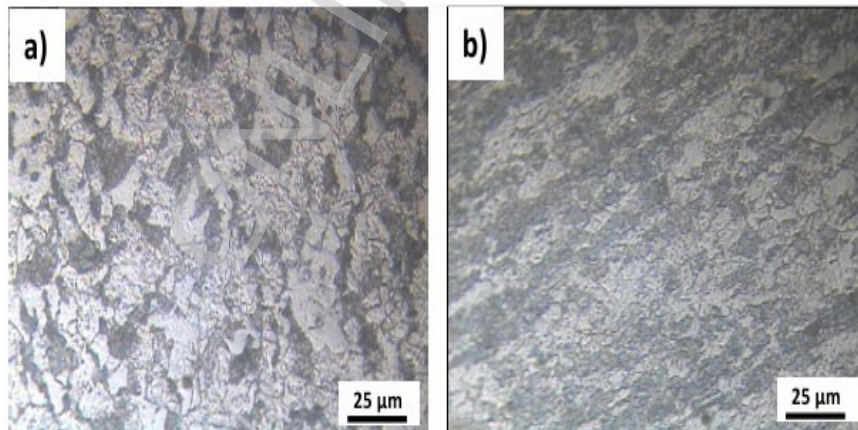


Figure 3. Microstructure of 41Cr4 steel in the delivery state: (a) transversal view (section perpendicular to the surface); (b) longitudinal view (section parallel to the surface).

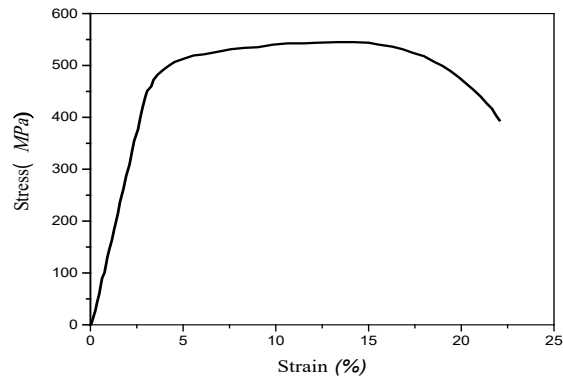


Figure 4. Stress / strain curve of 41Cr4 steel in the delivery state.

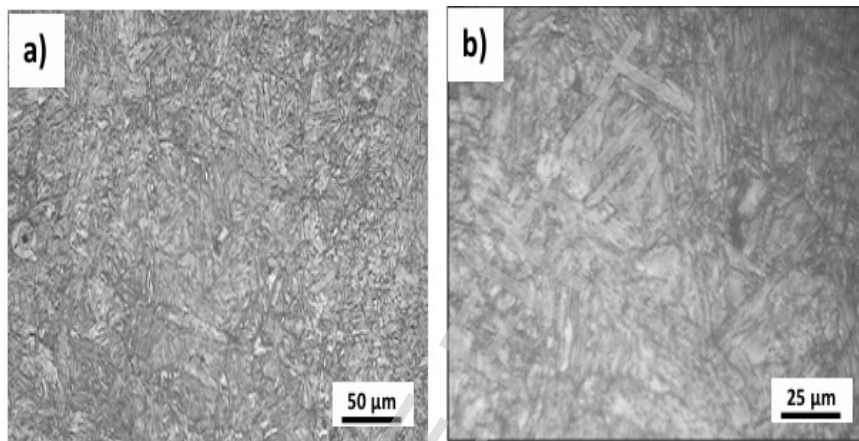


Figure 5. Structure after quenching + tempering of 41Cr4 steel: (a) martensitic structure; (b) zoom on a martensitic grain.

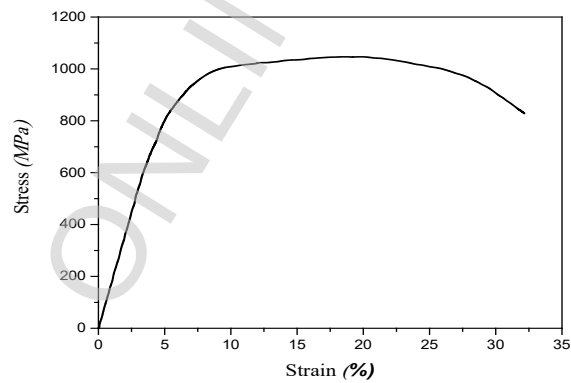


Figure 6. Stress / strain curve of 41Cr4 steel after quenching and tempering.

Table 2. Mechanical Properties of 41Cr4 Steel after Heat Treatment.

Re (Mpa)	Rm (Mpa)	A (%)	Hardness (HV 0,3)
830	1040	32,42	366

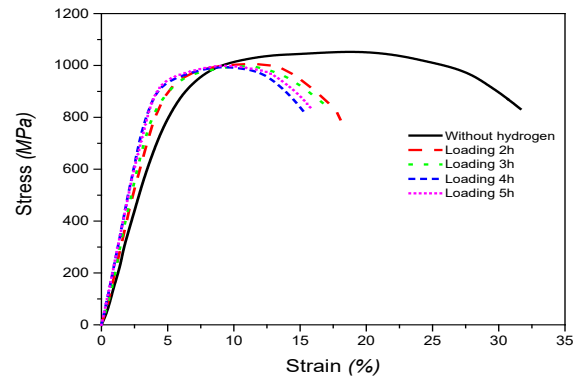


Figure 7. Stress / strain curves for different loading durations.

We have evaluated the susceptibility to hydrogen embrittlement (*HE*) by a comparative study of the ductility between a material loaded and a material non-responsive. Figure 7 clearly demonstrates that the steel 41Cr4 is very sensitive and this sensitivity also depends on its metallurgical condition. Among the specimens are complementary tested, the steel in the delivery state has a drop of ductility reduced. Thus, one is interested only in

the study of the susceptibility of the steel heat treated state (table 3).

We observe that the main mechanical properties (breaking strength and elastic limit) of the steel do not undergo significant modification for the different hydrogen loadings, thus we study the embrittlement index (E_i , %) (Figure 8) based on the elongation at break.

Table 2. Mechanical characteristics for 41Cr4 steel after different hydrogen loading.

Loading	Re (Mpa)	Rm (Mpa)	A, %	Hardness HV
Not loaded	830	1040	33%	290/320
Loading 2h	830	1002	18%	290/320
Loading 3h	820	996	17%	290/320
Loading 4h	830	996	16%	290/320
Loading 5h	830	1002	15%	290/320

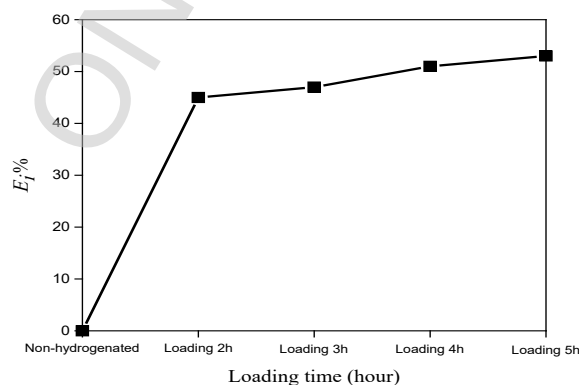


Figure 8. Embrittlement index (E_i) of steels as a function of loading time.

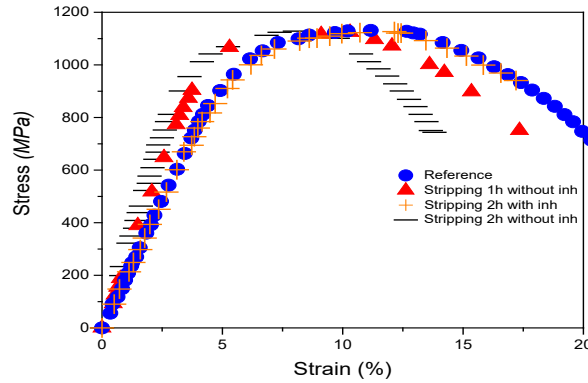


Figure 9. Influence of the corrosion inhibitor on the mechanical properties of hydrogenated 41Cr4 steel.

The analysis of the embrittlement index (E_i) graph (figure 8) shows that the highest embrittlement index is found in the specimen that underwent the longest hydrogen loading (5 hours). Thus, we can conclude that the charging time, i.e. the amount of hydrogen charged, leads to a reduction in elongation of almost 50%.

4.2.4. Influence of the inhibitor

During hydrogen loading, a small amount of inhibitor (parcolite 7) was added to the loading medium, the figure 9 shows the tensile tests for the hydrogen-loaded specimens with this inhibitor. It is observed that the corrosion inhibitor generally limits hydrogen penetration into steel very well for the different loads. The embrittlement index (E_i) decreases by almost half (for example: for a loading time of 2 hours with inhibitor: $E_i=15\%$ and on the other hand without inhibitor: $E_i=32\%$).

5. Recovering of mechanical properties

After characterizing the hydrogen embrittlement of 41Cr4 steel and analyzing the influence of loading time and microstructure on hydrogen embrittlement, it had been studied the possibility of restoration, i.e., recovering the mechanical properties by degassing carried out at different temperatures. First, degassing was carried out at a temperature of 200°C during 20 and 30 minutes. In order to determine the variation in mechanical properties, tensile tests were carried out on loaded and outgassed specimens. In figure 10, it can be seen a recovery of the elongation at break.

The mechanical tests (elongation (A, %), necking (Z, %)) were performed at the end of the different stages of the galvanizing process to determine whether each stage could induce significant hydrogen embrittlement. The results obtained (Figure 10) confirm that only acid pickling can pose a risk.

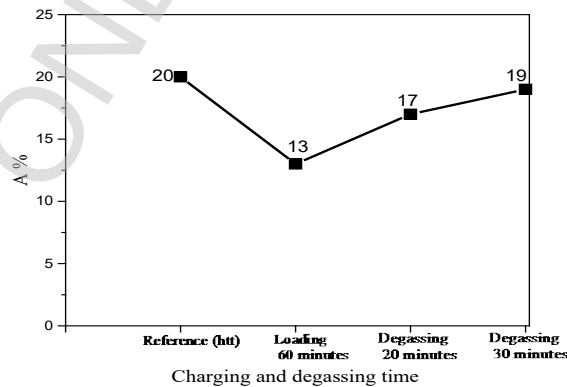


Figure 10. Evolution of the strain at break after loading and degassing at 200°C, for different degassing times.

6. Synthesis and discussion of results

All tests carried out during this study are aimed at studying and thus simulating the susceptibility to the hydrogen embrittlement phenomenon (HE) under industrial conditions of 41Cr4 steel. We note that the structure of the steel plays an important role in the

diffusion of hydrogen, and the steel to the state treaty, with a martensitic structure, presents a weakening high in comparison with the steel in the delivery state. Another important parameter, it is the time of loading, which reflects the concentration of hydrogen. In agreement with the literature, there is a field of speed of deformation for which the interactions hydrogen / plasticity in the same way that the enrichment / depletion of hydrogen in certain

areas of the material for the benefit of others who may be more sought mechanically will induce a failure by *HE*. The competition between the mobility of dislocations, the diffusivity of hydrogen and the energy trapping at the level of the different crystalline defects in the material, in particular dislocations, are these interactions hydrogen/dislocations, a parameter of complex study. Based on the results of this study, as well as on those of the literature [11,12,14], it is legitimate to consider the hydrogen concentration as a parameter of the first order. Thus, the definition of a critical content of hydrogen inducing the *HE* established on the basis of dosage, doesn't actually correspond to the critical content of hydrogen necessary and sufficient to the disbanding of the local area.

The trials of degassing could completely restore the mechanical properties, and therefore the trapping of hydrogen in the case of our steel is very low. This result confirms the results obtained by other authors of the work [9,13,14]. The tests were done using the corrosion inhibitor has revealed its effectiveness vis a vis the risk of *HE*.

The objective of this study was to characterize experimentally the effects of hydrogen during the galvanizing process and analyze the results to assess the damage induced by hydrogen. Our approach was to compare experimentally the behavior of the steel for bolts 41Cr4 under the influence of the hydrogen. This steel has all the metallurgical characteristics that can lead to a high susceptibility to embrittlement by hydrogen (*HE*): martensitic structure, high mechanical strength, presence of non-metallic inclusions, etc. Our study focused on the sensitivity to the *HE* steel during pickling acid, which also presents a detailed characterization of the damage associated with the *HE* and quantification of such damage by tensile tests.

7. General Conclusion

This work, essentially experimental, has made it possible to highlight the sensitivity of 41Cr4 steel to *HE* in acidic environment at ambient temperature. This study, carried out by mechanical tensile tests and metallographic analysis, showed that the microstructure of the material and certain experimental parameters (strain rate, hydrogen loading time) are first-order parameters involved in the *HE* of 41Cr4 steel during acid pickling.

This study also allowed, through the execution of tensile tests with the degassing, to show that the trapping of hydrogen in steel is very low and the mechanical properties could be restored completely. Based on this study on galvanizing and the risks of *HE* it has been shown that only acid pickling can induce hydrogenation risks, so it is recommended to minimize the pickling time, by the use of a corrosion inhibitor reducing the diffusion of hydrogen in the steel.

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Uticaj hemijskog napada na vodoničnu krtost čelika za vijke

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Apstrakt

Ovaj dokument se bavi uticajem hemijskih napada i vodonične krtosti na čelike za vijke, sa posebnim naglaskom na tehnike cinka koje se koriste u sektorima kao što su aeronautika i automobilska proizvodnja. Istraživanje ističe kako apsorpcija vodonika tokom površinske obrade može negativno uticati na mehanička svojstva čelika, što dovodi do strukturnih kvarova. Jedna od glavnih metoda o kojima se raspravlja je vruće cinkovanje, koje pruža zaštitu od korozije nanošenjem cinkovog preмаза на челик. Овај процес укључује потапање челика у растопљени цинк, поступак који може ненамерно олакшати апсорпцију водоника, што резултира потенцијалном кртошћу материјала. Водонична кртост (ВК) је критична брига јер се јавља када водоник инфилтрира метал, узрокујући његову кртост и повећавајући ризик од ломова, посебно код вијка и причвршћивача који су изложени механичком напрезању. Студија је спровела експериментално истраживање користећи челик 41Cr4 како би се анализирано његово понашање под различитим условима оптерећења водоником. Коришћене методе су укључивале термичку обраду, микроструктурну анализу и испитивање затезања ради процене својстава челика. Истраживачи су користили индекс кртости како би квантификовали осетљивост челика на водоничну кртост. Резултати су указали на значајно смањење дуктилности са повећањем трајања оптерећења водоником. Закључно, истраживање наглашава да процес цинковања, посебно током фазе декапирања киселином, представља значајан ризик за апсорпцију водоника. Да би се ублажили ови ризици, препоручује се употреба инхибитора корозије. Штавише, студија сугерише да се опоравак механичких својстава може постићи контролисаном дегазацијом, наглашавајући важност управљања изложеношћу водонику током обраде челичних компоненти.

**Originalni naučni rad,
Pregledni rad**
Mašinstvo,

Ključne reči:

Galvanizacija;
Vodonična krtost ;
Vijci;
Čelični vijak;
Površinska obrada;