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Experimental investigation of hydrogen embrittlement to stainless steel charged by sulfuric acid at ±20°C

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ABSTRACT

The penetration and trapping of hydrogen within metallic materials can lead to damage and degradation of mechanical properties during the operating phase. Much more in the energy, chemical, and petrochemical industries sectors, these types of metallic materials, widely used in aggressive hydrogenated environments, undergo degradation. In this present experimental work, we studied the influence of charged hydrogen on the mechanical behaviour in tensile of a highly alloyed austenitic stainless steel of the commercial grade AISI304L was prepared in the form of standardized cylindrical type specimens of 8mm diameter were manufactured by machining. Their hydrogen loading was carried out electrolytically according to industrial conditions adopted at different pre-charging times in hours in a Pyrex glass enclosure containing an aqueous solution of purely sulfuric acid H2SO4 at 0.1N and equipped with two electrodes, one cathode connected to the test specimen, an unattackable platinum anode with a chosen current density equal to 100mA/Cm^2. The mechanical fracture tests were carried out using an instrumented tensile machine with a maximum load of 400KN and a nominal displacement speed of 23mm/min. The microscopic analyses of the samples were carried out by different techniques: optical microscopy (OM), x-ray diffraction (XRD), and scanning electron microscopy (SEM), the results obtained in agreement with the work of the different authors who showed loss of ductility due to martensitic transformation of austenite caused by deformation and sensitivity by diffusion and hydrogen trapping.

1. Introduction

Austenitic stainless steels with chromium and nickel are widely used in the energy industries. During their operation in an aggressive hydrogen environment [1], these materials undergo an alteration of their mechanical characteristics, which causes their rupture [2], [3].

The diffusion of hydrogen in its steels can lead to a deterioration of their mechanical properties which generally results in a drop in ductility which causes a reduction in their reliability, this is the phenomenon of hydrogen embrittlement known as the FPH phenomenon [4], [5]. Several research works have been carried out and theories deduced from this phenomenon [6], [7], [8], [9], [10]. Recent work has clearly shown that the hydrogen atom, due to these physicochemical characteristics [11], [12], can easily diffuse in metallic materials even in humid environments [13] and at low or high temperatures [14]. In the material, hydrogen can interact with cavities and microstructural defects, as well as interstitial sites in the metal lattice [15]. This hydrogen defect interaction leads to a drop in the ductility of the material which causes its weakening [16], [17], [18], [19], [20] and consequently its sudden rupture.

Furthermore, other authors [21], [22], [23], [24] have experimentally studied the mechanical behavior of stainless steels by different methods and have arrived at different theories in order to explain this harmful phenomenon of material degradation.

Recently other in-depth work has been published on this study of the internal structure of stainless steels [25] and the reduction in mechanical properties due to the hydrogen embrittlemnt (H.E) phenomenon. These authors choose as hydrogen charging methods [26], [27] the electrochemical method [15], [28] at constant current intensity at room temperature with a variation in the time factor. This method has the advantage of providing pure hydrogen [21], [23].

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On the other hand, defects in the crystal lattice such as dislocations and vacancies exert a great influence on diffusion processes [27], [29]. The transport of hydrogen by dislocations has received numerous experimental proofs [15], [29], [30], [31] likewise it has been demonstrated that dislocations could constitute traps for hydrogen. Hydrogen in the form of a proton (H+) can be transported by dislocations and contributes to the creation of a microcrack at the level of preventing dislocations [32]. Interfaces such as grain boundaries [15], [32] or secondary phases [29] should be considered as rows of dislocations, and are thus likely to accumulate hydrogen in solute form, very possible in molecular form, thus weakening the interface. According to Y. Murakami and H. Matsunaga [15] non-metallic inclusions, which by optical observation appear as dark zones ODA (Observation of the dark area) see (Figure 10) (a) and (b), play a very important role in the process of fatigue crack propagation.

In order to make a contribution to this phenomenon, this present work was carried out using mechanical tensile testing [32] and 304L austenitic stainless steel electrolytically charged with hydrogen at room temperature and at different times.

2. Materials and methods

2.1. Chemical composition

The material used in this experimental part is AISI304L austenitic stainless steel having the chemical composition given in the (Table 1) below:

Table 1: Chemical composition by mass (%) of AISI304L steel.

Fe%	C%	Cr%	Ni%	Si%	Mn%
69,50	0,0652	17,97	9,63	0,0126	1,64
P%	**S%**	**Nb%**	**Mo%**	**Al%**	**Co%**
< 0,00030	0,0283	0,0496	0,367	0,0078	0,0992
B%	**V%**	**Ti%**	**Cu%**	**W%**	**Pb%**
0,0152	0,0887	0,0054	0,717	0,0317	0,0037

2.2. Mechanical characteristics

The mechanical characteristics of the material in its raw state are shown in (Table 2) below:

Table 2: Mechanical characteristics of the steel studied.

Material	Rm (Air) (MPa)	Ré (Air) (MPa)	Rr (Air) (MPa)	A (Air) (%)	Z (Air) (%)	HV (30) (Air) (N/mm^2)
AISI304L	686.80	589.0	414.0	41.0	78.0	227/232

2.3. Specimens

The experimental part consists of using standardized tensile specimens DIN50125 [33] having the cylindrical shape and dimensions shown in (Figure 1). These test pieces are obtained by machining using a semi-automatic lathe.

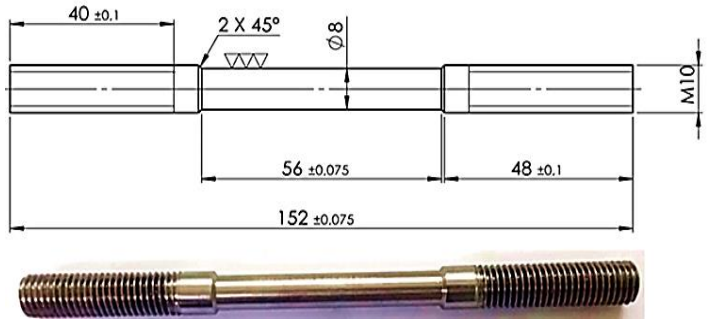


Figure 1: Shape and dimensions of tensile test specimen type in (millimeters); used according norme to DIN50125 [34].

2.4. Heat Treatment (Quenching, tempering)

After the manufacture of the specimens, an austerization treatment was carried out according to the thermal cycle given in (Figure 2) [34].

It consists of heating for 30minutes at 1050°C, followed by water quenching (Rapid water cooling). To obtain a homogeneous and stable austenite structure (γ Austenizing), the next step consists of applying tempering at 700°C for 35minutes and then cooling in ambient air (Open air cooling).

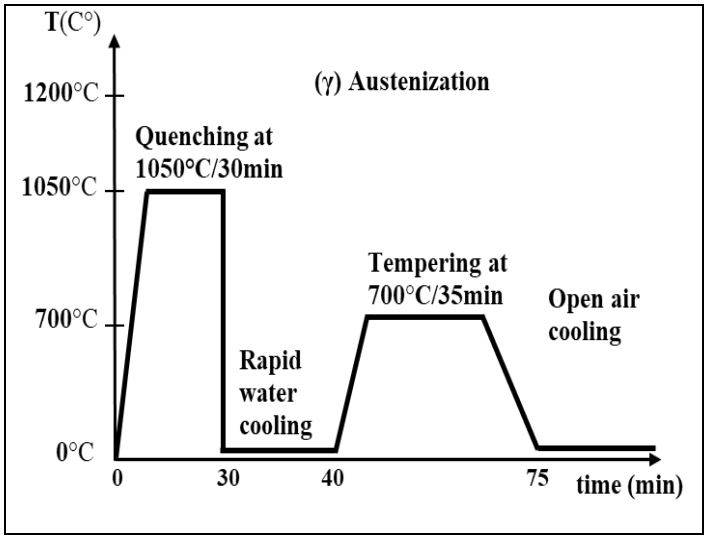


Figure 2: Heat treatment cycle applied [34].

2.5. Cryogenic treatment at -196°C

After the austenization treatment cycle, the specimens are cold quenched at a temperature of -196°C of liquid nitrogen (N2), (Cryogenic treatment), following the thermal cycle presented in (Figure 3) [34]. The test specimens are kept in the liquid nitrogen tank for 35minutes, followed by a slow heating in ambient air for approximately 45 minutes.

This cold treatment cycle of the test specimen was repeated twenty times (x20).

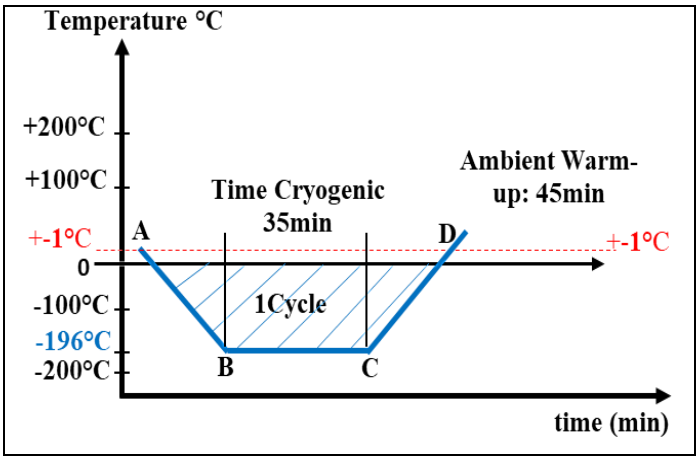


Figure 3: The cryogenic quenching cycle used at -196°C [34].

2.6. Conditions of hydrogenated

After the cyclic cryogenic quenching treatment, the specimens are electrolytically (hydrogenated) pre-charged with hydrogen. The method consists of using a pyrex glass cell enclosure containing a 0.1N H2SO4 solution acid sulfuric equipped with two electrodes, an unattackable platinum anode, and a cathode connected to the specimen, see (Figure 4).

Charging was carried out at room temperature with a with a conditions industrial adopted [13], [14], [21], [23], [26], [28], [30], [31], current density equal to 100mA/Cm^2 for different durations of the pre-charging time from 01hours to 13hours with a step of 01hours.

Figure 4: Pyrex glass cell (hydrogen pre-loaded specimen in solution aqueous 0.1N H_2SO_4, /100mA/Cm^2/pH=1.32)/ [13], [14], [21], [23], [26], [28], [30], [31].

2.7. Mechanical fracture tensile

Immediately after loading, the specimens undergo mechanical tests at room temperature using an instrumented tensile [21], [23], [30], [32] machine model Frank karl GMBH with a maximum load equal to 400KN and a nominal speed of 23mm/min equipped with a stress curve plotter (displacement force) see (Figure 5).

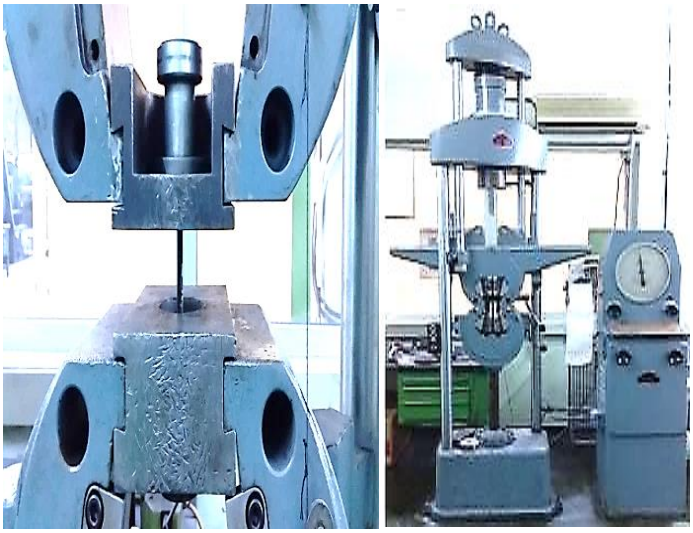


Figure 5: Tensile model testing machine Frank Karl GMBH [32].

Table 3 presents the mechanical properties after the pre-loading protocol and the mechanical test by tensile fracture at room temperature:

Table 3: Mechanical characteristics of the steel studied.

Data specimen (Air)	Stress $Rm_{(Air)}$ (Mpa)	Yield $Ré_{(Air)}$ (Mpa)	Failure $Rr_{(Air)}$ (Mpa)	Elongat $A_{(Air)}$ (%)	Strictures $Z_{(Air)}$ (%)
Refence0 (Air)	686.8	589.0	414.0	41.0%	78.0%
Hydrogen loaded in (hours)	**Stress Rm_H (Mpa)**	**Yield $Ré_H$ (Mpa)**	**Failure Rr_H (Mpa)**	**Elongat A_H (%)**	**Strictures Z_H (%)**
1hour	532	286	302	58%	79%
2hours	553	254	334	57%	78%
3hours	553	246	336	56%	79%
4hours	549	238	350	61%	81%
5hours	541	238	342	58%	80%
6hours	551	238	358	59%	80%
7hours	545	230	358	57%	80%
9hours	551	238	350	58%	80%
8hours	541	230	358	57%	80%
10hours	543	230	334	69%	82%
11hours	525	230	350	57%	80%
12hours	515	222	342	64%	81%
13hours	515	191	350	57%	80%

3. Results and discussions

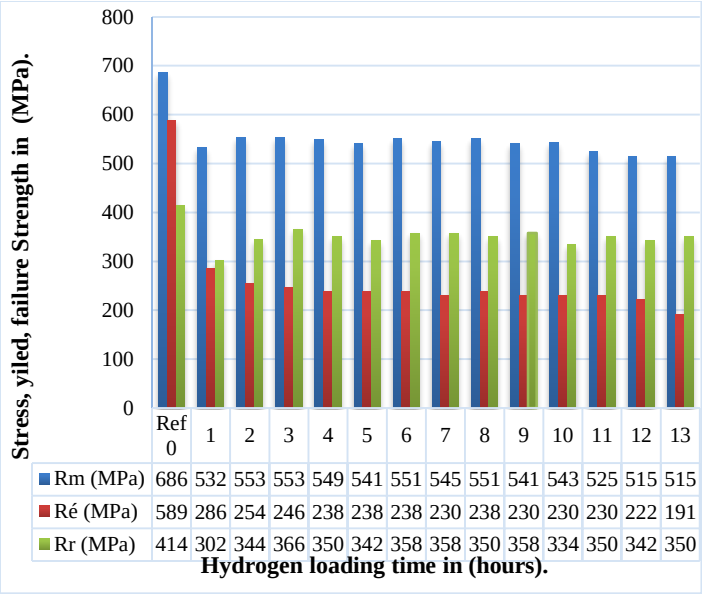
Analyzing the numerical results obtained, deduce the mechanical characteristics after the fracture tests of all the test specimens and which are indicated in (Table 3) in the form of experimental curves see the figures below (see Figures 6 and 7), in order to deduce the following remarks:

The analysis of these curves shows a slight decrease in the resistance of the material as a function of the quantity of absorbed hydrogen represented by the loading duration, see (Figure 6) (a) and (b). This is in agreement with the work carried out by various researchers who stipulate that hydrogen, by diffusing in the form of (H+) cation in the material, regroups in the form of H2 gas to occupy the existing defects and consequently exerts internal pressures [15], [21], which reduces resistance [23], [29].

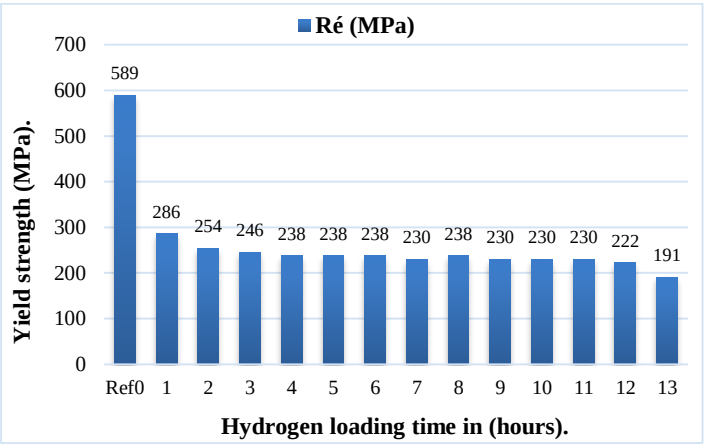
Firstly, (Figure 6) (a), represents a decrease sensitive to the values of the mechanical resistance stress strength (Rm) as a function of hydrogen pre-charging time in hours. In parallel, a comparison of these values with the reference sample which presents a significant value is greater of the order of $Rm_{(Air)}$: 686(MPa) to Rm_H: 532(MPa) or the mechanical resistance passes through a minimum value of the order of Rm_H: 515(MPa), see (Table 3).

Secondly, a behavior expected by an yield strength (Ré) in (MPa) as a function of the level of number of hours of hydrogen pre-charging, which represents a progressive reduction at these yield compared to the Reference0(Air) is of the order of $Ré_{(Air)}$: 589(MPa) to $Ré_H$: 286(MPa), up to the duration of 13 hours or this yield strength passes through less significant values of the order of $Ré_H$: 191(MPa) see (Figure 6) (b). According to the two parameters of the variation of the mechanical resistance: stress strength (Rm_H), yield strength ($Ré_H$) in (MPa), which undergo drops and progressive reductions compared to the reference0 (Ref0 Air) value.

The result is that this type of AISI304L alloy studied is fragile under the effect of the industrial conditions adopted [13], [14], [21], [23], [26], [28], [30], [31], the environmental medium used and the different loading times, thus the value of the density of the applied polarization is: 100mA/Cm^2 in order to carry internal microcracks induced by the influence of these mechanical properties due to the absorption of charged hydrogen see (Figure 8) (b).



(a)



(b)

Figure 6: (a): (Rm, Ré, Rr) stress, yield, failure strength variation of resistance in (MPa), (b): ($Ré_H$) Yield strength in MPa of AISI304L steel with hydrogen loading time in hours.

Observing (Figure 7), we notice a slight improvement in plasticity as a function of hydrogen loading times. This result is in agreement with the work of [24], the latter were able to conclude that the reduction in resistance yield strength (Ré) [23], and failure strength (Rr) can be caused by increasing the temperature of the tempering treatment, which induces an increase in the ductility of the material studied and significantly improve their plasticity which is associated with the elongations A(%) in percent, comparing with the reference0(Air) i.e. from 41.0(%) to A_H: 58.0(%) in order to achieve a value of being stable throughout the hydrogen loading stage in hours up to 13 hours, i.e. by a value that was important is: A_H: 57.0(%). In parallel, the data on the Z strictures (%) in percent explained by significant increases are even significant compared to the reference is of the order of Z(Air): 78.0(%) to Z_H: 79.0(%), or these strictures go through improvements in order to be stable at Z_H: 80.0(%). The result is that the plasticity of the steel studied increases significantly and remains almost constant throughout the hydrogen loading stage in hours, due to the sets of heat treatments of tempering at 700C°, thus the use of the protocol immersion in succession following the cycle applied, see (Figure 3) by cryogenic quenching at -196°C.

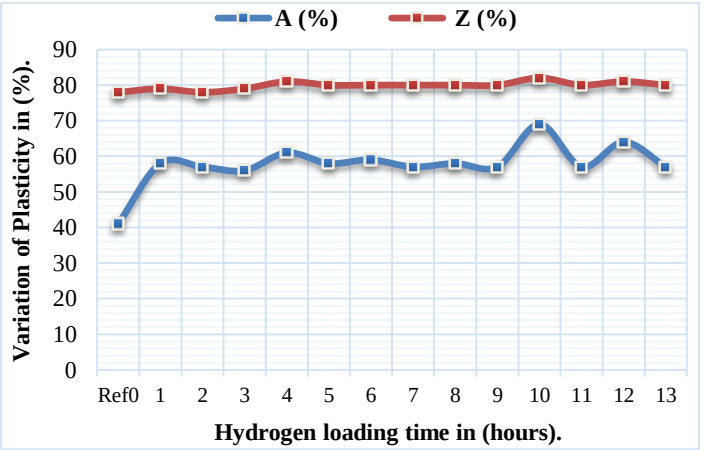


Figure 7: Influence of hydrogen loading on the plasticity A, Z in (%) of AISI304L stainless steel.

(Figure 8), represents the cracking mechanism which linked to the observations of the facies, resulted that these test samples showed facies characterized by two different zones after plastic deformation by fracture, one is charged by hydrogen during a loading period of 13hours, is fragile plane oriented with an angle of 90° relative to the stress exerted see (Figure 8) (b), and the second in raw state without heat treatment, shows a ductile rupture with an angle of orientation is 45° with the applied stress, see (Figure 8) (a). This experimental work validated by the authors [16], [18], [19].

After the mechanical tests by fracture, the study of the facies of the broken specimens, see (Figure 8) (a) and (b) as well as the microstructure of the AISI304L steel was carried out by different techniques: Optical microscopy (OM), x-ray diffraction (XRD), and scanning electron microscopy (SEM). Observation under an optical microscope, after mechanical polishing and chemical attack with the 10% solution of oxalic acid ($C_2H_2O_4$), including the composition of 10grams of oxalic acid in 100milliliters of distilled water with an attack duration of 10 to 45 seconds under a voltage of 6 Volts to reveal the microstructures represented using a Leica DM4 GMBH brand optical microscope (MO), see (Figure 9) (a) and (b).

(a) **(b)**

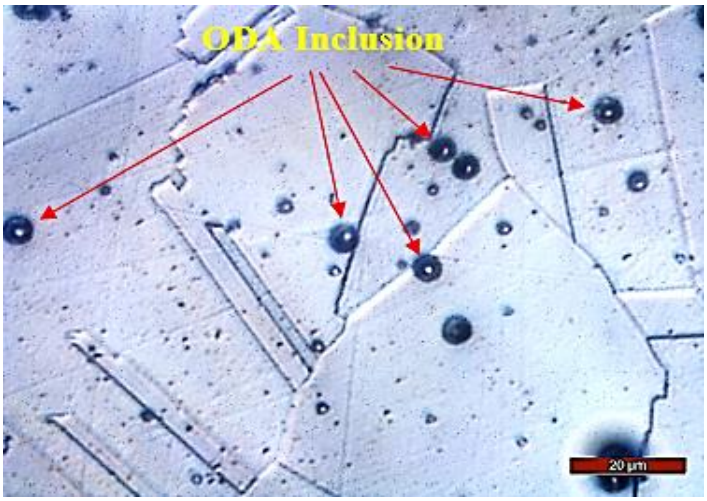
Figure 8: Observation of the specimen facies after mechanical testing (a): crude state; (b): hydrogen loaded state.

AISI304L steel is a low carbon austenitic stainless steel. During the electrolytic hydrogen charging, ODA dark zones are created at the grain boundaries [15], [32], see (Figure 10) (a) and (b).

Under the effect of deformation stresses, the hydrogen charged in the material will be transported by the dislocations towards the plastic zone of maximum stress. The accumulation of hydrogen in defects causes crack growth. The formation of molecules along a row of adjacent hydrogen atoms causes its dislocations to anchor. This causes the creation of a plastic zone of maximum stress at the same time as we create a fragile zone and develop maximum stress, which reduces the breaking resistance of this zone and consequently gives the creation of microcracks [21], [23].

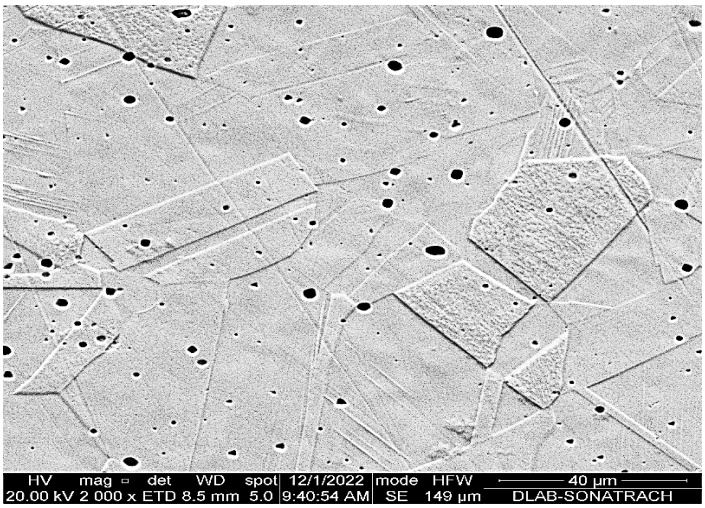
The coalescence of its microcracks under the effect of the high pressures of hydrogen molecules H2 into a large crack causes the displacement and advancement of this crack. Precipitations of carbides in the grain boundaries [31] in square format [27], [29], due to the penetration of the hydrogen molecule form what is called the fish-eye [15] view (Figure 10) (a) and (b).

(a)

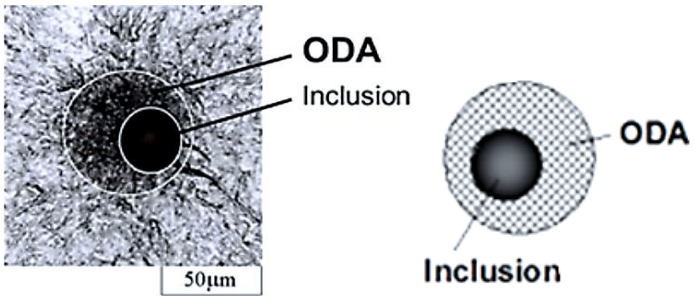


(b)

Figure 9: Optical microscopy images of, (a): the rough microstructure of AISI304L stainless steel (x20µm), (b): of the AISI304L stainless steel loaded time in 13hours with hydrogen (x20µm).



(a)



(b) [15].

Figure 10: (a): SEM microstructure of hydrogen loaded AISI304L stainless steel, (b): SEM Observation of the Dark Area (ODA) [15].

The EDAX analysis of the reference SEM (FEI Quanta FEG 650), that the observation of the dark area (ODA) shows a reduction in the quantities of carbon, iron and chromium compared to the raw material without treatment [15], [21], see (Figure 11) and (Figure 12) below. Comparing the two figures we notice the total absence of sulfur in the raw structure while the microanalysis of the ODA [15] shows the appearance of this element which, by interacting with hydrogen, can form H2S which can be responsible for the phenomenon of hydrogen embrittlement (H.E) of AISI304L stainless steel. The result is that the sulfur (S) element clearly appears to be harmful elements with respect to resistance to hydrogen embrittlement (H.E).

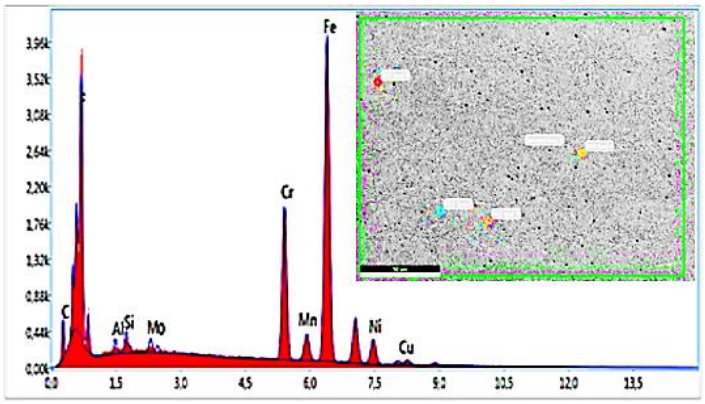


Figure 11: Scanning electron microscopy by (EDAX), microanalysis of AISI304L Crude.

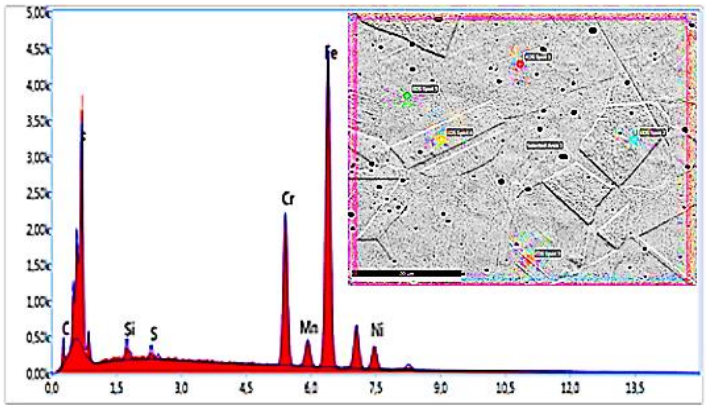


Figure 12: Scanning electron microscopy by (EDAX), microanalysis of ODA of hydrogen-loaded of AISI304L stainless steel.

4. Conclusions

In this experimental study, we have shown the sensitivity of AISI304L steel to both quenching and tempering heat treatments in the ranges of applied temperatures between 1050C° and 700C°, which followed by cryogenic quenching at -196C° to the presence of pre-charging of induced hydrogen at ambient for different durations of charging times. Based on the results obtained, we can draw out the following points:

The variation of mechanical resistances stress strength (Rm), yield strength (Ré), and failure strength (Rr) is very sensitive and associated with the microstructure depending on the loading conditions polarization density, the environmental medium, loading time, can lead to a strong difference in concentration content of hydrogen between the surface and the core of the internal structure of the matrix.

In charging by the cathodic route, the quantity of desorbed hydrogen increases with the duration of cathodic charging in hours, which negatively influences the resistance of this material such as its yield strength (Ré).

The experimental curves of the resistance variation show that the hydrogenated mechanical resistances (Rm_H, $Ré_H$, Rr_H) go through a significant reduction compared to the references resistances $Rm_{(Air)}$, $Ré_{(Air)}$, $Rr_{(Air)}$.

The effects caused by the hydrogen embrittlement (H.E) phenomenon on AISI 304L stainless steel are characterized by a decrease in its ductility, sometimes undergoing sudden weakening.

Heat treatments and electrolytic hydrogen charging can significantly improve the plasticity of AISI304L stainless steel.

ODA black points are points where hydrogen is concentrated either in molecular form H2 or combined with sulfur in form H2S. These two gases are responsible for the (H.E) phenomenon in AISI304L stainless steel.

5. Conflict of Interest

The authors declare no conflict of interest.

6. Acknowledgment

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