



Analysis of fatigue behaviour of stainless steels under hydrogen influence

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ABSTRACT. Three stainless steels – ASTM 304, 316 and 316L - used in hydrogen utilization equipment are under investigation at conditions of tension-compression, rotating-bending and fretting fatigue. Fatigue tests are carried out with hydrogen charged and uncharged specimens. Hydrogen charging includes cathodic type of charging and exposure to high pressure hydrogen gas. The experiments under rotating bending and tension-compression fatigue are conducted under different frequencies in three different laboratories: at The University of Chemical Technology and Metallurgy, Sofia, Bulgaria; at Sandia National Laboratory, California and The University of Tufts, Medford, Massachusetts, USA; The HYDROGENIUS Institute at Kyushu University, Japan. The fretting fatigue tests are presented by The HYDROGENIUS Institute at Kyushu University, Japan. The obtained results are presented in Wöhler curves complemented by plots "Short fatigue crack length–Number of cycles" and "Tangential force coefficient–Stress amplitude". The found fatigue characteristics are analyzed and compared at different loading conditions, showing the best performance of Steel 316L.

KEYWORDS. Tension-compression fatigue; Rotating-bending fatigue; Fretting fatigue; Stainless steels; Short fatigue crack; Hydrogen influence.

INTRODUCTION

There are many investigations done into one of the most attractive alternative energy technologies, the hydrogen technology, including hydrogen produce, and hydrogen storage and infrastructure. Although over the last years hydrogen vehicles and utilization machines are in active use across the world, there are still questions to be answered about hydrogen influence on fatigue and fretting fatigue of alloys used in hydrogen fuel cells, engines, compressors, storage tanks, pipes and different members of hydrogen transportation elements. The most frequently used alloys are austenitic stainless steels. It is known that hydrogen environment can affect steel microstructure having changed steel crystal lattice mechanical properties and fatigue life.



Our paper presents the results of research on fatigue behavior of austenitic stainless steels at different fatigue and fretting fatigue conditions, which will be used for future characterization, analysis and predictions of fatigue and fretting fatigue in products made of these steels, and subjected to hydrogen-environment influence.

MATERIALS AND EXPERIMENTAL WORK

Materials

Studies of our team on hydrogen influence on metals are focused on analyzing fatigue and fretting fatigue characteristics of three austenitic stainless steels ASTM 304, 316, 316L (SUS304, SUS316, SUS316L by Japanese Standard) Tab. 1 [1-8].

Tests

A. Fretting fatigue tests

The tests are carried out in the HYDROGENIUS Institute, Kyushu University, Japan

- Kondo et al [3] examine the hydrogen influence on Steels 304, 316, 316L by two types of specimens: hydrogen charged and uncharged ones

The fretting fatigue tests are carried out under the following conditions:

1. Using of an assembly shown in Fig. 1a and specimens represented in Fig. 1b;
2. Pressing contact pads (Fig. 1c) against the specimen by tightening the bar springs through clamping bolts;
3. Inducing fretting by the difference of deformation between the specimen and contact pads, when a constant amplitude cyclic bending moment applies to the assembled fatigue specimen;
4. The specimens and pads used in the tests are made of the same steel.

Some specific characteristics of this fretting fatigue testing are connected with the following details [3]. The pads used are bridge type ones with 3mm contact length of each foot in the relative slip direction. The contact edge of each pad is square without chamfer. The 0.5mm foot length is chosen in order to generate a large relative slip range as the effect of

Chemical compositions of steels (wt %)								
Steel	C	Si	Mn	P	S	Ni	Cr	Mo
304	0.06	0.51	0.92	0.033	0.004	8.08	18.8	–
316	0.05	0.49	1.31	0.030	0.027	10.22	17.0	2.04
316L	0.012	0.19	1.64	0.031	0.012	12.19	16.6	2.22

Mechanical properties of steels				
Steel	Condition	Yield stress R _e (MPa)	Ultimate stress R _m (MPa)	Elongation A (%)
304	Solution heat-treated	285	637	60
316	Solution heat-treated	286	598	59
316L	Solution heat-treated	212	530	59

Table 1: Characterisation of used steels (wt %).

hydrogen on fretting can be observed clearer at large amount of fretting wear. The contact surfaces of both, the specimen and the pad, are finished by 400 Emery paper. A part of an experiment is carried out when the contact surfaces are finished only by grinding for investigating the effect of machining process on fretting fatigue strength. Before test starting the contact pressure between the specimen and the contact pad is set up at 100 MPa. It is found that after 10 million cycles of fretting the initial contact pressure drops roughly by 5%. The amount of relative slip is measured by using a small displacement sensor attached at the end of contact pad. The tangential force is measured by a strain gage pasted between the pad feet. The strain of pad is transferred into tangential force by elastic finite element stress analysis. Nominal stress

amplitude applied to the specimen is measured by a strain gage pasted in each specimen. In order to restrict the cracking location, the fretting damage of one pair of the pads is prevented by inserting a thin polyamide film between the contact surfaces.

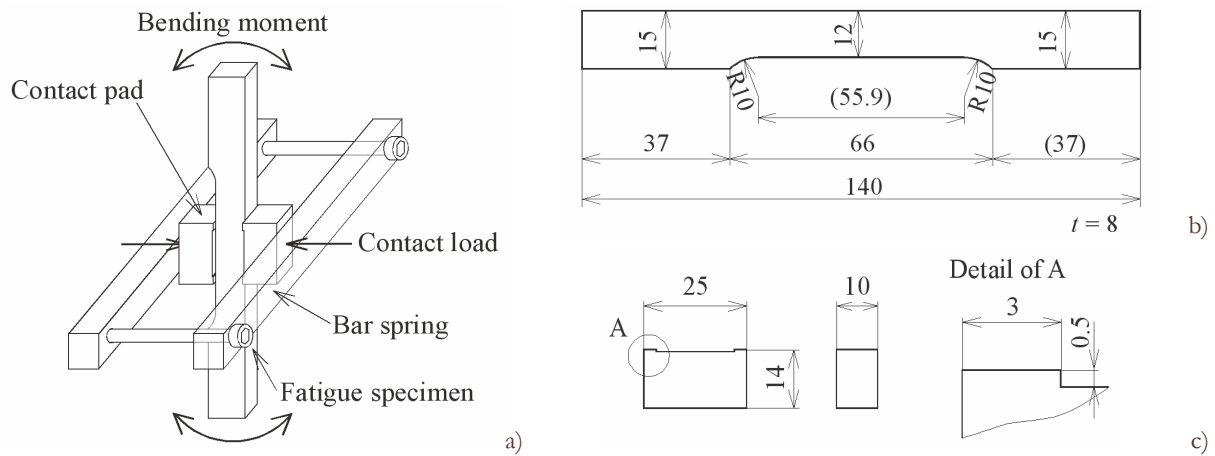


Figure 1: Fretting fatigue test [3]: a) Test assembly; b) Specimen geometry (in mm); c) Bridge type contact pad.

B. Fatigue tests

The tests are carried out in: The University of Chemical Technology and Metallurgy Sofia, Bulgaria; The HYDROGENIUS Institute, Kyushu University, Japan; Sandia National Lab, California & the University of Tufts, Medford, Massachusetts, USA

- Fatigue research team at the University of Chemical Technology and Metallurgy, Sofia, Bulgaria [4] investigates and analyses the hydrogen influence on fatigue behaviour of Steel 316L

The fatigue tests are carried out under the following conditions:

1. Using a table model Rotating Bending Machine FATROBEM 2004 with a corrosion testing box for environment-assisted short-fatigue-crack-growth investigations and specimens shown in Fig. 2a;
2. Applied loading conditions: cyclic rotating-bending at room temperature and laboratory air; testing frequency of 11 Hz; stress ratio $R = -1$; stress ranges σ_a and registered corresponding fatigue lifetimes N_f presented in Tab. 2.

- Skipper [5] examines the hydrogen influence on Steel 316L by two types of specimens: hydrogen charged and uncharged ones.

The hydrogen charging of specimens is performed in the following sequence:

1. Thermal precharging at 573K in 138 MPa hydrogen gas for more than 30 days; specimen's freezing before, after tests for minimizing hydrogen loss;
2. Keeping each charged specimen at room temperature for 1 hour before tests; measuring its hydrogen content by inert gas fusion at a commercial vendor.

The tests are performed on R. Moore Rotating Beam Fatigue Testing Machine at room temperature, frequency of 50 Hz; constant stress range, σ_a ; number of cycles to failure, N_f , determined by specimen fracture or when sufficient deformation precluded rotation.

- Murakami [6-8] investigates the influence of hydrogen on Steel 304, 316, 316L using mainly two types of specimens: hydrogen charged and uncharged ones.

The fatigue tests are carried out under the following conditions:

1. Cathodic and gas environment hydrogen charging;
2. Applying of special heat treatment Non-Diffusible Hydrogen Desorption Heat Treatment (NDH-HT) to some specimens for removing non-diffusible hydrogen reaching a level of 0.4 wppm;
3. Drilling of small hole with diameter and depth 100 μm into the specimens;
4. Tension-compression tests at stress ranges 260, 280 MPa, stress ratio $R = -1$; frequencies 0.0015, 1.5, 5 Hz; a specimen with its hole is shown in Fig. 2b;



5. Surface replicating of short fatigue crack growth.

RESULTS AND DISCUSSION

A. Fretting fatigue tests

The results of fretting fatigue tests are shown in Fig. 3a. The effect of hydrogen on tested steels is different: in 304 fretting fatigue limit is lesser in hydrogen gas (by 13 %) than in air, while in 316 - only slightly lesser; in 316L this limit is almost

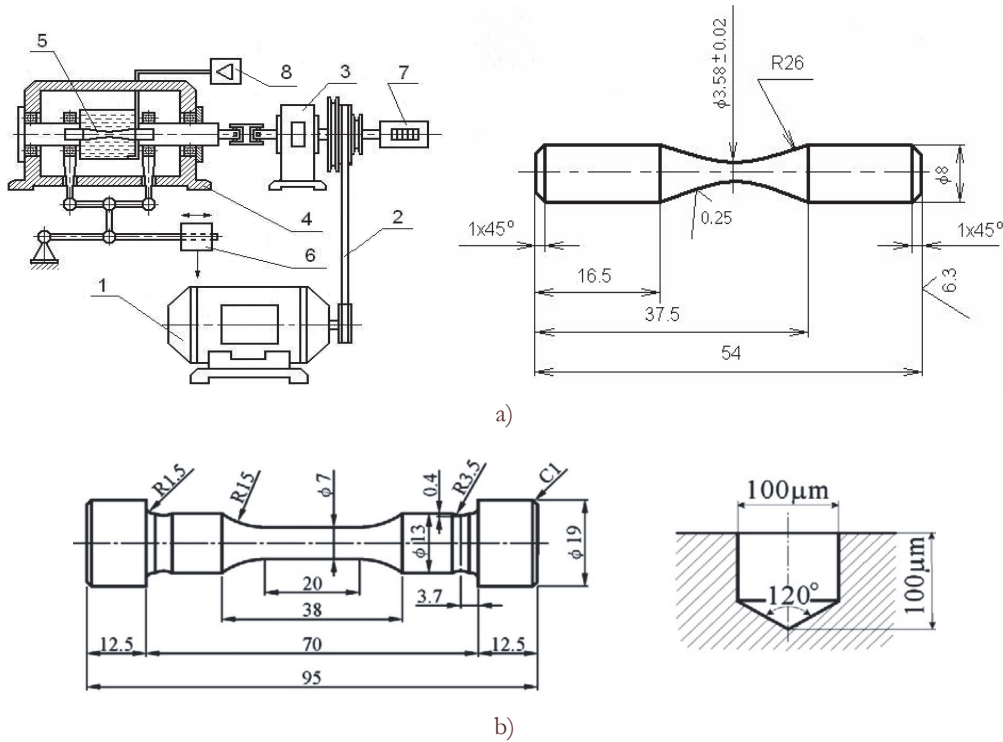


Figure 2: Fatigue test: a) FATROBEM 2004 - electric engine 1, driving belt 2, ball-bearing unit 3, test box 4, specimen 5, loading device 6, counter 7, circulation-aeration device 8; b) Murakami specimen with a drilled hole of 100 μm; all dimensions in mm.

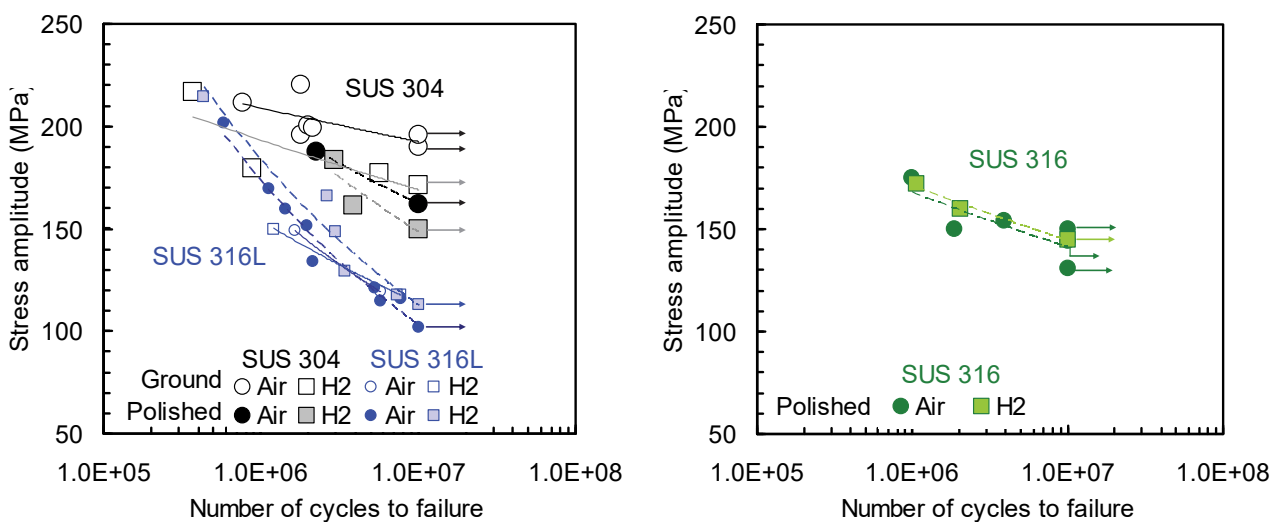
	σ_a (MPa)	N_f (cycles)		σ_a (MPa)	N_f (cycles)		σ_a (MPa)	N_f (cycles)
1	260	3433100	5	340	39600	8	400	129910
2	280	208670	6	360	154000	9	440	38940
3	300	315150	7	380	52030	10	460	9900
4	320	340010						

Table 2: Fatigue life at different stress ranges.

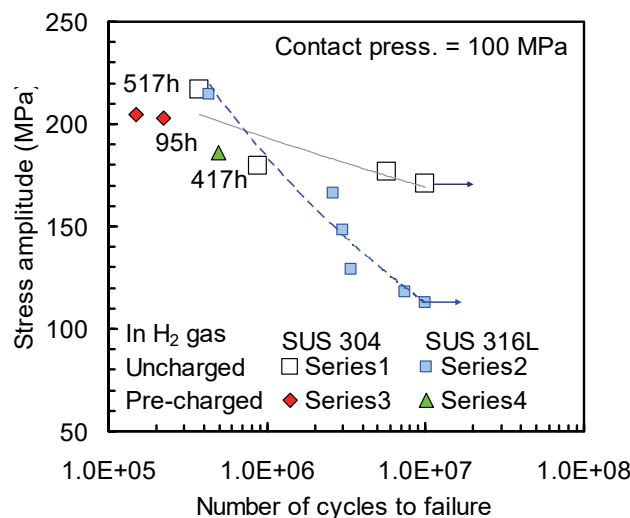
the same as that in air [3]. At the same time there was no reduction of fatigue strength of Steel 304 in case of plain fatigue in hydrogen gas at low gas pressure condition [9]. On the whole austenitic stainless steels are relatively less affected by hydrogen gas (in terms of fretting fatigue strength) in comparison with aluminum alloys which strength decreases by nearly 60% in hydrogen gas [10]. The best “hydrogen immune” steel is 316L.

There is a difference in the effect of machining process on the steels; in 304, fretting fatigue strength of polished specimen is lower than that of ground one, while in 316L, there almost is no difference [3]. Tab. 3 shows micro Vickers hardness of the contact surfaces, measured before and after fretting fatigue test; the hardness of Steel 304 is considerably increased by the fretting, while that of 316L is only slightly increased. Apart of the different hardening of Steels 304 and 316L, there is a correlation between their fretting fatigue strength and hardness, shown in Fig. 4a [11]. When the fretting fatigue strength of Steels 304 and 316L is evaluated by this diagram – Fig. 4a, the hardness effect is evaluated by using the hardness of fretted surface rather than of original surface; the influence of fretting fatigue strength on the hardness might be caused by the heavy work hardening of stainless steels [3].

The tangential force coefficient of Steels 304 and 316L is shown in Fig. 3b [3]. The effect of absorbed hydrogen in these steels is clarified by their hydrogen charging (before fretting fatigue test) which uses cathodic polarization in dilute sulfuric acid [3]. The results are shown in Fig. 4b. In 304, fretting fatigue life is substantially reduced compared to that of uncharged specimen. An increase of charging time reduces fretting fatigue life: the fatigue life of 517-hours pre-charged specimen is reduced to a half of that of an unfretted specimen. Such a noticeable reduction cannot be found in 316L. It is important to know that during the fretting a hydrogen absorption into steels is observed.



a)



b)

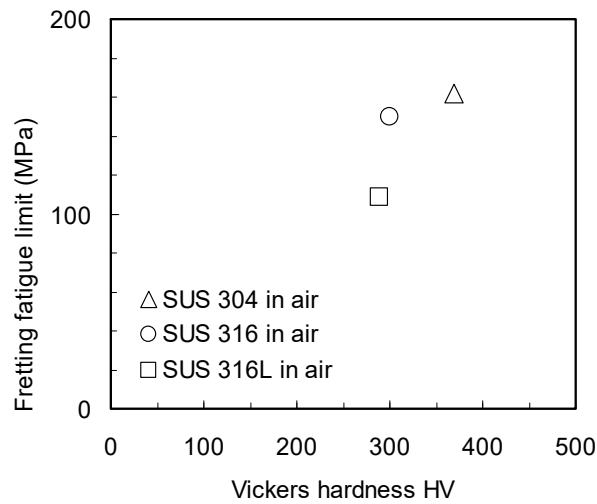
Figure 3: Tests [3]: a) Fretting fatigue strength; b) Effect of hydrogen pre-charging.



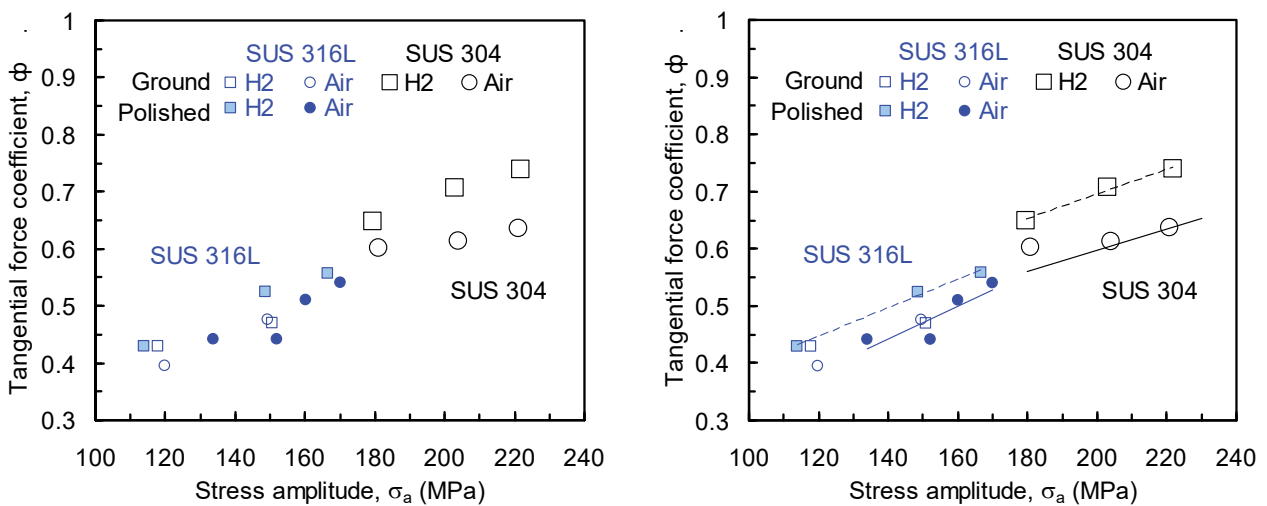
Steel	Mirror finished surface	400 Emery polished surface		Ground surface	
		Unfretted	Fretted in air	Unfretted	Fretted in air
304	192	293	369	330	416
316	182	294	301	–	–
316L	172	271	290	283	285

Table 3: Micro Vickers Hardness of fretted surface (Indentation load = 0.245N)

Formation of martensite due to fretting is observed in Steel 304 and not in 316L; it is found as well that hydrogen absorption in 304 contributes to decrease of fretting fatigue strength through: (a) hardening; and (b) formation of martensite due to fretting [3].



a)



b)

Figure 4: Fretting fatigue [3]: a) Dependence “Fretting fatigue limit – Vickers hardness”; b) Tangential force coefficient.

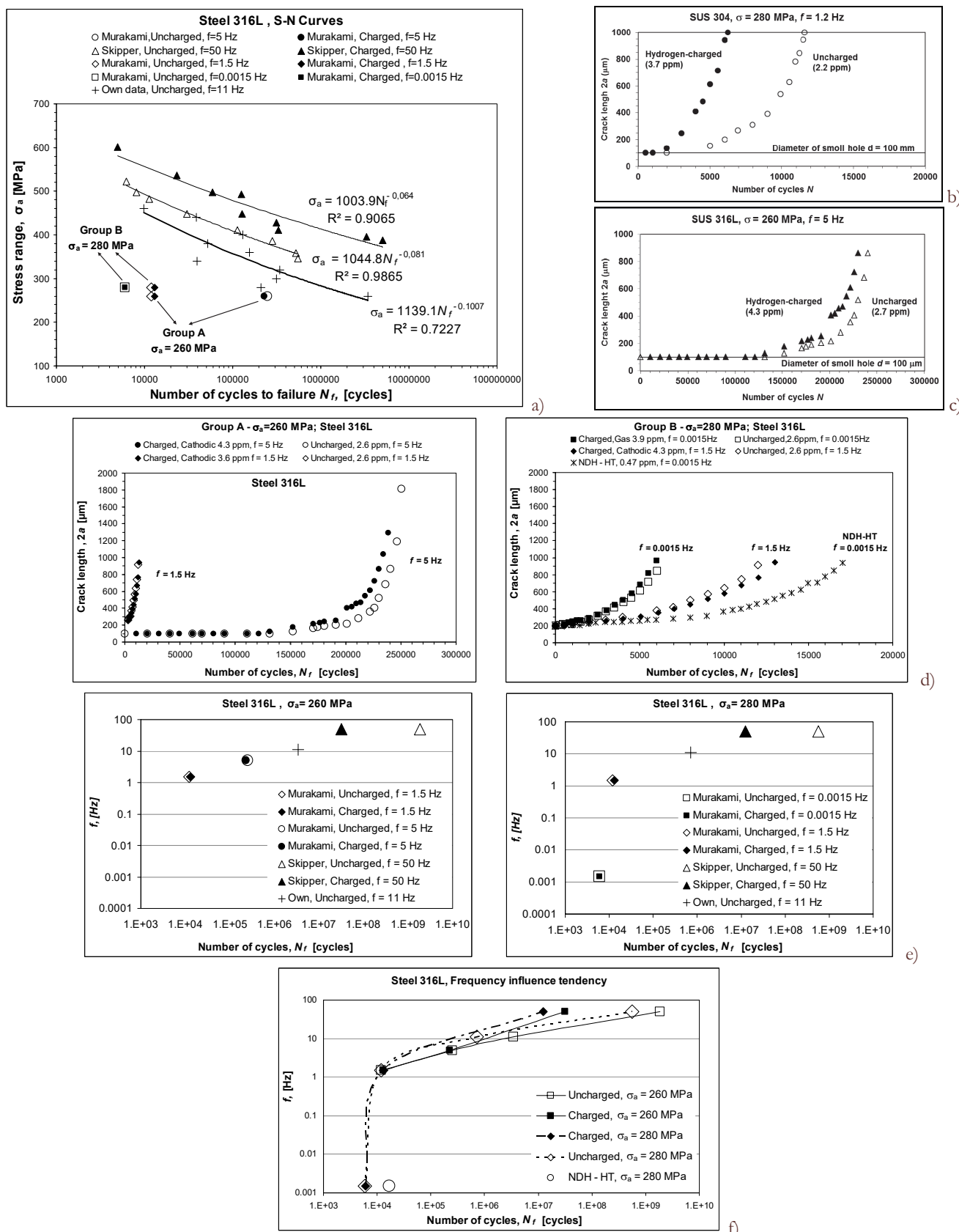


Figure 5: Tests: Wöhler curves, a); Curves a-N for Steel 304, 280MPa, b) & Steel 316L, 260MPa, c); Hydrogen influence on fatigue lifetimes at different frequencies, e-f).



B. Fatigue tests

A comparative analysis between our own results and those obtained by Skipper and Murakami for Steel 316L can be made considering the Wöhler curves shown in Fig. 5a. The data of Skipper and Murakami [5-8] presented in Fig. 5a show the opposite tendencies in the behavior of the same steel taking into consideration hydrogen charged and uncharged specimens. Obviously, in the case of Skipper there is classical hydrogen embrittlement while in the case of Murakami hydrogen affects the steel microstructure transforming it into martensitic one at the tip of propagating crack – a phenomenon much more pronounced in the case of another austenitic stainless steels as Steel 304 and 316 which show more martensitic transformation while stressed than Steel 316L, Fig. 5b-d [7, 8]. Undoubtedly one of the most important factors for this effect is the frequency of testing which assists the penetration of hydrogen on microstructural level. The very low frequency provokes a definite effect of decreased lifetime, Fig. 5e-f. At the same time Murakami noticed [7] that at very low frequencies even uncharged specimens showed a decrease in their lifetimes due to non-diffusible hydrogen (2-3 wppm) trapped in the stainless steel during its production. This type of hydrogen is different from the diffusible hydrogen charged into steel by electrochemical methods or gas environment. Murakami's studies on non-diffusible hydrogen show that this hydrogen can be removed by a special heat treatment NDH-HT, which definitely increases uncharged specimen lifetimes - the single round symbol in Fig. 5f. Murakami marks that the non-diffusible hydrogen has not been considered in the previous classical hydrogen embrittlement studies. In both cases of 260, 280 MPa (Fig. 5e-f) the hydrogen charged and uncharged specimens of Steel 316L show almost the same lifetimes at lower frequencies (0.0015- 5 Hz) and smaller lifetimes for the charged specimens at frequencies above 5 Hz. So, frequency is the most important factor of influence for steels fatigue in hydrogen media. We should note as well that in Fig. 5f the fatigue loading condition above 5 Hz changes from tension-compression to rotating-bending.

CONCLUSIONS

The hydrogen-energy technology still shows many unsolved problems connected with *hydrogen* utilization machines, storage tanks, infrastructure, all using austenitic stainless steels; here Steels 304, 316, 316L are investigated in hydrogen gas and air, and at pre-charged and uncharged state. Now it becomes clear that hydrogen gas *influences* fretting fatigue of these steels at different machining process and hydrogen pre-charge, *changes* absorption of hydrogen during fretting, tangential force coefficient and steel fatigue strength, *transforms* their microstructure to martensitic one. The plain fatigue of the same steels shows some different behaviour of hydrogen charged and uncharged specimens, and the importance of frequency factor which in combination with hydrogen media at high pressure leads to microstructure transformation in martensitic one and diminishes fatigue life of metal members. Under both, fretting and plain fatigue, Steel 316L shows best characteristics. On the whole more deep knowledge is needed for clarifying hydrogen influence on different steels at different fatigue loading conditions.

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