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Research Article

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HYBELLO, Pneumatically Powered Material Testing Device for Pressurized Hydrogen Atmospheres

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Abstract

A new material testing device, Hybello, was developed to characterize the behavior of material in demanding environments such as pressurized hydrogen atmosphere. The Hybello utilizing pneumatic loading was developed and calibrated to test the fatigue properties of standard sized fatigue test specimen. Quenched and tempered low alloy steel, typically used for pressure vessels, has been tested in 10-13 MPa hydrogen pressure. The Hybello produced new and important material fatigue data, which is required to improve the safety, design, and performance of components used in hydrogen gaseous environments.

Keywords: Hybello; Hydrogen atmosphere; Fatigue test; Pressure

Introduction

Modern high-tech and cutting edge technologies put high demands on the development and testing of materials. One example of this is the storage and transportation of gaseous hydrogen, which is a very demanding environment for the materials that are used. Instead of traditional material design parameters, such as yield and ultimate tensile strength, designers need to know more about specific characteristics and performance in real environmental conditions, such as the fatigue endurance in a realistic environment that the material experiences. The field of materials and their properties is wide and includes aspects such as Environmentally Assisted Cracking (EAC), fatigue, and corrosion. In many cases, materials are used in different environments and, for example, the operation environment can affect the materials durability more than what the yield and breaking strength-based calculations can take into account. Life cycle considerations also have a marked bearing on today's materials development.

Testing in a gaseous hydrogen environment is demanding, and new solutions are needed to increase the safety and diversity of testing. The pneumatically powered material testing technology can play an important role in the current and future material testing programs focused on material qualification that are needed to ensure the proper design of components that experience fatigue loading in hydrogen gaseous environments.

International and national rules for fatigue design of components is often based on the tensile strength of the material. This includes the design of components to be used in transport and storage of gaseous hydrogen. The approach of the rules and standards for pressure vessel design is to make a safe combination between the wall thickness and maximum allowable tensile strength. Some standards like the ASME section VIII allow the use of a fracture mechanics approach that uses an accelerated crack growth rate to take the gaseous hydrogen atmosphere into consideration. One clear deficiency is that the current standards used for design do not specifically address the effects of the gaseous hydrogen atmosphere on fatigue crack initiation and early growth that can limit the fatigue life time considerably. Up to now, relatively little test data is available to assess this aspect.

In order to perform material testing in highly pressurized gas environment and other very demanding environments, the technological development path from single bellows pneumatic load device towards the more demanding Double Bellows (DB) loading apparatus for fatigue and combined tension/compression/internal pressure system has been developed and tested. This testing device provides the testing capabilities needed to determine the effect of pressurized gaseous hydrogen environments on the fatigue properties of materials [1].

Objectives and Requirements

The primary objective of the Hybello development and verification is to test a developed pneumatic material test device suitable for 8 mm type of the fatigue specimen under pressurized gaseous hydrogen environment. Key factors and requirements for the miniature autoclave testing system are the size of the loading frames with the pneumatic loading units with metal bellows, the size of the autoclave chambers, the structure of the autoclave with the sealing elements, the accuracy of the displacement rate and the accuracy of the load. The strength of the test material and the cross-section of the test specimen determines the amount of load needed in the tests. It was determined that a test load of ± 15 kN would be sufficient for most tests on hydrogen pressure vessel materials. The requirements for the test set-up for the Hybello are as follows: Constant load, constant displacement rate and fatigue (R=-1) tests shall be possible to perform under high-pressure gaseous hydrogen environment (up to 50 MPa). Further, excellent quality specimen alignment is required and is performed using thin metal slides [2].

The design parameters for the device are thus: maximum hydrogen pressure 50 MPa, the hydrogen vessel volume <~0.3 dm3 the amount of hydrogen is kept to a minimum due to safety factors, the maximum displacement range for the DB apparatus is 2 mm. The number of needed feed troughs is eight and the sealing element type is a double conical metal sealing ring. Safety calculations according to the pressurized vessel von Mises stress distributions was performed. The total length of the device is 245 mm and the maximum diameter is 145 mm, the maximum dimensions of the hydrogen chamber is 74.8 mm in diameter with a maximum length of 117.6 mm. The required properties of the data acquisition system, PLC (Programmable Logic Control) controlling system, LVDT-sensor’s attachments etc., were evaluated and subjects for further studies were also identified [3].

Figure 1 shows the main structure of the device. The specimen to be tested is supported in a separate cassette as shown in the figure.

The fixed end of the specimen is attached to the cassette by the nut. The moveable end of the specimen is supported by thin metal slides and connected directly to the secondary bellows by the load post.

The separate cassette system is fixed by the bolts to the body of the autoclave as shown in the figure. In this manner the volume of gaseous hydrogen is minimized.

The LVDT (linear variable differential transformer) sensor is used to measure the movement of the primary and the secondary bellows via the loading post.

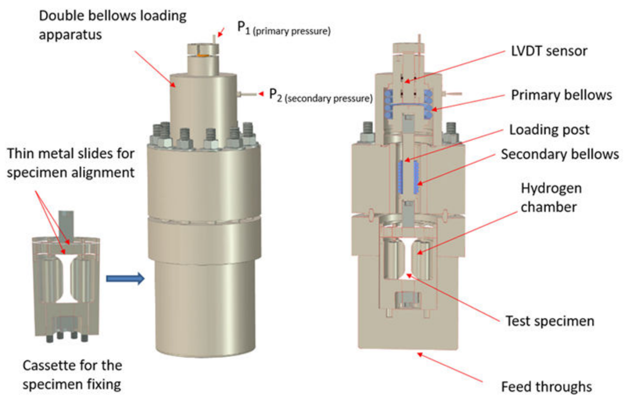

Figure 1: The main structure of the Hybello.

Pressure Integrity Analysis

Stress analyses for the pressure vessel of the Hybello are carried out using commercial finite element program Abaqus.

The computation is performed for the pre-stressing of the vessel bolts and pressurizing of the vessel (Figure 2).

Additionally, two different sealing elements are studied. This analyses was carried out using linear elastic material properties.

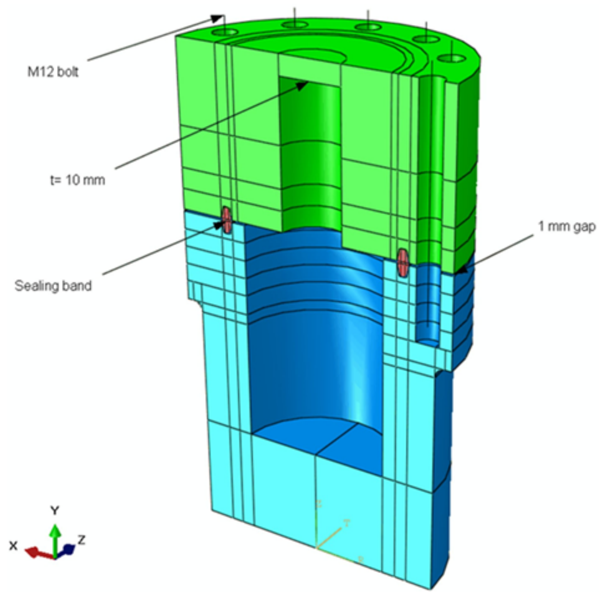

Figure 2: Structure of the pressure vessel.

The results from the analyses of the two design cases with two operating loading are presented below. The reference stresses obtained in the finite element simulations were compared with the European Standard specified requirements for the design of unfired pressure vessels. Figure 3 shows the locations studied for the primary membrane and bending stresses. The stresses (Von Mises) were obtained from the finite element analysis and linearized in order to determine the average stress (i.e. primary membrane stress Pm) over the wall thickness [4]. The primary membrane bending stresses were approximated visually from the stress distribution results obtained in the computation. Both stresses and displacement were investigated. The main results are listed in Tables 1 and 2.

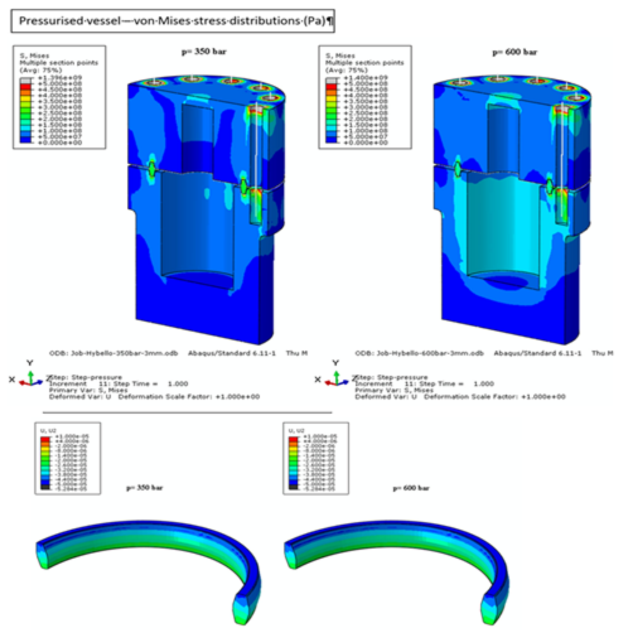

Figure 3: The stress distribution on the structure.

Location	Stress category	Reference stress (Mpa)	Limit	Allowed stress (Mpa)	Criteria fulfilled
Vessel cylinder	P_m	79 (84)	f	400	Yes
	P_{m+b}	100 (120)	1.5f	600	Yes
Sealing channel edge, cylinder	P_m	52 (144)	f	400	Yes
	P_{m+b}	72 (154)	1.5f	600	Yes
Sealing channel edge, cover	P_m	38 (57)	f	400	Yes
	P_{m+b}	90 (111)	1.5f	600	Yes
Cover	P_m	65 (55)	f	400	Yes
	P_{m+b}	94 (80)	1.5f	600	Yes
Bolts	P_m	650	f	<1200	Yes

Table 1: Stress comparison operating pressure 350 bar thin vs thick sealing. Results of the vessel with the thick sealing band are presented in parenthness.

Location	Stress category	Reference stress (Mpa)	Limit	Allowed stress (Mpa)	Criteria fulfilled
Vessel cylinder	P_m	115 (116)	f	400	Yes
	P_{m+b}	152 (175)	1.5f	600	Yes
Sealing channel edge, cylinder	P_m	98 (119)	f	400	Yes
	P_{m+b}	152 (147)	1.5f	600	Yes
Sealing channel edge, cover	P_m	50 (67)	f	400	Yes
	P_{m+b}	135 (68)	1.5f	600	Yes
Cover	P_m	69 (60)	f	400	Yes
	P_{m+b}	93 (83)	1.5f	600	Yes
Bolts	P_m	650	f	<1200	Yes

Table 2: Stress comparison operating pressure 600 bar thin vs thick sealing. Results of the vessel with the thick sealing band are presented in parenthness.

Double Bellows Loading Apparatus

To manage the high pressure of the environment (up to 50 MPa) the Double Bellows (DB) apparatus, is equipped with additional secondary bellows as shown in Figure 4. The primary bellows (working bellows) is installed into the pressure chamber and generates the test load needed [5]. The secondary bellows can eliminate the effect of the hydrogen gas pressure applied. The operational principle and load generation of the DB loading apparatus is described in the following. The LVDT (Linear Variable Differential Transformer) sensor is placed into the upper piston of the primary bellows.

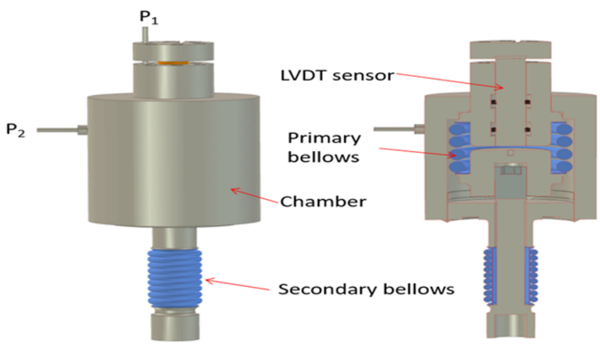

Figure 4: The DB loading apparatus.

The DB loading apparatus has two different pressure boundaries, i.e., (a) between working bellows (p1) and chamber (p2) and (b) between secondary bellows (p2) and environment (p3), see equation 1. Inner pistons are needed for three reasons; to act as a support element for the corrugated bellows elements, to connect the two bellows together and to minimize the gas volume of the bellows. Figure 5 shows the load generation of the DB load apparatus as a function of different pressure variations.

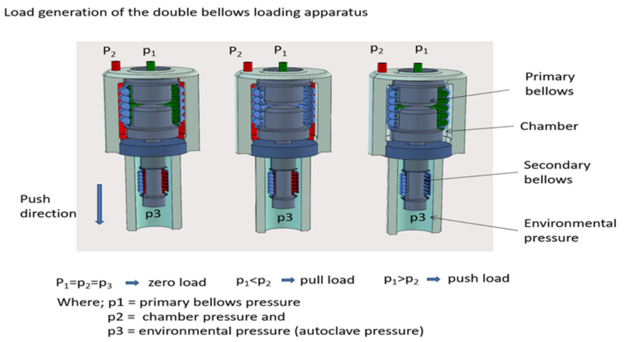

Figure 5: Load generation of the DB loading apparatus.

Load Calibrations for DB Loading Apparatus

Commercial load sensors are typically designed for low temperature gas environments, and they cannot be used in high temperature water environments. The load determination of the pneumatic loading apparatus is based on developed calibration methods where the metal bellows' intrinsic stiffness while the effective cross-section is determined for the true load calculations. A special calibration frame was used to calibrate the applied gas pressure in the DB (working bellows) and pressure chamber with the actual load acting on the load cell (see Figure 6a). The LVDT sensor is placed into the DB loading apparatus to measure the compliance of the whole system during the calibration. This is because the intrinsic stiffness of the DB loading apparatus is relatively high, and thus affects the load calibration accuracy [6].

The DB's (working and secondary bellows) intrinsic stiffness and effective cross-section are needed for calculation of the load acting on the test specimen. The bellows intrinsic stiffness value reported by the bellows manufacturer, e.g. spring rate cannot be applied directly, and the true intrinsic stiffness of the complete pneumatic loading unit has to be determined experimentally. The simplest method to measure the true intrinsic stiffness and the friction factor of the pneumatic loading unit is to perform a test with steadily increasing load using the loading unit without any test specimen. The interior pressure of the bellows, the true intrinsic stiffness, and the friction factor of the pneumatic loading unit with DB are needed to calculate for the force acting on the test specimen. The force induced by the pneumatic loading unit can be calculated from the following equation:

$$F = \left[\left(\Delta p_{b(A)} A_{effwb} \pm \Delta p_{b(B)} A_{effsb} \right) - ownst \right] \qquad (1)$$

Where, F is load, Δpb(A) is the pressure difference on boundary (A), Aeffwb is the effective cross-section of the working bellows, Δpb(B) is the pressure difference on boundary (B) and Aeffsb is the effective cross-section of the secondary bellows and owns is the intrinsic stiffness values of the primary and the secondary bellows. The preliminary calibration tests and calculations for the DB loading unit were performed at a load level of ~3 kN, and with and without environmental pressure [7].

In the first calibration test, the pressure loss arising from the DB intrinsic stiffness (working and primary bellows) and the amount of friction in the internal parts of the pneumatic loading apparatus were determined over the working range. The DB loading apparatus was installed into the autoclave without a test specimen. Then p1 was increased as a linear way up to 0.3 MPa pressure level. The intrinsic stiffness values for the DB loading apparatus was 0,157 MPa/mm at 23°C as shown in Figure 6b.

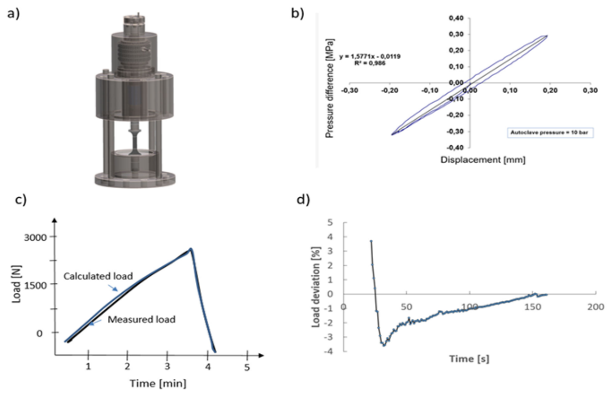

Figure 6: Intrinsic stiffness of the DB loading apparatus at 23°C.

The effective cross-section of the working bellows is 2092 mm^2 see Figure 6 c. There is a small deviation between calculated and measured load curves as shown in Figure 6d. Most probably, this is due to the DB loading apparatus intrinsic stiffness and the internal friction factor of the moving parts. The effective cross-section of the secondary bellows can be calculated by the following way:

$$F = (\Delta p - Own_{st} \times disp)\, A_{eff\,sec} - (\Delta p - Own_{st} \times disp)\, A_{eff\,pri} \qquad (2)$$

where Δp=pressure difference of the working bellows and chamber pressure, F=measured load level, Aeffsec=effective cross-section of the secondary bellows, Aeffpri=effective cross-section of the working bellows, Ownst=Own (intrinsic) stiffness and disp=movement of the secondary and primary bellows. The calculated effective cross section for the secondary bellows was 410 mm^2. Figure 6d summarizes the accuracy of the calibration of the DB loading apparatus. The accuracy of the load and pressure calibration over the tested range was approximately ± 2 %. According to this data, the biggest deviation of the calculated load is at the beginning of the calibration curve. The compliance of the calibration (furnace) frame, initial load value and free clearance of the parts affects the calibration results [8].

Programming of the Motion Axis Control Results

As presented above, the intrinsic stiffness and the effective cross section of the primary and secondary bellows determines the test load together with the pressure differences of the pressure boundaries. To avoid pressure fluctuation of the autoclave the primary bellows pressure and chamber pressure are synchronized together with the autoclave pressure. The synchronization is performed by connecting the primary, chamber and autoclave pressures together with the ratio command in the Motion Axis Control (MAC) program. When the autoclave pressure is changed, the pressure synchronization system can automatically affect the primary and chamber pressures, and thus compensate for the pressure variations between them. The starting and stopping of the test with the pressure synchronization system is easy to perform because when the autoclave is pressurized up to 50 MPa the primary and chamber bellows pressures can automatically follow the autoclave pressure changes and keep test load at zero. Figure 7 shows an example of the visualization of the test parameters. All needed test parameters can be displayed and adjusted on the screen during the test.

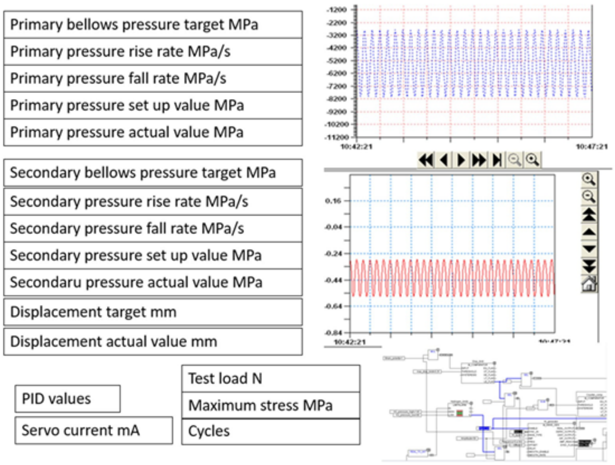

Figure 7: The visualization of the MAC program.

Pneumatic Servo Controlled Pressure Adjusting Loops

The Hybello is designed to test the effects of gaseous hydrogen atmosphere on the fatigue performance of steels or other materials using standard sized round test specimen. The schematic layout for the material testing under pressurized gaseous hydrogen environment is shown in Figure 8. The pressure of the double bellows loading apparatus is regulated by means of a pneumatic servo-controlled pressure control loop which is controlled by the MACS program. With the help of two separate pressure adjusting loops, the double bellows loading apparatus can be adjusted as desired. The pressure loops operates with a constant gas flow and with the servo valves. The maximum pressure for the

system is 20 MPa. The hybello was pressurized with a helium gas bottle as shown in the figure below. The LVDT sensor was used to measure the displacement of the main load post (connected into the primary bellows as shown in Figure 1). The Moog MACS was used to control the test load by the DB loading apparatus. Furthermore, the MACS was used together with the Agilent data logger to collect all needed data during the test.

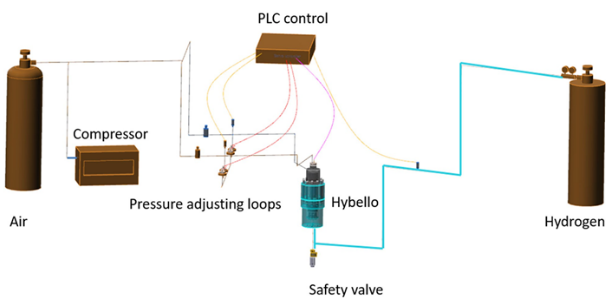

Figure 8: Layout for the Hybello material testing device setup.

Preliminary Test Results under 20 MPa Pressurized Air

The first test with the pneumatically powered Hybello was a reference test at 23°C in air under 20 MPa environmental pressure.

The material used was a martensitic stainless steel, and the specimen geometry is given in Figure 10. In the first test, the loading frequency was 0.3 Hz and a loading ratio R of 0.32 was used during the experiment.

In the second test, the loading frequency was reduced to 0.01 Hz and the loading ratio was changed to -1. A typical set of raw data from the fatigue test is shown in Figure 9.

The designed pressure control program worked as intended during the pressurization of the autoclave (keeping automatically the test load at zero) and during the test.

The measured load and displacement accuracy was ± 1% from the measured value. The long term stability was good during the test. The number of fatigue loading cycles was 30000 during the test.

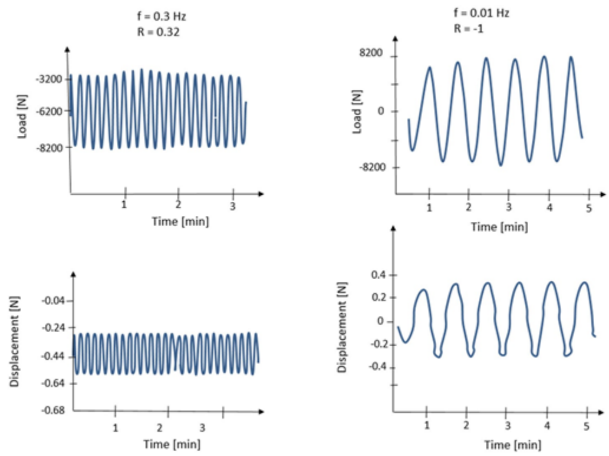

Figure 9: Displacement and load curves as a function of time (f=0.3 Hz, R=0.32 and f=0.01 Hz, R=-1).

Fatigue Tests under Gaseous Hydrogen Atmosphere

The Quenched and Tempered (QT) steel that was tested is a 25 CrMo4 QT steel according to EN 10083-3. The chemical composition of the steel is given in Table 3.

	C	Si	Mn	Cr	Mo
Standard requirements	0.22-0.29	Max 0.4	0.6-0.9	0.9-1.2	0.15-0.30
Product analysis	0.26	0.24	0.76	0.98	0.2

Table 3: The chemical composition (wt %) of the steel studied.

The initiation and growth of fatigue cracks from defects was studied by drilling small holes into the test gauge portion of the specimen.

These holes were either 200 or 500 µm in diameter, with a depth that is equal to the diameter.

An illustration of these holes and their location on the test specimen is shown in Figure 10.

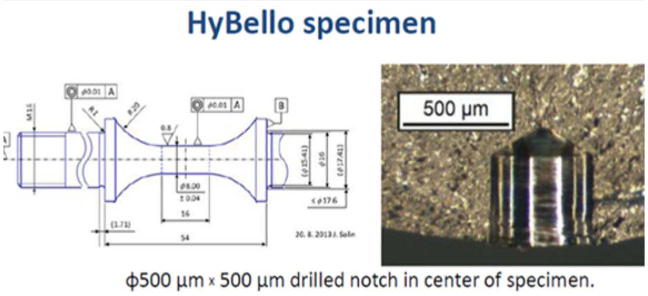

Figure 10: An illustration to show the location and type of small drilled hole used in the research. The measurements are given in mm unless marked as µm.

After the holes were drilled, the test specimens were cleaned in an ultrasound ethanol bath to remove any dirt or grease from the test specimen. The test specimens were then placed into the cassette, which was fixed into the hydrogen chamber of the Hybello, see Figure 1. The device then was closed and sealed, after which the chamber was pumped to a vacuum and then flushed with nitrogen gas twice. This flushing took place by filling the chamber up to the testing pressure with gaseous nitrogen and then removing the gas and pumping the chamber back down to a vacuum. After this, the same flushing procedure was used with ultra-pure (99.99999%) hydrogen gas.

Once the hydrogen gas is removed and a vacuum was pumped the chamber was again pressurized to the test pressure with the ultra-pure hydrogen gas for testing. This flushing procedure is done to ensure the purity of the testing environment. The pressure that was used for the testing as well as the flushing of the testing chamber was 10 to 13 MPa, and the loading frequency was 0.05 to 0.1 Hz. The tests were force controlled and once the desired number of loading cycles was achieved, the testing was stopped and the specimen was removed from the Hybello to record by camera the size of the crack that had initiated from the small drilled holes. Similar testing for comparison was done on the same steel batch in air. The cracks that initiated and grew in the tests performed in air were monitored in situ using a high speed video microscope (Figure 11).

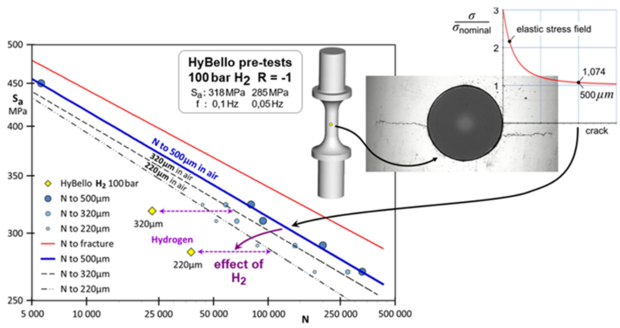

Figure 11: The results of the fatigue tests performed in the Hybello.

On the x-axis the crack size is scaled to the length required for the crack to grow out of one side of the stress gradient of the hole, in this case for a hole with equal diameter and depth of 500 µm. The two legends in the figure provide the stress level used, as well as the number of cycles for the test.

Conclusion

A prototype DB based pneumatically powered Hybello with a miniature sized autoclave capable of performing loading for tensile type testing in hydrogen environment has been designed, built and pre-tested.

The DB loading device with internal LVDT sensor is based on new technology, it operates successfully, and gave reliable test results. The calibrations for the DB loading device were performed with the following results: intrinsic stiffness of the two bellows was 1.26 MPa/mm over the 0.3 mm bellows working range at 23°C, the shape of the intrinsic stiffness curve was linear, and the effective cross-sections of the secondary bellows and working bellows were 410 mm2 and 2089 mm2, respectively.

The accuracy of the load and pressure calibration over the tested load range was approximately ± 2.5 % from 1000 N to 3000 N load level.

The first test with the pneumatically powered Hybello was a reference test at 23°C in air under 20 MPa environmental pressure. The material used was a martensitic stainless steel. The designed pressure control program worked well during the pressurization of the autoclave (keeping automatically the test load at zero) and during the test.

The measured load and displacement accuracy was ± 1% from the measured value. The fatigue test loading ratio was R=-1 with load level ± 7.3kN with a loading frequency of 0.01 Hz. The long term stability was good during the test. The maximum number of loading cycles was 30000 during the test. Metallic double cone sealing of the main lid and feedthroughs worked well and no leaks occurred during the test.

The material selected for the gaseous hydrogen environment tests is 25 CrMo4 QT steel, which was tested at 23°C and 10-13 MPa hydrogen pressure level(s). The specimen type was fatigue pre-holed specimen and notched fatigue specimen.

The designed MAC PLC programs worked well and reliably during the pressurizing period and testing period of the miniature sized autoclave. The effect of the hydrogen atmosphere was measured in the initiation and crack growth from small holes and notches. The acceleration caused by the hydrogen atmosphere was observed clearly when compared to tests performed in air.

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