

Miniature autoclave and double bellows loading device for LWR and SCW material testing at VTT – Calibration, testing and results

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Summary

The pneumatic loading technology provides important potential benefits and has already been successfully applied for materials testing in different test environments. The pneumatic servo-controlled material testing system has earlier been used to perform fracture toughness, corrosion fatique, tensile and electrochemical measurements in gas, high temperature aqueous and irradiation environments. The main advantages of a pneumatic testing system are sensitivity and the possibility to separate the material testing and control system by long (> 30 m) pressure tubes without decreased the test load accuracy. Furthermore, the system can be made compact so that several testing systems can fit simultaneously into one test chamber. Thus the total cost of testing is decreased and the reliability of the test result is considerably increased.

The present work consists of a test set-up study of a new pneumatic loading apparatus based on double-bellows and with miniature autoclaves, enabling applications at pressures up to and higher than 200 bar. It has been demonstrated that it is technically feasible to carry out well defined and controlled material testing in the SCW environment using this set-up. This makes it possible to investigate the intrinsic role of the applied stress on the deformation behaviour of material in light water reactor (LWR) and harsh supercritical water (SCW) environments. The compactness and versatility of the setup makes it particularly attractive for deployment in a hot-cell for testing of irradiated materials.

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Foreword

This report summarizes the development work carried out by VTT to develop a pneumatic loading device of miniature size for supercritical water (SCW) condition together with the Inconel 625 double-bellows loading apparatus. Calibration and pressure controlling systems were designed, constructed and verified for the double-bellows loading apparatus. The testing system was further installed into a miniature SCW autoclave at VTT and the first tests have been performed. Furthermore, the new kind of PLC (Programmable Logic Control) programs for the test load controlling was designed and successfully tested. The funding of this work by VTT is highly appreciated.

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1. Introduction

Modern-day high technologies such as nuclear power plant and fusion reactor technologies have strongly affected the development and testing of their construction materials. Instead of traditional material design parameters, such as yield and ultimate tensile strength, designers have to know much more about specific characteristics of the construction materials. The field of materials and their properties is very wide, including aspects such as environmentally assisted cracking (EAC), fatigue, corrosion and the effect of irradiation. In many cases materials are used in different environments and, for example, the operation environment can affect the material's durability more than simply yield and fracture strength based calculations can take into account. Life cycle considerations also have a marked bearing on today's materials development work.

In recent years the use of fracture mechanics based approaches have become more popular in the research of environmentally assisted cracking (EAC) in SCW environment. The main factors of interest are the susceptibility of the material to stress corrosion cracking (SCC) and stress corrosion propagation rate, $(da/dt)_{EAC}$ or $(da/dt)_{SCC}$, as a function of stress intensity factor KI or J integral. Also, the threshold stress intensity factor for SCC, K_{IEAC} or K_{ISCC} , is of interest. Furthermore, the electric and electrochemical properties of the oxide films formed on construction material surfaces have a significant influence on the susceptibility of materials to general and localized corrosion, such as in EAC in high-temperature aqueous environments. According to the results on modeling high-temperature aqueous corrosion, the properties of oxides are influenced by temperature, potential, electrolyte composition (presence of oxidising, reducing and/or complexing agents) and electrolyte flow rate, in addition to the material composition.

The pneumatically powered material testing technology can play a very important role in the current and future material testing programmes focused on material qualification needed for the extension of the operation licence of the current Gen II and III LWRs, as well as for future advanced fission Gen IV concepts like Supercritical Water Reactors (SCWR), and for construction of the fusion experimental reactor ITER and the demonstrator DEMO. This is particularly true when materials need to be tested at very high temperatures, in environmental conditions, and/or under irradiation in in-pile/in-reactor facilities. As already mentioned above, successful expansion of the pneumatic bellows based technology can occur via utilization in future experiments in high temperature liquid lead facilities or future hot-cell and in-pile facilities for which design is under way. In order to perform material testing under liquid re-circulation loop up to 650°C and other very demanding environments, the technological development path from single bellows pneumatic load device towards the more demanding double bellows apparatus for fatigue and combined tension and compression internal pressure system is under development.



2. Objectives and requirements

The primary objective of the miniature autoclave project is to test a developed pneumatic material crack growth test device suitable for 5DC(T) type specimens (width of 5 mm) under light water reactor (LWR) and supercritical water (SCW) environments. Reliable operation of the loading module, control and data acquisition systems are to be verified by out of reactor tests (in autoclave), followed by possible in-core experiments. The 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 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 determine the amount of load needed in the tests. It was determined that a load of 500 N, producing a stress intensity factor (K) in the range of 8 MPa√m for a 5 DC(T) specimen, would be sufficient for most tests on austenitic stainless steel materials (plane strain condition).

The requirements for the test set-up for the pneumatic material crack growth test device are that it should be possible to perform constant load, constant displacement rate and fatigue (R<1) tests, all using the same pneumatic crack growth test device.

The requirements for the controlling and data acquisition systems were to be approached by evaluating previously used control and data acquisition systems, and assessing the required properties of the data acquisition system, controlling system, LVDT-sensor's attachments etc. of the new device. The topics for further studies were then to be identified.

3. Double bellows loading apparatus

To manage the high pressure of the environment (250...350 bar), the double bellows (DB) loading apparatus is equipped with additional secondary bellows, as shown in Figure 1. The primary bellows (working bellows) is installed into the pressure chamber and generates the needed test load. The secondary bellows can eliminate the effect of the environmental pressure. The operational principle and load generation of the DB loading unit with double bellows is described in the following.

3.1 Theoretical load calculations of the DB loading apparatus

The DB loading apparatus consist of the working and secondary bellows, inner pistons and pressure chamber, as shown in Figure 2. Furthermore, the system has two different pressure boundaries, i.e., (A) between working bellows and chamber and (B) between secondary bellows and environment. Inner pistons are needed for the following reasons; to act as a support element for the corrugated bellows elements, to connect the two bellows together, and also to minimize the gas volume of the bellows. The test load can be calculated by using the following equation:

$$F = \Delta p_{b(A)} A_{effwb} \pm \Delta p_{b(B)} A_{effsb}$$
 (1)

Where **F** is load, $\Delta p_{b(A)}$ is the pressure difference on boundary (**A**), A_{effwb} is the effective cross-section of the working bellows, $\Delta p_{b(B)}$ is the pressure difference on boundary (**B**) and A_{effsb} is the effective cross-section of the secondary bellows



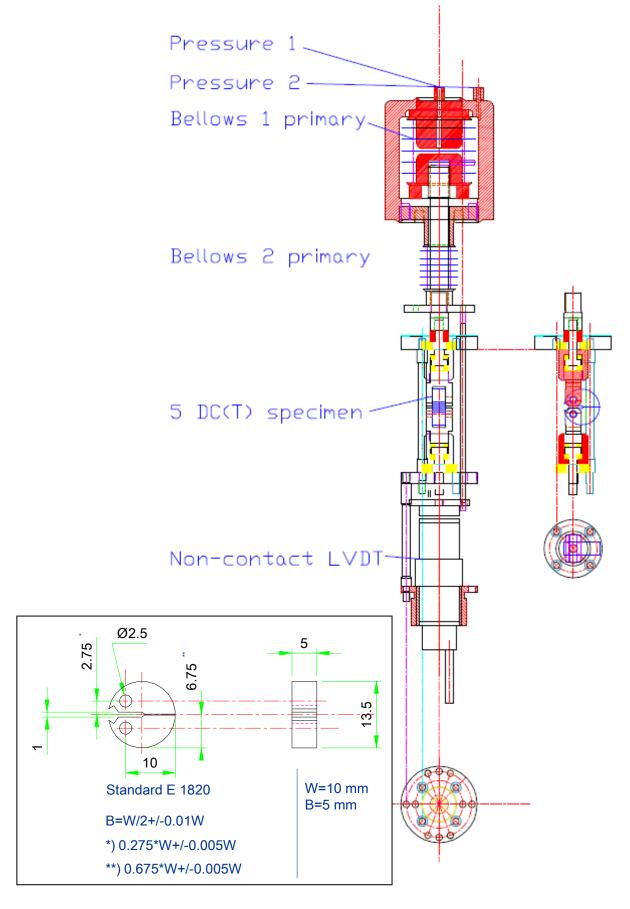
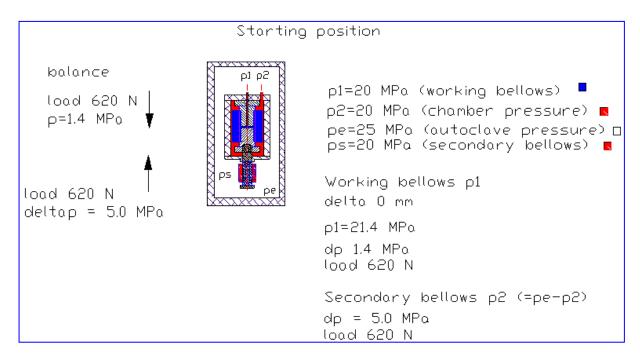


Figure 1. The loading frame with the double bellows, for the 5 DC(T) specimen (inset).





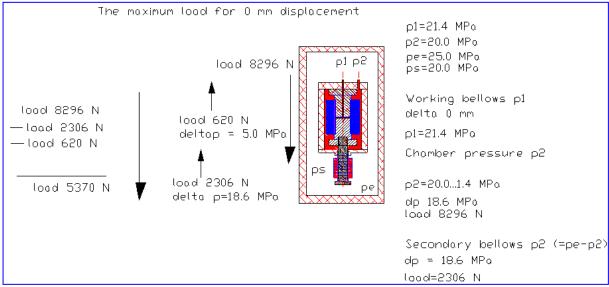


Figure 2. The operation principle of the DB loading apparatus with the double bellows.

The load generation of the DB loading apparatus is based on the operation of pressure boundaries between working bellows, chamber, secondary bellows and environment pressure. In the starting position (see upper schematic of Figure 2), the chamber, working bellows, secondary bellows and environment are pressurized up to 20 MPa pressure level by using pressure pipes p1 and p2. The pressure of the chamber is the same as the secondary bellows pressure. Because of zero pressure difference for both boundaries (A and B), the test load is zero. After that the environmental pressure can be increased up to 25 MPa (working bellows and chamber pressures remain at the same level), and the pressure difference at boundary (B) becomes 5 MPa.

According to equation (1), a 5 MPa pressure difference with the secondary bellows effective cross-section of, for example, $A_{\text{effsb}} = 124 \text{ mm}^2$, will generate 620 N of load, as shown in the upper schematic in Figure 2. To put the DB loading unit into a balanced situation (zero load and displacement), the pressure of the working bellows has to increase up to 21.2 MPa, i.e., the pressure difference for boundary (A) is then 1.2 MPa, which generates the same 620 N load (in the opposite direction) compared to the secondary bellows load.



Now the system is set for a zero load and zero displacement situation, and the test load generation can be started.

The test load can be generated by changing the pressure difference at boundary (A), i.e. between the pressure of the working bellows and the chamber. The maximum pressure difference at pressure boundary (A) can be 18.6 MPa, and according to equation (1) the maximum load for the working bellows is then 8296 N, as shown in the lower schematic of Figure 2. Because the pressure of the chamber is decreasing from 20 to 1.2 MPa, the pressure of the secondary bellows is also decreasing from 20 to 1.2 MPa generating a pressure difference of 18.6 MPa for the pressure boundary (B). This pressure difference provides an opposite load of 2306 N, counteracting the test load as shown in the lower schematic of Figure 2. So, the maximum theoretical test load for this pneumatic loading apparatus with double bellows is 5370 N.

3.2 DB loading apparatus with miniature autoclave

The main operation principle and main dimensions of the SCW autoclave need to be verified according to the DB loading apparatus with the loading frame. The safety calculation for the autoclave is performed under 350 bar and 650°C environment.

The following basic requirements for the SCW autoclave need to be fulfilled:

- 1. SCW type of autoclave, pressure 350 bar and temperature 650°C,
- 2. Material: Nimonic 80A.
- 3. The maximum outer diameter 64 mm.
- 4. Conax type feed through for the LVDT, pressure, electrodes and PD wires, 4 places on the lid of the autoclave,
- 5. Stainless steel type seal on the lid with two contact points,
- 6. Inductive heating system and
- 7. Water inlet pipe is installed into the outlet pipe.

The fulfilment of these requirements is possible though a set-up like that show in Figure 3.

3.3 Operation principle of the pneumatic servo-controlled pressure loop

The testing setup of the pneumatic material crack growth device consists of the loading frame with the pneumatic loading unit and a pneumatic pressure adjusting loop. The main parts of the pneumatic pressure adjusting loop and its four pressure boundaries (A), (B), (C) and (D) are shown in Figure 4. At the pressure boundary (A), the gas flow needed for the servo valve (6) is provided by the fully automatic high pressure compressor (1). An operating pressure of 200 bar is achieved with the compressor. During the tests, the pressure at the pressure boundary (A) may be varied between 175 and 200 bar.

From the pressure accumulator (2) the gas is led to the pressure reducer (3) and then through the manual flow valve (4) by using pipes with an outer diameter of 6 mm. The pressure at the pressure boundary (B) is adjustable with the pressure reducer (3) according to the test environment and specimen size. According to the manufacturer, the maximum operating pressure of the servo valves is 200 bar.



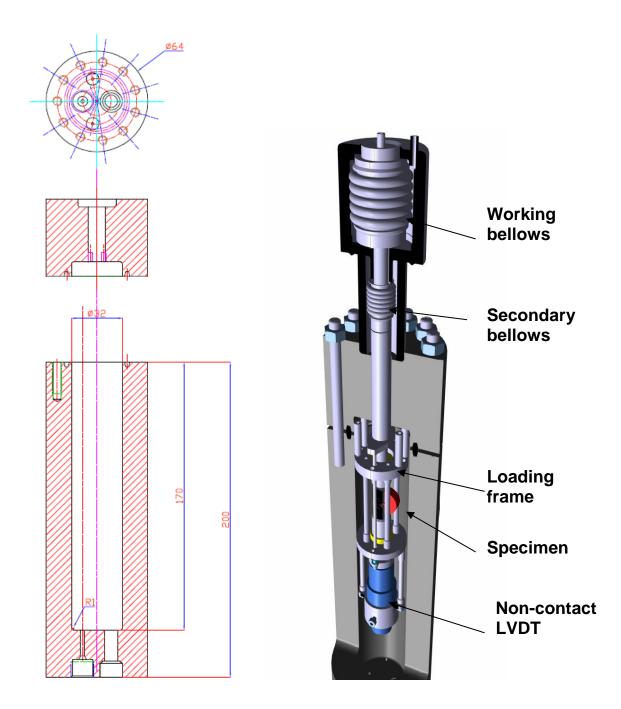


Figure 3. DB loading unit with loading frame installed in SCW autoclave.

From the flow valve (4) the gas is led to the bellows (5) and to the servo valve (6) (pressure boundary (C)) by using pipes with a diameter of 3 mm. The gas is let out from the pressure boundary (C) via the servo valve. A suitable initial pressure for the pressure boundary (C) is adjusted with the pressure reducer (3) and the flow valve (4) for each test. In the previously mentioned example case, the initial pressure for the pressure boundary (C) could be adjusted to 7 bar before the test. In that case, the gas flow through the servo valve would be approximately $8-10 \, l/min$.

During the tests, the pressure of the pressure boundary (C), and therefore also the bellows (5), is adjusted by controlling the continuous gas flow through the flow valve (4) and the servo valve (6). Only one mechanical flowing valve (4) and one servo valve (6) (or electrically controlled closing valve) are needed to provide the adjustable pressure for the pressure boundary C. With this kind of a setup, a two-way pressure controlling system is



created. By opening the servo valve the pressure in the bellows (5) decreases, while throttling the servo valve causes the pressure to increase. The load that the bellows induces to the specimen is directly proportional to the pressure of the bellows (5) (pressure boundary (C)).

The displacement that the bellows produces to the specimen is measured with the LVDT-sensor (7). The LVDT sensor gives a feedback signal to the servo controller. The servo controller compares the LVDT sensor's feedback signal to a pre-set base signal. The servo controller adjusts the servo valve so that the two signals become equal.

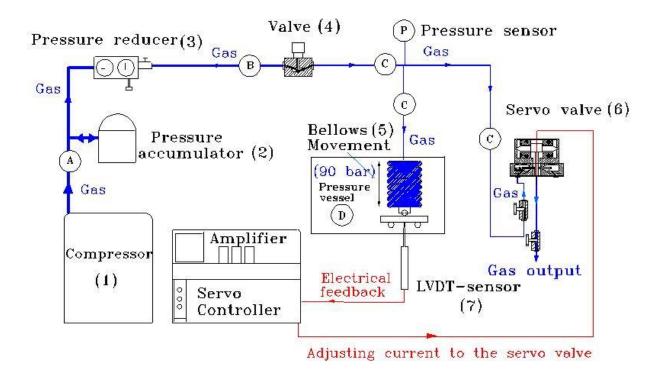


Figure 4. The operating principle of the pressure adjusting loop.

4. Load calibrations for DB loading apparatus

Commercial load sensors are typically designed for low temperature gas environments, and they cannot be installed for high temperature water environments. The load determination of the pneumatic loading apparatus is based on developed calibration methods where the metal bellows' intrinsic stiffness and effective cross-section are determined for the true load calculations. High temperature calibration of the pneumatic loading unit has been performed in a gas environment in a furnace, as shown in Figure 5.

4.1 DB-bellows calibration

The bellows (working and secondary bellows) intrinsic stiffness and the 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 inside pressure of the bellows, the true intrinsic stiffness and the friction factor of the pneumatic loading unit with double bellows are needed to calculate for



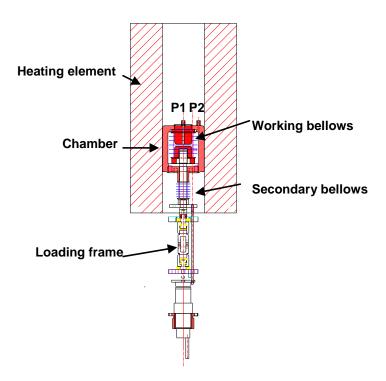


Figure 5.Intrinsic stiffness measurement for the DB loading apparatus in a high temperature furnace.

the force acting on the test specimen. The force induced by the pneumatic loading unit can be calculated from the following equation;

$$F = p A_{eff} - F_{\mu} - F_{s} \tag{2}$$

$$F_s = \frac{c_\delta \, \delta_b}{n_w} \tag{3}$$

where $\mathbf{F} = \text{load}[N]$

 \mathbf{F}_{s} = bellows intrinsic stiffness loss [N]

 \mathbf{F}_{μ} = friction factor [N] \mathbf{p} = pressure [MPa]

 A_{eff} = bellows effective cross-section [m²] c_{a} = axial spring rate for the bellows [N/mm]

 δ_{b} = required axial movement for the whole bellows [mm]

 n_w = number of corrugations

The preliminary calibration tests and calculations for the DB loading unit were performed at a load level of ~500 N and without environmental pressure. The objective of this pre-calibration procedure was to find out how the intrinsic stiffness and the effective cross-section determinations of the DB loading unit should be performed.



4.1.1 Intrinsic stiffness of the DB loading apparatus

In the first calibration test, the pressure loss arising from the two metal bellows' intrinsic stiffness (working and primary bellows) and internal parts friction factor of the pneumatic loading apparatus were determined over the working range (see Figure 6). Note that only the working bellows is pressurized (p1) during the intrinsic stiffness calibration tests. The intrinsic stiffness of the double bellows loading apparatus is decreased as a function of temperature, as shown in Figure 6. The intrinsic stiffness values for the DB loading apparatus were 20.6 bar/mm at 23°C and 17.9 at 550°C.

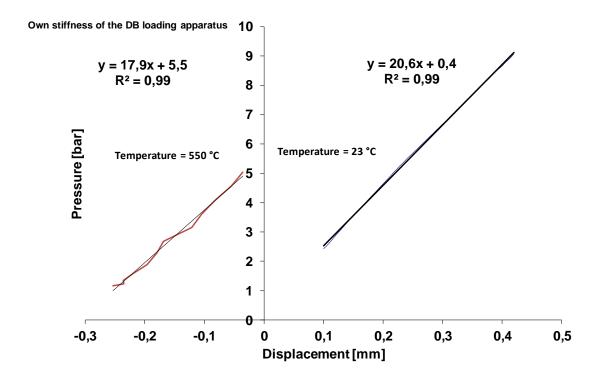


Figure 6. Intrinsic stiffness of the DB loading apparatus at 23°C and 550°C.

4.1.2 The effective cross-section of the secondary bellows

A special calibration furnace was used to calibrate the applied gas pressure in the double bellows (secondary and working bellows) and pressure chamber with the actual load acting on the load cell, see Figure 7. The LVDT sensor is placed in the furnace to measure the compliance of the whole system during the calibration. This is because the intrinsic stiffness of the DB loading unit is relatively high, and thus affects the load calibration accuracy.

The effective cross-section of the secondary bellows was determined by pressurizing working bellows p_1 and chamber pressure p_2 at the same time. The load is calculated by the following way:

$$F = (\Delta p - Own_{st} \times disp)A_{eff_{sec}} - (\Delta p - Own_{st} \times disp)A_{eff_{nri}}$$
(4)

where Δ_p = pressure difference of the working bellows and chamber pressure, \mathbf{F} = measured load level, \mathbf{A}_{effsec} = effective cross-section of the secondary bellows, \mathbf{A}_{effpri} = effective cross-section of the working bellows, \mathbf{Own}_{st} = Own (intrinsic) stiffness and \mathbf{disp} = movement of the secondary and primary bellows.



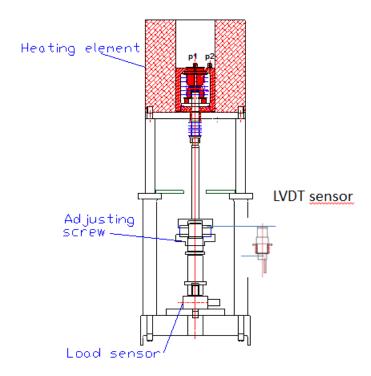


Figure 7. The effective cross-section determination in a high temperature furnace.

The effective cross-section of the working bellows was 360 mm². There is small deviation between calculated and measured load curves. Most probably this is due to the double bellows loading unit's intrinsic stiffness and the internal friction factor of the moving parts. The effective cross section for the secondary bellows was 81 mm².

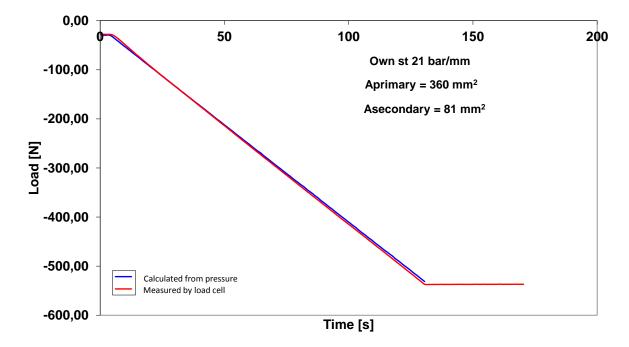


Figure 8. The effective cross-section for the working bellows.



4.1.3 Theoretical tensile load level under SCW environment

The theoretical tensile test load calculations for the DB loading unit are:

$$F_{sec} = (p_3-p_2) A_{effsec} - (F_{own})(5)$$

$$F_{work} = (p_2-p_1) A_{effwork} - (F_{own})$$
(6)

where, \mathbf{F}_{sec} = secondary bellows load [N], \mathbf{p}_3 = environment pressure [bar], \mathbf{F}_{own} = bellows own (intrinsic) stiffness of the working and primary bellows [N/mm], \mathbf{F}_{work} = working bellows load [N], \mathbf{p}_2 = chamber pressure [bar], \mathbf{p}_1 = working bellows pressure [bar] and \mathbf{A}_{eff} = effective cross-section of each of the bellows [mm²].

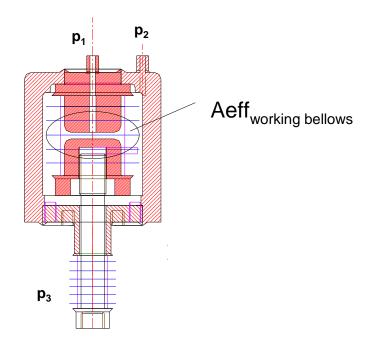


Figure 9. Tensile load generation of the DB loading apparatus.

In the case of a tensile test, $F_{\text{sec}} > F_{\text{work}}$, and, thus, the decrease of F_{work} can determine the needed test load for the specimen. For the test load calculations the effective cross-section of the working bellows and pressure difference between p_1 and p_2 are needed. Based on the test load calculations above and the calibration results from section 4.1.2, Table 1 summarises the needed pressure levels for the primary bellow pressure p_1 and the chamber pressure p_2 so that the needed test load is zero under 250 bar environment pressure. One of the greatest benefits of using the DB loading apparatus is that it can work with relatively low pressure levels under high environmental pressure, as shown in Table 1.

The estimated maximum load to fulfil the requirement for plain strain conditions for the precracked 5 DC(T) type of specimen is calculated according to the ASTM E 399 standard. According to Figure 10, the maximum load level for an AISI 316 stainless steel specimen is around 400-500 N, depending on the initial length of the pre-crack. Table 2 summarises the needed pressure levels for the 5 DC(T) specimen testing under SCW condition. Note that only the primary bellows pressure is changed during the test.





Table 1. Required pressure levels of the double bellows loading apparatus for zero testing load generation in a SCW environment.

Environment				
pressure		250		0,36
Chamber pressure	p2	90		
Primary bellows	p1	126		0,504
LOAD		0 N		
LOAD	Primary load		positive	
LOAD	Primary load Secondary load	1296	positive negative	

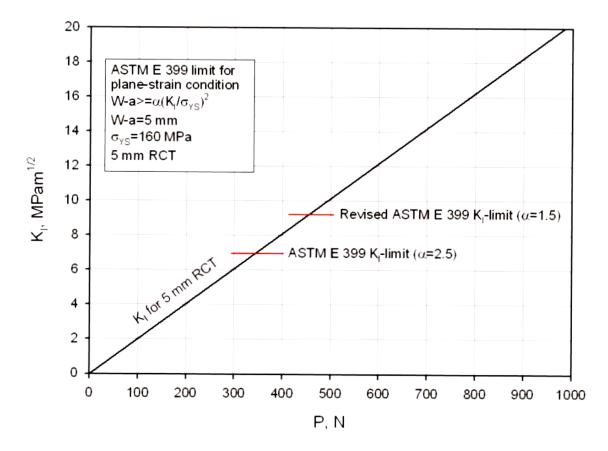


Figure 10. Plain strain condition under different load levels for 5 DC(T) specimen



Table 2. Required pressure levels of the double bellows loading apparatus for the generation of a 468 N testing load in a SCW environment.

Supercritical Water Environment		bar	pressure ratio
pressure		250	0,3
Chamber pressure	p2	90	
Primary bellows	p1	113	0,45
1045		-468 N	
LOAD			
LUAD	Primary load		positive
LOAD	Primary load Secondary load	828	positive negative

5. Programming of the MAC results

As presented above, the intrinsic stiffness and the effective cross sections of the primary and secondary bellows determines the test load together with pressure differences of the pressure boundaries. To avoid the pressure fluctuation of the autoclave, the primary bellows pressure and chamber pressure shall be synchronized together with the autoclave pressure.

The synchronization is performed by connecting the primary, chamber and autoclave pressures together with the ratio command in a MAC (Motion Axis Control) program as shown in Figure 11. When the autoclave pressure is changed the pressure synchronization system can automatically adjust the primary and chamber pressures and thus compensate for the pressure variations between them. The starting and stopping of the tests with the pressure synchronization system is easy to perform, because when the autoclave is pressurized up to 250 bar the primary and chamber bellows pressures can automatically follow the autoclave pressure changes and keep test load at zero value.

Figure 12 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.



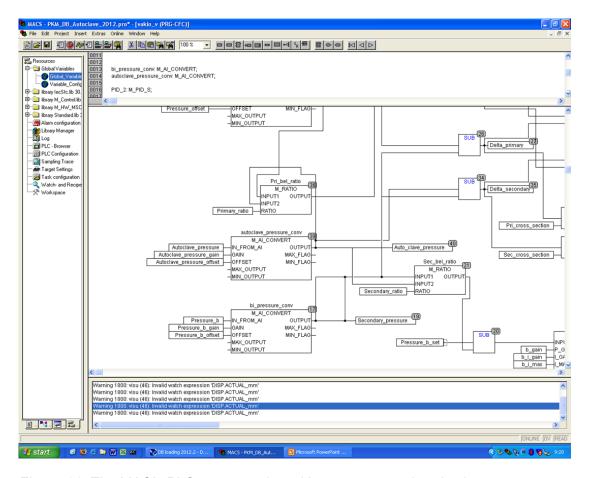


Figure 11. The MAC's PLC programming with pressure synchronization structure.

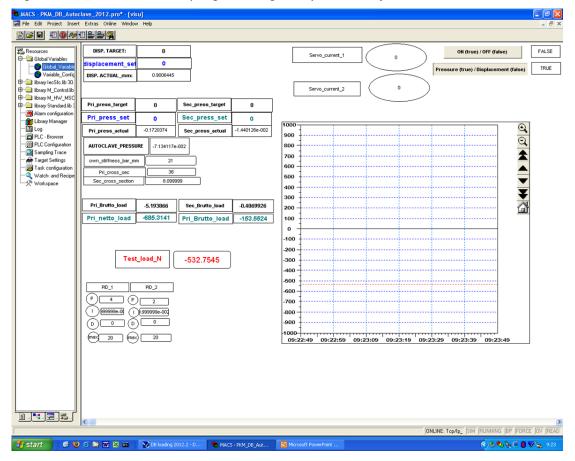


Figure 12. The visualization of the MAC program.



6. Reference tests

The reference tests were performed in air followed by the autoclave tests at LWR and SCW conditions. The material used was an AISI 316 stainless steel, and the specimen was a precracked 5 DC(T) specimen.

6.1 Reference test in air at 550°C

The first test with the pneumatically-powered miniature autoclave testing system was a reference test at 550°C in air. The 5 DC(T) specimen was installed into the double bellows loading frame with the potential drop (PD) wires as shown in Figure 13. The non-contact type of LVDT sensor was used to measure the displacement of the main load post (connected to the top of the secondary bellows). The tensile load for the test specimen was created by changing only the chamber pressure during the test. The primary (working) bellows pressure was constant (7 bar) during the test. Figure 14 shows the heating element and the miniature autoclave with the main feedthrough. The PD wires and LVDT sensor's wires were placed into the main feedthrough with ceramic and graphite insulators. A typical set of raw data from the constant rising load test is shown in Figure 15. The starting point of the specimen loading was easy to determine from the curve (load line slope is changing when load is applied to the specimen).

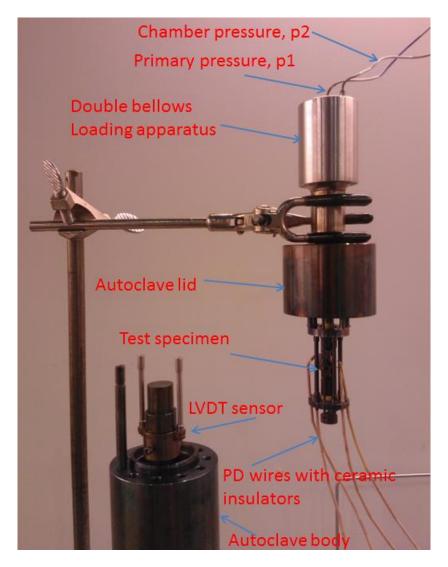


Figure 13. The miniature autoclave material testing system and its main components.



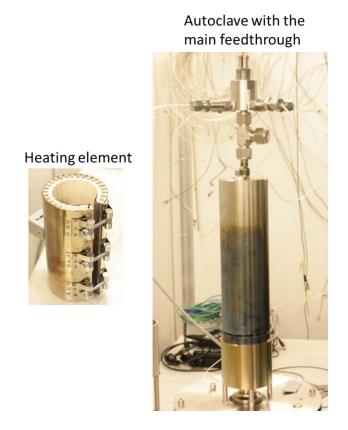


Figure 14. The test set-up for the reference test in high temperature environment.

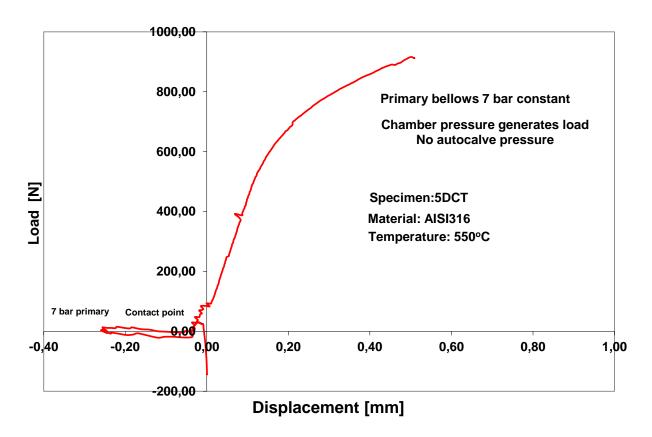


Figure 15. Load as a function of displacement at 550 °C in air for AISI 316 specimen.



6.2 SCW autoclave tests at 550°C

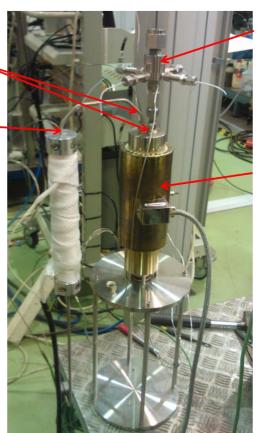
The second test with the pneumatically powered miniature autoclave testing system was a reference test at 550°C in SCW conditions. The autoclave was connected to the recirculation loop with the preheater, as shown in Figure 16. The target water flowing value was ~6 ml/min. The material used was an AISI 316 stainless steel, and the specimen was a pre-cracked 5 DC(T) specimen.

The designed pressure synchronization program worked well during the pressurization of the autoclave and during the test. The primary and secondary pressures followed autoclave pressure variations, as shown in Figure 17. The fluctuation of the autoclave pressure was around ±2 bar during the test. Figure 18 illustrates the pressure variation during the test start up procedure. The primary and secondary bellows pressures can automatically follow the changes of the autoclave pressure, i.e., the test load was constant (and zero) during the pressurizing period of the autoclave.

The miniature autoclave testing system was pressurized up to 250 bar pressure. Unfortunately, the LVDT sensor failed at around 230 bar pressure of the autoclave. Due to this failure the test was stopped.

Thermocouples

Water in with preheater



Feedthrough for the PD and LVDT wires

Autoclave with the heating element

Figure 16. The test set-up for the material testing in SCW condition.



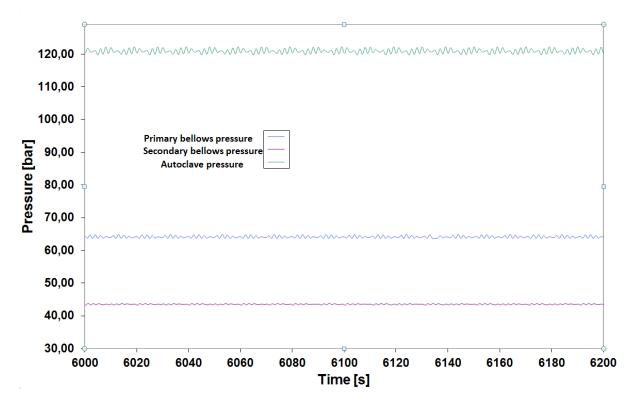


Figure 17. The pressure synchronization system for the double bellows test load generation.

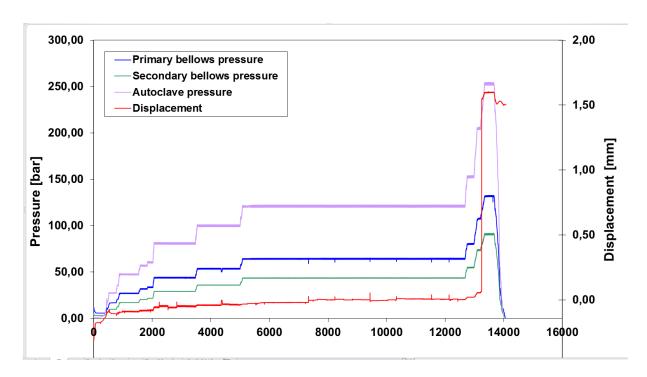


Figure 18. The primary and secondary pressures as a function of autoclave pressure.

6.3 Reference tests at LWR and SCW conditions

The third test with the pneumatically powered miniature autoclave testing system was a reference test at 288°C in LWR and 500°C in SCW conditions. The autoclave was connected to the re-circulation loop with the preheater, as shown in Figure 16.



The target water flowing value was ~16 ml/min which was approximately 2.5 times higher compared to previous test at 550°C in SCW conditions. The dissolved oxygen content was maintained between 150 and 200 ppb in the inlet flow. The material used was an AISI 316 stainless steel, and the specimen was a pre-cracked 5 DC(T) specimen as in the earlier tests.

As mentioned above due the sycronization of the pressures the primary and secondary pressures followed automatically autoclave pressure variations, as shown in Figure 19a. The fluctuation of the autoclave pressure was around ±2 bar during the test and even then the test load accuracy under LWR and SCW coolants was ±2N as shown in Figure 20.

The primary and secondary bellows pressures can automatically follow the changes of the autoclave pressure as well as the fatigue control signal as shown in Figures 19b and 20b.

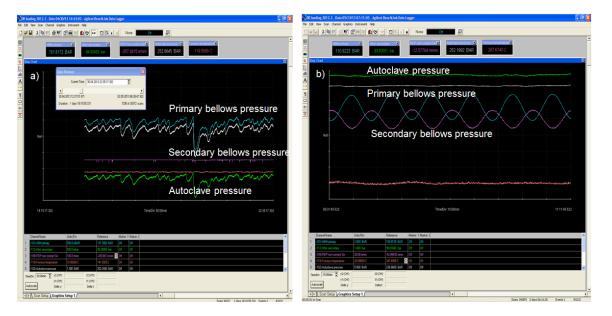


Figure 19. The pressure syncronization during the test, a)constant load and b) cyclic load.

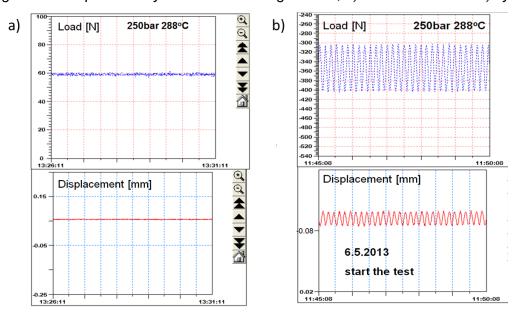


Figure 20.The constant a) and cyclic b) test load and displacement accuracy under LWR coolant.



The maximum load for the AISI 316 stainless steel specimen was 518 N at LWR coolant (and 420 N at SCW coolant) corresponding K-level around 10 MPa(m)^2 for 5mm DC(T) precracked specimen. The test load was increased from the initial maximum value of 400 N by 38 N steps up to 518 N (the maximum peak value) during 7 days cycling as shown in Figure 21. The crack started to growth when the maximum test load reached the value of 518N.

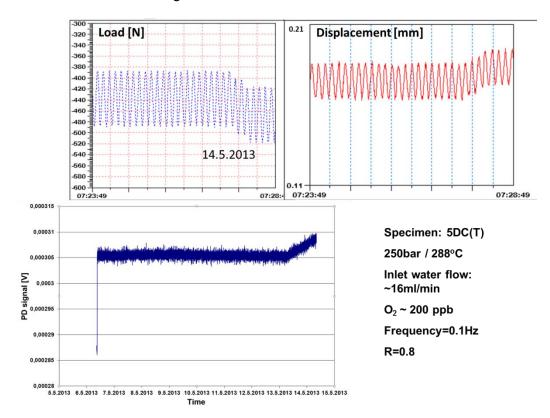


Figure 21. Load, displacement and PD-signal response after the test load was increased by 38 N.

After few days cycling the fatigue frequency was changes from 0.1 Hz to 0.001 Hz during the test. Figure 22 shows how the crack growth rate is decreased as a function of the slower fatigue frequency. Note, that the total displacement of the double bellows loading apparatus was increased as a function of the crack growth rate. This means that the Intrinsic stiffness of the double bellows loading apparatus was decreased the maximum test load level around 30 N.



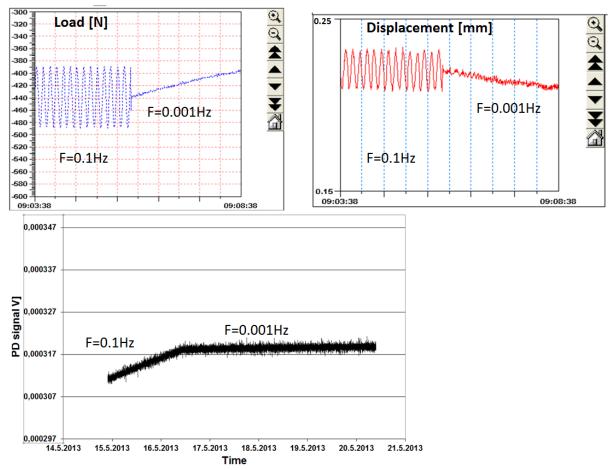


Figure 22. The influence of the slower fatigue frequency for the crack growth rate.

After 15 days crack growth period at LWR coolant condition the autoclave temperature was increased up to 500°C, i.e. SCW condition as shown in Figure 23. The test load was increased by 38 N steps with the several times under SCW condition without clear crack growth observation.

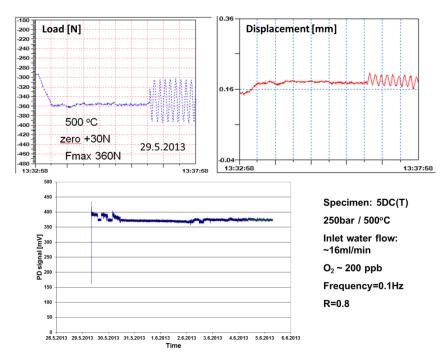


Figure 23. Load, displacement and PD-signals under SCW conditions.



The SEM characterization was performed on the 5DC(T) specimen after the test at LWR (288°C) and SCW (500°C) conditions. The crack tip with the IG and TG crack growths at LWR coolant and interrupted crack growth by plasticity (stripes) at SCW coolant were observed, Figure 24.

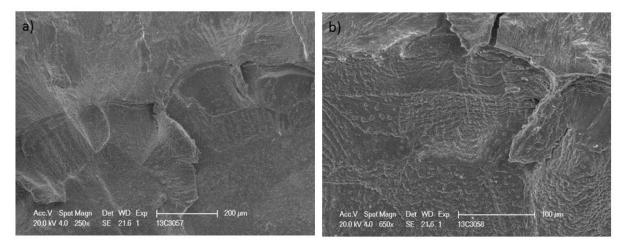


Figure 24. The crack tip with the (a) IG and TG crack growth at LWR coolant and (b) interrupted crack growth by plasticity (stripes) at SCW coolant.

The elastic load lines for the 5 mm DC(T) AISI 316 stainless steel specimen were measured under different coolant temperatures as shown in Figure 25. The test load can be adjusted and controlled with very high accuracy under different coolants by the pneumatically powered testing devices. The maximum load for the AISI 316 specimen was 500 N at LWR coolant (and 420 N at SCW coolant) corresponding K-level around 10 MPa(m)^2 for 5 mm DC(T) pre-cracked specimen. Most probably the deviation of the elastic lines in case of unloading 1 and 2 can occurred from the plasticity of the test specimen's crack tip (during increased the test load by 58 N).

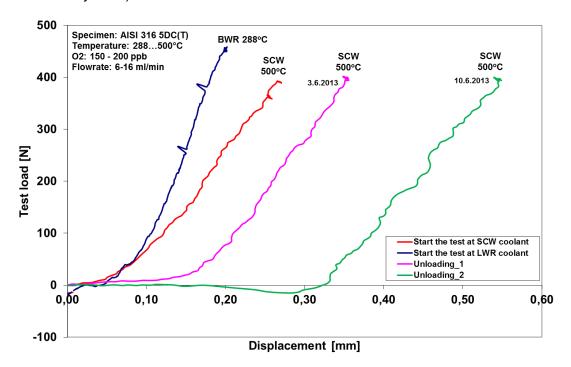


Figure 25. The elastic load lines for the 5 mm DC(T) specimen under high tempeture coolants.



7. Summary

The pneumatic loading technology developed provides important potential benefits and has already been successfully applied for many kinds of materials testing in different test environments. A pneumatic servo-controlled material testing system has been used to perform fracture mechanic, corrosion fatigue, tensile, and electrochemical measurements in gas, high temperature aqueous and irradiation environments. The greatest advantages of this new testing system are increased sensitivity and possibilities to perform material testing inside a reactor core. Furthermore the system can be made compact so that several testing systems can be fitted simultaneously in one test chamber. Thus the total cost of testing is decreased and the reliability of the test result is considerably increased.

- A prototype double bellows (DB) based pneumatic loading unit with miniature size of the autoclave capable of performing loading for tensile type testing in SCW environment has been designed, built and pre-tested.
- The DB loading unit was based on new technology, it operated well, and gave reliable pre-test results. The matching and welding procedures of the main parts for Inconel 625 and Nimonic 80A materials were investigated and performed.
- Main parts and feedthroughs for the miniature autoclave were designed, manufactured and tested.
- The calibrations for the pneumatic loading unit were performed with the following results; intrinsic stiffness of the two bellows was 17.9 bar/mm over the 0.3 mm bellows working range at 550°C, the shape of the intrinsic stiffness curve was linear, the effective cross-section of the secondary bellows was 81 mm² and the effective cross-section of the working bellows was 360 mm².
- The verification tests using developed miniature autoclave material testing system with 5 DC(T) specimens was performed at a temperature of 550°C. The designed MAC PLC programs worked well and reliably during the pressurizing period and testing period of the miniature size of the autoclave.
- The specimen type was 5 DC(T) pre-cracked specimen (AISI 316) which tested under LWR (288°C) and SCW (500°C) coolants.
- Clear crack growth (both type IG and TG) was achieved under LWR coolant at 288°C.
- Very little or no crack growth (only plasticity) was achieved under SCW coolant at 500°C.
- The compactness and versatility of the device makes it particularly amenable to implementation in a hot-cell setting for testing of irradiated materials.

References

- 1. Singh. B.N., Tähtinen. S., Moilanen. P., Jacquet. P., Dekeyser. J. "In-reactor uniaxial tensile testing of pure copper at 90 °C", Journal of Nuclear Materials, 2003. Pp. 12
- 2. Moilanen, Pekka, "Pneumatic servo-controlled material testing device capable of operating at high temperature water and irradiation conditions", 2004, VTT Industrial Systems, Espoo, VTT Publications: 532. ISBN 951-38-6384-0; 951-38-6385-9, Pp. 154
- 3. Moilanen, Pekka, "Preliminary design work for the pneumatic materials testing system capable of working at Super Critical Water environment" VTT Publications: VTT-R-03258-06, Pp. 19
- 4. Moilanen Pekka, "Development of loading device for SCW environment. Construction and calibration", VTT Publications 31.3.2006: VTT-R-04720-08, Pp. 10