SCW facilities Installed in JRC Petten –Past, Present and Future

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Abstract

Currently there are three SCW loops installed and operated in JRC IE Petten for different kinds of tests such as SSRT (Slow Strain Rate Tests), Crack Growth Rate and CER (Contact Electric Resistance). In summer 2010, a new SCW loop, built by Cormet, was installed in JRC IE Petten. The main difference with the first generation of SCW loops manufactured by Cormet is in new two phase heat exchanger and pre-heater system, which allows water flows up to 20 l/h to be reached even at the maximum parameters. The first tests focused on evaluation of chemistry control at the maximum reachable parameters the loop can achieve, in particular to determine, how effectively the oxygen dosed into the system can be controlled. Conclusions comparing tests under the same conditions using the first generation of SCW loops and the new one are presented.

A by-pass system installed in the high pressure part of the loop allows attachment of different loading devices based on pneumatic bellows technology. The first prototype built, which was developed during the common JRC&VTT exploratory research project "Miniature Size Autoclave Bellows", is described in this paper.

Keywords: SCW Circulating loop, SCC, Corrosion, Pneumatic Bellows Bases Loading Device

1. Introduction

The European concept of a supercritical-water-cooled reactor (SCWR) called a High Performance Light Water Reactor (HPLWR), refers to a Light Water Reactor (LWR) in the 1000MWe class with a direct cycle operated at supercritical coolant pressure (p>22.1 MPa). Inside the reactor core, water at a pressure of 25 MPa is heated from 280°C to 500°C. Due to the supercritical coolant, a net plant efficiency of about 44% and, due to much lower mass flow rate, a reduction of component sizes are envisaged, which are expected to reduce the electricity generation and capital costs of this innovative nuclear power plant [1]. To assess the viability of the HPLWR concept and to determine its potential in a future electricity market, the European Project HPLWR-Phase 2 has been co-funded by the European Commission within the 6th Framework Program [2]. Detailed investigation of the most detrimental processes like corrosion and stress corrosion cracking (SCC), embrittlement, creep resistance etc., significantly influencing the behavior of materials in a specific SCW chemistry environment, are of fundamental importance [2].

Since 2005, JRC IE Petten has put a lot of effort in building and installing experimental facilities working under SCW conditions in order to investigate different corrosion processes in SCW. This paper summarizes the experimental facilities built in JRC IE Petten; following that, some recent results are presented, and finally, development work and future plans are briefly introduced.

2. SCW experimental loops with Autoclaves

2.1 SCW loop 1, 2

The first SCW loop with autoclave was installed in 2005 with the primary target of carrying out general corrosion tests of prospective materials evaluated within the HPLWR Phase 2 EUROATOM FP6 project. In 2006, an identical loop with identical SCW autoclave was installed equipped with a tensile loading machine primarily designated for stress corrosion cracking (SCC) susceptibility tests. Some of the results were published by Mathis et al. [3] and Novotny et al. [4] in 2010.

A flow chart of these loops including the autoclave is shown in Figure 1. The loops consist of a low and high pressure part and an autoclave. Chemical parameters pH, conductivity and dissolved oxygen concentration are measured continually in the low-pressure part of the loop at the outlet from the autoclave. The maximum flow rate achieved was limited to 3.5 l/h due to insufficient heating power.

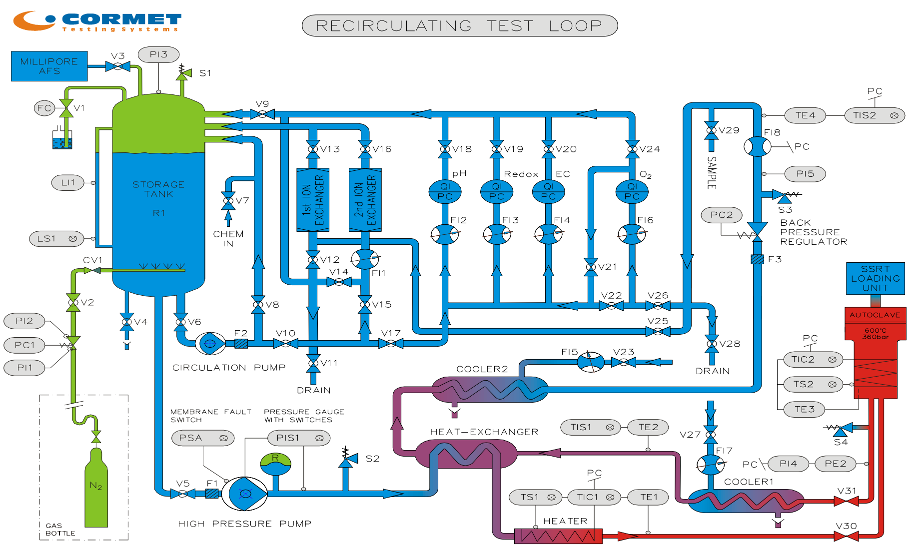
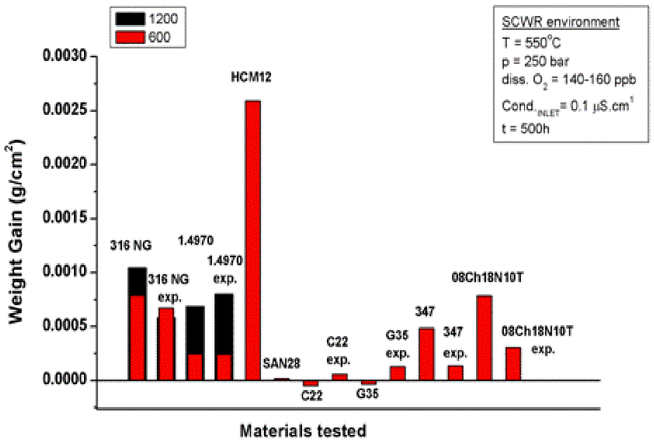
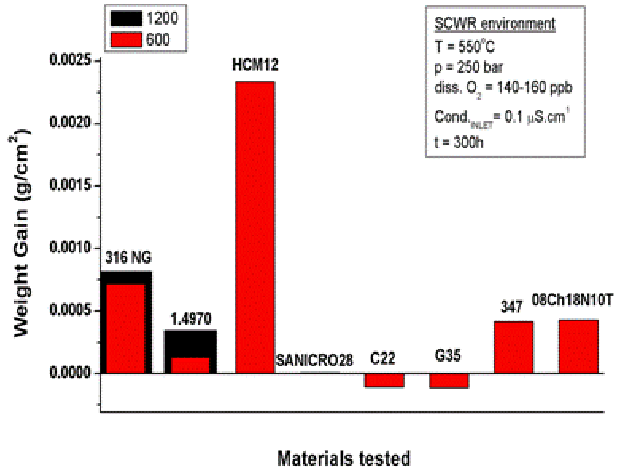


Figure 1 Flow chart of the SCW loop with autoclave 1,2.

Besides the above mentioned SSRT tests, the main emphasis was on general corrosion resistance tests of prospective materials for the European SCWR concept. General corrosion resistance, (the corrosion rate) was studied in frame of HPLWR Phase 2 in SCW at 400 - 650^{o}C using weight change measurements. Oxide thicknesses were determined from sample cross-sections. The compositions of the oxide layers were analyzed using scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS). The layers of selected samples were also analyzed by X-ray diffraction (XRD). The results were published by Pentillä et al. [5] and Novotny et al. [4]. Aside from SSRT tests carried out at later stage of HPLWR Phase 2, additional corrosion tests have been done in order that newly available materials and the influence of parameters such as surface finish or pre-exposure in SCW could be evaluated. Some of unpublished results obtained from these exposures are given in Figure 2a, b.



a)



b)

Figure 2a,b Weight gains/losses of 316 NG, 1.4970, 347 H, HCM12, SAN28, C22, G35 and 08Cr18Ni10Ti specimens after 300 and 500h exposure at 550°C in SCW with 200 ppb O_2 (Exp. – specimens pre-exposed for 300 h under SCW conditions described in Figure 2b).

The materials studied included austenitic stainless steels 316 NG, 347 H, 1.4970, 08Cr18Ni10Ti, Sanicro 28, high chromium martensitic steel HCM12 and Ni-base alloys C22, G35. All the studied alloys were of commercial quality. Some of the specimens (see Figure 2a, b) were exposed for a total of 800 h under the SCW conditions described in Figure 2a,b. 316 NG and 1.4970 specimens were polished to two different surface finishes by using 600 and 1200 grinding paper.

The results in Figure 2a,b shows that the 1.4970, SAN28 and 347 H specimens exhibited the lowest corrosion rate and HCM12, 316 NG and 08Cr18NiTi the highest corrosion rate measured by weight gain, after 300 h and 500 h exposures. Ni-base alloys C22 and G35 showed entirely different behavior compare to the other materials; since the specimens were dissolving in SCW resulting in weight reduction.

Utterly different properties were observed for the tested materials exposed to SCW in two steps: first 300 h exposure, followed by 500 h exposure. 1.4970 pre-exposed specimens exhibited significantly higher corrosion rates compared to as-received ones. On the other hand, 347 H and

08Cr18Ni10Ti pre-exposed specimens showed considerable decrease of corrosion rate, which was rather moderate for 316 NG. C22 and G35 pre-exposed specimens exhibited a complete change of corrosion mechanism compare to as-received ones since observed weight gain was comparable to pre-exposed 347 H (G35) and SAN28 (C22) specimens.

Surface finish had more pronounced influence on corrosion rate values for 1.4970 specimens (see Figure 2a, b) because specimens polished up to 1200 grit exhibited a steep increase of corrosion rate compare to the ones polished to 600 grit. The influence was even more distinct for pre-exposed specimens. The effect of surface finish was rather moderate for 316 NG at the same time not having any effect for previously pre-exposed specimens (see Figure 2a).

2.3 SCW loop 3

In 2008, a new SCW loop was built by CORMET and installed in JRC IE allowing testing of materials at even higher SCW temperature, but more importantly, due to the considerably higher flow rate, better control water chemistry during the tests. The schematic drawing is shown in Figure 3. The main parameters of both JRC IE SCW loops are compared in Table 1.

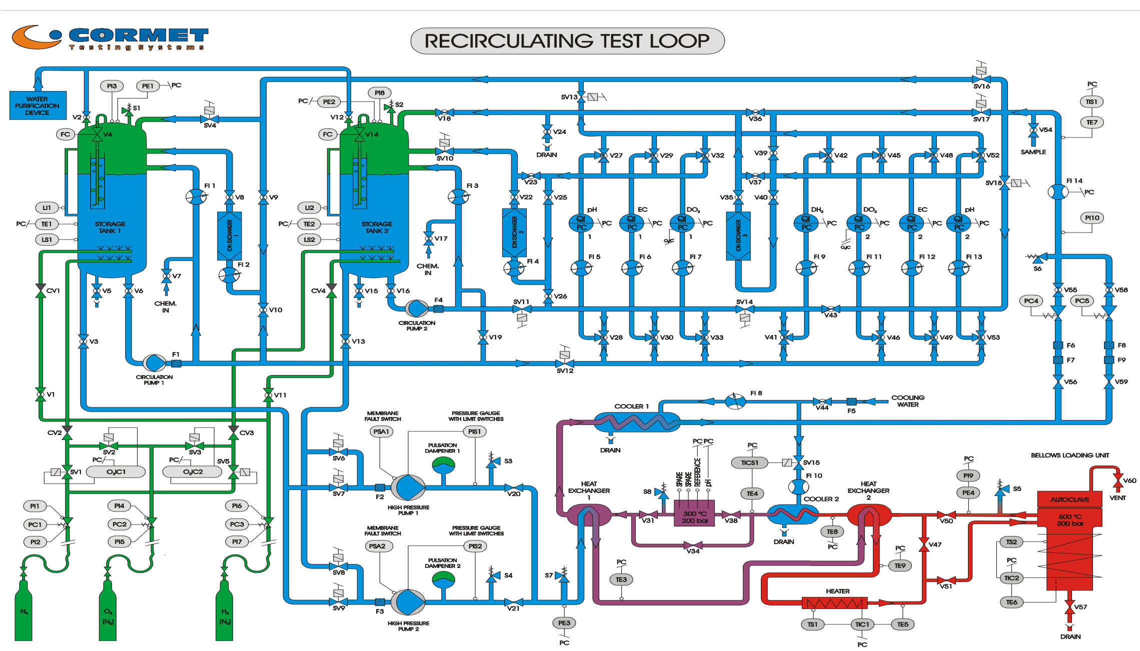


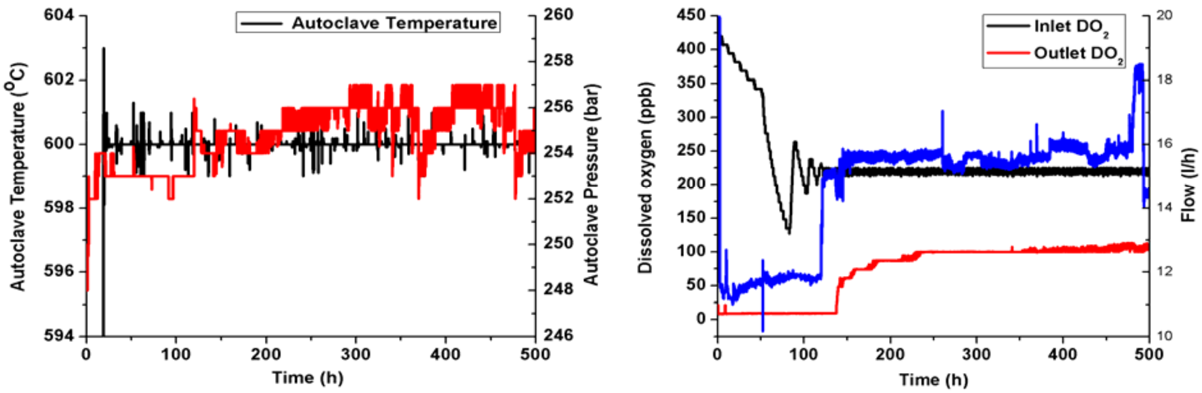
Figure 3 Flow chart of the SCW loop with autoclave 3.

Parameter	**SCW Loop 1,2**	**SCW Loop 3**
Operating pressure [bar]	300	350
Operating temperature [^{o}C]	600	650
Max. flow rate (600^{o}C) [l/h]	3.5	20
Heating	Heat exchanger electrical pre-heater Autoclave heater	Two stage heat exchanger electrical pre-heater Autoclave heater
High pressure pumps (max. flow [l/h])	1xLDC1 Lewa (3.5)	2 x LDC1 Lewa (17)
Autoclave volume [l]	0.8	1.7
Autoclave material	Nimonic 80A	Nimonic 80A
Water chemistry control	pH, cond. and dis. O_2 inlet	pH, cond. and dis. O_2 continually inlet/outlet, H_2

Table 2 Comparison of operating parameters for the SCW loops with autoclaves installed in JRC IE Petten.

Two high pressure pumps were placed in parallel with a view to either guarantee reliable operation during more than 2000 h exposures or provide for independent connection to another autoclave system which could be built in. The new loop also includes a high temperature by-pass section in order to allow an additional autoclave system e.g. a miniautoclave bellows based loading device developed by JRC&VTT to be connected.

The first test using the new SCW loop 3 was in fact a repetition of an exposure carried out at 600 ^{o}C and 250 bar SCW in SCW loop 1 in 2009, however, a flow rate almost six times higher compared to the 3.5 l/h in previous test was used. The temperature and pressure as well as flow rate and dissolved oxygen concentration at the inlet/outlet of the autoclave were continually monitored and recorded. The results of monitoring are given for illustration in Figures 4a and b.



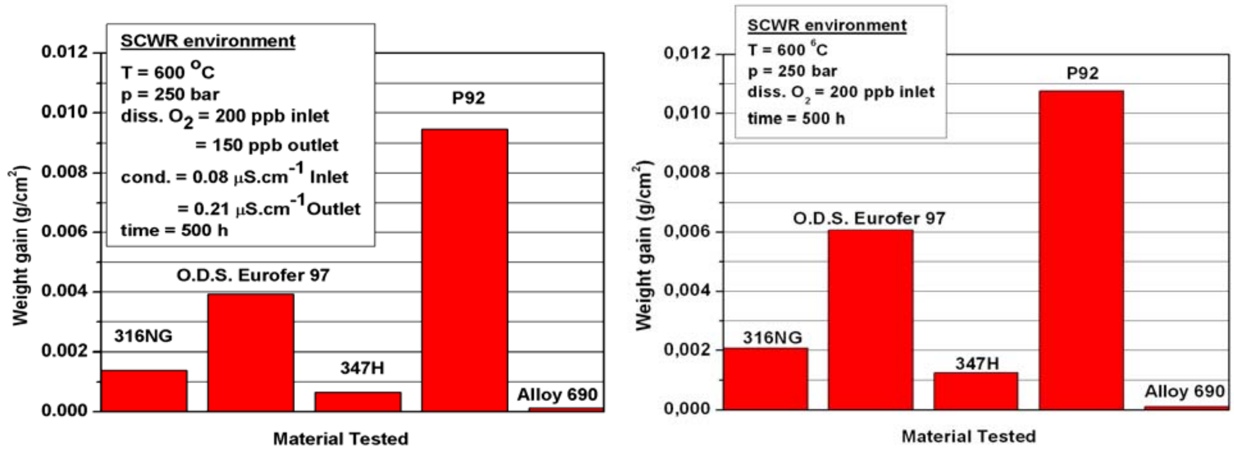
a) b)

Figure 4a, b a) Temperature and pressure recorded during the first test in SCW loop 3. b) Flow rate and dissolved oxygen concentration at the inlet/outlet measured during the first test in SCW loop 3.

The data shown in Figure 4a, b demonstrate good temperature and pressure stability of the SCW loop 3 despite of the much higher flow rate (16 l/h). The main diference between these two SCW loops is related to oxygen concentration monitoring and water chemistry control generally. Dissolved oxygen concentration measured at the autoclave outlet was always much lower than expected even at the maximum flow rate achievable (3.5 l/h), therefore the oxygen concentration dosed into the SCW autoclave was always controlled by the oxygen analyzer connected to autoclave inlet. The data shown in Figure 6 clearly demonstrate that:

1. The increased flow rate from 12 to 16 l/h resulted in an increase in the oxygen concentration measured at the autoclave outlet
2. The oxygen concentration measured at the autoclave outlet slowly increased up to the saturation value around 130 ppb.

After the first corrosion test, the results of weight gain measurement were compared to the ones achieved in SCW loop 1. The results of the calculated weight gain for both exposures in SCW loop 1 and in SCW loop 3 are given in Figure 5a, b.



a) b)

Figure 5a, b Weight gains of 316 NG, 347 H, P92, O.D.S Eurofer 97 and Alloy 690 specimens after 500 h exposure at 600°C in SCW with 200 ppb O_2 obtained for exposures in a) SCW loop 3 and b) SCW loop 1

The studied materials included austenitic stainless steels 316 NG and 347 H, ferritic/martensitic steel P92, oxide dispersion strengthen steel O.D.S. Eurofer 97 and Ni-base Alloy 690. All the studied alloys were of commercial quality. The results in Figure 5a, b show that Alloy 690 and 347H specimens exhibited the lowest weight gain and P92 and O.D.S. Eurofer 97 the highest, which is in agreement with the results published by Penttilä et al. [5]. One would expect the weight gain to rise due to the higher flow rate, however, except for Alloy 690, the weight gaines obtained from exposure in new SCW loop 3 are systematically lower compare to the ones calculated from the results in SCW loop 1. On the other hand higher flow rate can considerable increase susceptibility to layer spallation from the specimen surface and also can increase oxide dissolution due to increased mass transfer. Further detailed SEM and EDX analysis should answer whether this difference is caused by water chemistry or if it is just the scatter of experimental data.

3. SCC crack growth rate tests in SCW

3.1 Pneumatic bellows based loading device installed in SCW autoclave 1

This section is a summary of preliminary results of crack growth rate SCC tests measured in SCW. An essential requirement for crack growth tests is the ability to monitor cracking response on-line in conjunction with the possibility to vary the load that is applied to the samples. SCC crack growth rate tests in SCW pose a very challenging task for obvious reasons. A pneumatic bellows based loading device represents an attractive possibility since these systems have been developed in the past e.g. by VTT [6] or OECD Halden Reactor Project [7] for BWR/PWR applications. Several BWR/PWR systems have already been installed in JRC Petten working up to 340°C and 160 bar high temperature water and results have been published by Novotny et al. [8]. In 2008, JRC Petten installed a pneumatic bellows loading device built by VTT designed for operation in SCW. The basic scheme of the device is shown in Figure 6.

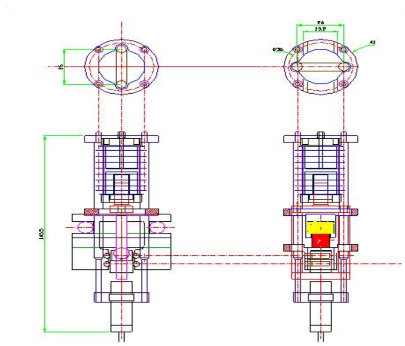


Figure 6 Pneumatic bellows based loading device designed for SCW applications.

The basic description and principle of bellows operation is given in Figure 7. The inner built-in pistons were installed inside the bellows to restrict the bellows to uniaxial movement. The bellow was then closed from the outside environment by welding. Pressurized air, which serves as a driving force for our loading system is dosed via 1.8 mm O.D. stainless steel pressure tube to the bellows. It was decided to design the loading system in the opposite way to that used for BWR and PWR conditions due to the high pressure SCW conditions.

Applied load is adjusted by pressurizing the bellows and autoclave at the same time up to the pre-defined pressure. Zero position, corresponding to zero applied load, is determined by a LVDT placed at a lower temperature in external cell due to the fact that a sufficiently reliable LVDT working in SCW does not exist. The applied load is increased by decreasing the pressure in the bellows, at the same time keeping the autoclave pressure controlled by the back pressure regulator as stable as possible. The accurate level of load applied by the bellow is controlled by a pressure-adjusting loop. The basic scheme of the loop is shown in Figure 8.

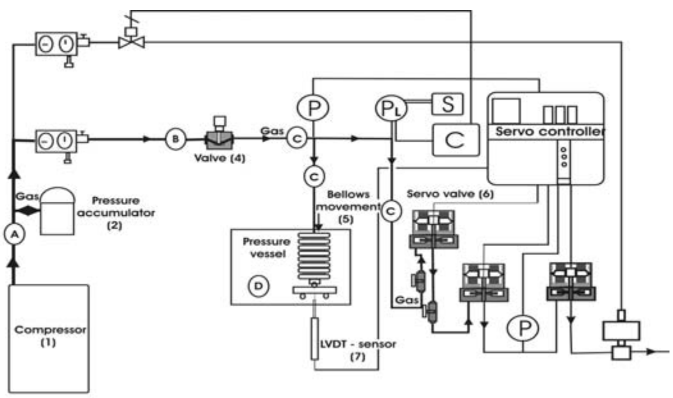


Figure 7 Pressure adjusting loop – basic scheme.

The pressurized air is produced and delivered by a high pressure compressor via the pressure accumulator (part A) to the pressure-adjusting loop. Pressure accumulator was inserted into the loop in order to increase the volume of pressurized air available for the loop and at the same time to dampen the pressure oscillation before the loop inlet. The high pressure reducer (B) and very accurate flowing valve (C) then forms further elements of the loop which should stabilize the air flow to the bellows (D). The air flow is directed into the bellows and parallel to the Moog servo-valves together with pressure transducers as the main controlling elements. In the controlling mode there is constant air flow through the servo-valves. The configuration shown in Figure 8, with two controlling servo-valves and a buffer one, was applied in the crack growth rate SCC tests in SCW.

3.2 Preliminary results

The 08Cr18Ni10Ti type stainless steel (equivalent to AISI 321) used in this set of experiments was supplied by OAO Saint-Petersburg, Russia. The average grain diameter was from 0.031 to 0.015 mm and yield stress $R_{p0.2}$ at 550°C was extrapolated based on tensile test results between 180-200 MPa. The chemical composition is given in Table 3.

Material	C	Si	Mn	S	P	Cr	Ni	Ti	Mo
08Cr18Ni10Ti	0.085	0.45	1.07	0.015	0.011	18.0	10.0	0.64	≤0.1

Table 3 Chemical composition, in mass%, of the 08Cr18Ni10Ti type stainless steel (from mill certificate).

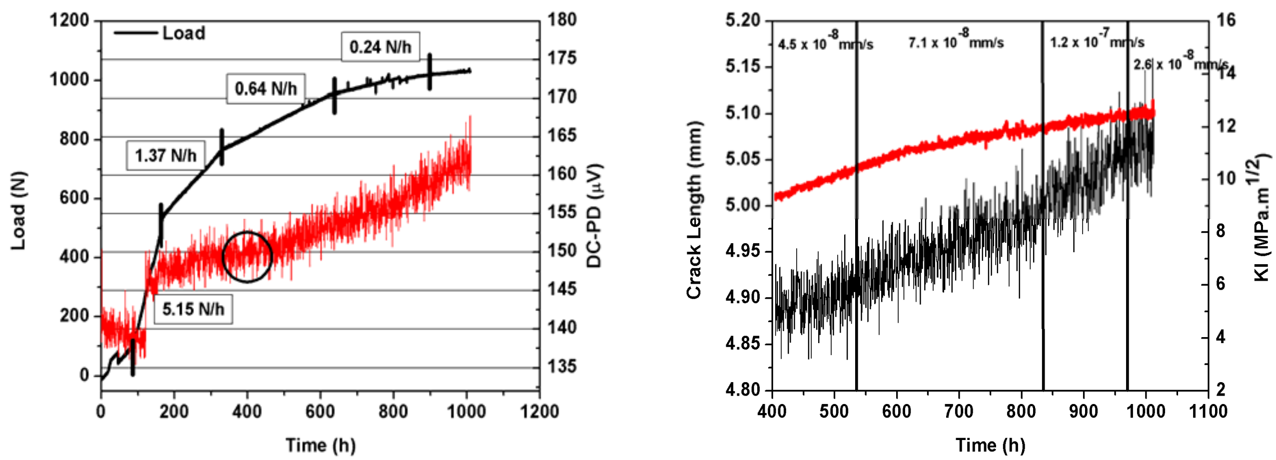
Single Edged Notch Bend SEN(B) specimens were manufactured according to ASTM E 1820-01 standard. The SEN(B) specimens were cut out in T-L orientation. All the SEN(B) specimens were fatigue pre-cracked in air up to a/W = 0.5 following that side grooved to the 2 mm depth. The tests were executed in simulated SCWR conditions; the parameters are described in Table 4. The dissolved oxygen concentration as well as conductivity and pH were measured continually at the inlet.

Temperature [^{o}C]	550
Pressure [bar]	230
Inlet Conductivity [μS.cm^{-1}]	0.09
Outlet Conductivity [μS.cm^{-1}]	0.15 - 0.2
Inlet Dissolved O_2 [ppb]	0, 200, 2000; 8000

Table 4 Water chemistry parameters applied in crack growth rate SCC tests in SCW.

The first SCC tests were conducted by a very slow rising load test method from two reasons:

1. The LVDT sensor placed in an external cell does not allow direct control of the test.
2. Pre-cracking in the environment by fatigue to transition the crack morphology could be critical for the lifetime of the bellows loading device. Contrary to common practice, specimens were loaded by very slow rising load as presented in Figures 8a, b.

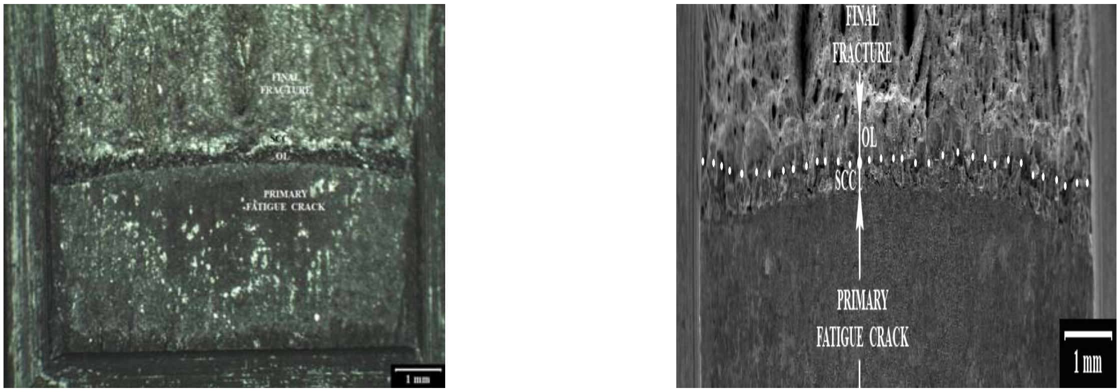


a) b)

Figure 8a,b a) Loading history, PD signal vs. time and b) crack growth rates, K_I vs. time plots obtained for specimen AC23 exposed in the very slow rising load test executed while exposed in 550°C, 250 bar and 8000 ppb of dosed oxygen SCW test.

The loading rate was gradually decreased during the test (see Figure 9a) towards zero, adjusted in the last approximately 200 h long period. The results of PD measurement presented in Figure 9a show that crack growth initiated approximately at 400 h followed by linear crack growth. The accuracy (± 2.5 µV) of PD measurement was acceptable considering the test conditions. The data in Figure 9b show that starting from approximately 700 h, crack growth rate was almost constant and loading rate independent. However, the final switch to constant applied load caused quite a significant decrease of crack growth rate down to 2.6 x 10^{-8} mm/s. A comparison was made with the crack growth rates results published by Karlsen et al. [9] for AISI 347 CT specimens exposed in BWR SCC tests due to the absence of similar data in the literature. The comparison showed that the crack growth rate 2.6 x 10^{-8} mm/s measured at constant load ($K_I \approx$ 13 MPa.m$^{1/2}$) is almost one order of magnitude lower than values measured by Karlsen et al. [9] for the same stress intensity factor under BWR conditions.

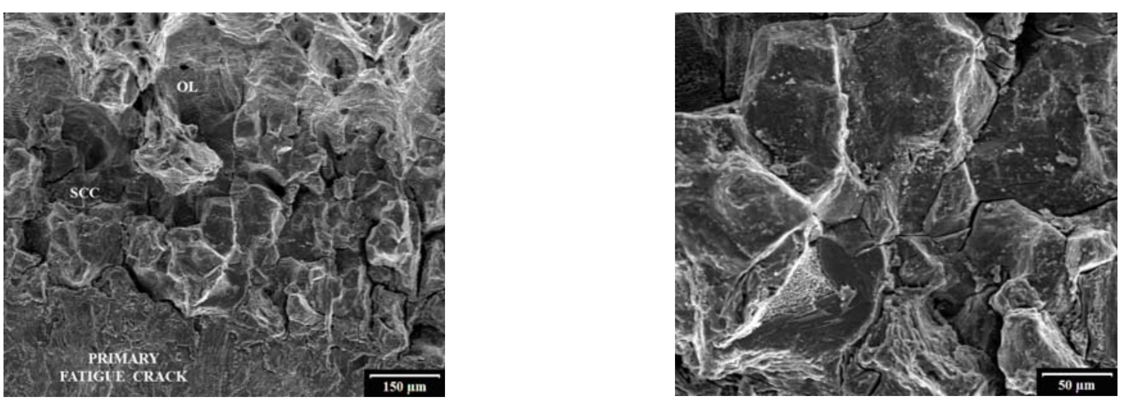
Preliminary results of fractographic analysis are presented in Figure 9a, b and 10a, b. Macroscopic analysis of the fracture surface area made by optical stereoscope and SEM is given in Figures 9a, b.



a) b)

Figure 9 Fracture surface of AC23 specimen exposed to very slow rising load test executed under SCW (550°C, 250 bar and 8000 ppb of dosed oxygen) a) before b) after using the cleaning method developed at CTU FNSPE Department of Materials in Prague [10].

The fracture surface was covered with a black Fe oxide layer (top layer probably magnetite Fe_3O_4) therefore, the fracture surface had to be cleaned by the special method developed at CTU FNSPE Department of Materials in Prague for analysis of AISI 304L specimens exposed in corrosion fatigue tests in PWR primary water [10]. Four fractographically different areas can be distinguished on the fracture surface: Primary crack developed by fatigue pre-cycling in air followed by an approximately 300 μm wide area of intergranular stress corrosion cracking IGSCC passing in the area of overload and final fracture. Detailed SEM analysis of the fracture surface morphology is presented in Figures 10a and b.



a) b)

Figure 10a, b Micromorphology corresponding to mechanism of Intergranular Stress Corrosion Cracking for specimen AC23 exposed to very slow rising load test executed under SCW (550^{o}C, 250 bar and 8000 ppb of dosed oxygen).

The detailed analysis of fracture surface morphology in Figure 10a, b showed almost purely intergranular IGSCC. Preliminary results show that despite of the different chemical and physical properties of SCW and BWR the mechanism of the IGSCC crack growth in SCW can not be fundamentally different from that measured for the similar material (AISI 347 Nb stabilized stainless steels) under BWR conditions e.g. by Karlsen et al. [9].

4. Conclusions and future plans

The newly installed SCW loop demonstrated much better control of water chemistry in the course of SCW experiments than the previous loops. On the other hand, a comparison of the results achieved from exposures at the same SCW conditions in SCW loops 1 and 3 did not show significantly different results.

In order to understand the fundamentals of oxidation behaviour of the candidate materials under high temperature and high pressure conditions, a combination of in-situ investigations and ex-situ analytical studies of the oxide film forming processes is needed. The CER system (Contact Electric Resistance) installed in JRC IE Petten enables in-situ monitoring of the kinetics of oxide growth in high temperature and pressure SCW. Future experiments will also be focused on characterization of corrosion mechanism in SCW for two O.D.S steels PM2000 and MA956 as the most prospective cladding materials.

Preliminary crack growth rate SCC tests demonstrated sufficient reliability and accuracy of the newly developed pneumatic bellows based loading device in SCW. Subsequent work will concentrate on development of an LVDT working in SCW and further improvement of the loading device for possible in-pile IASCC application. The key factors and requirements for in-core testing are the size of a load frame and its pipe length, accuracy of DCPD, LVDT and load measuring system, therefore, a new miniature autoclave double bellows concept was introduced. The first prototype applying load by using double bellows technology has already been tested in JRC IE Petten laboratories.

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