FACILITY FOR THE STUDY OF ENVIRONMENTALLY ASSISTED CRACKING

Mônica Maria Abreu Mendonça Schvartzman
Centro de Desenvolvimento da Tecnologia Nuclear – CDTN; R. Prof. Mario Werneck, s/nº, 30123-970 Pampulha, Belo Horizonte, MG, Brasil
monicas@cdtn.br

Célia de Figueiredo Cordeiro Neves
Centro de Desenvolvimento da Tecnologia Nuclear – CDTN; R. Prof. Mario Werneck, s/nº, 30123-970 Pampulha, Belo Horizonte, MG, Brasil
caf@cdtn.br

Wagner Reis da Costa Campos
Centro de Desenvolvimento da Tecnologia Nuclear – CDTN; R. Prof. Mario Werneck, s/nº, 30123-970 Pampulha, Belo Horizonte, MG, Brasil
wrcc@cdtn.br

Pedro Augusto Pinheiro Moreira.
Centro de Desenvolvimento da Tecnologia Nuclear – CDTN; R. Prof. Mario Werneck, s/nº, 30123-970 Pampulha, Belo Horizonte, MG, Brasil
palm@cdtn.br

Abstract: The study of environmentally assisted cracking (EAC) involves the consideration and evaluation of the inherent compatibility between a material and the environment under conditions of either applied or residual stresses. EAC is a critical problem because equipment, components and structure are subject to the influence of mechanical stresses, water environment of different composition, temperature and different material history. Testing for resistance to EAC is one of the most effective ways to determine the interrelationships among these variables on the process of EAC. Up to now, several experimental techniques have been developed worldwide, which address different aspects of environment caused damage. Slow strain rate tests, constant loading of CT specimens and rising displacement tests are typical examples of tests which are used for the estimation of parameters of stress corrosion cracking. To assess the initiation stages and kinetics of crack growth, the testing facility should allow active loading of specimens in the environment which should be close to the actual operation conditions of assessed component. Under cooperation of CDTN/CNEN and International Atomic Energy Agency a testing system was developed by Nuclear Research Institute Rez (NRI), Czech Republic, and it is being used since the end of 2002 for the environmentally assisted cracking testing at CDTN/CNEN. The facility allows high temperature testing in well-defined Light Water Reactor (LWR) water chemistry using constant load, slow strain rate and rising displacement techniques. The facility consists of autoclave and refreshing water loop enabling testing at temperatures up to 330°C. Active loading system allows the maximum load on a specimen as high as 60 kN. The potential drop measurement is used to determine the instant crack length and its growth rate. The paper presents the facility and describes the potential drop technique, that is one of the most used techniques to monitor crack growth in specimens under corrosive environments. It also shows the results of an initial test on unsensitized AISI 316L stainless steel, carried out to check the working of the facility and to test the methodology of measurement of crack growth rate.

Keywords: Facility for stress corrosion cracking, reversed direct current potential drop method, crack growth rate, fracture.

1. Introduction

Environmentally Assisted Cracking (EAC) encompasses Stress Corrosion Cracking (SCC), Corrosion Fatigue (CF), Hydrogen Embrittlement (HE), and Creep-Crack Growth (CCG). EAC of materials used in industrial components is a potentially critical issue concerning safety of plant operation and plant life extension. The EAC behavior is determined by material, mechanical and environmental factors which affect individually or have synergetic effects in cracking response.

Stress corrosion cracking (SCC) is one of the most subtle corrosion-related causes of premature fracture of structures. SCC is a term used to describe service failures in engineering materials that occur by slow, environmentally induced crack propagation. The observed crack propagation is the result of the combined and synergistic interaction of mechanical stress and corrosion reactions. Susceptibility to SCC is not a property of a material in the same sense as a mechanical property, but is nonetheless a very important factor in determining the serviceability of metallic materials. (Jones, 1992).

Several parameters are known to influence the rate of crack growth in aqueous solutions, these include: material type, process history, product form, active cracking mechanism(s), loading configuration and geometry and service environment conditions.

Two basic electrochemical corrosion reactions, anodic and cathodic, dominate the SCC process in conjunction with mechanical tensile stress. The chemical composition of the environment, including pH and the presence of hydrogen

recombination poisons that affect the cathode reaction product, and the composition of the metal determine which of the two partial reactions is dominant. In most cases, SCC has been associated with the process of active path corrosion whereby the corrosive attack or anodic dissolution initiates at specific localized sites and is focused along specific paths within the material (Jones, 1992).

SCC failures frequently are difficult to reproduce and study in the laboratory but testing for resistance to SCC is one of the most effective ways to determine the interrelation of material, environmental and mechanical variables on the cracking process. The aim of SCC testing is usually to provide information more quickly than can be obtained from service experience, at the same time attempting predict service behavior. It is important, however, that this procedure be controlled in such a way that the details of the failure mechanism are not changed.

Stress corrosion cracking is one of most severe damage mechanisms influencing the lifetime of components in the operation of nuclear power plants. When a pressurized water reactor (PWR) has been running over a long period, SCC might occur in the components and structure due to the effect of the pressurized high temperature water environment. Consequently fundamental understanding of the mechanism and countermeasures against SCC are the important issues with regard to the long-term operation of the PWR. To assess the initiation stages and kinetics of crack growth as the main parameters coming to residual lifetime determination it was acquired in 2002, under cooperation of CDTN/CNEN and International Atomic Energy Agency, a testing facility developed by Nuclear Research Institute Řež (NRI), Czech Republic, that will be used for the assessment of degradation of selected materials of PWR nuclear power plant (Ruscak, 1998).

The facility is used to carry out stress corrosion tests under simulated PWR environment, where temperature, water pressure and chemistry are controlled, which are considered the most important factors in SCC. Test specimens are subjected to constant or varying loads and displacements, depending on test objectives.

The paper gives technical description of the facility and presents potential drop technique that is used to determine the instant crack length and its growth rate under corrosive environments. Over the last years the potential drop technique has gained increasingly wide acceptance in fracture research as one of the most accurate and efficient methods for crack length determinations in a wide range of testing environments. It also presents the results of an initial test on unsensitized AISI 316L stainless steel, carried out to check the working of the facility and to test the methodology of measurement of crack growth rate.

2. Facility for stress corrosion cracking testing

The facility has been developed as an universal testing system for all principle kinds of stress corrosion tests according to ASTM, ISO and DIN standards. The facility consists essentially of the following components (Fig. (1)): autoclave; water loop; RDCPD (reversed DC potential drop technique) unit.

Figure 1 - Facility for stress corrosion cracking testing.

The testing facility is a refreshing water loop autoclave with active servo hydraulic loading controlled by displacement or load. Measurements of load, displacement, crack growth, water chemistry (dissolved oxygen concentration, condutivity), thermohydraulic and electrochemical parameters are obtained during the test and stored in computer. Constant load, cyclic corrosion fatigue and slow strain rate tests with fatigue pre-cracked, notched or smooth specimens can be performed over a wide range of loading and environmental parameters. Corrosion-assisted crack growth is monitored on-line by a reversed direct-current potential-drop system (Vomáčka, 2002).

2.1. Autoclave and its measurement and control system

The autoclave is a stainless steel pressure vessel used to keep the testing environment around the tested specimen and to load the specimen which can be tensile or CT types. A pull rod is connected with external piston which is controlled by a servo valve. Several outputs and inputs in the autoclave wall are used for the water loop, RDCPD instrumentation, thermocouple, pressure gauge and high temperature electrode. It is heated by an electric furnace in stainless steel continuously controlled by PID system.

The hydraulic oil pump gives high pressure to drive both water loop pump and loading piston in autoclave. (Vomáčka. 2002).

The control system is responsible for controlling the pull rod position, defining the displacement range, the load-displacement regime, the water pump and the autoclave heating. Data acquisition and its graphic representation are provided using an application software created in LabVIEW® environment. Three screens can be chosen from program menu:

- main screen: used to initiate and to finish the test;
- screen of the parameters adjustment (Fig (2));
- measuring screen: continuous recording of the data being measured. (Fig. (3)).

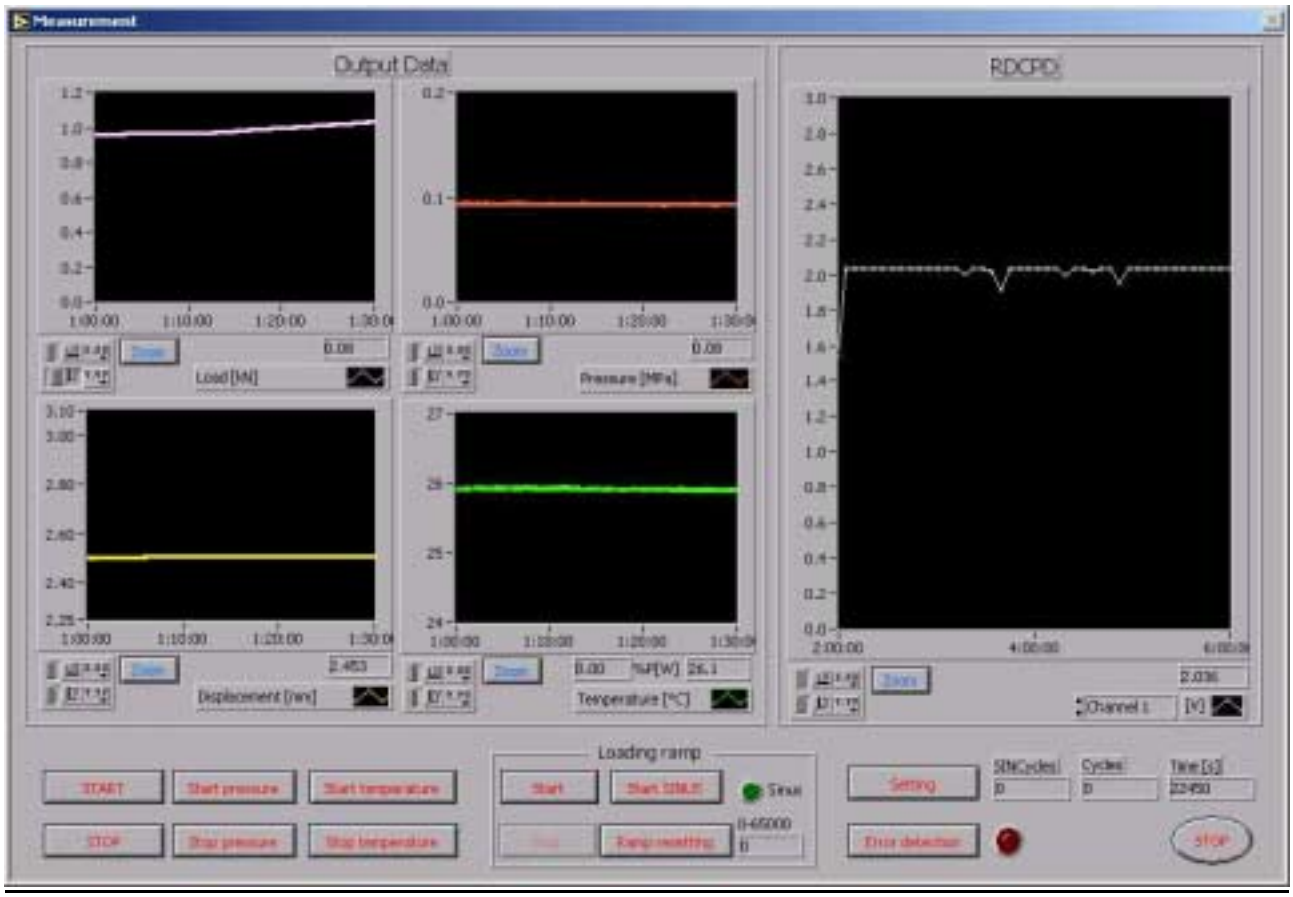


Figure 2. Screen of the parameters adjustment.

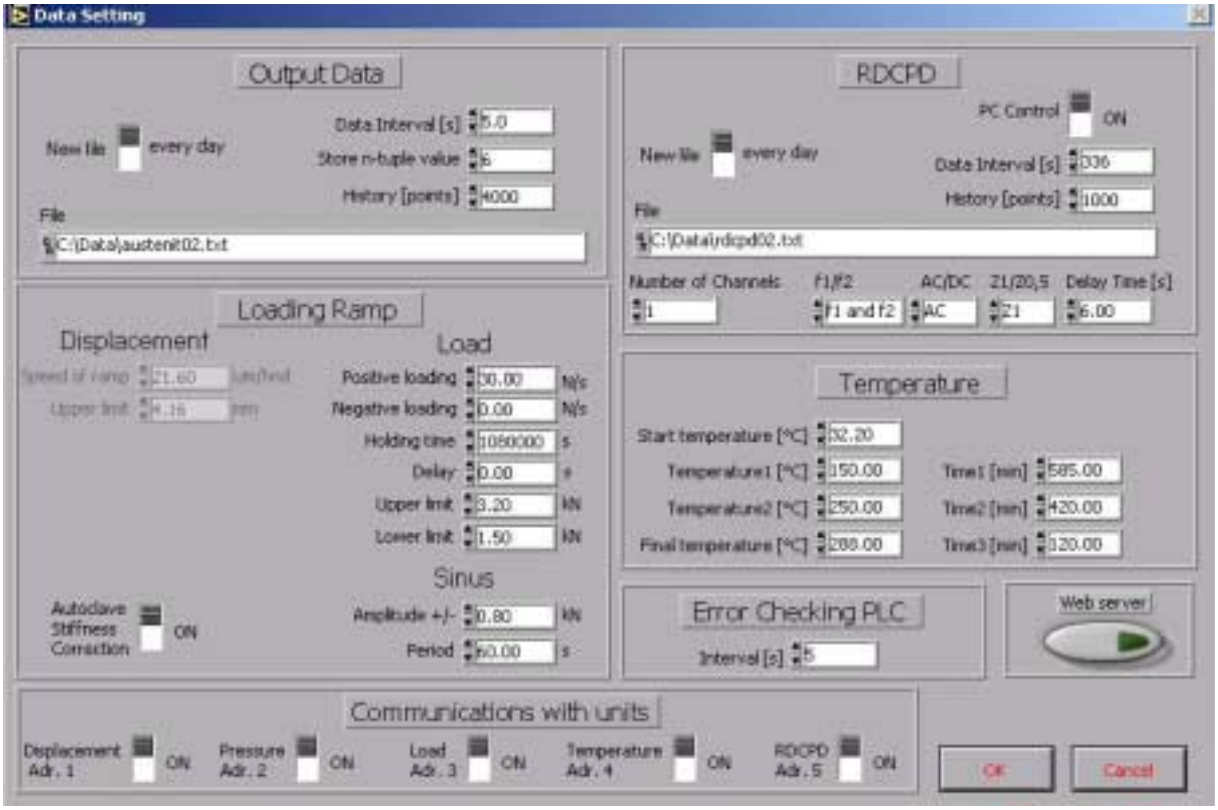


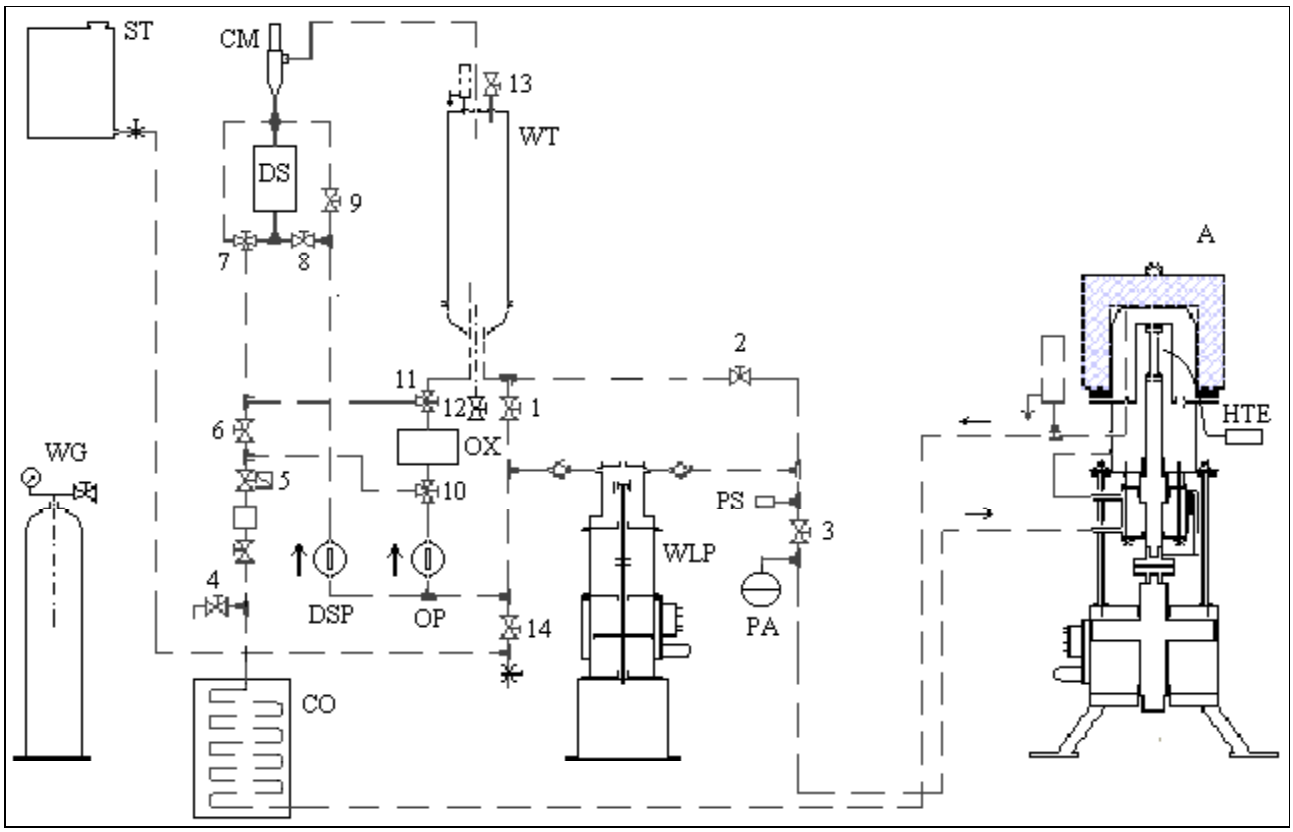
Figure 3. Measuring screen.

2.2. Water loop

The water loop of autoclave is designed to feed the equipment with working fluid and to treat its chemical parameters. Water loop block diagram is given in Fig. (4). There were used stainless steel pressure pipes, unions and valves to distribute the fluid. Fluid distribution system consists of three independent circulating loops equipped with own pumps:

- Autoclave feeding loop – working tank, water loop main pump, autoclave, cooler, control needle valve, working tank
- Oxymeter loop – working tank, oxymeter pump, oxymeter, working tank
- Fluid chemical treatment loop (demineralisation station) – working tank, demineralisation station pump, ion exchanger (mixed bed), working tank

Desired water chemistry is achieved in several steps of operation. First, the working tank should be filled through the ion exchange filter. This resource of clean water is then used to clean out all of the system including the autoclave and specimen. Water is returned to the working tank through the filter again. Once the conductivity measured on the output is acceptable, an experiment can start. During the experiment, circulating water is again cleaned before entering the working tank. Before or during experiment, these changes of water chemistry can be done: by bubbling of nitrogen or oxygen in the working tank and keeping an overpressure of gas mixture, an enrichment of oxygen, or removal of oxygen from water can be reached. According to the experimental plan other species can be added to the working tank before the experiment.



A - Autoclave
CM - Conductivity meter
CO - Cooler
DS - Demineralisation station
OX - Oxymeter
OP - Oxymeter circulating pump
EP - Exchange filter circulating pump
HTE - High temperature electrode
PA - Pressure accumulator
OS - Pressure sensor
ST - Service tank
WG - Working gas
WLP - Water loop pump
WT - Working tank

Figure 4. Water loop block diagram.

2.3. Technical parameters of the system

The main technical parameters of the facility are listed in Tab. (1):

Table 1 - Technical parameters of the facility for stress corrosion cracking.

Item	**Parameter**	**Limit value(s)**
Autoclave	Volume Max. temperature Max. pressure	1.5 ℓ 325ºC 12.5 MPa
Crack growth measurement	RDCPD	0 – 100 mm
Water loop	Max. flow rate	2 $\ell.h^{-1}$
Loading system	Max. load Max. displacement Min. displacement rate Max. displacement rate	60 kN 50 mm 10^{-10} m.s^{-1} 10^{-4} m.s^{-1}
Water cleaning plant	Max. output conductivity Oxygen content	0.06 µS.cm^{-1} 0.5 – 15,000 ppb O_2

3. RDCPD

The potential drop method relies on the fact that the electrical potential distribution in the vicinity of a crack in a current carrying body changes with crack growth. The magnitude of the change depends directly on the size and shape of the crack.

During the crack growth monitoring, a constant current passes through a cracked test specimen and the change in the electrical potential across the crack is measured as it propagates. With increasing crack length, the uncracked cross-sectional area of the test specimen decreases, its electrical resistance increases and thus the potential difference between the two points spanning the crack rises. By monitoring this potential increase and comparing it with some reference potential, measured elsewhere on the test piece preferably in a region unaffected by crack growth, the crack length (a) or the crack length-to-specimen width ratio (a/W) may be determined through the use of the relevant calibration curve for the particular test piece geometry concerned.

Both direct current (DC) and alternating current (AC) have been used to measure crack size in test specimens. The reversed DC potential drop technique (RDCPD) utilizes the rectified square wave alternating current. This method has an advantage against the classic direct-current method in the smaller input current. In comparison with the classic alternating-current method, there is a possibility to eliminate the skin effect interference with this method. The frequency of switching on the single polarity current signal is variable. It ranges from 12 to 960 Hz and can be adjusted for materials sensitive to skin effects, like ferromagnetic materials.

Several fracture mechanic specimens configurations are suited to the potential drop method. In measuring the crack length in a CT specimen, it is used an arrangement of cables and measuring electrodes shown in the Fig. 4. Two conductors serve as an input of the electric current I and two pairs of potential electrodes (Ni wires), welded to opposite corners of the crack mouth, are used for the electric potential pickup U (M_1) and U (M_2). All the wires are connected by spot welding.

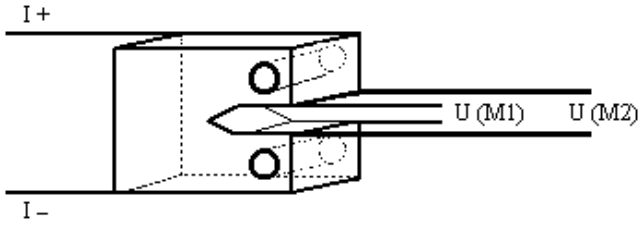


Figure 4. Scheme of CT specimen instrumentation.

The two pairs of potential electrodes give two values of potential for the crack length measurements. The best results are achieved using an average of eight potential values recorded at two different frequencies and in each case at two input current polarities [Řež, s/n/t]:

$$U = \begin{bmatrix} U(M_1 f_1 +) + U(M_1 f_1 -) + U(M_2 f_1 +) + U(M_2 f_1 -) + \\ + U(M_1 f_2 +) + U(M_1 f_2 -) + U(M_2 f_2 +) + U(M_2 f_2 -) \end{bmatrix} / 8 \quad (1)$$

The crack length calibration relationship has to be fit for each specimen and material type as a function f:

$$a/W = f(U/U_0) \quad (2)$$

where U e U_0 are the potential values. U_0 is a normalized constant that depends on specimen and material types. It is calculated from initial values of the crack length a_i and the potential U_i as:

$$U_0 = U_i / g(a_i/W) \quad (3)$$

The functions $f(U/U_0)$ and $g(a_i/W)$ are fourth power functions with experimentally fit parameters [Řež, s/n/t, Brožová, 1996]:

$$f(U/U_0) = \sum_{k=0}^{4} \alpha_k (U/U_0)^k, \qquad g(a_i/W) = \sum_{k=0}^{4} \beta_k (a/W)^k \tag{4}$$

where

α_0 =	0.0194		β_0 =	- 0.327
α_1 =	0.138		β_1 =	7.19
α_2 =	0.335	and	β_2 =	- 16.80
α_3 =	- 0.187		β_3 =	25.32
α_4 =	0.032		β_4 =	- 12.46

The reversed potential drop method has been used for the SCC crack length measurements in hot pressurized water of PWR and BWR environments. Accuracy of measurements of the crack length depends on the stability of the measurement condition, especially temperature and force. In optimized conditions, the expected accuracy is 20 μm for ferritic steel and 10 μm for austenitic steel.

4. Stress Corrosion Testing

It can be said that there is no single perfect testing technique for the evaluation of EAC. However, the evaluation of materials for EAC typically involves the use of the specimen and technique that takes into account as many necessary factors as possible for the particular material and environment under consideration. In some cases, this may mean the use of

- More than one type of test specimen;
- Various alternative configurations of the same specimen;
- Alternative test techniques with the same specimen (e.g. constant load, constant displacement and slow strain rate).

Tests on statically loaded smooth specimens are usually conducted at various fixed stress levels, and the time to failure of the sample in the environment is measured. These experiments can be used to determine the maximum stress that can be applied in service without SCC failure, to determine an inspection interval to confirm the absence of SCC crack growth, or to evaluate the influence of metallurgical and environmental changes on SCC.

Tests on statically loaded precracked specimens are usually conducted with either a constant load or with a fixed crack opening displacement, and the actual rate or velocity of crack propagation, da/dt, is measured. (Jones, 1992). The magnitude of the stress distribution at the crack tip is quantified by the stress-intensity factor, K, for the specific crack and loading geometry. As result, the crack-propagation rate, da/dt, is ploted versus K.

Slow strain rate (SSR) testing is a tensile test conducted at extremely low crosshead speeds (10^{-4} to 10^{-7} cm.s^{-1}). In this case, the constant load has been replaced by a slow extension of the specimen until failure. The main benefit of the SSR test is that it allows the evaluation of the effect of metallurgical variables such as alloy composition, heat treatment and processing and/or environmental parameters (e.g., aeration, concentration, inhibition, etc.) in a relatively short period of testing. Evaluation for susceptibility to EAC is normally obtained through the comparison of the results of tests conducted in a corrosive environment vs. corresponding data obtained in an inert environment. In most cases these tests are conducted in air. Direct examination of the specimen gage section for EAC and documentation of fracture mode are also important to a full interpretation of the SSR test results. The SSR test results that are used include time to failure, plastic elongation to failure, reduction in area, ultimate tensile strength, and load at fracture. These data are usually presented in terms of their ratios vs the corresponding value from a test conducted in an inert environment.

Most of all, it is important to provide a link between the results of laboratory evaluations and real-world service applications. This is often developed through studies involving:

- Integrated laboratory and field or in-plant tests;
- Correlation of laboratory data with service experience;
- Reviews of published literature on the service performance of similar materials.

5. Component lifetime management: inputs from the test facility

The lifetime of components working in water environment should be evaluated regarding the SCC degradation mechanisms. The presented facility enables wide range of tests which provide crucial parameters for the evaluation:

- assessment of sensitivity of material to the SCC in modeled environment;
- time to crack initiation,
- threshold values of stress intensity factors both under static and cyclic loading;
- crack growth rates as a function of stress intensity factors and the resulting time of crack growth;

- possibility to test influences of water chemistry, type of loading and temperature on the damage processes.

5. Experimental

The material used in the first study was AISI 316L stainless steel, in the as received condition (cold worked) in order to assess the crack growth rate in demineralized water at elevated temperature and pressure.

The composition of the alloy is given in Tab. (1). The test under constant load was performed with 25 mm thick 1T-C(T) specimen manufactured in accordance with ASTM E399. The specimen was pre-cracked by fatigue in air at room temperature using a load ratio R of 0.1 and a final $K_I$max of 15 MPa·$m^{1/2}$.

The experiment was performed in refreshed, high-pressure (8MPa), high-temperature (288ºC) water loop under "low-flow" conditions (ca. 0.4 liters per hour).

On-line crack growth was continuously monitored during the tests by a reversed dc potential drop technique (RDCPD). The specimen was tested in constant load mode with an initial fatigue transition period (30.5h). The load conditions for initial fatigue period is summarized in Tab. (2).

Table 1. Chemical composition of the steel used in this study (composition in wt%, balance: Fe).

Alloy	C	Mn	Cr	Ni	Mo	Others
316	0.03	1.66	20.7	12.1	1.35	<1

Table 2. Load conditions for initial sinus fatigue period.

	Conditions
Upper limit	14.08 kN
Lower limit	4.22 kN
Amplitude	4.5 kN
Period	60 s

Figure (4) shows the graphic for sinus fatigue in the interval 29.2 to 30.2 h. The recorded potential data during nearly the same time interval are shown in the same figure (results achieved using an average of eight potential values recorded at two different frequencies and in each case at two input current polarities). The mean, pre-fatigue crack length $<a_0>$ was assigned to the potential drop at the point of crack-growth initiation during initial loading in the test. The crack growth increment was calculated by the GE's formula. The calculated crack length at the end of the fatigue was 298 μm.

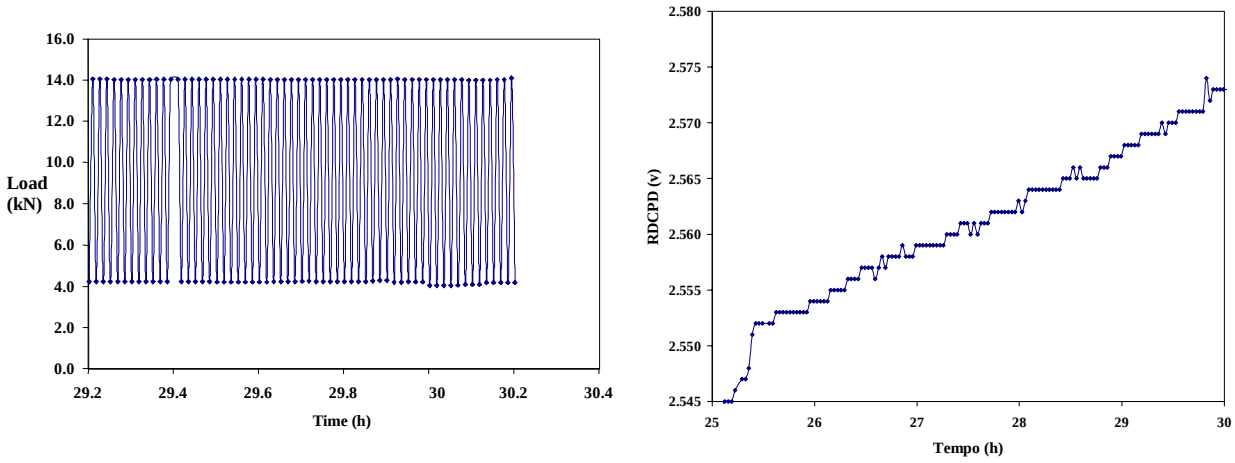


Figure 4. Load conditions for initial fatigue and crack propagation evaluated by RDCPD technique.

The results of the constant-load SCC test are shown in Fig. (5). The specimen was loaded at 14.55 kN stress intensity value. Data indicate that no crack growth was observed in demineralized water at elevated temperature and pressure when tested under constant load during 320 h.

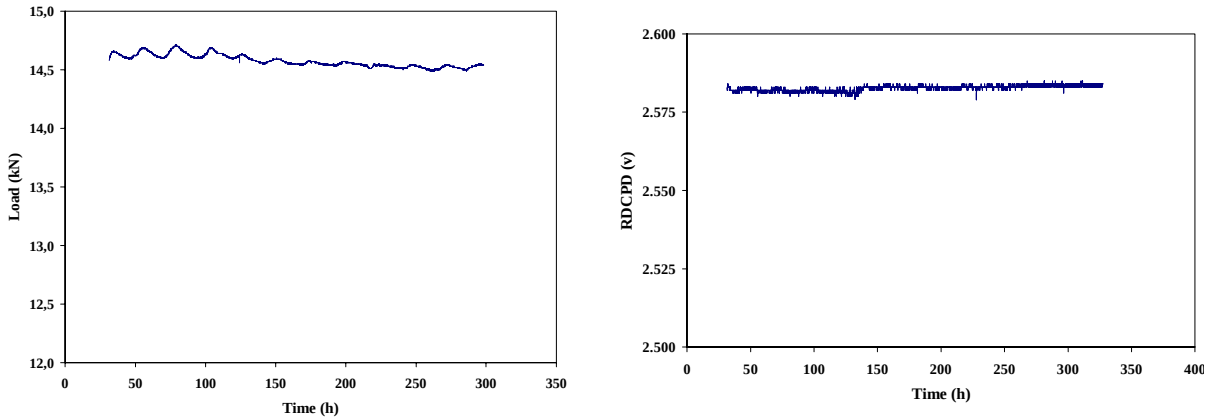


Figure 4. Load conditions for initial fatigue and crack propagation evaluated by RDCPD technique.

6. Conclusion

Under cooperation of CDTN/CNEN and International Atomic Energy Agency a testing facility was developed by Nuclear Research Institute Řež (NRI), Czech Republic, that is being used for the environmentally assisted cracking testing at CDTN/CNEN. By means of this facility, it will be possible to determine all crucial parameters for the assessment of lifetime of components subjected to environmental degradation. The facility allows high temperature autoclave corrosion mechanical testing in well-defined LWR water chemistry using constant load and slow strain rate tests. The facility consists of autoclave and refreshing water loop enabling testing at temperatures up to 330^{o}C. Active loading system allows the maximum load on a specimen as high as 60 kN. The potential drop measurement is used to determine the instant crack length and its growth rate. This is one of the most used techniques to monitor crack growth in specimens under corrosive environments. An initial test carried out on unsensitized AISI 316L stainless steel showed that the facility is working well and it will permit to study some stress corrosion problems and give support in SCC field for reliable operation of our nuclear power plants.

7. Acknowledgement

The authors thank the International Atomic Energy Agency (IAEA) for its valuable support and the financial resources, that allowed the development of this work.

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