Instrumentation for SCC testing in low pressure superheated hydrogen steam environments

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Abstract. This contribution is focused on the issue of Environmentally Assisted Cracking (EAC). For EAC testing in low pressure superheated hydrogen steam environments a special corrosion apparatus is being built in cooperation with University of Manchester and Škoda JS. The whole concept is being prepared for the MEACTOS project. One section concerns the problems with determining an environment with a mixture of hydrogen, argon and steam. The presented equations are necessary for controlling the environment and for the settings of the area of test interest. The test instrumentation is described.

1 Introduction

Environmentally assisted cracking (EAC) is the dominant issue in determining the reliability of most commercial equipment and applications which are controlled by the interactions between structural materials, cooling environments and operating stresses [1, 2]. The goal of the MEACTOS project (Mitigating Environmentally-Assisted Cracking Through Optimisation of Surface Conditions) [3] is to improve the safety and reliability of Generation II and III nuclear power plants (NPPs) by increasing the resistance of critical locations, including welds, to EAC through the application of optimised surface machining and improved surface treatments. This project will quantify the effects of various surface machining and treatment techniques on the EAC behaviour of specific structural materials in the primary circuit of NPPs. The gained knowledge will be summarized in practical guidelines, which can be incorporated into key nuclear design and manufacturing codes.

Pressurized Water Reactor (PWR) nuclear power plants operate with primary cooling water in the proximity of the Ni/NiO electrochemical transition. This is also where Ni based alloys are most susceptible to EAC. Hydrogen is often added to high temperature water to maintain low levels of dissolved oxygen, thereby minimizing corrosion of the structural metals. Primary water stress corrosion cracking (SCC) of Alloy 600 and similar materials is a form of EAC that can occur in essentially pure hydrogenated water at high temperatures [4]. Hydrogen gas produces rapid growth of EAC in high strength steels, and this EAC initiates on surfaces which are absolutely smooth and
requires no previous defects such as pits, intergranular penetrations or mechanical defects [2]. These facts and a lack of clarity has led to careful, detailed investigation. The area of interest is focused on significant acceleration of the EAC process in a low pressure H2-steam environment. In fact, Economy et al. [5] showed a monotonic dependence of crack growth rate (CGR) and EAC initiation time in both water and steam, suggesting that the EAC initiation mechanism is similar for both environments. A special corrosion apparatus built in cooperation with University of Manchester will be used within the MEACTOS project. The final implementation is performed by Škoda JS.

2 Definition of the Environment
It is necessary to perform accelerated testing to determine the PWR structural material resistance to EAC initiation. That is why this device, where tests can be carried out in a hydrogen atmosphere simulating the primary circuit of PWR at temperatures up to 480 °C, was built. Material specimens will be especially tested at 480°C in a steam-hydrogen mixture where the oxygen partial pressure (\( p_{O_2} \)) will be controlled. This control will be obtained by manipulating the steam to hydrogen ratio (\( R_{steam/H_2} \)).

Equilibrium oxygen partial pressure is achieved by the following equation

\[
p_{O_2 \text{Ni/NiO}} = \exp \left( \frac{\Delta G^0_{\text{NiO}}}{RT} \right)
\]  

(1)

where \( R = 8.314 \) J.mol\(^{-1}\).K\(^{-1}\) is the gas constant (also known as the molar, universal, or ideal gas constant), \( T \) corresponds with testing temperature, and \( \Delta G^0_{\text{NiO}} \) is the Gibbs free energy which can be calculated as

\[
\Delta G^0_{\text{NiO}} = A + B T \log T + CT
\]  

(2)

where constants \( A \), \( B \) and \( C \) are shown in Table 1 for NiO.

|          | NiO       | H2O      |
|----------|-----------|----------|
| A        | -232 450  | -239 648 |
| B        | 0         | 18.75    |
| C        | 83.435    | -9.25    |

The area of interest is the ratio between the equilibrium oxygen partial pressure and the oxygen partial pressure in the system in a range of 0.078 up to 50. The partial pressure of oxygen in the system is calculated similarly to equation (1) except for the following details

\[
p_{O_2} = R_{\text{steam/H}_2} \exp \left( \frac{\Delta G^0_{H_2O}}{RT} \right)
\]  

(3)

where the hydrogen ratio (\( R_{\text{steam/H}_2} \)) can be calculated as

\[
R_{\text{steam/H}_2} = \frac{\rho_\text{steam} \cdot T_\text{room}}{\rho_\text{H}_2 \cdot \rho_\text{H}_2 \cdot T_\text{test}}\]  

(4)
In equation (4) there are variables which depend on the system and which can be selected. The flow of water \( F_{H_2O} \) is provided by a pump in millimetres per minute. The flow of hydrogen \( F_{H_2} \) depends on the mixture (for example if the mixture is 4% \( H_2 \) + 96% Ar, the flow of hydrogen will be 0.04*total flow \( F_{H_2+Ar} \)). The density of steam can be determined by the following relationship

\[
\rho_{steam} = AT^0 + BT^1 + CT^2 + DT^3 + ET^4 + FT^5 + GT^6
\]

where constants \( A, B, C, D, E, F, G \), are presented in Table 2.

**Table 2. Constants for density of steam determination.**

|   | A     | B     | C     | D     | E     | F     | G     |
|---|-------|-------|-------|-------|-------|-------|-------|
|   | 8.26774 | -2.83640 | 7.75890 | -1.48420 | 1.79870 | -1.22340 | 3.50000 |
|   | E-04  | E-06  | E-09  | E-11  | E-14  | E-17  | E-21  |

The Gibbs free energy for \( H_2O \) is established in the same way as in equation (2) but different numbers in the \( H_2O \) column in Table 1 have to be used as constants.

### 3 Configuration of the Experimental Device

The environment defined above requires building a new corrosion cell. This technological system consists of a test chamber, a fluid filling system, an organized leaks system and a system invoking the loading of the tested samples.

The new cell for low pressure superheated hydrogen steam environment is currently installed on an electromechanical creep testing machine Kappa SS-CF (see Figure 1) with a load capacity of up to 100 kN and with a speed range from 1 \( \mu \)m/h to 100 mm/min. The creep machine consists of four columns to which the test chamber is attached. The upper crosshead is fixed and the bottom one is sliding. The creep machine invokes the necessary load for carrying out the experiments by moving the bottom crosshead.
Figure 1. A) Electromechanical creep testing machine Kappa SS-CF and B) its modification with corrosion cell for low pressure superheated hydrogen steam environment.

The experiments are performed in the test chamber. The test chamber is shown in Figure 2. It can be seen from the figure that the test chamber consists of two main parts – a cover and a vessel. The test chamber vessel primarily serves as a pressure envelope. The test chamber cover is tightened to the vessel using strength bolts and packed with a torus seal. The tested sample is placed in the test chamber cover with prismatic reductions. A spring-bellows provides the sealing of the test chamber during linear movement. The prismatic attachment of the sample reduction allows testing of different types of samples - cylindrical samples for micro-tensile test such as in [6] or flat samples. The strain gauge, thermocouples and the filling mixture system are located in the test chamber cover.

Figure 2. External and internal assembly of test chamber.

The filling mixture system consists of a storage tank, a dosing pump, a steam generator, Ar+H₂ gas dosing system and a blender. A special chromatographic pump sucks demi water from the storage tank and doses it into the steam generator. Water evaporates in the steam generator and it passes to the blender as steam. The Ar+H₂ gas is heated in a parallel pipe and proceeds to the blender where it is mixed with steam. The mixture is mixed in the blender and heated to 200°C at which it enters into the test chamber. Using a coiled heat exchanger the mixture can be heated up to 480°C and aimed at the tested sample.

Since it is a continuous fluid dosing system, the steam with the Ar+H₂ gas will further be drained from the chamber through the drainage system. The drainage system consists of a cooler, a condenser, an air-leak chamber and a waste demi water storage tank. The cooler and condenser form a piping system where the gaseous mixture is cooled down and as the temperature drops below boiling point it liquidizes and proceeds to the air-leak chamber. The air-leak chamber has a free level at a height of approximately 1 metre by which it keeps hydrostatically internal overpressure of approximately 0.1 bar. Once bubbled through the Ar+H₂ gas, the mixture separates from the water. Subsequently, the Ar+H₂ gas flows to the air condition exhaust and water through the overflow to the waste demi water storage tank.
The entire system is controlled autonomously using a control system controlled by thermocouples and it also controls the equipment using feedback. The system also controls the Ar+H₂ gas and demi water dosing. All the analysed data will be completely backed-up.

4 Conclusions
A low pressure superheated H₂-steam system is used to accelerate the oxidation kinetics while keeping the conditions similar to PWR primary water. One of the most important requirements for this environment is that it needs to replicate the Ni/NiO transition. Nevertheless, despite several studies being carried out by different research groups in H₂-steam environments, there is still a considerable level of uncertainty over the thermodynamics of the oxidation process. The new corrosion cell is being built for this purpose. Two materials will be investigated in the MEACTOS project: Alloy 182 weld metal and type 316L stainless steel. It is planned to test the EAC with two types of specimen – tapered and single-edge notch tension (SENT) with U-notch [7]. Each tapered specimen will have two flat surfaces treated with different final treatments. One flat surface will be a reference and the other one will be treated with one of the investigated surface techniques. A Single Edge Notch Tension specimen will be used for active constant load testing to study EAC crack initiation on a limited area of the surface - only on the notch surface. Testing will be performed at temperatures of 360, 400 and 440°C.

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