ENVIRONMENTALLY ASSISTED CRACKING OF 316L AUSTENITIC STAINLESS STEEL IN A HYDROGENATED STEAM ENVIRONMENT

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ABSTRACT

A low pressure superheated hydrogen steam system has been used to accelerate the oxidation kinetics while keeping the electrochemical conditions similar to those of the primary water in a pressurized water reactor. A special corrosion apparatus was built in cooperation with University of Manchester and Škoda JS for the purpose. The initiation is investigated using a Constant Extension Rate Test (CERT) with flat tapered specimens made from Type 316L austenitic stainless steel in a reducing vapour environment at 350 °C. Two different surface finishes were used: as-ground and as-polished. Constant extension rate tests with a test rate of 2×10^{-6} ms^{-1} were performed on 4 specimens - at room temperature and at an elevated temperature of 350 °C and as a more oxidizing environment was chosen $R = 1/6$, on the contrary as a more reducing environment was chosen $R = 6$, where parameter R represents the ratio between the oxygen partial pressure at the Ni/NiO transition and the oxygen partial pressure. Post-test evaluation using SEM was applied.

KEYWORDS

Environmentally assisted cracking, stress corrosion cracking, hydrogenated steam, oxidation.

INTRODUCTION

The 300 series austenitic stainless steels (ASS) are widely used in the nuclear industry due to their reliable long-term performance in high-temperature water. On the other hand, some cases of Environmentally Assisted Cracking (EAC) have occurred in the components of boiled water reactors (BWRs) and recently in pressurized water reactors (PWRs) owing to a hardened layer under the surface induced during the fabrication process [1, 2].

The sensitivity to EAC of various surface treatments applied to ASS has been studied elsewhere [3, 4, 5]. It was proven that it is connected to the high residual stress as well as to a nano-crystalline microstructure layer under the surface several microns thick. The actual stress needed to initiate an EAC crack is likely a sum of the applied and residual stresses.

It has been recognized that EAC initiation of 304L/316L high resistance SS in water cooling systems operating around 300 °C is accelerated through increasing the water temperature up to 360 °C. Accelerated oxidation leads to the formation of a superficial Cr-rich and inner Ni-rich oxide layers including grain boundary oxidation. A typical intergranular crack initiates after fracture of the oxide or metal-oxide interface [2, 6]. With increasing temperature, higher acceleration of EAC and shorter time to initiation is expected. Under laboratory conditions, one can use temperatures up to 480°C with steam vapour. The conditions have been successfully applied to stimulate EAC in Ni-base alloys [7, 8].

In the paper, the EAC tests are accelerated by applying three factors: strain rate, steam environment and by increasing the temperature. Dominant acceleration via a constant extension rate test (CERT) is employed. Moreover, tapered-shape specimens are used, which allows us to examine a range of stresses and strains simultaneously on one specimen. This type of accelerated EAC testing had been developed in the past [9] and then recently updated within the "Mitigation of Crack Initiation" (MICRIN) project [10, 11]. The effect of surface treatment on EAC crack initiation is studied on two different surface finishes – ground and fine polished, which are typically used for laboratory studies.

EXPERIMENT

This study was performed using 316L ASS produced by Industeel, Alcelor group for the IP EUROTRANS project (Table 1, Table 2). The steel was delivered as 15 mm thick hot rolled and heat-treated plates. The solution annealing was done at 1050-1100 °C. Microstructure in as-received state consisted of austenite grains and about 5% of δ-ferrite stringers oriented in the rolling direction.

Table 1. Chemical composition of the experimental material (wt. %).

Fe	Cr	Mo	Ni	C	Si	Mn	P	S	Al	Cu	Ti	V	N
Bal.	16.69	2.08	9.97	0.018	0.64	1.84	0.027	0.004	0.018	0.23	0.006	0.07	0.029

Table 2. Mechanical properties of the experimental material.

T [°C]	E [GPa]	YS [MPa]	UTS [MPa]	Ag [%]	A30 [%]	Z [%]
25	202	251	567	44	60	80
350	143	168	421	26	38	66

Flat tapered specimens (see Fig. 1) were cut using electrical discharge machining (EDM) with the longer side parallel to the rolling direction of the plate. The specimen's parallel flat surfaces were subjected to different surface finishes: one was manually ground to 500-grid in direction parallel to the load axis, and the other one was polished using 1 μm diamond paste.
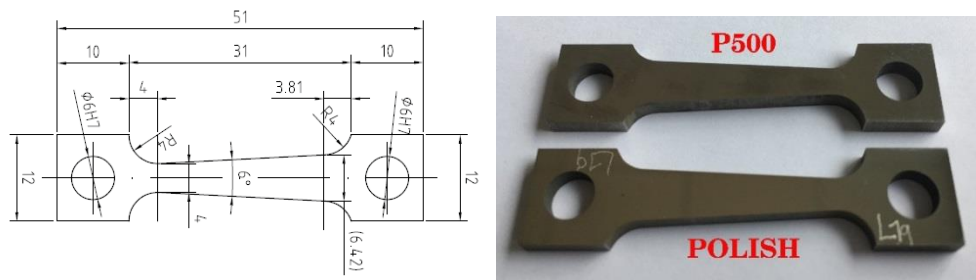

Figure 1. Dimensions of tapered test specimen and two different surface finishes: ground and polished.

The arithmetical mean roughness (Ra) of the polished and ground surfaces were 0.005 μm and 0.032 μm respectively. The HV0.01 micro-hardness measured at about 20 μm below the polished and ground surface was 1.94 GPa (198HV0.01), and 2.05 GPa (209HV0.01) respectively.

The cell for the low pressure superheated hydrogen steam environment is currently installed on an electromechanical creep testing machine Kappa SS-CF (see Figure 2) with a load capacity of up to 100 kN and with a speed range from 1 μm/h to 100 mm/min. 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 filling mixture system consists of a storage tank, a dosing pump, a steam generator, Ar+H$_2$ 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+H$_2$ 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+H$_2$ 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+H$_2$ gas, the mixture separates from the water. Subsequently, the Ar+H$_2$ gas flows to the air condition exhaust and water through the overflow to the waste demi water storage tank. This oxidation system was originally developed by Scenini et al. [12] and subsequently used by several laboratories.
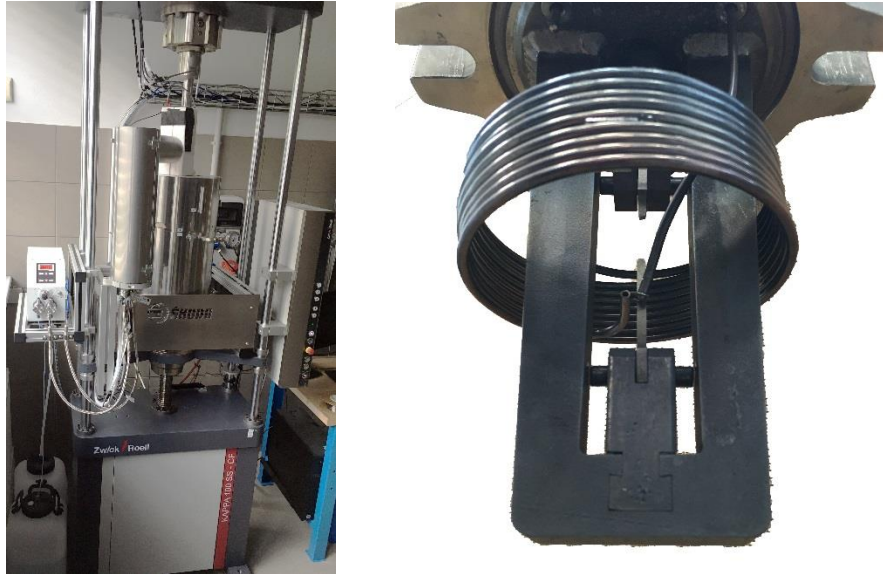
Figure 2. Modification of electromechanical creep testing machine Kappa SS-CF with the corrosion cell for low pressure superheated hydrogen steam environment and inserted broken specimen after CERT experiment.

The CERT corrosion-mechanical test technique was applied. This technique uses uniaxial tensile testing performed with a very slow constant extension rate. Here, it was a test rate of 2×10^{-6} ms^{-1}, corresponding to maximum strain rates of about 10^{-4} s^{-1} at the minimum dimensions of the tapered specimen. The tapered parts of specimens were observed using a field emission gun (FEG) SEMs TESCAN MIRA3.

The oxygen partial pressure was controlled by manipulating the steam to hydrogen ratio (R_{steam/H_2}). The relationships for this method are described in [13]. The mixture 6% H_2 + 94% Ar was used. To simulate different oxidizing and reducing environments with respect to the Ni/NiO transition: the oxygen partial pressure (p_{O2}) could be varied by changing the H_2 partial pressure (e.g. by increasing the H_2 flow and hence the H_2 partial pressure, the redox potential decreases). The complete thermodynamics of the H_2-steam environment can be described using the parameter R which represents the ratio between the oxygen partial pressure at the Ni/NiO transition ($p_{O2\ Ni/NiO}$) and the oxygen partial pressure p_{O2} according to equation (1). For values lower than 1, NiO is stable (oxidizing environment), while for values higher than 1, Ni is stable (reducing environment).

$$R = \frac{p_{O_2\ Ni/NiO}}{p_{O_2}} \qquad\qquad (1)$$

RESULTS

The specimens were tested in a low-pressure H_2-steam environment at a temperature of 350 °C up to rupture. $R = 1/6$ was chosen as a more oxidizing environment, which corresponds to a water flow rate of 2.22 mL/min, a gas mixture (6% H_2 + 94% Ar) of 50 cc/min, a steam-to-H_2 ratio of 983 and an oxygen partial pressure of 2.48×10^{-30} atm. On the contrary as a more reducing environment was chosen $R = 6$ which corresponds to a water flow rate of 0.37 mL/min, a gas mixture (6% H_2 + 94% Ar) of 50 cc/min, a steam-to-H_2 ratio of 164 and an oxygen partial pressure of 6.90×10^{-32} atm. Ultrahigh purity water with a conductivity of 0.055 μmS/cm was used. The tapered specimens were exposed to the steam for one day before CERT loading.
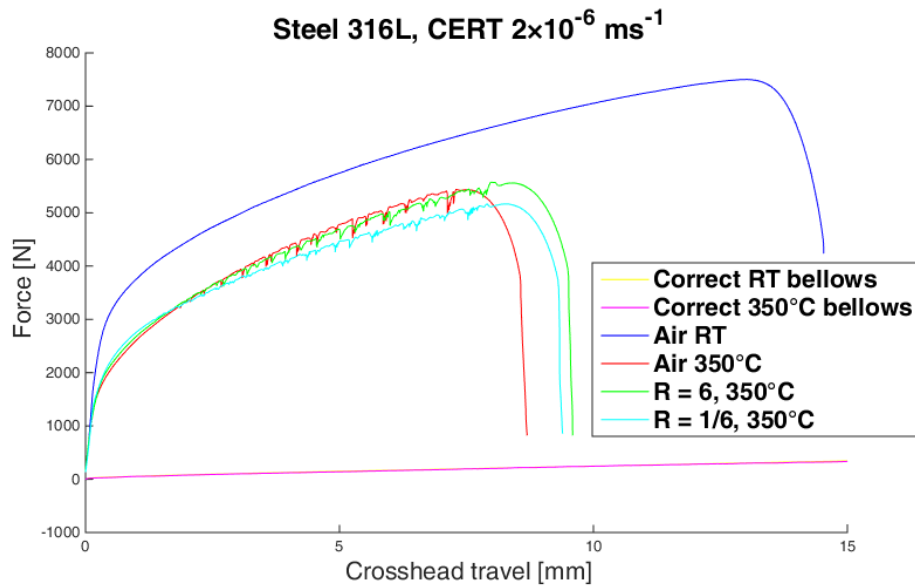
Figure 3. Comparison of measured curves of the CERT tests. Correction curves for room temperature and for elevated temperature 350 °C are identical.

Fig. 3 shows the complete results of the tests. Every curve corresponds to one sample. The first tensile test with a rate of 2×10^{-6} ms^{-1} was performed on the same rig at room temperature and 350 °C. To correctly interpret the results, it was necessary to perform tests without a sample at both temperatures. These correction curves have to be subtracted from the curves with the specimens because the correction curves represent the resistance of the spring bellows. It can be seen that both correction curves are the same, which means that the temperature does not play any role for the spring bellows, because this part is outside the chamber.

The corrected curves of CERT with specimens at 350 °C are plotted in Fig. 4. For the tapered specimen tested at room temperature, the maximum stress at the minimum cross section was evaluated as 603 MPa (note Table 2 UTS value is 567 MPa). For the specimen at 350 °C the maximum stress was 426 MPa (Table 2 UTS value is 421 MPa). It can be said that the CERT curves taken in the two different steam environments lay under the curve in air. Moreover both curves in the steam environments showed longer elongation. The effect is likely higher for the curve in the oxidizing environment R = 1/6, than for the one with R = 6. All three curves at 350°C show oscillations of load, which are typical for dynamic strain ageing.

Small coupons of pure nickel (99.5 wt%) were used in each test to confirm the environment. However, it was found that an exposure of one day seems to be insufficient for the material to be affected by the environments.
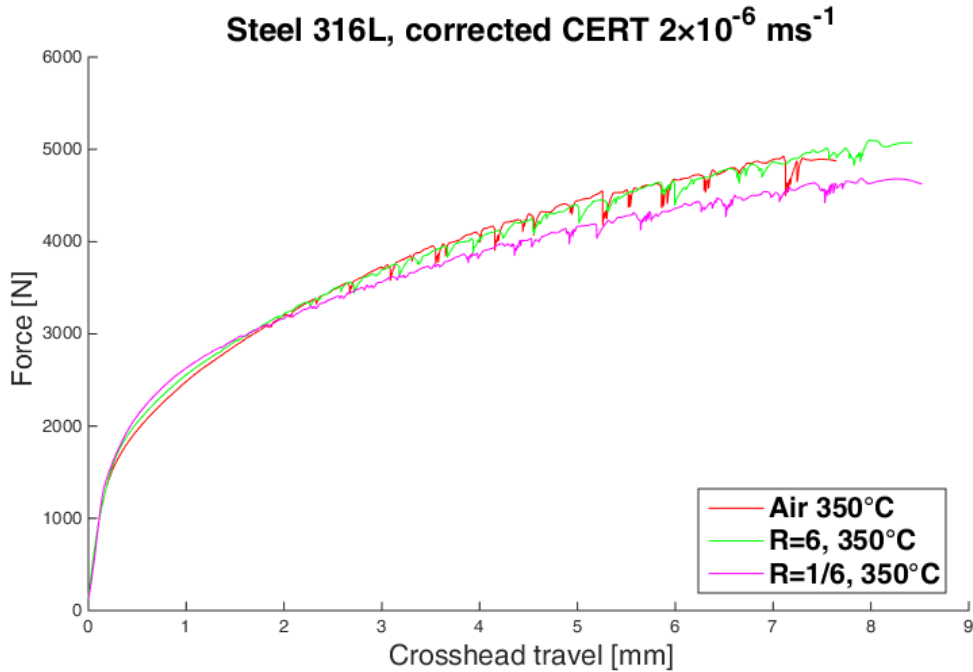
Figure 4. The CERT test curves of the tapered specimens in air and steam at 350 °C after subtraction of the correction curve.

All three specimens failed by ductile fracture with extensive plastic deformation. Post-test evaluation using SEM showed that the flat surfaces were clean, practically without any oxide particles. The polished and ground surfaces behaved similarly. The SEM observation of the fracture shows typical ductile fracture dimples (Fig. 5). It proved that a one-day exposure is likely insufficient for the material to be affected by the environments.
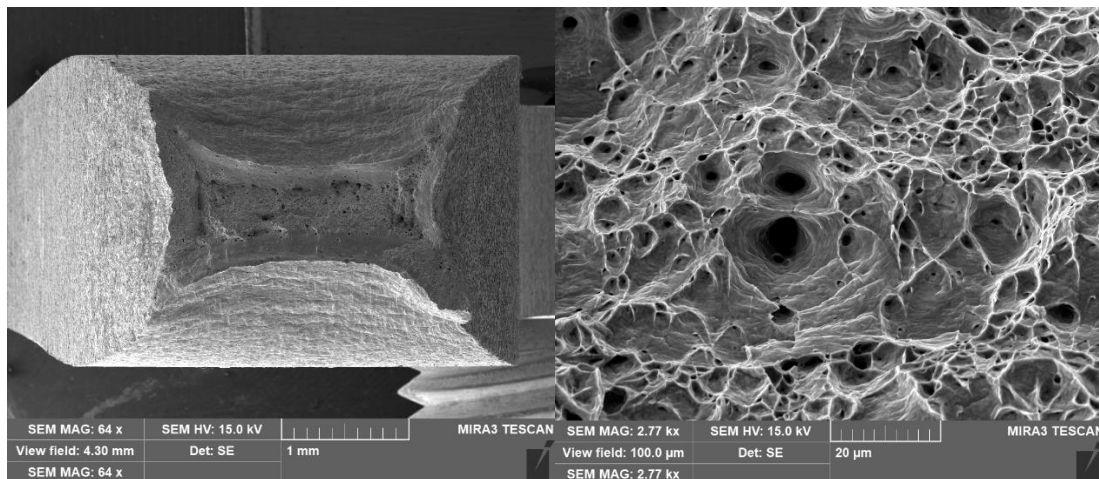

Figure 5. Fracture surface of the tapered specimen after CERT with rate of 2×10^{-6} ms^{-1} in the steam environment of R = 6 at 350 °C.

CONCLUSIONS

A new low pressure superheated hydrogen steam system was used to perform Constant Extension Rate tests at room temperature and 350 °C. The special corrosion apparatus was built in cooperation with University of Manchester and Škoda JS for the MEACTOS EU project.

Four flat tapered specimens made from austenitic stainless steel 316L, each with one side polished and the other ground, were investigated. Oxidizing and reducing environments with R = 1/6 and 6 respectively were applied.

The exposure of the 316L steel to both steam environments at 350 °C and loaded with a rate of 2×10^{-6} ms^{-1} did not lead to initiation of EAC cracking.

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