ELECTROCHEMICAL STUDIES ON STRESS CORROSION CRACKING OF INCOLOY-800 IN CAUSTIC SOLUTION
Part II: Precracking samples

by

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Stress corrosion cracking (SCC) in a caustic medium may affect the secondary circuit tubing of a CANDU NPP cooled with river water, due to an accidental formation of a concentrated alkaline environment in the areas with restricted circulation, as a result of a leakage of cooling water from the condenser. To evaluate the susceptibility of Incoloy-800 (used to manufacture steam generator tubes for CANDU NPP) to SCC, some accelerated corrosion tests were conducted in an alkaline solution (10% NaOH, pH = 13). These experiments were performed at ambient temperature and 85 C. We used the potentiodynamic method and the potentiostatic method, simultaneously monitoring the variation of the open circuit potential during a time period (E_{corr}/time curve). The C-ring method was used to stress the samples. In order to create stress concentrations, mechanical precracks with a depth of 100 or 250 m were made on the outer side of the C-rings. Experimental results showed that the stressed samples were more susceptible to SCC than the unstressed samples, whereas the increase in temperature and crack depth lead to an increase in SCC susceptibility. Incipient microcracks of a depth of 30 m were detected in the area of the highest peak of the mechanical precrack.

Key words: caustic stress corrosion cracking, Incoloy-800, CANDU nuclear power plant, steam generator

INTRODUCTION

It is known that stress corrosion cracking (SCC) is due to a process involving conjoint corrosion and straining of a metal due to residual or applied stresses [1]. SCC is an insidious form of corrosion, because it produces a marked loss of mechanical strength with little metal loss. Although the damage is not obvious by casual inspection, stress corrosion cracks can result in a rapid growth of fracture and catastrophic failure of components and structures. Fortunately, the occurrence of SCC depends on the simultaneous compliance of three requirements: susceptibility of material to SCC, an environment that causes SCC of a specific material and sufficient tensile stress to induce SCC.

The SCC mechanism can affect both components of the primary and the secondary side of the nuclear power plant, depending on the existing system metal/environment and predominantly in areas containing concentrated impurities [2]. In the case of the secondary side of a CANDU NPP cooled with river water, an accidental formation of a concentrated alkaline medium in areas with restricted circulation, as a result of a leakage of cooling water from the condenser, can lead to the damage of steam generator tubes made of Incoloy-800 (type UNS N08800) [3]. Another reason for corrosive environment occurrences may be the abnormal water chemistry in the circuits of a CANDU NPP. Stress can appear in the rolling expansion regions of steam generator tubes, at the joint with the tube plate and in the region of tube supports. It may also result

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from vibrations due to the high flow rate of heavy water, or may be generated by the formation of corrosion products in limited areas.

Because steam generator damage involves the shutdown of the reactor and extremely high costs of repairs, it is necessary to find methods to avoid SCC occurrence in steam generators tubes.

EXPERIMENTAL

The material tested in our experiment was Incoloy-800, which is an austenitic Fe-Cr-Ni alloy (UNS N08800), manufactured according to ASTM SB 407. In tab. 1, the chemical composition determined by the supplier is presented. Values of yield stress $\sigma_{0,2\%}$ and ultimate tensile stress σ_r of uniform elongation A and of elastic moduls E for Incoloy-800 tubes, obtained in the circumferential direction, are shown in tab. 2 [4].

Table 1. The chemical composition of Incoloy-800 [wt %]

Fe	Ni	Cr	C	Si	P	S	Mn	Ti	Cu	Al
Rest	30-35	19-23	0.02	0.52	0.009	0.004	0.50	0.47	0.01	0.31

Table 2. The mechanical properties of Incoloy-800 at room temperature [4]

$\sigma_{0,2\%}$ [MPa]	σ_r [MPa]	A [%]	E [kN/mm²]
170-205	450-500	35	198

The C-ring method [5] was chosen as a mode of stressing Incoloy-800 tubes. The C-rings were obtained by cutting rings 12 mm in height from the tubes of the generator. The outer diameter of these tubes is 15.9 mm, wall thickness 1.13 mm. The C-rings were then compressed using a screw (fig. 1). To obtain a concentrated stress in a certain zone, some samples were mechanically precracked. The mechanical cracks had two depths: 100 and 250 mm. The length of these mechanical precracks is 12 mm (the height of the C-rings).

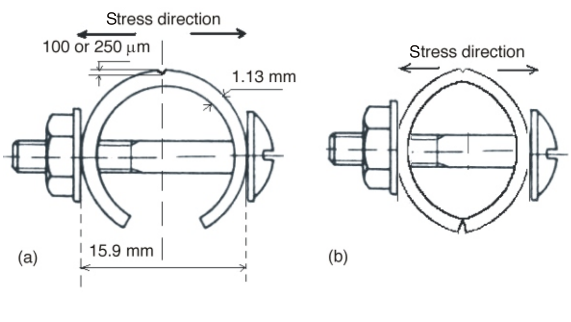


Figure 1. The C-ring used to test the susceptibility of Incoloy-800 to SCC; (a) unstressed sample, (b) stressed sample

The stresses and deformations induced in Incoloy-800 rings were evaluated by using the ANSYS code. This code is a finite-element analysis package, widely used in industry to simulate the response of a physical system to structural loading, thermal and electromagnetic effects. The estimation made at room temperature shows that the maximal value of stress at the highest point of the mechanical crack is around 614 MPa in both cases (precrack depth 100 and 250 mm) [6]. Because the stress becomes relaxed at ambient temperature after the tightening of the C-rings, the applied stress stabilizes at a value known as the “internal stress”. Studies performed on Incoloy-800 showed that, practically, the stress was stabilized at 80% of the value theoretically applied [7]. Therefore, the value of the real stress at the highest point of the mechanical crack was stabilized below the value of ultimate stress.

The concentration of NaOH in the area of crevices formed in the hot zone of the steam generator tubes may be 3% after 10,000 hours of exposure and increase up to 50%, after a very long time of exposure [8]. A solution of 10% NaOH (pH = 13) was used in these experiments.

Since it takes a long time to initiate SCC-type cracks (approximately 10,000 hours [9]), accelerated electrochemical corrosion tests were performed using the Princeton Applied Research Model 273 Electrochemical System. In the electrochemical cell, containing tree electrodes (work electrode, reference electrode, and auxiliary electrode [3]), an alkaline solution (10% NaOH, pH = 13) was introduced. The tests were carried out at ambient temperature and 85 C (the maximum temperature at which the electrochemical cell can operate).

When the potentiodynamic (PD) method was used, the potential values ranged between –800 mV *vs.* E_{cor} and +1600 mV *vs*. SCE (Saturated Calomel Electrode), with a scan rate of the potential of 0.2 mV/s. The samples potentiostatically tested at 100 mV were maintained between 24 and 48 hours at 85 C in a 10% NaOH solution (pH = 13). This potential value was chosen from the active region of the potentiodynamic curve. Another method that permitted the assessment of the susceptibility to SCC consisted of recording the variation of the open circuit potential over a time period (E_{corr}/time curve).

The samples tested potentiostatically were examined by the optical microscope NEOPHOT2.

RESULTS AND DISCUSSIONS

In the case of precracked samples with 100 μm crack depth, the PD curves for unstressed and stressed samples, respectively, both tested at 25 C,

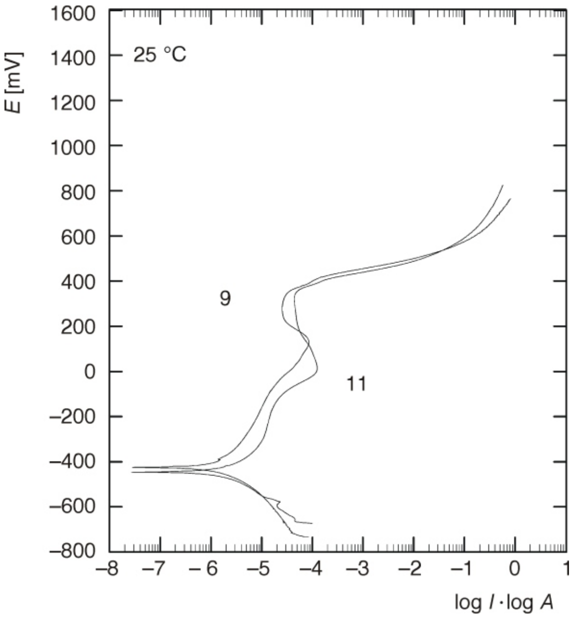


Figure 2. PD curves of the precracked samples with 100 μm depth of precrack tested at 25 C; 9 – unstressed, 11 – stressed

are shown in fig. 2. The potentiodynamic (PD) curve shows the evolution of corrosion potential *E* function of the corrosion current *I*. The displacement of anodic current to higher values indicates that the stressed sample is more susceptible to corrosion. Figure 3 presents the influence of the temperature increase on the susceptibility to SCC of stressed samples. It is noticed that the samples

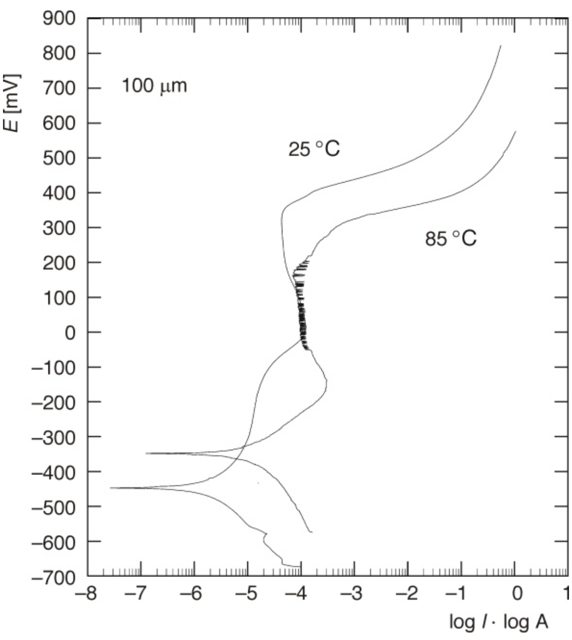


Figure 3. PD curves of the stressed and precracked samples tested at room temperature and 85 C (100 μm depth of precrack)

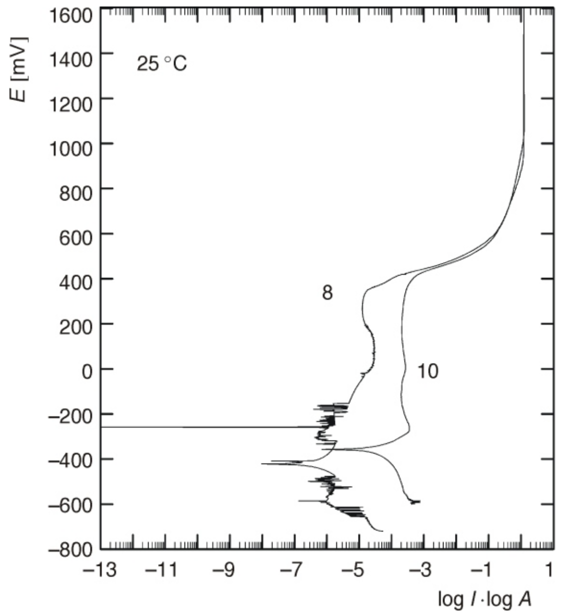


Figure 4. PD curves of the precracked samples with 250 μm depth of precrack; 8 – unstressed, 10 – stressed

tested at room temperature have a greater passivity tendency than the samples tested at 85 C. At the same time, the passive region of the samples tested at room temperature is larger than the passive region of the samples tested at a higher temperature. Therefore, the susceptibility to SCC increases with the increase of temperature and stress.

The same results were obtained in the case of mechanically precracked samples with a 250 μm depth of crack. Figure 4 shows the PD curves corresponding to these samples, stressed and unstressed, tested at room temperature. Figure 5 displays the overlap of the PD curves, corresponding to the stressed samples tested at room temperature and at 85 C, respectively.

Because the anodic current is displaced to higher values in the case of samples with a 250 μm depth of precrack, we can state that the increase in crack depth led to an increase in SCC susceptibility (fig. 6).

The stressed samples with the depth of mechanical precracks of 100 μm were tested potentiostatically in two stages: the duration of the first stage was 14 hours, of the second one 20 hours. Between these stages, the sample remained into the solution at room temperature; hence, the total duration of the real test was 34 hours. Potentiostatic curves, which represent the evolution of corrosion current function of the time, are presented in fig. 7. The establishment of the anodic current on a certain constant value (about few nA) in the first stage, shows that a passive layer was formed on the sample surface. Also, 2.5 hours after the start of the experiment (fig. 7a), the occurrence of an anodic current peak could be observed, which

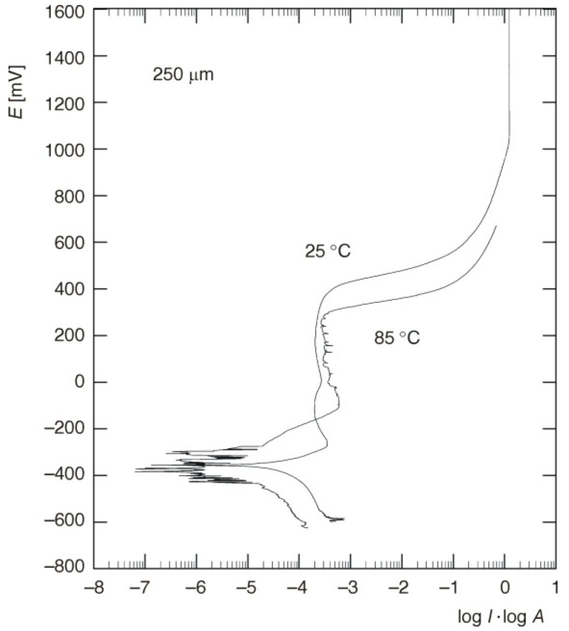


Figure 5. PD curves of the stressed and precracked samples tested at room temperature and 85 C (250 µm depth of precrack)

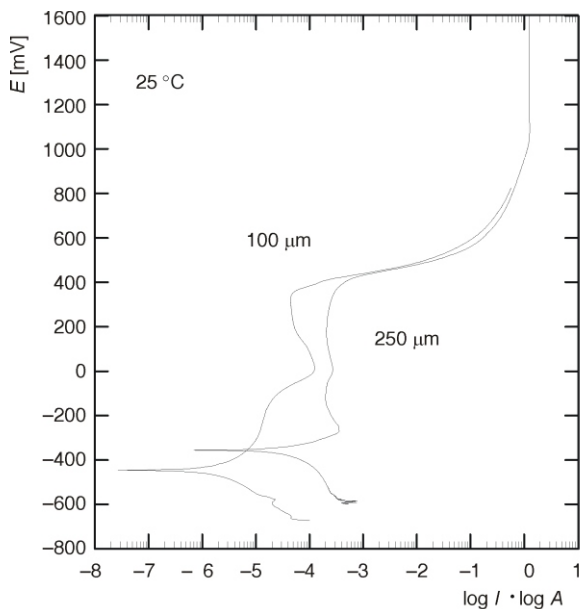


Figure 6. PD curves of the precracked and stressed samples tested at 25 C

could represent the initiation of SCC cracks. During the second stage, one could observe certain increases of the anodic current, due to the breaking of the passive layer and to the propagation of SCC cracks (fig. 7b). The first important peak of the anodic current occurs 14.5 hours after the beginning of the experiment (14 hours the duration of the first stage and 0.5 hours after the start of the second stage fig. 7b).

In the case of the samples with the depth of the mechanical precrack of 250 µm, the potentiostatic test was performed in three stages, *i. e*. for 5.5 hours (first and second stage) and 10 hours (third stage). The total duration of this test was 21 hours. In this case, SCC cracks initiation occurred 1 hour after the start of the experiment (fig. 8a) and the first important increase of the anodic current occurred 8 hours after the beginning of the test (fig. 8b). In the last stage, numerous peaks developed representing the propagation of SCC cracks (fig. 8c).

The E_{corr}–time curves corresponding to the stressed samples tested at 85 C in a solution of 10%NaOH are presented in fig. 9. The variations of potential values show a process due to the growth of the passive layer and its subsequent breaking. In the case of the samples with a depth of the mechanical precrack of 250 µm, the potential decreased dramatically down to about 629 mV, which can represent the breaking of the passive layer and the start of

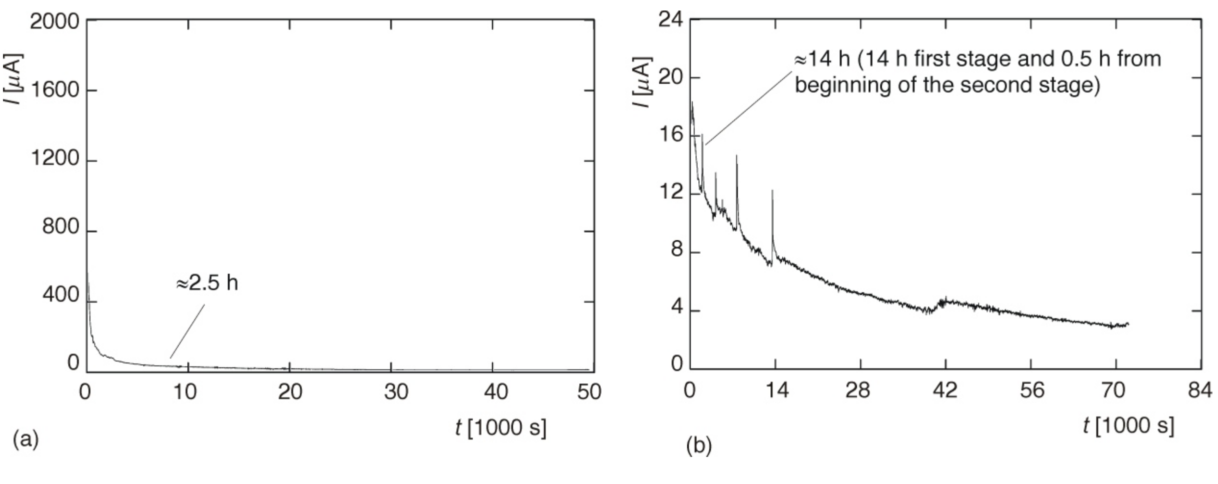


Figure 7. The potentiostatic curves obtained on a stressed sample with the depth of the mechanical precrack of 100 µm; (a) first stage (14 hours); (b) second stage (20 hours)

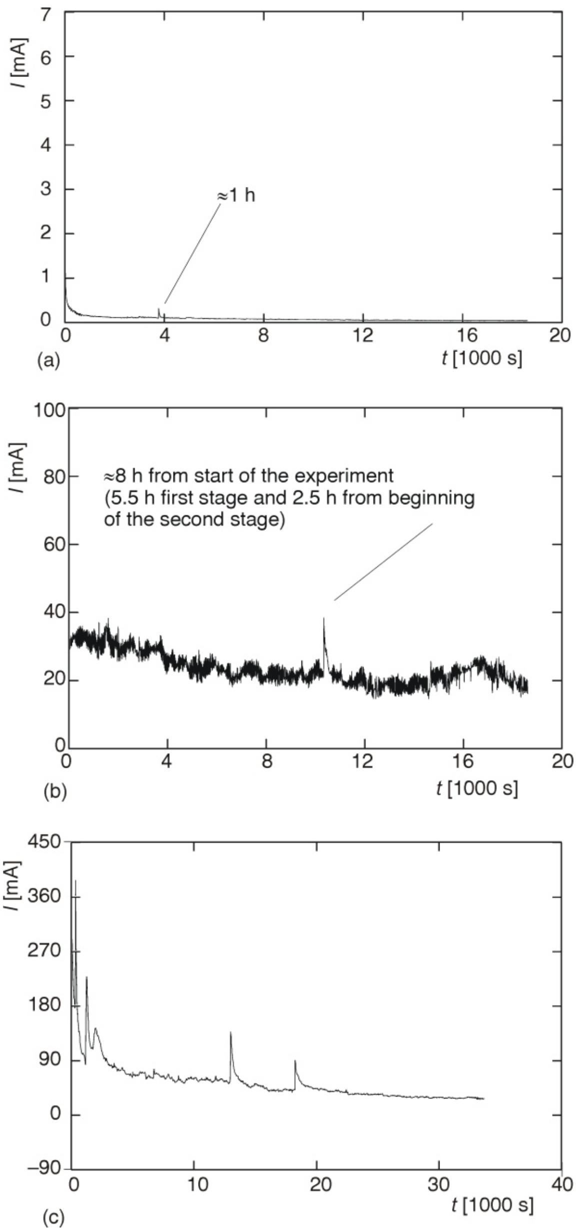


Figure 8. Potentiostatic curves obtained on a stressed sample with the depth of mechanical precrack of 250 mm;
(a) first stage (5 hours),
(b) second stage (5 hours),
(c) third stage (10 hours)

SCC cracks. The values of the potential had more negative values in the case of the sample with a depth of the mechanical precrack of 250 μm. Therefore, this sample is more susceptible to SCC.

In the case of the samples tested potentiostatically, optical microscopy emphasised the presence of incipient SCC cracks. The cracks propagated up to the depth of approximately 30 μm. It was noticed that these incipient cracks occurred in the area of the deepest point of the mechanical precracks. These cracks initiated lat-

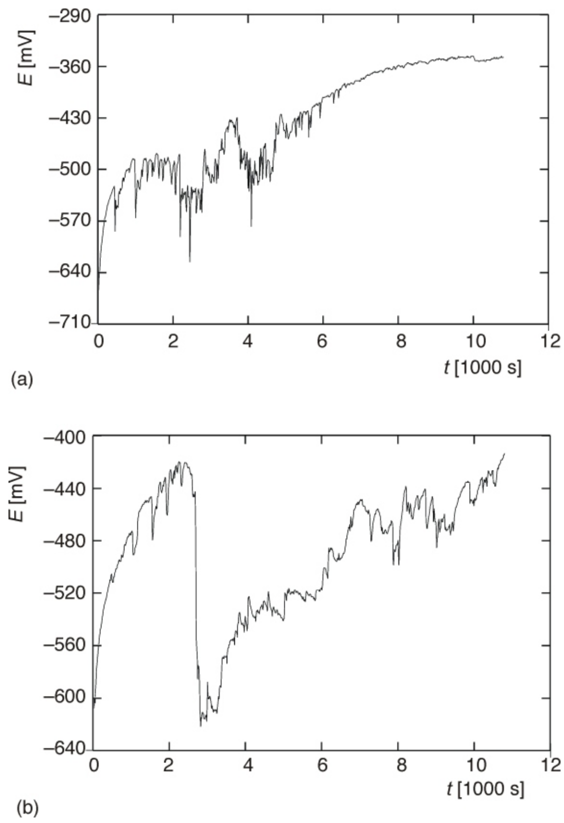


Figure 9. E_{corr}-time curves of the stressed samples tested at 85 C in a 10% NaOH solution with the depth of mechanical precrack; (a) 100 μm, (b) 250 μm

erally in the area with the maximum stress estimated by ANSYS code (fig. 10).

CONCLUSIONS

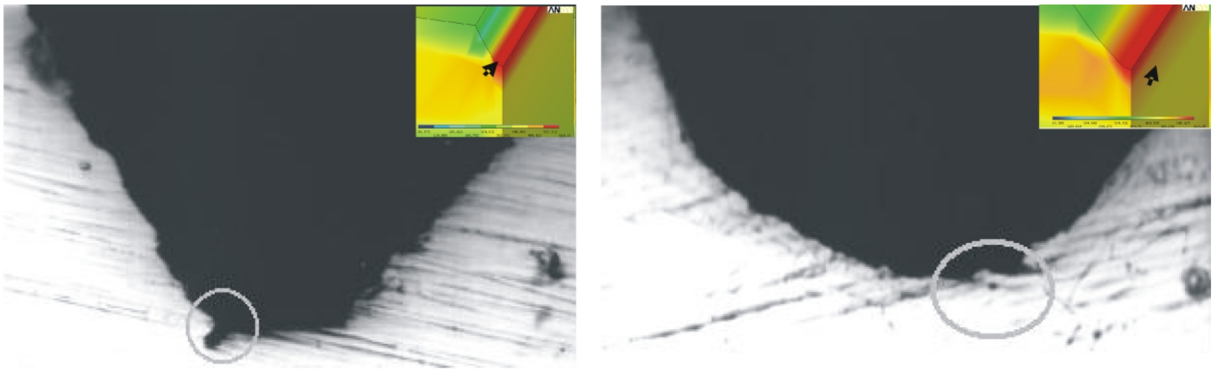
The potentiodynamic tests which allowed SCC evaluation showed that:

(a) the stressed samples are more susceptible to SCC than the unstressed samples. The passive range is shorter in the case of the stressed samples than in the case of those unstressed at room temperature and at 85 C,

(b) the increase in temperature led to the increase in SCC susceptibility of the stressed samples. At 850 C, the passivity tendency is lower than at room temperature, and

(c) the increase in the depth of the mechanical precrack determined an increase in SCC susceptibility.

Potentiostatic tests indicate that the chosen value of corrosion potential (100 mV) is able to initiate and propagate SCC microcracks at the surface of the tested samples and that the time of SCC microcracks initiation decreases with the increase in the depth of the mechanical precrack.



(a) (b)

Figure 10. SCC microcracks in the deepest point of the mechanical precrack of stressed samples tested potentiostatically in a 10% NaOH at 85 C and 100 mV; (a) depth of mechanical precrack 100 μm, (b) depth of mechancal precrack 250 μm (500)

Using the metallographic method, it was possible to detect the presence of incipient SCC microcracks in the area of the highest point of the mechanical precrack. The average depth of these incipient microcracks is approximately 30 μm.

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ЕЛЕКТРОХЕМИЈСКО ПРОУЧАВАЊЕ ПУЦАЊА ЛЕГУРЕ INCOLOY-800 УСЛЕД НАПОНСКЕ КОРОЗИЈЕ У РАСТВОРУ НАТРИЈУМХИДРОКСИДА ДЕО ДРУГИ: УЗОРЦИ СА ПРЕТХОДНОМ НАПРСЛИНОМ

Пуцање цеви услед напонске корозије и цурење хладне воде из кондензатора последица су случајног нагомилавања базног садржаја у зонама ограничене циркулације, што може да поремети систем секундарног кола нуклеарне електране типа CANDU хлађене речном водом. Спроведена су испитивања убрзане корозије у базном раствору (10% NaOH, pH = 13) која су имала за циљ да се оцени осетљивост на пуцање услед напонске корозије легуре Incoloy-800 (коришћене за израду цеви парогенератора нуклеарне електране CANDU). Експерименти су обављени на собној температури и 85 C. Коришћене су потенциодинамичка и потенциостатичка метода уз истовремено праћење промене потенцијала отвореног кола током једног временског интервала (крива E_{corr}/време). Узорци су претходно деформисани применом методе C-прстена. Да би се остварила концентрација напрезања, на спољашњој страни C-прстена вештачки су изазване напрслине дебљине 100 и 250 μm. Експериментални резултати указују да су претходно деформисани узорци осетљивији на пуцање него недеформисани, као и да пораст температуре и дубине напрслина доводи до пораста осетљивости на напонску корозију. У околини највеће деформације првобитне напрслине запажено је образовање почетних микронапрслина дубине 30 μm.

Кључне речи: пуцање услед напонске корозије у базним растворима, Incoloy-800, CANDU нуклеарна електрана, парогенератор