CORROSION SCIENCE AND TECHNOLOGY, Vol.3, No.1(2004), pp.14~19

Stress Corrosion Cracking in the Pre-Cracked Specimens of Type 403 Stainless Steel

Jong Jip Kim

Materials Evaluation Center, Korea Research Institute of Standards and Science, 1 Doryongdong, TaeJeon 305-606, KOREA

Crack growth rate and threshold stress intensity factor for stress corrosion cracking(SCC), K_{ISCC} were measured for type 403 stainless steel in 3.5% NaCl solution at room temperature and SCC was monitored by electrochemical noise technique during K_{ISCC} testing.
In rising load test, pits were formed at the tip of pre-crack for the pre-cracked compact tension specimen unlike in smooth round specimen in which only unstable pits were observed and hence immune to SCC. Micro-cracks were found to initiate from the pits in the former specimen, and initiation of micro-crack as well as macro-crack was detected by electrochemical noise technique in rising load K_{ISCC} tests. Crack growth rate increased with increasing either displacement rate or stress intensity factor at crack initiation and was higher in rising load K_{ISCC} test compared to constant load K_{ISCC} test at given stress intensities. In addition, K_{ISCC} value was lower in the former test, indicating the test condition of rising load test was more severe than that of constant load test.

Keywords *: crack growth rate, K_{ISCC}, rising load test, crack initiation, electrochemical noise*

1. Introduction

Because of crack-like defects introduced during either manufacture or subsequent service, the tests have been developed that employ pre-cracked specimens for the stress corrosion cracking of materials. Furthermore, such tests provide information on the susceptibility to stress corrosion cracking which may not be evident from smooth specimens in some materials. It is also well known that major parameters such as crack growth rate and threshold stress intensity factor for stress corrosion cracking, K_{ISCC} can be measured accurately only by these tests.[1]

Especially for K_{ISCC}, there exist at present several standard test methods[2)-3)] developed based on either constant load or constant deflection testing. However, they have major limitation, that is, testing times of the order of hundreds to thousands of hours are required normally to establish a meaningful K_{ISCC} value. Because of this limitation, an accelerated test method is currently being standardized based on rising load testing[4)] that can reduce the testing time remarkably.

Though much efforts have been made for the standardization of the rising load test, still more information is needed for the further improvement or elaboration of the test method. So far, not sufficient data have been reported on the K_{ISCC} values in wide variety of material, environment and test method combinations and the details in the progress of stress corrosion cracking during rising load K_{ISCC} test.

In this work, the K_{ISCC} is measured by rising load test for the type 403 stainless steel and the result is compared with that determined by constant load testing. In addition, electrochemical noise is measured for the monitoring of the stress corrosion cracking during pre-loading and main rising load testing.

2. Experimental

Commercial 45.0 mm thick AISI 403 stainless steel plates were used in this investigation. The plates were austenitized at 1000 ℃ for 1.5 h, air-quenched and then tempered for 2 h at 390 ℃. Specimens were cut from the plates in SL orientation and then pre-cracked for the measurement of critical stress intensity factor, K_{IC} and SCC threshold stress intensity factor, K_{ISCC}. Prior to K_{ISCC} testing, the specimens were pre-loaded in 3.5% NaCl solution at a constant load which corresponds to a value of 5% of K_{IC} in accordance with ISO FDIS 7539-9. The rising load K_{ISCC} tests were performed at room temperature at the displacement rates ranging from 1×10^{-7} to 1×10^{-5} mm/sec. For this work, a horizontal loading frame was used that allows the immersion of only the crack tip and

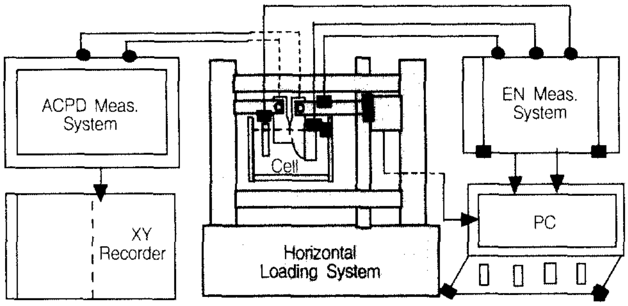

Fig. 1. Schematic drawing of the experimental set up.

anticipated crack growth region of the specimen in test solution to avoid the galvanic or crevice corrosion in contact areas between grip and specimen.

Crack growth rate and electrochemical noise were measured simultaneously in addition to load and displacement during the tests using the test set up shown in Fig. 1. The crack growth rates were measured by alternating current potential drop method(ACPD), and EN measurements were performed under open circuit conditions in a three electrode set-up consisting of a working electrode(tensile specimen under load), a platinum counter electrode and a saturated calomel electrode. The noise data were acquired at a frequency of 10 Hz using a Gamry Instruments Inc. PC 4 potentiostat and associated software.

3. Results and discussion

Fig. 2 shows the result of rising load test for a pre-cracked compact tension specimen at a displacement rate of $1x10^{-5}$ mm/sec in 3.5% NaCl solution. This test was performed after pre-loading for 40 h in the same solution at a constant load of 280 kgf which corresponds to a value of 5% of K_{IC} of the specimen. The maximum or crack initiation load is 1189.6 kgf and the displacement at this load is 0.192 mm. The values are much lower than those obtained during K_{IC} testing in air, indicating that stress corrosion cracking occurs for this specimen in 3.5 % NaCl solution at room temperature. In air, the maximum load and the displacement at this load are 5510 kg and 15.5 mm, respectively. This result is in contrast with that of smooth round bar tensile specimen with no pre-crack tested at the same displacement rate. As reported previously,[5] the ratio between the elongation to failure in 3.5 % NaCl solution and that in air was 0.964 for this specimen, and it was concluded that the specimen is immune to SCC in this solution at room temperature since the ratio was greater than 0.8, an index of immunity to SCC.[6]

For the further investigation, the fracture surface was

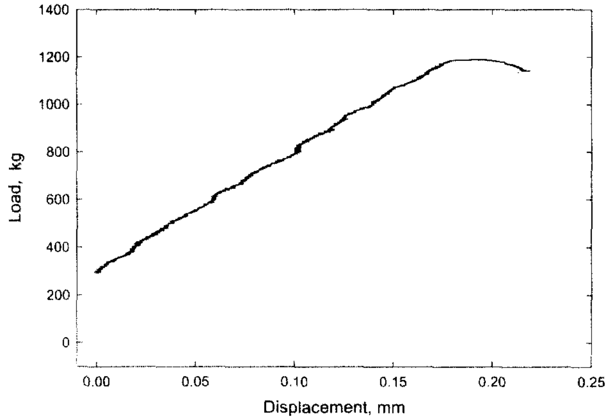

Fig. 2. Load vs displacement curve plot during rising load test at a displacement rate of $1x10^{-5}$ mm/sec.

examined by SEM. As can be seen in Fig. 3, the fracture surface of the tip of pre-crack consists of small dimples and large pits. Large stable pits are believed to be formed during pre-loading as can be confirmed later from electrochemical noise data. Except the pits observed on the pre-crack, the fracture surfaces are similar for both pre-cracked compact tension and smooth round tensile specimens. Final or mechanical fracture zone consists of small dimples, and inter-granular fracture is observed in the SCC zone indicating that SCC mechanism of both type of specimens are the same regardless of the specimen geometry. Considering only the difference in fracture surfaces between two specimens is the presence of the pits formed during pre-loading, stress corrosion cracks are presumed to initiate from stable pits at the pre-crack. The formation of stable pits may be assisted by stress intensified at the tip of the pre-crack of compact tension specimen, though the pre-load applied is a low level.

For the determination of K_{ISCC}, rising load tests were performed at lower displacement rates as shown in Fig. 4. The stress intensity factor at crack initiation, $K_{I,init}$. decreases with decreasing displacement rate, and the $K_{I,init}$. vs displacement rate curve appears to be asymptotic to a value, which is K_{ISCC}, 22.9 MPa · $m^{1/2}$. This K_{ISCC} value determined in this work is much lower than that by a constant load testing using a canti-lever beam type tester[7] as can be seen in Figs. 5. The K_{ISCC} value obtained with the latter test is 39.1 MPa · $m^{1/2}$ implying that the test condition of the latter test is less severe than the former test. Note in the latter test a constant load is applied at the beginning of the test without any pre-loading until the specimen failure occurs. The highest load initially applied or the corresponding stress intensity factor resulting in no failure or no evidence of crack growth after a predetermined period of test time, usually 1000 hr for steels, is

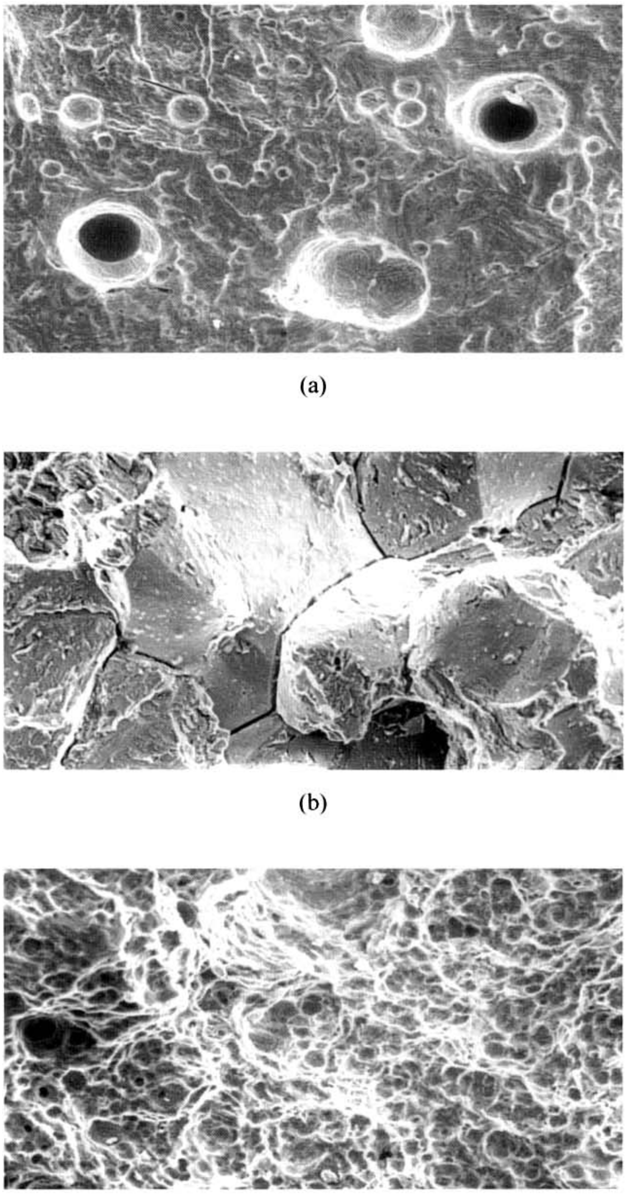

Fig. 3. Typical fracture surfaces of compact tension specimen after rising load test; (a) pre-cracked zone, (b) stress corrosion crack propagation zone and (c) mechanical fracture zone.

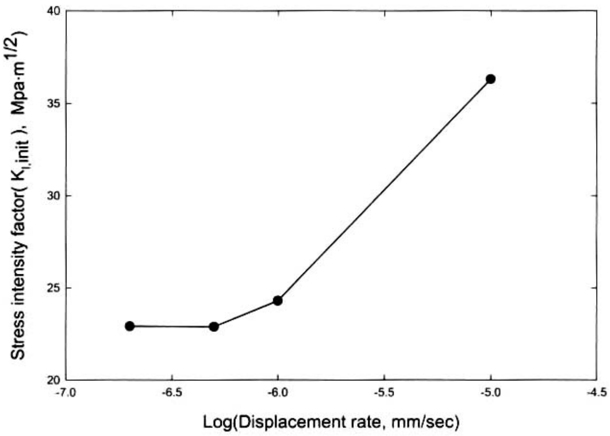

Fig. 4. Effect of displacement rate on the stress intensity factor at crack initiation in rising load test.

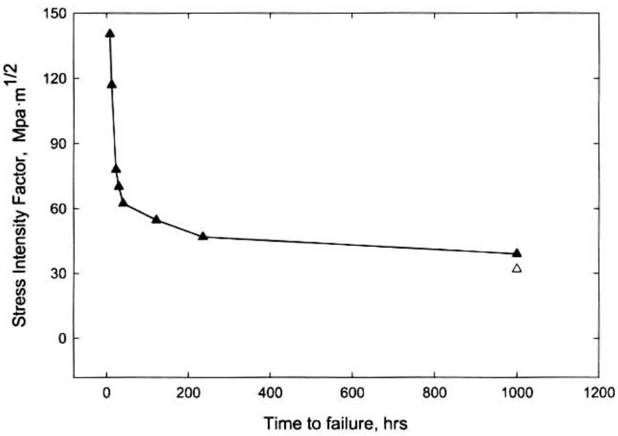

Fig. 5. Applied stress intensity factor vs time to failure plot in constant load test.

used to determine K_{ISCC}. Thus, much higher K_{ISCC} value obtained in constant load testing implies that the proposed test duration of 1000 hrs[3] is not adequate for this material.

The results of stress corrosion crack growth rate measurements are shown in Fig. 6. The crack growth rates varies remarkably with time after the initiation is detected(Fig. 6(a)). The crack propagation is slow immediately after the initiation and then the growth rate increases rapidly followed by a remarkable decrease. Only the slow crack growth observed immediately after initiation is believed to be associated with stress corrosion cracking, which can be confirmed from the record of load-displacement curve. In the time period where the rapid increase in crack growth rate is observed, the load drops very rapidly indicating unstable crack growth occurs by the mechanical fracture, and in the time period of the slow crack growth followed the size of un-cracked ligament is less than 5 mm and the load is almost zero indicative of the final stage of fracture.

The stress corrosion crack growth rate increases with increasing stress intensity factor at crack initiation or increasing displacement rate(Fig. 6(b)). This is expected because the balance between the chemical reaction or the film formation and the crack tip strain rate is lost with the increase in displacement rate or strain rate. Similar observations have been made in earlier investigations[8),9)] on rising load tests. It is also notable that the crack growth rate determined in rising load test is much higher than those in constant load tests as can be seen in Figs. 6 and 7. This reflects the severity of the test condition and confirms an earlier reasoning that the test condition of the rising load test is more severe compared to the constant load test.

For the monitoring of SCC, electrochemical noise were measured during rising load K_{ISCC} tests. During pre-loading at a constant load, the sharp positive going current transients coupled with negative going potential transients are observed repeatedly after 28 hrs. of test as can be seen in Fig. 8. Since the transients are typical of stable pits, this confirms the formation of stable pits at the tip of pre-crack during pre-loading as was expected from fracture surfaces in Fig. 3.

During the main or rising load test followed by pre-loading, fluctuation of mean current and potential is large after 7 hrs of test as shown in Fig. 9. This may be associated with the repeated appearance of anodic dissolution and passivation of newly formed surfaces during micro-crack initiation or propagation as reported in inter-granular stress corrosion cracking.[10] Following the observation of large fluctuation, mean current appears to increase accompanied by a general decrease of mean potential. The sustained increase in current and accompanying sustained decrease in potential has been interpreted as due to the sustained growth of macro-crack.[10]

The value of standard deviation of current as well as potential starts to increase remarkably after 7 hrs of test reflecting the fluctuation of current due to initiation of

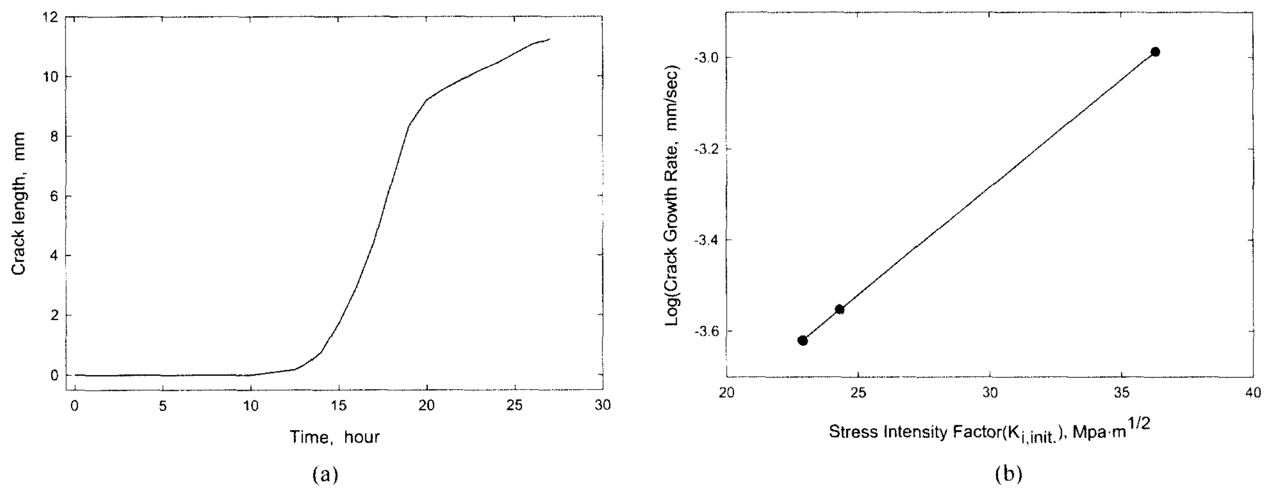

Fig. 6. Plots of crack growth vs time(a) and crack growth rate vs stress intensity factor at crack initiation(b) in rising load test.

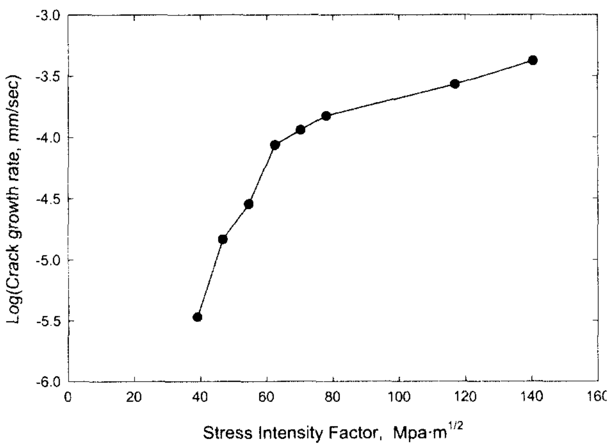

Fig. 7. Crack growth rate vs applied stress intensity factor plot in constant load test.

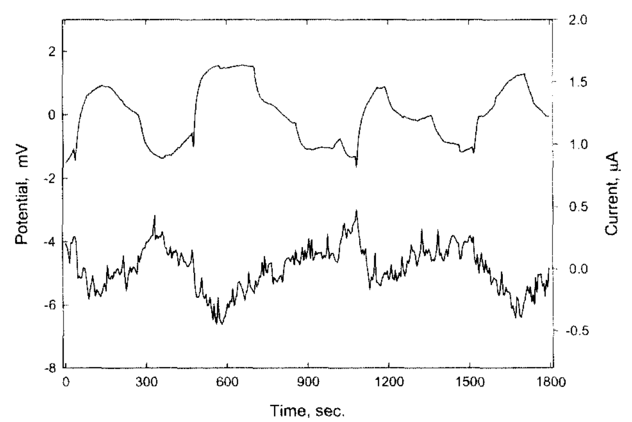

Fig. 8. Current and potential transients typical of stable pit formation observed after 28 hrs of pre-loading.

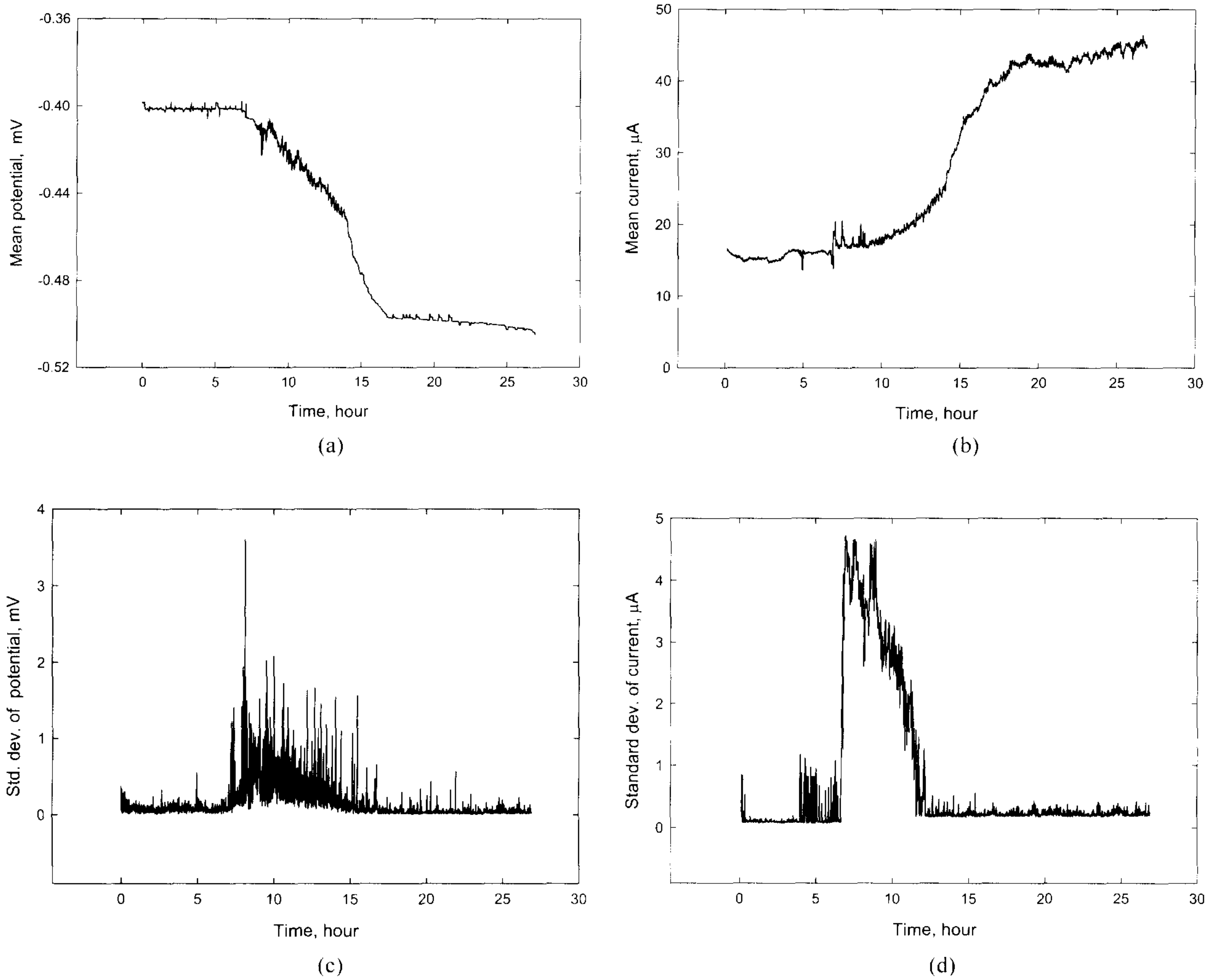

Fig. 9. Typical electrochemical noise parameters vs time plot; mean potential(a), mean current(b), standard deviation of potential(c), and standard deviation of current(d) during rising load testing.

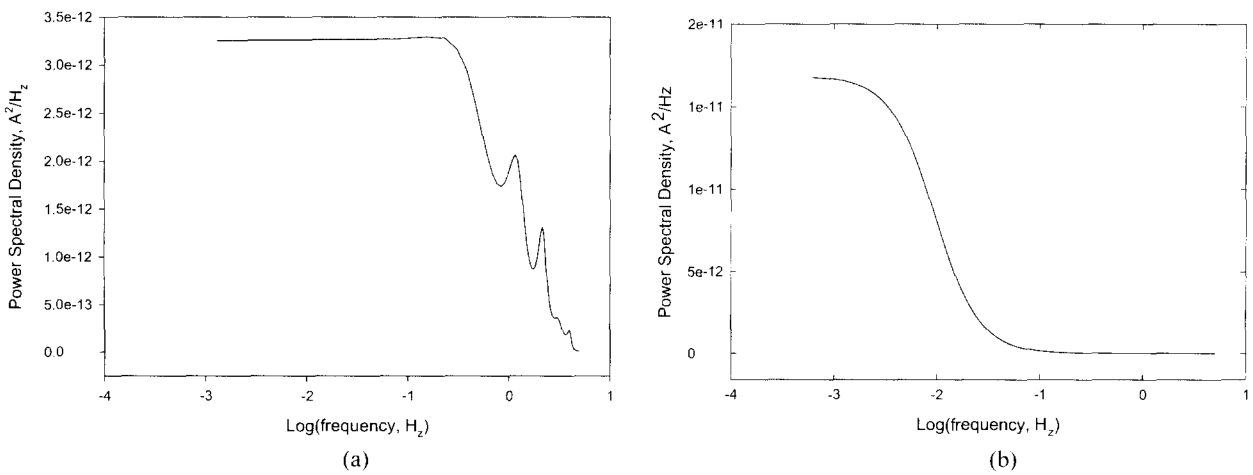

Fig. 10. Results of MEM analysis of electrochemical noise recorded during 30 min after 10(a) and 15 hours of rising load test.

micro-cracks noted above, then starts to decrease after the macro-crack initiation is detected in the load-displacement curve and ACPD data after 12.5 hrs of test. The macro-crack initiation is also marked by a decrease in the load in the load-displacement curve, but as a increase in the potential drop in ACPD data. The decrease in the value of standard deviation indicates the change in mechanism of corrosion, that is, from localized corrosion to general or uniform corrosion[5] after initiation of macro-crack.

For the further analysis, the current-time records were processed with an maximum entropy method(MEM) of order 10. The result obtained during 30 min after 10 hrs and 15 hrs of test are as shown in Fig. 10. The roll-off slope of the PSD plot from the time periods of 30 min after 10 hr is -1 decade/decade, and that after 15.0 hr is -2 decade/decade. In addition the roll off frequency in the former time period is 0.1 Hz and that in the latter time period is 0.001 Hz. This also indicates the occurrence of localized corrosion in the former time period, but general corrosion in the latter time period. Thus the micro-crack initiation as well as macro-crack initiation or propagation is detected during rising load K_{ISCC} test by electrochemical noise technique.

4. Conclusions

Conclusions drawn from the work on the stress corrosion cracking of the pre-cracked specimen of type 403 stainless steel can be summarized as follows.

1) In rising load test, pits were formed at the tip of pre-crack for the pre-cracked compact tension specimen unlike in smooth round specimen with no pre-crack in which only unstable pits were observed and hence immune to SCC.

2) The growth rate of stress corrosion crack increases with increasing stress intensity factor at crack initiation or increasing displacement rate, and micro-crack initiation as well as macro-crack initiation or propagation is detected by electrochemical noise technique during rising load K_{ISCC} test.

3) Lower K_{ISCC} but higher crack growth rate is measured in the rising load K_{ISCC} test compared to constant load K_{ISCC} test indicating test condition of rising load test is more severe than that of constant load testing.

References

1. S. T. Rolfe and J. H. Barsom, Fracture and Fatigue Control in Structures, p.292, Prentice-Hall, Englewood Cliffs, 1983.
2. ASTM E1681-99, Test method for determining a stress intensity factor for environment assisted carcking of metallic materials.
3. ISO 7539-6, Preparation and use of pre-cracked specimens for tests under constant load or constant displacement .
4. ISO/FDIS 7539-9, Preparation and use of pre-cracked specimen for tests under rising load or rising displacement.
5. J. J. Kim, *J. Corros. Sci. Soc. of Korea*, **30**(6), 293 (2001).
6. A. Mysaka and H. Ogawa, *Corrosion/90 paper No 67*, NACE, Houston (1990).
7. J. J. Kim, S. J. Cho, H. Moon, B. S. Han, and H. K. Chang, *J. Mat. Sci. Lett.*, (9) 1 (1990).
8. R. A. Mayville, T. J. Warren, and P. D. Hilton, *Trans. ASME*, **109**, 188 (1987)
9. W. Dietzel and J. Mueller-Roos, *Mat. Sci.*, **37**(2), 264 (2001)
10. D. B. Wells, J. Stewart, R. Davison, P. M. Scott, and D. E. Williams, *Corr. Sci.*, **33**(1) 39 (1992).