TIME DEPENDENT FACTORS IN CORROSION FATIGUE TESTING OF CATHODICALLY PROTECTED LOW ALLOY STEELS

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The effect of long term pre-exposure on the corrosion fatigue behaviour of low alloy steels has been studied in order to assess the reliability of short term testing. Tests were conducted in aerated 3.5% NaCl at a potential of -1100 mV(SCE), using a sinusoidal loading wave with a stress ratio of 0.25 and a frequency of 0.167 Hz. Pre-exposure of AISI 4340 for 21 weeks enhanced the crack growth rates by up to a factor of 7 relative to data from 4 week tests. There was some indication of a small effect for BS4360 50D but the scatter in the data prevented a definite conclusion. Nevertheless it is clear that the magnitude of the effect depends on the sensitivity of the material to hydrogen content. Time dependence of hydrogen uptake must be accounted for in designing tests and relating the data to service conditions.

INTRODUCTION

Testing to evaluate the corrosion fatigue resistance of materials is expensive so there is considerable advantage if data from short term tests can be extrapolated to predict long term behaviour. However this may not be reliable when failure is due to absorbed hydrogen atoms. The uncertainty arises because the main source of hydrogen atoms can be the external surface (bulk charging) rather than the crack tip under a range of conditions (1). Since laboratory test times are usually short in relation to the time required for hydrogen transport through the thickness of the specimen, measured crack growth rates may be lower than those occurring in practice, for which there is sufficient time for full hydrogen charging. The purpose of this study is to determine experimentally the importance of exposure time and to give recommendations for reliable testing based on measurements of the diffusivity of hydrogen in the material. Two low alloy steels with varying strength level and microstructure were tested in 3.5% NaCl solution. This environment was used rather than seawater to eliminate complications associated with calcareous scale and thus allow clarification of the effects due solely to hydrogen uptake.

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EXPERIMENTAL

The materials investigated were AISI 4340 steel with a composition in wt% of 1.82 Ni, 0.77 Cr, 0.75 Mn, 0.43 C, 0.30 Si, 0.25 Mo, 0.02 P, 0.01 S, bal. Fe and BS4360 50D steel with a composition in wt% of 1.42 Mn, 0.35 Si, 0.18 C, 0.03 Nb, 0.02 S, 0.02 P, 0.01 V, bal. Fe. The AISI 4340 steel was heat treated at 840 °C for 1 hour, oil quenched, tempered at 550 °C for 1 hour and air cooled which resulted in a 0.2% proof stress of 1209 MPa and an UTS of 1263 MPa. The BS4360 50D steel was used in a normalised condition with a 0.2% proof stress of 366 MPa and an UTS of 529 MPa. The compact tension specimens were prepared from the parent material with the working direction perpendicular to the plane of the crack. The specimen surface was finely ground except for a central area which was diamond-polished to allow visual measurement of the crack length. The specimen was pre-cracked in air at a frequency of 50 Hz and the load was gradually reduced to the value appropriate to the corrosion fatigue test using a standardised procedure (2). The crack length was increased by at least 0.5 mm at this final load.

The corrosion fatigue tests were conducted in accordance with the draft ISO standard (2). The specimen was enclosed in a perspex cell through which aerated 3.5 wt.% NaCl was recirculated from a 25 l reservoir giving an approximate linear flow velocity of 1.6×10^{-4} ms^{-1} past the specimen. The temperature of the solution was maintained at 22 ± 1°C, and the pH at 8.5 ± 0.2 using a pH regulator. The potential of the specimen was controlled by a potentiostat with a large platinum counter electrode. A two compartment cell separated by fritted discs was used with the solution in the counter electrode compartment vigorously aerated to limit any effects caused by the products of the anodic reaction. The specimen was insulated from the epoxy-coated grips and pins using acetal sleeves and Tufnol inserts. The crack length was measured using a 4 probe DC potential-drop technique.

The specimen was loaded on a servo-fatigue machine. 3.5 wt.% NaCl, prepared from analytical grade reagent in distilled water, was pumped through the cell and a potential of -1100 mV (SCE) was applied on immersion of the lower part of the specimen. A sinusoidal loading wave with a frequency of 0.167 Hz and a stress ratio of 0.25 was applied on immersion of the notch. The tests in air were conducted at a frequency of 50 Hz with the relative humidity in the range 40-45%.

In order to investigate the importance of bulk charging, three sets of experiments were conducted. The first type was a conventional test with a cyclic load applied as soon as the specimen was immersed. The second type involved pre-charging the specimen prior to testing by polarisation at -1100 mV (SCE) in the test environment. On removal from the solution the specimens were immediately immersed in ethanol in an ultrasonic bath then dried in acetone. Within 5 hours the specimens were immersed in 3.5wt.% NaCl and the test was started. The third type of test involved coating the sides of the specimen to limit hydrogen entry.

An estimate of the "K_{ISCC}" of AISI 4340 polarised at -1100 mV (SCE) in 3.5 wt.% NaCl was obtained using the step loading procedure with the stress intensity factor increased daily by 2 MPa $m^{1/2}$. The minimum increase in crack length that could be resolved was 0.08 mm. Hence, for this particular test, crack velocities below 10^{-9} ms^{-1} would not be detected because of the short periods at specific loads, and the threshold is defined in that context.

RESULTS

AISI 4340

The approximate K_{Iscc} of 4340 steel in 3.5 wt.% NaCl at a potential of -1100 mV (SCE) was 22 MPa $m^{1/2}$. Therefore, for a stress ratio of 0.25, K_{max} will be greater than the static load K_{Iscc} for ΔK values above 15 MPa $m^{1/2}$.

The results of the corrosion fatigue tests are shown in Fig. 1 with the broken lines indicating occasions when the cyclic loading was unintentionally interrupted for less than 24 hours. During this time the applied load was less than 1 kN so no crack growth occurred. The crack growth rates were calculated from the crack length vs cycles data using the secant method (2). The incremental polynomial method, also recommended (2), gave equivalent results. The conventional tests in 3.5 wt.% NaCl produced crack growth rates up to an order of magnitude greater than those in air in the Paris region. At higher values of ΔK, the crack growth rates in solution were independent of ΔK and at a ΔK of 24 MPa $m^{1/2}$ they were enhanced by at least a factor of 4 relative to air. Examination of the fracture surfaces of the specimens tested in air showed that the crack front was concave but the crack fronts of the specimens tested in solution were convex ie the crack length was greater towards the sides of the specimen. The standard (2) specifies that the maximum and minimum crack lengths must not differ by more than 10% of the average crack length but this requirement was not met by the specimen from the test that lasted for 4 days. Therefore, ΔK will vary through the thickness of the specimen at least towards the end of the test so the values measured must be considered as average values. The convex crack front suggests that the crack growth rates in 3.5% NaCl are enhanced towards the sides of the specimen. This is consistent with a greater concentration of hydrogen near the external surfaces induced by bulk charging.

Pre-exposing the specimen for 70 days enhanced the crack growth rates in the Paris law region by up to a factor of 3 relative to the conventional tests. At higher values of ΔK, a plateau region was observed and the crack growth rates were increased by a factor of 5. Pre-exposing the specimen for 147 days had a greater effect with crack growth rates increased by up to a factor of 7 in the Paris law region and a factor of 6 in the plateau region. It can be demonstrated using the student t test that despite the small number of tests the differences are statistically significant at a confidence level greater than 95%. The tests with pre-charged specimens were not halted before unstable fracture because the crack growth rates were higher than anticipated. Hence, the shape of the crack front was not observed.

One test involved coating the sides of the specimen with a marine epoxy followed by silicone rubber to limit hydrogen entry. However in practice this did not affect the crack propagation rate and the crack front was convex implying that the epoxy coating was not effective in limiting hydrogen entry.

BS4360 50D

The crack growth rates plotted in Fig. 2 were calculated from the crack length vs cycles data using the incremental polynomial method (2).The conventional tests in 3.5% NaCl produced crack growth rates up to 20 times greater than those in air in the Paris region (Fig. 2). At higher values of ΔK, a plateau region was observed with crack growth rates increased by at least a factor of 30 relative to air at a ΔK of 24 MPa $m^{1/2}$. The crack growth rates of BS4360 50D in 3.5% NaCl were up to an order of magnitude lower than those of AISI 4340 in the Paris region but in the plateau region the crack growth rates were similar for the two alloys. All the specimens of BS4360 50D had concave crack fronts.

The crack growth rates of the specimens pre-exposed for 59 and 60 days were increased by up to a factor of 2 relative to the conventional tests in the Paris law region but there was no effect for the specimen pre-exposed for 111 days. In the plateau region the crack growth rates of the specimen pre-charged for 60 days were enhanced by up to a factor of 3 but there was no increase for the specimens pre-charged for 59 and 111 days. These results indicate that pre-exposure may have a small effect but the scatter in the data prevents a definite conclusion. The final test involved coating the external surfaces of a specimen with gold to limit hydrogen entry during the test but this did not affect the crack propagation rate.

DISCUSSION

The results for AISI 4340 demonstrate that bulk charging significantly increases crack growth rates. An alternative explanation based on hydrogen entry at the crack tip during pre-exposure would seem untenable. The pre-crack would result in a highly alkaline solution with a significant potential drop (3), which would give rise to low charging currents in the absence of strain-induced film disruption (1).

In contrast to AISI 4340 steel the crack growth rates of BS4360 50D are not enhanced significantly by pre-exposure. The effective diffusion coefficient for hydrogen in BS4360 50D in 3.5% NaCl at a potential of -1100 mV(SCE) is 9.0×10^{-11} m^2s^{-1} (4) compared to 8.9×10^{-12} m^2s^{-1} (5) for AISI 4340. Hence the time to charge a compact tension specimen of 22 mm thickness to steady state is 16 days for BS4360 50D relative to 360 days for AISI 4340 steel. Therefore, for BS4360

50D the conventional tests with durations of 21 and 42 days would have been sufficiently long to allow diffusion of hydrogen through the specimen. However, the test with a duration of 4 days produced similar crack growth rates to the longer tests (Fig. 2) and coating the surfaces of the specimen with gold did not affect the crack propagation behaviour. This indicates that the difference in hydrogen content resulting from bulk charging is insufficient to affect significantly the corrosion fatigue behaviour of BS4360 50D. The measured hydrogen uptake in 3.5% NaCl solution at a potential of -1100 mV(SCE) is similar for BS4360 50D and AISI 4340 steels (4),(5). Therefore, the magnitude of the effect of bulk charging on crack growth rates reflects the sensitivity of the material to the hydrogen content.

CONCLUSION

Pre-exposure for 21 weeks prior to testing increased the fatigue crack growth rates of AISI 4340 steel in 3.5% NaCl at a potential of -1100 mV(SCE) by up to a factor of 7 relative to results from 4 week tests. There was some indication of a small effect for BS4360 50D but the scatter in the data prevented a definite conclusion.

The reliability of corrosion fatigue data depends critically on the exposure time. Time dependence of hydrogen charging must be accounted for in designing laboratory tests and relating the data to service conditions.

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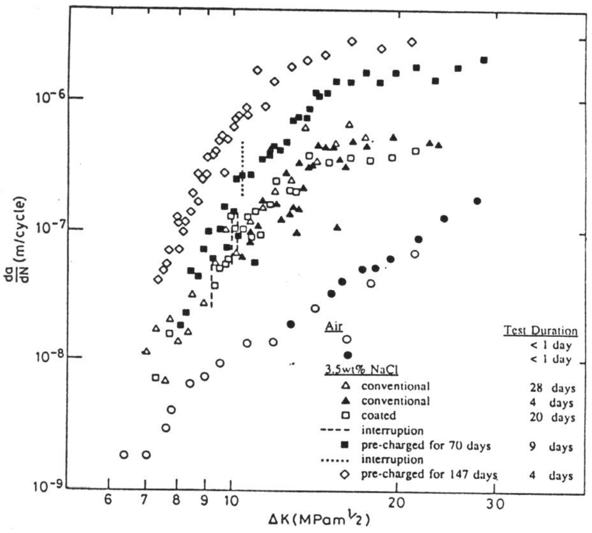

Figure 1 Effect of bulk charging on the corrosion fatigue behaviour of AISI 4340 steel in 3.5wt% NaCl at -1100 mV(SCE) for f=0.167 Hz and R=0.25

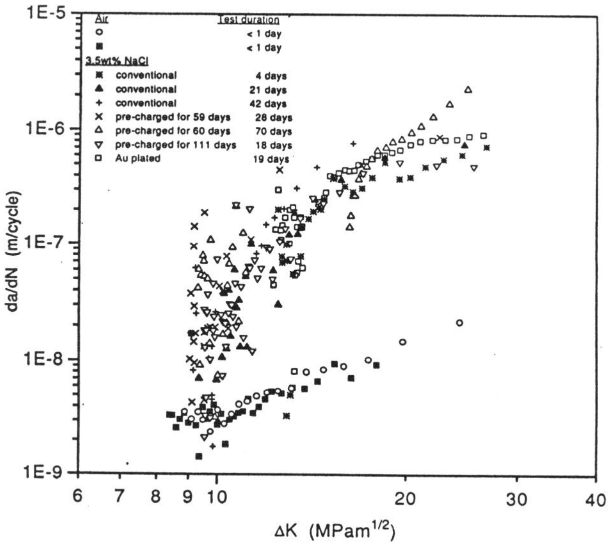

Figure 2 Effect of bulk charging on the corrosion fatigue behaviour of BS4360 50D steel in 3.5wt% NaCl at -1100 mV(SCE) for f=0.167 Hz and R=0.25