THE ROLE OF INCLUSIONS IN CORROSION FATIGUE CRACK INITIATION IN Q1N

R.A. Cottis and A. Markfield*

The role of inclusions in the initiation and early stages of growth of fatigue cracks has been studied for Q1N in sodium chloride solution. The test conditions are such that the effect of corrosion is exclusively the dissolution associated with the crack, with no hydrogen embrittlement. Oxide inclusions are found to initiate fatigue cracks in air, while sulphides are more effective in corrosion. This is attributed to the effect of sulphur species produced by dissolution of the inclusions in enhancing the rate of corrosion of the steel around the inclusion. The addition of thiosulphate spreads the enhanced corrosion over a larger surface area, and thereby reduces the local rates of attack, and transfers the initiation site back to the oxide inclusions. Corrosion is thought to accelerate the growth of the short cracks by the dissolution of microstructural barriers, and by the elimination of closure effects.

INTRODUCTION

The effect of moderately corrosive environments, such as seawater and sodium chloride solution, on the growth of long fatigue cracks in steels has been widely studied, and the observed accelerations have been clearly attributed to the damaging effect of hydrogen produced by the cathodic reaction within the crack. Conversely, the corrosion fatigue crack initiation behaviour, as determined by the measurement of S–N curves on plain specimens, has long been associated with the metal dissolution effects of corrosion, and particularly the effects of pitting corrosion. Recently the actual formation of a crack nucleus in air fatigue has been shown to occur very early in the fatigue life. Thus the results of S–N tests, and even the fatigue limit itself, have been shown to be controlled by the growth of very short cracks. Being of size comparable to the grain size and/or the size of the reversed plastic zone, these cracks do not follow normal long-crack growth laws. Cracks grow rapidly in favourably oriented grains, but are held up at high angle grain boundaries and other microstructural barriers. The absence of closure effects allows short cracks to grow relatively fast, although plasticity- and roughness-induced closure develops quite quickly. As a result of the work of Ritchie, Suresh, Miller, Lankford and others, we now have a reasonably clear view of the factors governing fatigue crack growth from the beginning of an S–N test.

* Corrosion and Protection Centre, UMIST, P.O. Box 88, Manchester, M60 1QD.
Rather little work in this area has considered the effect of corrosion on these early stages of the growth of the crack, notable exceptions being the work of Congleton et al.\(^1\) and Ray et al.\(^2\).

In the work presented here the early stages of growth of corrosion fatigue cracks in Q1N have been investigated, and the results observed related to the pitting behaviour of the steel.

**MATERIAL**

The material used for this study was a sample of Q1N, a steel to a British Naval specification, which is for most practical purposes identical to HY80. The material was supplied in the form of 30 mm plate. It was tested in the as-received, quenched and tempered condition. The material analysis and mechanical properties are given in Tables 1 and 2.

**TABLE 1** — Chemical Composition of Q1N Test Plate

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>S</th>
<th>P</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>Va</th>
<th>Cu</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.15</td>
<td>0.35</td>
<td>0.32</td>
<td>0.007</td>
<td>0.010</td>
<td>2.61</td>
<td>1.62</td>
<td>0.39</td>
<td>&lt;0.05</td>
<td>0.12</td>
<td>0.021</td>
</tr>
</tbody>
</table>

**TABLE 2** — Mechanical Properties of Q1N Test Plate

<table>
<thead>
<tr>
<th>0.2% PS</th>
<th>UTS</th>
<th>Elongation</th>
<th>R of A</th>
</tr>
</thead>
<tbody>
<tr>
<td>610 MPa</td>
<td>730 MPa</td>
<td>23%</td>
<td>69.5%</td>
</tr>
</tbody>
</table>

**EXPERIMENTAL PROCEDURE**

**Test Machine and Specimen Configuration**

The fatigue tests were performed in an Avery—Denison Type 7303 reversed bend fatigue testing machine. This applies reversed bending loading to the specimen, the configuration of which is shown in Figure 1. This machine uses displacement—controlled loading through an eccentric, but a relatively soft spring attached to the other end of the specimen converts the loading into approximately constant stress. The spring also serves as a dynamometer for the measurement of the load applied to the specimen, the displacement of the spring being measured by two dial gauges. The loading frequency for all of the tests reported here was 25 Hz, and the stress ratio was \(-1\).

**Corrosive Environments**

Two corrosive environments were studied:

(a) 0.6 M \(\text{NaCl}\) solution
(b) 0.6 M \(\text{NaCl} + 0.03 \text{ M } \text{Na}_2\text{S}_2\text{O}_3\).
In both cases the solutions were prepared with analytical grade reagents and deionized water. No pH adjustment was made, the initial pH typically being 6.7.

For corrosion experiments the solution was aerated, while for the fatigue tests the agitation induced by the fatigue cycling is assumed to have maintained the solution in a fully aerated condition. All experiments were conducted at room temperature (approximately 20°C).

The solution was maintained around the specimen in a silicone rubber cell. For tests in which the specimen potential was controlled the cell also contained a reference electrode and a platinum counter electrode.

**Crack Growth Measurements**

The initiation and growth of small cracks during testing was monitored by examination of the polished surface of the specimen under the optical microscope. Once cracks had been detected their development was monitored at regular intervals throughout the test. The rather more convenient replication technique was not used because previous work with stress corrosion cracking tests (3) had shown that the replication process seriously disrupted the corrosion behaviour.

**Micro-Corrosion Tests**

In addition to the fatigue tests, polished but unstressed samples of Q1N were exposed to the same test environments for periods of 5 seconds to 5 minutes in order to study the initial stages of the development of localized corrosion around the inclusions. These tests were based on the micro-corrosion tests developed by Noren—Brandel and reported by Wranglen(4). In order to obtain valid results care was taken to ensure that all surfaces of the specimen exposed to the solution were thoroughly polished. Even fine scratches showed a strong tendency for preferential attack.

**RESULTS**

**S—N Curves**

The S—N curves obtained in air and in the two corrosive environments are shown in Figure 2. The S—N curve in air shows the expected behaviour with a fatigue limit of \( \approx 440 \) MPa. In the 3.5% NaCl solution the fatigue life is markedly reduced, particularly at the longer lifetimes. There is no evidence of a fatigue limit, the \( 10^7 \) cycle endurance limit being \( \approx 155 \) MPa. The addition of sodium thiosulphate in the sodium chloride solution resulted, somewhat unexpectedly, in an improvement in the corrosion fatigue behaviour, the \( 10^7 \) cycle endurance limit being raised to \( \approx 190 \) MPa.

**The Effect of Applied Potential**

The endurance at an applied stress amplitude of 490 MPa is shown in Figure 3 as a function of applied potential. It can be seen that for potentials
below about $-900$ mV (SCE) the endurance is essentially the same as that in air. The scatter observed for the tests under cathodic polarization is rather large; the reason for this is uncertain, but it may in part have been attributable to difficulties in ensuring that the cathodic polarization was applied without interruption. The observed effect of applied potential implies that the damaging effect of corrosion does not involve hydrogen embrittlement. This is in agreement with the observations of many earlier workers.

**Crack Growth Rates**

Crack growth rates measured in air and in corrosion with an applied stress amplitude of 530 MPa are presented in Figure 4. Strictly this graph should be regarded as a plot of the crack growth rate at the surface as a function of the surface crack length. In order to provide a comparison with long crack data the maximum $K$ during the cycle ($K_{\text{max}}$) has been calculated on the basis of the simple relationship $K_{\text{max}} = \sigma_a \sqrt{a}$, where $\sigma_a$ = stress amplitude (or maximum stress for this fully-reversed loading). Detailed consideration of the correction factors applicable to the case of a semi-elliptical surface crack suggests that a more accurate value of $K_{\text{max}}$ will be 0.77 times that presented. The use of $K_{\text{max}}$ in place of $\Delta K$ also assumes that the compressive component of the cycle has no effect, i.e. closure occurs when the applied stress is zero.

**Crack Initiation Sites**

In fatigue in air the cracks initiated at persistent slip bands and at oxide inclusions. At high stress amplitudes initiation at persistent slip bands dominated; Figure 5 shows two stages in the development of a crack at a stress amplitude of 590 MPa (97% of the yield stress). An array of persistent slip bands leads to the development of a network of small cracks which propagate by growth of individual cracks, together with crack coalescence. Even in this example, however, inclusions provide a focus for the nucleation of some of the major cracks.

At low stress amplitudes oxide inclusions, primarily alumina, tend to dominate the crack initiation process. Figure 6 shows a crack forming from an alumina inclusion at a stress amplitude of 530 MPa. Cracks also initiated at duplex oxide—sulphide inclusions, but there was little evidence of initiation from sulphides alone.

In sodium chloride solution crack initiation transferred to the sulphide or duplex sulphide—oxide inclusions, with sulphide components of the inclusion generally showing signs of dissolution. Figure 7 shows a crack formed during a test at 490 MPa. The inclusion responsible for the right-hand crack has been lost by corrosion/dislodgement. The inclusion which has nucleated the left-hand crack is a duplex oxide—sulphide. The oxide phase is a rather angular Mg—aluminate, probably an MgO.Al$_2$O$_3$ spinel. The sulphide phase, much of which has been dissolved, is (Mn,Ca)S with a high manganese content. It can be seen that a shallow region of localized corrosion has formed around these inclusions, and relatively sharp, but open fatigue cracks have grown in this region.
In both cases the solutions were prepared with analytical grade reagents and deionized water. No pH adjustment was made, the initial pH typically being 6.7.

For corrosion experiments the solution was aerated, while for the fatigue tests the agitation induced by the fatigue cycling is assumed to have maintained the solution in a fully aerated condition. All experiments were conducted at room temperature (approximately 20°C).

The solution was maintained around the specimen in a silicone rubber cell. For tests in which the specimen potential was controlled the cell also contained a reference electrode and a platinum counter electrode.

**Crack Growth Measurements**

The initiation and growth of small cracks during testing was monitored by examination of the polished surface of the specimen under the optical microscope. Once cracks had been detected their development was monitored at regular intervals throughout the test. The rather more convenient replication technique was not used because previous work with stress corrosion cracking tests (3) had shown that the replication process seriously disrupted the corrosion behaviour.

**Micro-Corrosion Tests**

In addition to the fatigue tests, polished but unstressed samples of Q1N were exposed to the same test environments for periods of 5 seconds to 5 minutes in order to study the initial stages of the development of localized corrosion around the inclusions. These tests were based on the micro-corrosion tests developed by Noren-Brandel and reported by Wranglen(4). In order to obtain valid results care was taken to ensure that all surfaces of the specimen exposed to the solution were thoroughly polished. Even fine scratches showed a strong tendency for preferential attack.

**RESULTS**

**S—N Curves**

The S—N curves obtained in air and in the two corrosive environments are shown in Figure 2. The S—N curve in air shows the expected behaviour with a fatigue limit of \( \approx 440 \) MPa. In the 3.5% NaCl solution the fatigue life is markedly reduced, particularly at the longer lifetimes. There is no evidence of a fatigue limit, the \( 10^7 \) cycle endurance limit being \( \approx 155 \) MPa. The addition of sodium thiosulphate in the sodium chloride solution resulted, somewhat unexpectedly, in an improvement in the corrosion fatigue behaviour, the \( 10^7 \) cycle endurance limit being raised to \( \approx 190 \) MPa.

**The Effect of Applied Potential**

The endurance at an applied stress amplitude of 490 MPa is shown in Figure 3 as a function of applied potential. It can be seen that for potentials
below about $-900$ mV (SCE) the endurance is essentially the same as that in air. The scatter observed for the tests under cathodic polarization is rather large; the reason for this is uncertain, but it may in part have been attributable to difficulties in ensuring that the cathodic polarization was applied without interruption. The observed effect of applied potential implies that the damaging effect of corrosion does not involve hydrogen embrittlement. This is in agreement with the observations of many earlier workers.

**Crack Growth Rates**

Crack growth rates measured in air and in corrosion with an applied stress amplitude of 530 MPa are presented in Figure 4. Strictly this graph should be regarded as a plot of the crack growth rate at the surface as a function of the surface crack length. In order to provide a comparison with long crack data the maximum $K$ during the cycle ($K_{\text{max}}$) has been calculated on the basis of the simple relationship $K_{\text{max}} = \sigma_a^{-\pi a}$, where $\sigma_a$ = stress amplitude (or maximum stress for this fully-reversed loading). Detailed consideration (5) of the correction factors applicable to the case of a semi-elliptical surface crack suggests that a more accurate value of $K_{\text{max}}$ will be 0.77 times that presented. The use of $K_{\text{max}}$ in place of $\Delta K$ also assumes that the compressive component of the cycle has no effect, i.e. closure occurs when the applied stress is zero.

**Crack Initiation Sites**

In fatigue in air the cracks initiated at persistent slip bands and at oxide inclusions. At high stress amplitudes initiation at persistent slip bands dominated; Figure 5 shows two stages in the development of a crack at a stress amplitude of 590 MPa (97% of the yield stress). An array of persistent slip bands leads to the development of a network of small cracks which propagate by growth of individual cracks, together with crack coalescence. Even in this example, however, inclusions provide a focus for the nucleation of some of the major cracks.

At low stress amplitudes oxide inclusions, primarily alumina, tend to dominate the crack initiation process. Figure 6 shows a crack forming from an alumina inclusion at a stress amplitude of 530 MPa. Cracks also initiated at duplex oxide—sulphide inclusions, but there was little evidence of initiation from sulphides alone.

In sodium chloride solution crack initiation transferred to the sulphide or duplex sulphide—oxide inclusions, with sulphide components of the inclusion generally showing signs of dissolution. Figure 7 shows a crack formed during a test at 490 MPa. The inclusion responsible for the right-hand crack has been lost by corrosion/dislodgement. The inclusion which has nucleated the left-hand crack is a duplex oxide—sulphide. The oxide phase is a rather angular Mg—aluminate, probably an MgO.Al$_2$O$_3$ spinel. The sulphide phase, much of which has been dissolved, is (Mn,Ca)S with a high manganese content. It can be seen that a shallow region of localized corrosion has formed around these inclusions, and relatively sharp, but open fatigue cracks have grown in this region.
On the addition of thiosulphate to the sodium chloride solution the crack initiation site becomes less dependent on inclusion composition, with crack initiation at alumina and Ca−aluminate inclusions as well as sulphides and duplex oxide−sulphides. Figure 8 shows an example of crack initiation at an alumina inclusion. The corroded region is covered with a heavy scale of FeS, which makes detailed examination difficult.

Micro−Corrosion Experiments

The micro−corrosion experiments gave results which were consistent with the observations made during the fatigue tests. In 3.5% NaCl pits initiated around sulphide or duplex oxide−sulphide inclusions, particularly the more soluble calcium containing sulphides. On the addition of thiosulphate the pitting was more widespread, and nucleated on minor scratches as well as all inclusion compositions. The depth of attack tended to be less than that observed in the plain chloride solution.

DISCUSSION

Crack Initiation

The tendency for cracks to initiate at oxide inclusions in air may be explained in terms of the higher tessellated stresses (as compared to sulphide inclusions), on the angular inclusion shape. In the corrosive environments it seems clear that the corrosion process enhances crack initiation. In the plain sodium chloride the sulphide inclusions are most active in accelerating the corrosion of the adjacent steel, and consequently in initiating corrosion fatigue cracks. On the addition of thiosulphate the availability of sulphur from the solution leads to more widespread pitting attack, and the factors which control crack initiation sites in air re−assert their importance. However, because the area of active dissolution is increased and the total loss of metal is limited by the rate at which oxygen can be supplied to the metal from the solution, the local rates of corrosion are reduced, and the rate of crack initiation and the early stages of growth are less than for plain chloride solution.

Short Crack Growth

Considering the growth of cracks initiated from inclusions in air, there are known to be two factors tending to restrict growth rates. For microstructurally short cracks the microstructural barriers, principally grain boundaries, hold up or stop the crack growth process. For longer cracks the development of plasticity− and roughness−induced closure leads to a lower growth rate than would be predicted. For the experiments reported here there were indications of delays in the crack growth at intervals of the order of the grain size, particularly over the first few grain diameters. However the frequency of crack length measurement was too low to determine the instantaneous crack velocities in these early stages, and the crack velocities plotted in Figure 4 are averages.

It can be seen from Figure 4 that the corrosive environment affects the growth of fatigue cracks throughout the ΔK range. For very short cracks it is
suggested that the effect of corrosion is to facilitate the passage of the crack through the microstructural barriers. When a fatigue crack growing in air meets a grain boundary, the barrier to continued growth is provided by the misalignment of the crystal structures and the constraint at the crack tip. If the crack is broadened by corrosion the constraint on the crack tip will be reduced, the situation becoming more like that existing at the tip of a relatively sharp notch. This will facilitate the reinitiation of the crack in the new grain. An alternative view of this process is that pitting corrosion produces a defect which is sufficiently large to overcome the microstructural barriers. While this provides a useful quantitative description, as will be discussed below, it should be clear from Figure 7 that the pitting process is heavily modified by the applied cyclic stresses.

At higher crack growth rates, where the crack is no longer significantly influenced by grain boundaries and other microstructural features, it is thought that the effect of corrosion is to eliminate closure effects by dissolution of the crack walls. It can be seen from Figures 5 and 7 that the crack produced in corrosion is much wider than that produced in air. While the crack may be narrower below the surface, it is clear that at least at the surface closure is unlikely to occur, even at the maximum compressive stress. Assuming that the corrosion crack remains open throughout the cycle and the crack closure occurs at zero load in air, it is clear that the effective $\Delta K$ in corrosion will be twice that in air for the stress cycle studied in this work. It can be seen in Figure 9 that the corrosion curve is approximately coincident with the air data if it is displaced to double the $\Delta K$.

Threshold Behaviour

The link between the fatigue limit (or threshold stress amplitude) and the threshold stress intensity range can conveniently be presented in the stress—crack length diagram proposed by Kitagawa (6). Such a plot is shown in Figure 9. The value of the endurance limit, $\sigma_e$ in air from this work is used to define a stress amplitude threshold, while the threshold stress intensity range determined by James and Knott (7,8) is used to define the behaviour at lower stresses. A second line shows $\Delta K_{th}$ modified to allow for closure effects. The solid points plotted on this diagram correspond to the "pit" diameter measured on corrosion fatigue specimens at failure at a range of stresses below the air fatigue limit. It can be seen that these pit depths fit remarkably well on to the Kitagawa diagram. Micrographs of typical pits are shown in Figure 10.

CONCLUSIONS

1. In air fatigue cracks in Q1N initiate primarily at angular oxide inclusions. The crack growth rate is higher than that expected for long cracks in this material. Some p.s.b. initiation is observed at high stress amplitudes.

2. In 3.5% NaCl the fatigue cracks initiate at chemically-active sulphide inclusions and the accelerated initiation is attributed to strain-enhanced corrosion processes.

3. The growth of "chemically-short" cracks in 3.5% NaCl is initially

592
accelerated by the action of corrosion in overcoming microstructural barriers. As the crack grows, corrosion of the crack walls prevents the development of closure effects, giving a continued acceleration of crack growth rate.

4. Below the air fatigue limit the crack length at which there is a transition from the effect of dissolution on the initiation process (or the growth of the microstructurally—short crack), to the effect of dissolution on closure processes for longer cracks may be expressed in terms of the pit diameter necessary to reach the closure—free threshold stress intensity factor.

5. The addition of thiosulphate to the solution results in more widespread anodic regions, thereby limiting the severity of the attack at specific sites. This reduces the rate of corrosion fatigue crack growth.

SYMBOLS USED

\[ \sigma_e = \text{endurance limit (expressed as stress amplitude).} \]

\[ \sigma_a = \text{stress amplitude.} \]

REFERENCES


**Figure 1. Specimen Configuration**

**Figure 2. S-N Curves**

**Figure 3. The Effect of Applied Potential on Endurance**

**Figure 4. Crack Growth Rates**
Figure 5. Crack Development at $\sigma_a = 540$ MPa, air.

Figure 6. Crack Forming from an Alumina Inclusion, 530 MPa, air.

Figure 7. Crack at Oxide--Sulphide Inclusions, 490 MPa, NaCl.

Figure 8. Cracking at Alumina Inclusions, $\sigma_a = 450$ MPa, NaCl/Na$_2$S$_2$O$_3$. 
Figure 9. "Kitagawa" Plot for Q1N.

Figure 10. Pits Associated with Crack Initiation.