

THE EFFECT OF SOLUTION pH ON THE INITIATION AND GROWTH
OF SHORT FATIGUE CRACKS

R.Akid[†] and K.J.Miller[†]

Metals subject to a combination of static or cyclic loading and an aggressive environment may suffer accelerated degradation from the superposition of stress corrosion cracking or corrosion fatigue, respectively on mechanical processes. A comparison of appropriate S-N curves clearly reflects the increasing significance of chemical effects in controlling fatigue life as applied stress levels fall below that of the in-air fatigue limit. Evidence is presented showing the dependence of the important short fatigue crack growth phase on both the microstructure of the material and its test environment. Crack initiation modes are seen to depend upon solution pH. Furthermore assessment of fatigue life based on crack length evaluations alone appears unsuitable and a crack density approach is discussed.

INTRODUCTION

Many recent studies have concentrated on quantifying the behaviour of short fatigue cracks [1] and it is clear that it is this phase of the fatigue process which dominates long-life fatigue at low stress levels. For those materials with a fatigue limit, it has been shown that short fatigue cracks can develop at stress levels below the fatigue limit (2) which has two important characteristics, namely;

1. the limit is associated with the spacing of the dominant microstructural barrier as well as a stress range
2. the limit is only a feature of a constant stress amplitude type of cycle

Short fatigue crack growth behaviour is important when considering environmental effects since these can cause the fatigue limit to disappear, see Figure 1. The probable effect of an aggressive environment on short fatigue crack growth rate is shown schematically in Figure 2, where the growth rate in the environment is seen to be greater than in laboratory air tests. Figure 2 is based on an experimental

[†] Department of Mechanical and Process Engineering
University of Sheffield, Sheffield S1 3JD, England

data-based short fatigue crack growth model developed by Brown, e.g. see (3) using an Elastic Plastic Fracture Mechanics Analyses approach. In this Figure, equation 1, given below, represents growth within grains less than the major microstructural barrier spacing

$$\frac{da}{dN} = A \Delta\gamma^\alpha (d-a) \quad (1)$$

where $\Delta\gamma$ is the shear strain range, a is the crack length, d represents the spacing of major microstructural barriers, and A and α are material constants.

Where stress levels are above the 'in-air' fatigue limit, cracks may propagate beyond the microstructural barrier. In this case crack growth is represented by equation 2

$$\frac{da}{dN} = B \Delta\gamma^\beta a - D \quad (2)$$

In this case D represents a fatigue limit threshold crack growth rate term and B and β are material constants.

The contribution to crack growth through environmental effects has been modelled (4) using the following equation

$$\frac{da}{dN(cf)} = \frac{da}{dN(air)} + \frac{da}{dN(diss)} \quad (3)$$

and

$$\frac{da}{dN(diss)} = \frac{i_{corr} M}{zF\rho} \cdot \frac{1}{\omega} \quad (4)$$

where i_{corr} represents the anodic corrosion current for metal dissolution, ω is the cyclic frequency and z, F, ρ and M are constants. The subscripts (cf) and (diss) in equation 3 represent corrosion fatigue and dissolution respectively.

The most important aspect to be observed from Figure 2 is the removal of the microstructural barrier effect at stress levels below the fatigue limit.

For low stress cyclic loading the greatest proportion of a material's fatigue lifetime is concerned with very small cracks growing at very slow rates. The removal of the fatigue limit is probably associated with a 'tunnelling' effect due to the environment removing the obstruction to crack growth at the microstructural barrier. In Figure 2 this process is represented by a constant dissolution term represented through equation 4. In essence the zero crack growth rate associated with a fatigue limit is replaced by a finite crack growth rate due solely to the aggressive environment.

This paper presents the results of environmental tests conducted on a low-alloy structural steel which show the effects of aqueous chloride environments, of different pH values, on the behaviour of fatigue crack growth below the in-air fatigue limit stress.

MATERIALS AND EXPERIMENTS

The material chosen for this investigation was a low-alloy structural steel, BS4360 50B, its chemical composition (%wt) was C 0.18; Si 0.11; Mn 0.65; P 0.024; S 0.036; remainder Fe. The mechanical properties were as follows; σ_{uts} 540 MPa; σ_y 340 MPa, elongation 24%

Specimens were machined from normalised 'as-received' rolled bar. A shallow hour-glass shaped fatigue specimen was chosen to minimise the area to be analysed by surface replication, whilst providing a low stress and strain concentration at the minimum cross-section. Specimens were subject to fully reversed shear loading to produce surface stage I crack growth. The specimen calibration and replication procedures are recorded elsewhere (4). Environmental conditions consisted of circulating 0.6M NaCl solution, having variations in pH value of 6.0, 3.5 and 2.0, with specimens freely corroding at their rest potential. Evaluation of the electrochemical characteristics of the material were carried out using cyclic voltammetry techniques (5).

The material is composed of ferrite and 14% volume fraction pearlite. Texture was noted in the form of pearlite banding and grain elongation. Grain sizes, determined by mean linear intercept, were 15 μ m and 35 μ m in the longitudinal and transverse directions respectively.

RESULTS

Previous studies on this steel (4) showed a fatigue limit stress range around 325 MPa. Tests conducted at and below 320 MPa gave rise to an infinite lifetime where cracks were seen to initiate grow and arrest at crack lengths not exceeding 110 μ m. Deceleration and arrest of cracks was associated with ferrite-ferrite grain boundaries and ferrite-pearlite interfacial boundaries. The results of the torsional fatigue tests for both air and 0.6M NaCl test environments are given in Figure 1. The results of other tests conducted during this investigation notably variation in solution pH and two-stage air/corrosion and corrosion/air studies are summarised in Table 1

Crack length measurements were taken from acetate replicas and recorded against number of cycles. Metallographic examination revealed a different initiation mechanism for different test solution conditions, see Figure 3. At pH 6.0 cracking was associated exclusively with pit development at non-metallic inclusions. As test solution pH value decreased a mechanism involving dissolution at slip bands replaced the pitting mode of initiation. For near neutral pH conditions the failure crack developed from a single pit and grew in isolation of other cracks which ultimately arrested. At low pH values multiple crack initiation was seen and failure cracks developed as a result of crack coalescence. Detailed examination was made of a 500 μ m² area for tests conducted at pH 3.5 and 2.0. Figure 4 shows how cracks developed during early stages in tests conducted in low pH

solutions. Measurements of cracks in this area were also recorded and plotted as a density function against number of loading cycles, see Figure 5.

Type of test	Stress Range $\Delta\tau$ MPa	Applied cycles	Comments
AF	320	6×10^6 NF	$\Delta\tau < \text{fatigue limit}$
CF	320	915500	pH 6.0, failed
CF	315	960000	pH 3.5, failed
CF	319	529000	pH 2.0, failed
AF/CF	319	4×10^6 air + 950000 CF	Unfailed pH 6.0, failed pit development
CF/AF	289	130000 CF + 2×10^6 air	pH 6.0, unfailed failure in air at sub-fatigue limit stress
AF ; Air Fatigue : CF ; Corrosion Fatigue : NF ; Unfailed			

TABLE 1 Summary of fatigue test results

DISCUSSION

The microstructural characteristics of a material play a significant role in determining the origin and propagation rates of short fatigue cracks. For the material used in this study three different modes of initiation and growth were observed; (i) within ferrite grains, (ii) dissolution at slip bands within ferrite grains and (iii) initiation and growth after pit development at non-metallic inclusion sites. Which of these initiation and growth systems dominates appears to depend upon the nature of the environment, as illustrated in Table 2.

Table 2 shows that a gradual change in crack initiation mode occurs from pitting to slip band dissolution as the solution pH decreases. The reason for this transition may lie in an understanding of the chemical interaction occurring at the metal/solution interface. Since each solution contains the same concentration of chloride ions the effect may be attributed to the

difference in hydrogen ion concentration. Smialowska and Gust (6) showed that when a steel surface was in a passive condition pitting was favoured and that as the protective ability of the surface film decreased cracks nucleated on a smooth surface. In their studies this change was assigned to a modification in composition of the surface layer. It is generally acknowledged that crack nucleation occurs when steel is in its active state which occurs at the bottom of a pit where acidification of the crevice solution results through hydrolysis reactions. When the original solution pH is low, as represented by the test conducted in a solution of pH 2.0, two effects may contribute to a preference for slip band dissolution over that of pitting. Firstly the solubility of the surface film may increase and hence its integrity decrease and secondly an increase in the hydrogen ion concentration of the solution results in an increase in metal dissolution.

Initiation/growth mode	Environment
slip band cracking within ferrite	air
pitting	0.6M NaCl, pH 6.0
pitting/slip band dissolution	0.6M NaCl, pH 3.5
slip band dissolution	0.6M NaCl, pH 2.0

TABLE-2 Crack initiation and growth characteristics of BS4360 50B

Under 'inert' environmental conditions the fatigue lifetime of a smooth specimen is controlled by the applied stress level and the nature of the material's microstructure. The microstructural resistance to crack propagation occurs through barriers such as grain boundaries and interfaces between zones such as ferrite and pearlite. The fatigue limit in these cases is therefore defined as that stress which just allows for crack propagation beyond the dominant microstructural barrier, referred to as the microstructural unit length 'd'.

Where corrosion fatigue conditions prevail crack propagation rates are invariably higher and therefore the generation of a defect, ie a crack, of a size comparable with 'd' is likely to occur at an earlier stage compared to that under 'inert' conditions thereby resulting in a decrease in the fatigue lifetime. A further process observed from present studies is that of multiple crack initiation within adjacent grains which occurred in low pH solutions. In this case individual crack length no longer determines lifetime rather it is the distribution and density of these tiny cracks which needs to be considered in order to develop a predictive model. Figure 4 shows this process of

coalescence where cracking has occurred along the two complementary planes of maximum shear stress. As cracking continues, ie. beyond 300000 cycles it was observed that a major crack began to develop along the longitudinal direction, which, it has been suggested (7), is related to the inherent texture of the microstructure. Fatigue lifetime predictions, based on EPFM analyses (4), were made for both air and environmental tests (pH 6.0). This analysis was based on measured surface crack lengths. Where individual cracks do not grow to any appreciable length, ie. in low pH solution conditions, this approach is no longer suitable and another parameter is required. Exactly which parameter is the most appropriate is not yet clear. The results presented in this investigation do however suggest that where multiple crack initiation, growth and subsequent crack coalescence is observed an analysis based on crack density may be suitable. Figure 5 clearly shows a correlation between crack density and number of cycles₂. This data is based on the measurement of cracks within a 500 μm^2 area of the specimen at different intervals of lifetime. Further study is now necessary to assess the stage at which a crack density function changes to that of a normal crack length function.

Additional tests involving a two-stage approach has shown that pre-cycling in air at stress levels below that of the in-air fatigue limit has little or no effect on the subsequent corrosion fatigue lifetime and that pitting is preferred to slip band dissolution despite the fact that slip band cracks had developed and arrested during cycling in air. Two stage testing involving the application of corrosion fatigue conditions prior to air fatigue cycling suggests that environmental activity is required only to generate a defect having a length equal to or greater than the value of 'd' after which the environment plays a minor role, an observation highlighting the significant influence that the environment has on short fatigue crack growth.

CONCLUSIONS

Fully reversed torsional fatigue tests in both air and 0.6M NaCl solution were conducted using a low-alloy structural steel; BS4360-50B. Crack growth measurements based on a surface plastic replication method showed that the mode of crack initiation was dependent upon the test environment. Three modes of crack initiation were observed, notably, (a) crack growth within ferrite grains, (b) pit development followed by stage I shear crack growth and (c) slip band dissolution along planes of maximum shear stress. Crack growth measurements taken from tests conducted within low solution pH values suggest that a crack density analysis may be a suitable approach for incorporation into existing Elastic Plastic Fracture Mechanics short crack growth models.

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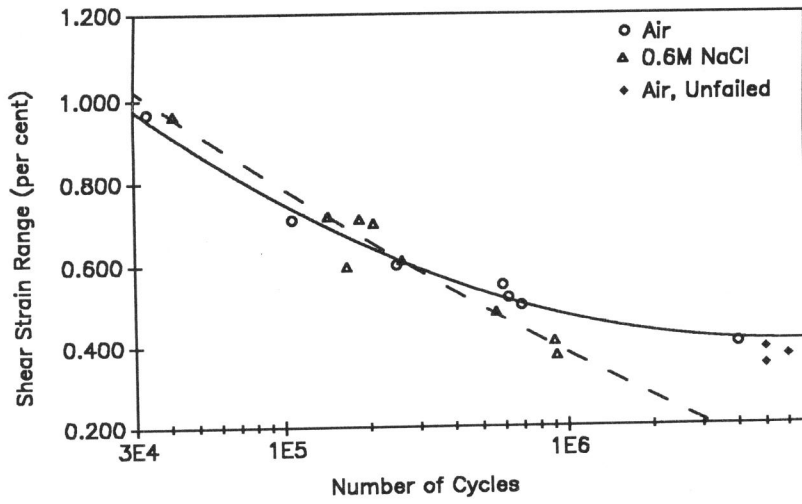


Figure 1 Fatigue Curves for BS4360 50B in air and 0.6M NaCl solution

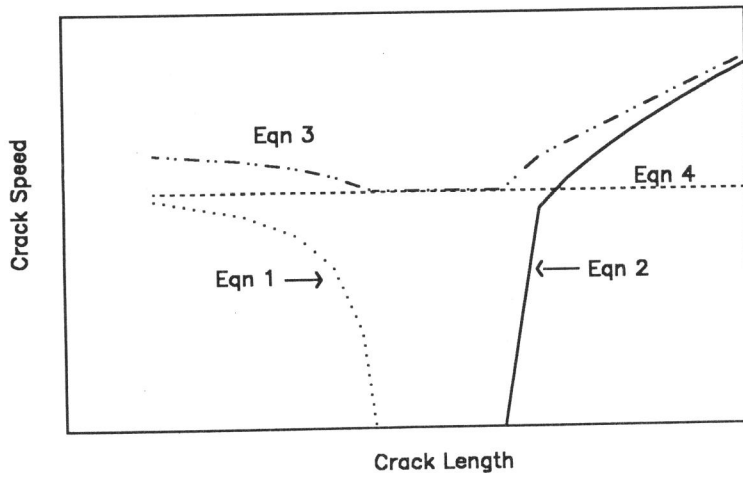


Figure 2 Schematic of models for air and environmental crack growth at shear stress levels below the in-air fatigue limit

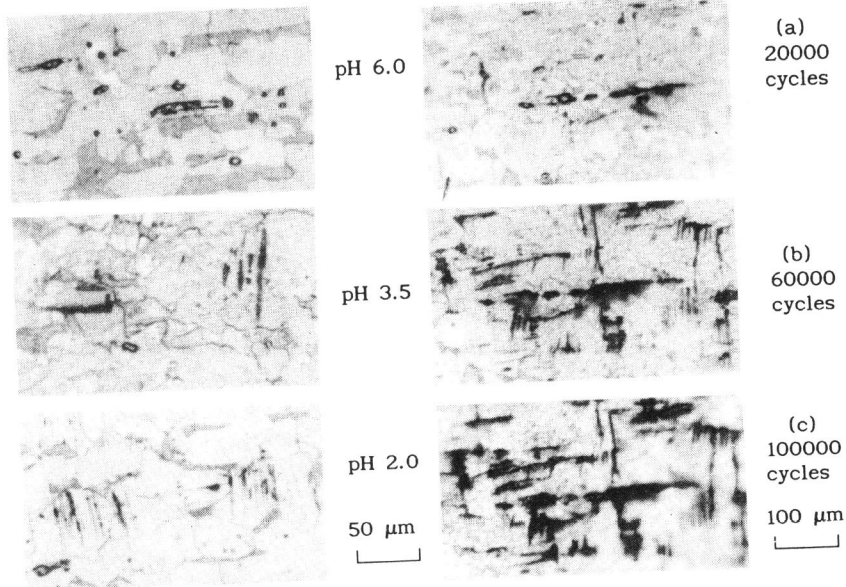


Figure 3 Initiation characteristics after 20000 cycles, $\Delta\tau = 320$ MPa.

Figure 4 Crack coalescence in 0.6M NaCl pH 2.0, $\Delta\tau = 319$ MPa.

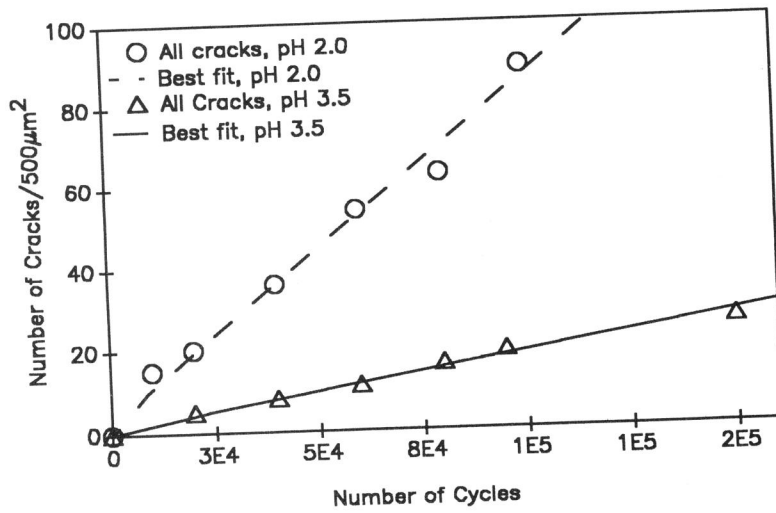


Figure 5 Plot of Crack Density versus Number of cycles at pH 2.0 and 3.5.