FATIGUE CRACK GROWTH RATE IN AIR OF A QUENCHED AND TEMPERED STEEL

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The research work undertaken on crack propagation in air of a structural quenched and tempered steel shows an important influence of the loading frequency. This unexpected result is explained by a strong difference in the thicknesses of the oxide layer formed on the crack surface.

From this work, it is obvious that a great care must be taken in using results obtained in conditions where oxide layers are present on the fracture surfaces, because they probably do not represent the "fail-safe" fatigue crack growth characteristics of the tested material.

INTRODUCTION

Crack propagation of a quenched and tempered steel was studied and tests with different experimental conditions were carried out, in controlled moist air environment. The so-called R-ratio effect was found strongly dependent on the testing frequency. This unexpected effect of frequency was already observed on structural steels by Yokobori and Sato (1), and Noack and Seifert (2). They both discussed this particular effect on the only mechanistic point of view. Recently, the authors (3) discussed the effect of test frequency taking into account of the environmental parameter and especially the effect of the oxide observed on fatigue crack surface, under some experimental conditions.

In the present study caracterisation of the oxide has been performed, with a Secondary Ion Mass Spectrometer (SIMS) and a comparison between crack closure and oxide thickness provide a better understanding of the crack propagation mechanism.

EXPERIMENTAL PROCEDURE

Material and specimens

The material investigated was a quenched and tempered structural steel E 550 in 20 mm thick plate used in the construction of marine structures. This material quenched after hot-rolling and tempered at 625°C for 20 mm has a sorbitic microstructure. Chemical composition and mechanical properties are given in table 1.

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TABLE 1 - Chemical composition (weight %) and mechanical properties.

	C	Mn	Si	S	P	Nb
Chemical analysis	0.145	1.41	0.36	0.001	0.020	0.018

	σ_{ys} (MPa)	U T S (MPa)	Elongation (%)	R in area (%)	KCV (- 40°C) (J/cm^2)
Mechanical properties	640	720	20	77	67

The specimens used for the fatigue tests were of CT type, W = 80 mm, B = 18 mm machined in the LT orientation.

Fatigue testing

Fatigue tests were carried out on an electro-servohydraulic testing machine, operating under load control. Each test was performed with a constant amplitude load (sine wave) and the influence of the two parameters, stress ratio: R, and test frequency : f, was investigated. Two R ratios were chosen : R = 0.1 and 0.7 and in each case two different tests were performed with different load signal frequencies, typically f = 65 Hz and 7 Hz. The following Table 2 indicates the fatigue tests conditions in each case.

TABLE 2 - Fatigue test conditions.

Stress ratio, R :	0.1	0.1	0.7	0.7
Load frequency * ; f (Hz):	65	7	65	7
$K_{initial}$ (MPa $\sqrt{m}$) :	9.5	9.5	6	6

* For the "65 Hz test", when the crack growth rate was higher than $\sim 2 \times 10^{-8}$ m/c the load signal frequency was lowered to 35 Hz.

The crack length was measured with a travelling microscope (x 40). Crack closure measurements were obtained, using a clip-on gage (operating frequency up to 200 Hz without distorsion) or back face strain gages (full bridge with 350 Ω gages) as proposed by Kikuwa et al. (4). Both techniques gave exactly the same results. A numerical oscilloscope allowed measurement at the test frequency. The environment was the air laboratory. Continuous records of temperature and relative humidity close to the specimen showed that for all the tests the temperature was 22°C and the relative humidity was 25 ± 5 %.

Crack surface analysis

The characterisation (nature and thickness) of the oxide was carried out with a Secondary Ionic Mass Spectrometer (SIMS). Although this ion analyser is normally used on polished sections, Benoit et al. (5) first used it successfully on fatigue crack surfaces. For these analysis a primary beam of argon ions was scanned over a 100 µm square, with an intensity I_p = 9 nA. The pressure inside the specimen chamber was 2 x 10^{-7} Torr.

CRACK GROWTH ANALYSIS

Influence of load ratio R and load frequency f

The influence of load ratio on fatigue crack growth for two different test frequencies (f = 65 Hz and 7 Hz) in moist air environment (RH = 25 ± 5 %, T = 22°C) is shown in figures 1 and 2. With the high frequency tests the so-called R effect is observed (figure 1). The higher R ratio give higher crack growth rate especially in the low ΔK region. On the contrary if the loading frequency is decreased enough (f = 7 Hz in the present study) the crack growth rate is largely unaffected, at least above 2 x 10^{-9} m/c, and for a large range of R (from 0.1 to 0.7) the growth rates are not significantly different (figure 2).

The exponent m of the Paris law is m = 2.8 for the R = 0.7 tests independently of the load-frequency. For the R = 0.1 tests the crack propagation law depends on the load-frequency : m = 4.3 for the high frequency test and m $\sim$ 3 for the low frequency test.

The macroscopic aspect of the crack surfaces of the R = 0.1 tests is quite different depending on load frequency as it is shown on figure 3. The micrograph 3 a) shows the specimen tested at low frequency and R = 0.1 with a bright grey fracture surface similar to these of the specimens tested with R = 0.7.

The micrograph 3 b) represents the specimen tested at high-frequency and R = 0.1, with a brown coloured fracture surface. The bright grey bands are related to crack propagation with a lower frequency (f = 1 Hz). The pictures 3 c) and 3 d) represent the specimens tested with R = 0.7 , f = 7 Hz and f = 65 Hz.

Crack closure

The crack closure measurements were carried-out during the propagation tests. Back face strain gages (full bridge with 350 Ω gages or clip-on gages) were used, and measurements at the test frequency were performed, using a numerical oscilloscope with buffer memories. A differenciation technique allowed us to determine accuratly the opening load, as shown figure 5. For the two experiments with R = 0.7, low frequency and high frequency, no closure was detected. For the R = 0.1 experiments, closure was no longer detected for the low frequency test (7 Hz) but for the high frequency test (65 Hz) a significant amount of closure was found. In this last case K_{op} was found constant, for the whole range of ΔK investigated $K_{op} \sim 4.2$ MPa$\sqrt{m}$.

By plotting da/dN versus ΔK_{eff} (figure 6) ($\Delta K_{eff} = K_{max} - K_{op}$) all the results lied down in the same scatter band. So the importance of crack closure on crack propagation mechanism was clearly demonstrated on this material. The crack closure phenomenon as we had already discussed in reference (3) is closely related with the occurrence of the oxide observed on fatigue fracture surfaces.

CRACK SURFACE ANALYSIS

Microscopic aspect

S.E.M.-work was carried out, on crack surfaces. For the R = 0.1 and high frequency test, evidence of a thick layer oxide is shown on figure 4. The microfractograph 4 a) corresponds to a crack growth rate of $\sim 10^{-9}$ m/c ($\Delta K \sim 10$ MPa $\sqrt{m}$). A very dense and partially crackled oxide covered completly the surface and it was impossible in these conditions to see the fracture morphology. Picture 4 b) represents the crack surface of the R = 0.1 and low frequency test for a crack growth of 10^{-8} m/c ($\Delta K \sim 14$ MPa $\sqrt{m}$). Fracture surface consisted of a fine scale transgranular mode with ductile striation.

Oxide caracterisation

A Secondary Ion Mass Spectrometer was used for the oxide caracterisation. The evolution of the ionic intensity versus sputtering time of $^{184}Fe_3O^+$ and $^{57}Fe^+$ that were characteristic of the mass spectra was studied. Typical records are shown figure 7. In a first step we compared our curves with the results obtained by Namdar (6) on a known iron-oxide which had the same ionic spectra that the oxide on the crack surface of the present study.

From this work we could say that a Fe_2O_3 layer was first crossed till the ionic intensity of Fe^+ reached a maximum which corresponds to the interface Fe_2O_3 - Fe_3O_4. Intensity of Fe_3O^+ reached its maximum deeper in the massive Fe_3O_4. Then both Fe^+ and Fe_3O^+ intensity decreased to a steady state level which corresponds to the metal.

Thus the oxide present on the crack surface of the material studied, consisted predominantly of Fe_3O_4 covered by a thin layer of FeO_3.

The second step was to estimate the thickness of the oxide. For this purpose the sputter rate was calibrated with a massive Fe_3O_4 oxide under the same experimental conditions, as discribed previously. The depth of a crater obtained after a given sputtering time was measured by Nomarski interferometry. The sputter rate in our experimental conditions was found to be 3.6 ± 0.5 Å/s in Fe_3O_4. To measure the oxide thickness, the oxide-metal interface was arbitrarily defined as a 90 % decrease of intensity between the maximum intensity and the steady state level (see figure 5).

According to White (7) and assuming only thickness-direction growth, the half of the oxide produced is in excess thickness, thus the oxide thickness measurements give the total excess material in the crack.

For the R = 0.1 and low frequency test the oxide thickness was equal to 400 Å all along the fatigue fracture surface, but for the high frequency test the oxide thickness on the crack surface depended on the crack growth rate or the ΔK level as shown is figure 8.

The uncertain $\Delta CTOD$ calculation and the arbitrary choose of the oxide-metal interface do not allow an accurate comparison of these both values. However if we compare the oxide thickness with the $\Delta CTOD = 0.49\ \Delta K^2/\ 2.\ \sigma_y.E$ (9) (figure 8), obviously for the high frequency test the excess of material inside the crack would strongly modify the crack-tip opening in the low ΔK region.

From this we can assess, that the differences in fatigue crack growth rates due to the change of test frequency are in close relationship with the excess oxide thickness.

DISCUSSION

Oxide formation

The oxide composition we had found, Fe_3O_4 covered by Fe_2O_3 is usual for the ambient air oxidation. In air environment at the atmospheric pressure the oxidation of iron lead to Fe_2O_3. Vernon et al. (8) had shown that for temperature below 200°C the transformation of Fe_2O_3 in Fe_3O_4 was very slow and incomplete ; above 200°C the transformation in Fe_3O_4 is complete and rapid. This last statement should lead us to conclude to a local elevation of temperature. During closure, friction due to the roughness of crack surfaces could rise the temperature at the very surface, just on a few Angström depth. However this hypothesis is very controversial and has not yet been verified.

The mechanism of the crack tip oxide thickening was already discussed by several authors. Benoit et al. (5) proposed an explanation based upon physisorption and chemisorption of molecular oxygen on the steps created by glide along slip planes. The thickening of the oxide film could then arise by a mechanism of fretting oxidation due to friction between the two crack walls in the crack closure zone. Ritchie et al. (10) proposed also that at low R ratio conditions of plasticity-induced closure amplify the fretting oxidation action and that in moist air environment the oxide-thickening enhances the crack-closure.

The crack closure and the oxide thickness measurements in this study, clearly show the relation which exists between closure and excess oxide thickness. Nevertheless if our results have shown that a thick layer of oxide exists only when evidence of closure arose, they have shown also that in air a low R ratio is not a sufficient condition for the crack-closure. The absence of closure and thick oxide on fracture surfaces for R = 0.1, low frequency test remains questionnable.

Frequency effect

In this study evidence of an effect of frequency has been found in conditions where crack closure could exist (i.e. R = 0.1), otherwise when the crack remained fully open we do not detected any significant effect on fatigue crack propagation, which ever the experimental conditions were (at least R and f). That means we have to look for the loading frequency effect through the occurrence of the mechanical crack closure. Modification in crack closure just by changing the test frequency could be explained by different approaches.

Atanaka et al. (11) have found by X-Rays stress measurements on fatigue fracture surfaces, that the level of the residual stress induced by crack tip plasticity was higher on a high strength steel than on a lower strength steel. This is in agreement with the usual results on residual stresses measurements. Noack and Siefert (2) have measured the increase in tensile and yield strength for an increase in strain-rate of a mild-steel and a quenched and tempered steel.

Magnin and Driver (12) also have measured the increase of the cyclic yield strength of b c c iron-based polycrystallin alloy with the increase of the strain-rate.

From these results we can assess than the yield strength of the volume of material at the crack-tip would be higher for the high frequency tests than for the low frequency ones. This rise of yield strength would conduct to higher residual stresses, due to the crack tip plasticity, which lead, behind the crack, to higher residual strains and then more closure.

On a different point of view a physico-chemical approach could also explain the effect of frequency on crack closure. Davidson and Lankford (13) proposed that hydrogen ion, from dissociated H_2O, transported into the lattice by diffusion or by dislocation "sweep-in" (14) reduced strain within the plastic zone. If we consider that this transport is time-dependent and also dependent of the easy motion of dislocations, it comes that a lower test frequency would lead to a lower ductility and a lower surface energy of the material at the crack tip. These assumptions are consistent with the reduction of crack-closure observed in our lower frequency test.

CONCLUSION

Fatigue crack propagation of a quenched and tempered steel has been studied in moist air environment and for different R ratio and test frequency. The rerults show an important influence of test frequency on crack closure and hence on the effect of the R ratio on the crack growth rate.

The fracture surface slide was composed of Fe_3O_4 covered by Fe_2O_3. Measurements of the oxide thickness shows that the crack closure is closely related with the thickening of the oxide.

The influence of the test frequency on crack propagation at R = 0.1 is discussed through to occurrence of a mechanical crack closure.

- The effect of an increased strain rate on strength of the material at the crack tip would lead to higher residual strain behind the crack and then more closure.

- Lower strain rate would enhance transport of hydrogen ion into the lattice and then reduce the surface energy of the material at the crack-tip which conduct to a reduction of the crack closure.

From this work it is obvious that great care must be taken in using results obtained in conditions where oxide layers are present on the fracture surfaces, because they probably do not represent the "fail-safe" fatigue crack growth characteristics of the tested alloy.

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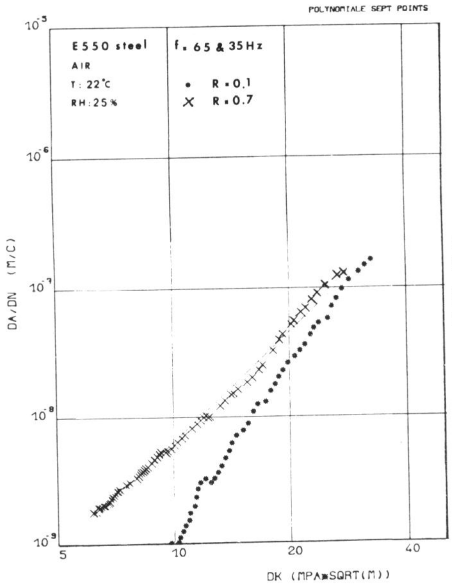


Figure 1 Fatigue crack growth rate at high frequency and different R ratios.

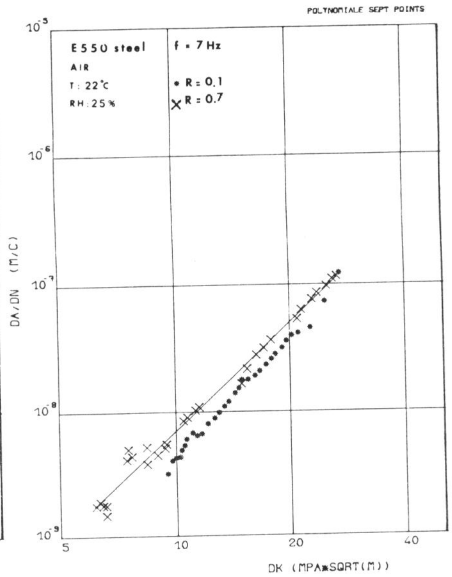
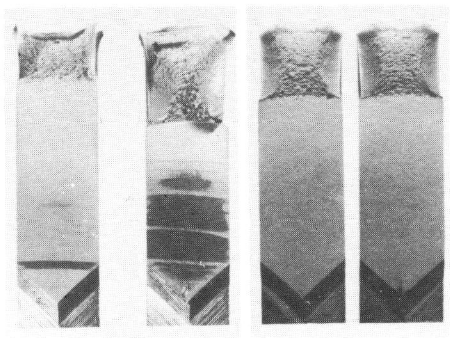


Figure 2 Fatigue crack growth rate at low frequency and different R ratios.

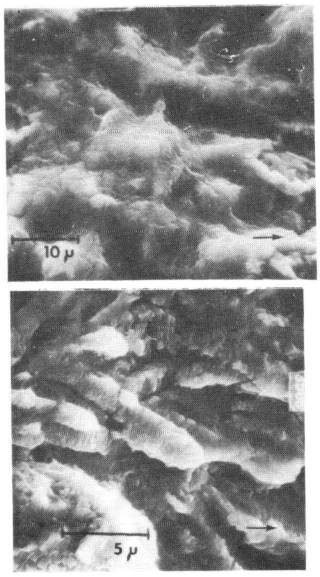


R = 0.1 R = 0.7

f = 7 Hz f = 65 Hz f = 7 Hz f = 65 Hz

a) b) c) d)

Figure 3 Fatigue fracture surfaces in E 550 tested in moist air with different loading conditions.



a)

b)

Figure 4 Fatigue fracture surfaces in E 550 tested in moist air at low K and R = 0.1, a) f = 65 Hz, b) f = 7 Hz.

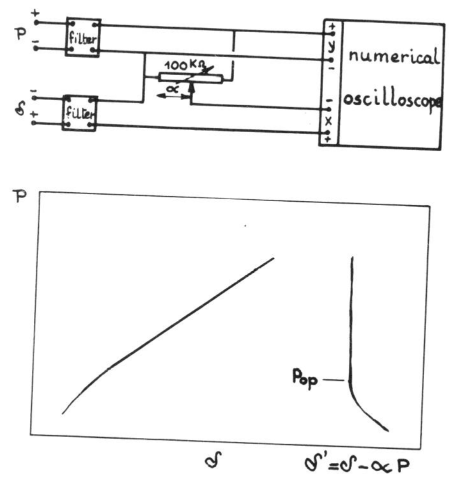


Figure 5 Schematic diagram of the crack closure measurement system.

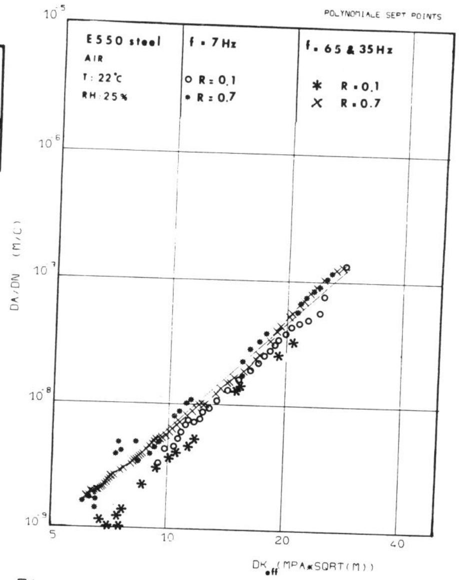


Figure 6 Fatigue crack growth rate, da/dN versus K_{eff} for different values of R and f .

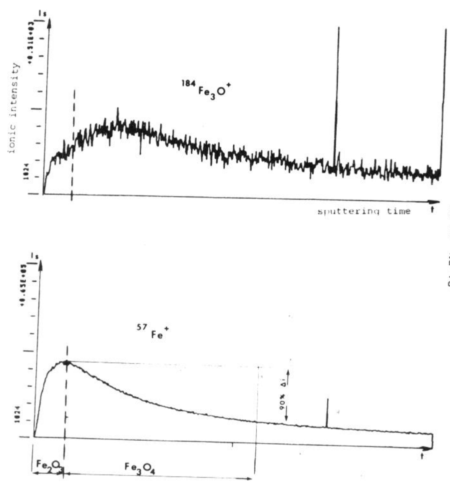


Figure 7 SIMS records and thickness measurement criteria.

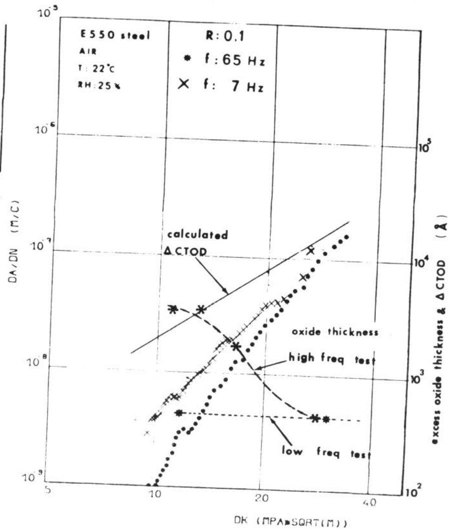


Figure 8 SIMS measurements of crack tip oxide thickness compared with propagation curves.