

FATIGUE OXIDATION INTERACTION IN A Cr-Mo STEEL

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The influence of fatigue cycling on the oxidation kinetic of a Cr-Mo steel has been studied. The number of cycles to crack formation in high temperature low cycle fatigue has been determined using a method based on the measurement of the penetration of oxide into the material, with the thickness of the initial oxide layer at the first cycle assimilated to a microcrack of equal length. It appears that the number of cycles to crack initiation correspond to a microcrack equivalent to the average size of the grain.

High temperature low cycle fatigue is one of the most important solicitation modes that appears in turbo machine parts and the environmental conditions are a key issue in the behaviour of materials against fatigue. The environmental conditions, e.g. an oxidant atmosphere, (1-4) will particularly affect life time, propagation speed, and failure-facies when compared with the same tests performed under vacuum or neutral atmosphere. Although several papers are available on the effect of various gaseous atmospheres, the fundamental principles of the effect of the environmental conditions have not yet been clearly defined. Several authors (5-7), suggested that grain boundaries weakened by oxidation could generate the triggering sites. Under strong distortions, shape alterations appear inside the grain boundaries that cut the free-standing surface of the sample. This results in a natural notch effect. The amplitude of this effect is directly related to the size of the grains. The purpose of this study is to define the fatigue-oxidation interactions by quantifying the influence of mechanical cycling on the oxidation process in high

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temperature low cycle fatigue, with particular emphasis on the influence of the environment on the crack initiation stage of fatigue cracks in relation to the microstructural evolution of the material.

EXPERIMENTAL PROCEDURE

Fatigue tests have been performed on a 10 tons capacity servo-controlled electro-hydraulic traction/compression machine. Test conditions are as follows :

- strain speed : $3.2 \cdot 10^{-4} \text{ s}^{-1}$
- total strain amplitude applied $\Delta \epsilon_t$: 0.74 %, 0.63 %
- temperature : 610 °C, 560 °C

A thermobalance is used to characterize the kinetics of oxidation in air as a function of temperature on materials that have either been exposed to mechanical cycling or not.

The composition of the material tested is listed in table I

Added element	C	Cr	Mn	Mo	Si	Ni	S	P
% of weight	0.22	2.47	0.54	0.42	0.19	0.14	0.025	0.016

Table I

EXPERIMENTAL RESULTS

- Oxidation test with no mechanical load.
 Isothermal oxidation tests have been performed without external mechanical load to determine the kinetics law and the Q_0 activation energy of the oxidation process.

T°C	T°K	k ₀ (g ² /cm ⁴ .s)	k(g ² /cm ⁴ .s)	D ₀ (cm ² /s)	D(cm ² /s)
914	1187	0.516	2.63X10 ⁻⁸	1,89	1.056X10 ⁻⁷
795	1068		3.872X10 ⁻⁹	1,89	1.4X10 ⁻⁹
705	978		6.36X10 ⁻¹⁰	1,89	2.45X10 ⁻⁹
610	883		7.1X10 ⁻²²	1,89	2.78X10 ⁻¹⁰

Table II

Tests results show that the Δm mass variation of the material as a function of time and temperature (figure 1) can be described by the $\Delta m^2 = kt$ law with :

$k = k_0 \exp -Q_0/RT$ and where k is a growth constant. The slope of the $\ln k = f 1/T$ curve gives the value of Q_0 which has been found to be 167 KJ (figure 2).

During this series of tests we have also measured the average thickness of the oxide layer (figure 3). The penetration of oxygen into the material follows a standard Arrhenius law :

$$x^2 = Dt \text{ with } D = D_0 \exp (-Q_0/RT)$$

where D_0 is a diffusion constant and D is the diffusion coefficient at a given temperature. The test results are listed in table II and they are compared with results from other authors (8,9).

- Oxidation test under mechanical load.

The study of the kinetics of oxidation under mechanical loading cannot be performed in situ. However, using the ERISMANN model, we will be able to correlate the number of cycles performed at 20°C with the number of cycles performed at 610°C and 560°C resulting in the same state of damage :

$$\frac{\Delta n_i}{\Delta N_i} = \frac{\Delta n_j}{\Delta N_j} \quad (1)$$

with N_i, N_j the number of cycles resulting in failure at 610°C, 560°C and 20°C being 610, 1400 and 7200 cycles respectively.

Δn_j is the number of cycles performed at 20°C and Δn_i is the equivalent number of cycles at 610°C or 560°C resulting in equal damage.

The evolution of Q , the activation energy as a function of Δn_j , the number of cycles that have been performed at 20°C (and corres-

ponding to $\Delta \epsilon_t$ cycles at 610°C or 560°C can be determined using the curve of figure 2.

Table III lists the available results :

Number of cycles at 20°C	Equivalent number of cycles	Activation energy Q (KJ/mole)
$\Delta \epsilon_t = 0.74 \% \quad T = 610^\circ\text{C}$		
12	1	167
591	50	161
1.180	100	157
2.360	200	146
4.721	400	136
6.492	550	128
$\Delta \epsilon_t = 0.63 \% \quad T = 560^\circ\text{C}$		
13	1	167
1.291	100	160
2.582	200	156
5.164	400	147
10.328	800	133
15.492	1.200	117

Table III

Variation of the activation energy during the mechanical cycling can be described as :

$$Q = Q_0 (1 - CN^\alpha) \quad (2)$$

where :

$$C = 1.8 \cdot 10^{-3} \text{ cycles}^{-1} \quad (T = 610^\circ\text{C} \quad \Delta\epsilon_t = 0.74 \%)$$

$$\alpha = 0.77$$

or

$$C = 1.7 \cdot 10^{-3} \text{ cycles}^{-1} \quad (T = 560^\circ\text{C} \quad \Delta\epsilon_t = 0.63 \%)$$

$$\alpha = 0.73$$

are characteristic constants of the material and N the number of equivalent cycles performed at 610° C and 560° C.

The penetration of oxide into the material can be represented by :

$$x = \left[[D_0 \exp -Q_0 (1 - CN^\alpha)/RT] \cdot t \right]^{\frac{1}{2}} \quad (3)$$

After application of a series of cycles at 610°C with 0,74 % total strain amplitude or at 560°C with 0.63 % total strain amplitude, the length of the largest crack as well as the average thickness of the oxide layers have been measured ; the latter value can be compared with the value obtained using equation (3) with respect to the 1 hour preliminary heating time of the sample in the furnace.

The results are listed in table IV

Number of cycles	Length of the maximum measured crack (μm)	Calculated oxide penetration (μm)
$\Delta\epsilon_t = 0.74\% - 610^\circ\text{C}$		
1	-	9.6
50	-	15.7
100	13	20.5
200	45	45.2
400	125	100
550	210	183
610	2.000	-
$\Delta\epsilon_t = 0.63\% - 560^\circ\text{C}$		
1	-	4.4
100	-	8.4
200	10	12.9
400	25	19.8
800	160	94.6
1.200	325	243

Table IV

The study of the kinetics of the oxidation laws during mechanical cycling has been complemented by metallurgical observations which prove the penetration of oxide into the material down to the bottom of the crack (figure 3-4).

DISCUSSION

In figure (4) it is noticeable that cracks originate from the oxide layer and then progress into the material. Our hypothesis will be that the oxidation layer can be assimilated to micro-cracks of equal thickness that will be cracked during the mechanical cycling. The length 'a' of the crack will be equal to the sum of 'a_i', the measured length of the crack and 'a_o', the initial oxide layer thickness. This value will be compared with 'x', the penetration of oxide into the material as calculated from (3) (figure 5).

The two curves are merged up to a number of cycles of around 70 (610°C - 0,74%) and 230 (560°C - 0,63 %) which corresponds to a thickness of the oxide layer of around 18 μm material. Beyond 70 cycles, the length of the crack becomes greater than the oxide thickness. This point where the two curves diverge can be considered as the crack initiation point.

Remarkably enough, many authors have defined the priming point as being a crack length equivalent to the average diameter of the grains. In addition, the evolution of the load F has also been recorded during the mechanical cycling (figure 6). As the variation of Q, the activation energy of the oxidation process as a function of the number of cycles applied is known from relation (2), (figure 7), it is possible to plot $F = f(Q)$ (figure 7).

The plot shows a slow decrease of the activation energy followed by a faster decrease. The slopes' intersection as determined on the curves $F = f(Q)$ corresponds to an energy of activation of around 160 and 156 KJ, 70(610°C - 0,74 %) and 230(560°C - 0,63 %). It also corresponds to a number of mechanical cycles of around 64° which is fairly close to the number of cycles to the point of crack initiation as defined previously (figure 8).

The curve on figure 9 shows the evolution of the length of the cracks to the rupture of the material. There are three different stages :

- stage 1 : initiation of fatigue cracks
- stage 2 : stable propagation
- stage 3 : unstable propagation.

CONCLUSION

The number of crack initiation cycles in high temperature low cycle fatigue has been determined using a method based on the measurement of the penetration of oxide into the material, with the thickness of the initial oxide layer at the first cycle being

assimilated to a microcrack of equal length. The number of cycles resulting in triggering corresponds to a microcrack equivalent to the average size of the grains.

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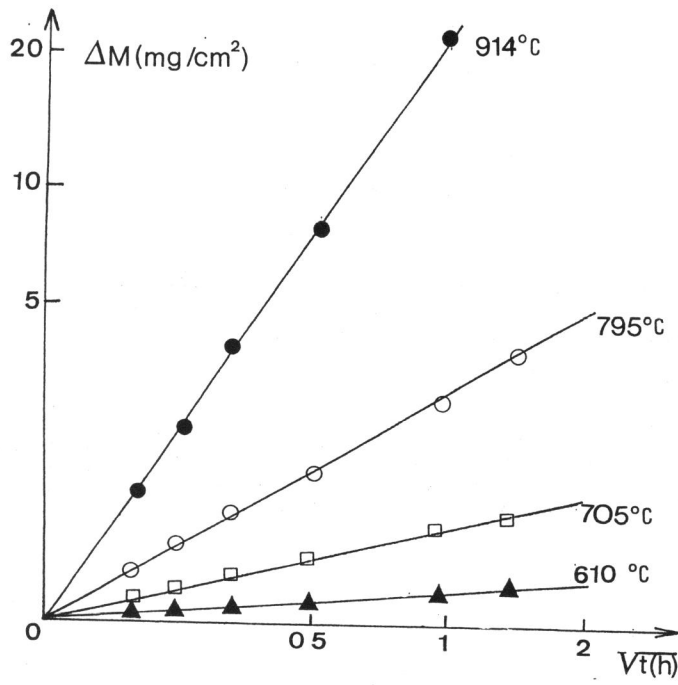


Figure 1: Typical results of oxidation.

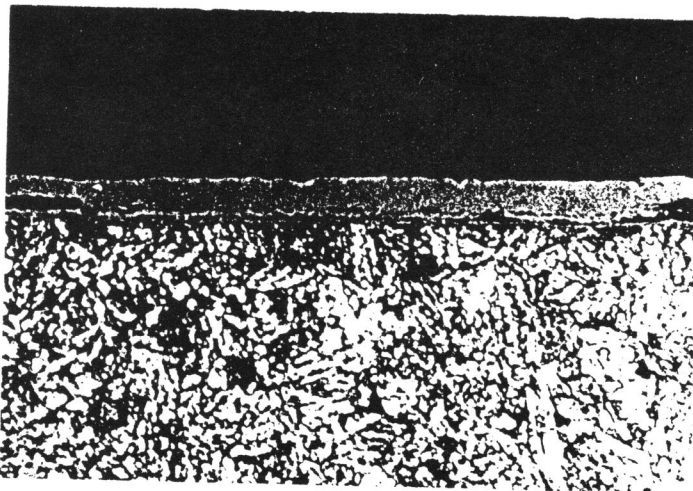


Figure3: Cross section of a specimen oxidized for 1 hour at 610°C

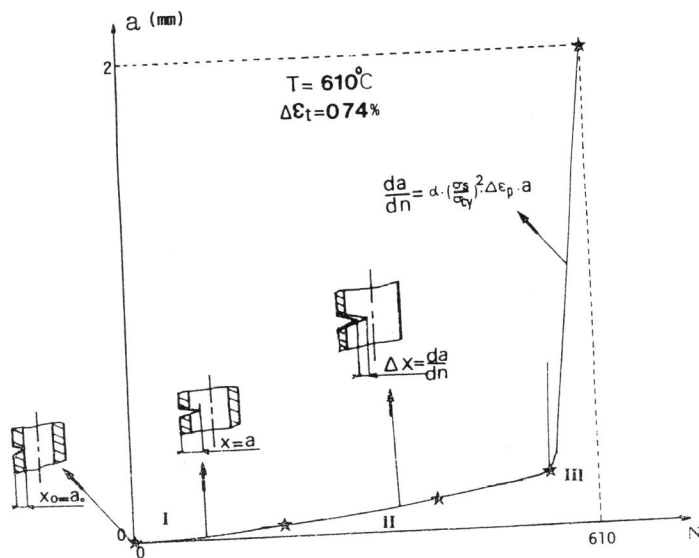


Figure 9: Evolution of crack length under mechanical cycling.