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Christophe Daffos, Pascal Lamesle, Farhad Rezai-Aria. Fatigue-oxidation interaction models for life prediction of hot forming tools steels under transient thermomechanical loadings. *International Journal of Microstructure and Materials Properties*, 2008, 3 (2-3), p.350-362. 10.1504/IJMMP.2008.018740 . hal-01851426

HAL Id: hal-01851426

<https://hal.science/hal-01851426v1>

Submitted on 28 Oct 2020

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Fatigue–oxidation interaction models for life prediction of hot-forming tools steels under transient thermomechanical loadings

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Abstract: This contribution deals with the life prediction of the virgin and nitrided hot-working tool steels X38CrMoV5 (AISI H11, 47 HRC) under Transient Thermomechanical (TMF) loading. Isothermal Low Cycle Fatigue (LCF) and thermomechanical (TMF) behaviour are investigated at different temperatures and various frequencies. It is shown that nitration can enhance the fatigue life only when the strain amplitude is lower than a critical value. Two Manson-Coffin laws govern the fatigue life of the virgin and nitrided steels for all temperatures and strain rates investigated. However, these models have shortcomings for predicting TMF life. Predictive fatigue life models take into account the interaction with oxidation. Regarding the second tempering temperature (550°C) and the steel surface (virgin or nitrided), a Paris-type ($T > 550^\circ\text{C}$) or a Tomkins-type ($T < 550^\circ\text{C}$) law is used. A modified Tomkins model considering the mean residual stress measured by X-Ray at high temperature in the nitrided layer is used. The model is applied for isothermal and thermal mechanical fatigue experiments performed and reported in literature.

Keywords: high-temperature fatigue-oxidation; life prediction; nitriding steels; hot-forming tools.

Reference to this paper should be made as follows: Daffos, C., Lamesle, P. and Rézai-Aria, F. (2008) 'Fatigue–oxidation interaction models for life prediction of hot-forming tools steels under transient thermomechanical loadings', *Int. J. Microstructure and Materials Properties*, Vol. 3, Nos. 2/3, pp.350–362.

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1 Introduction

The surface of hot-forming tools is a special area for damage accumulation and thus a limiting factor in the life of tools. The surface of tools is extremely solicited and damaged under wear–fatigue–oxidation interactions (Rézaï-Aria, 2002). It is thus necessary to get better insight into the damage mechanisms under cyclic transient thermal and mechanical loadings. The surface of the forging tools are in most cases nitrified in order to reinforce the surface tools. The nitriding consists in introducing the nitrogen on the surface of steels by appropriate thermochemical treatments (Daffos, 2004). Depending on the process and treatment conditions (time, temperature, *etc.*) two layers can be observed. On the extreme surface of tool steels a layer of about 15 μm thickness (white layer) with a hexagonal compact crystallographic structure and a second layer of about 250 μm (diffusion layer) are formed. Different carbo-nitrides with different crystallographic structures are formed. Very limited investigation has been devoted to the fatigue behaviour of 5% CrMoV-nitrified steels, widely used in forging for example. It is already reported that the oxidation plays an important role for crack initiation and propagation. Very little is known about the role of the nitride layer/oxidation interactions on fatigue behaviour and life. Within the framework of the French programme, SIMULFORGE, life-predictive models for transient thermal-mechanical loading were developed. This project was a national project with the forging industry and academic partners (with financial support from the French Ministry of Industry). Its goal was to develop new simulation tools for the optimisation of the forging process taking into account the parameters of the material (metallurgy, residual stress, heat treatments, *etc.*). This contribution deals with an investigation on high-temperature isothermal LCF behaviour and fatigue life prediction of a nitrified and virgin X38CrMoV5 (47 HRC). Isothermal LCF tests have been performed at temperatures ranging from 200°C to 700°C. Crack initiation and propagation mechanisms are studied by SEM. Oxidation kinetics on surface and on crack lips are also assessed.

2 Experiments and material

2.1 Material

A double-tempered martensitic steel X38CrMoV5 (AISI H11) with 47 HRC is investigated. Its composition, as shown in Table 1, and microstructure are investigated in Siaux (1994).

Table 1 Chemical composition of X38CrMoV5

<i>Chem. Elt.</i>	<i>C</i>	<i>Cr</i>	<i>Ni</i>	<i>Mn</i>	<i>Si</i>	<i>V</i>	<i>Mo</i>	<i>Other</i>	<i>Fe</i>
Wt%	0,4	5	0,2	0,5	0,9	0,5	1,2	2	bl.

Complex nitrated layer of 200 to 230 μm thickness is deposited by NH_3 gas cracking for 12 hours at 550°C . Metallographic investigations have shown that carbonitrides are precipitated parallel to the diffusion front of the nitrogen during the nitriding process (Daffos, 2004). It should be noted that the nitrated layer is actually not a monolithic but a gradient material in terms of its chemical composition and crystallographic structures (Barrallier, 1992). Moreover, one part of the nitrogen atom is consumed in chromium precipitates (Barrallier, 1992).

2.2 Experiments

Fatigue experiments are performed on a push–pull closed loop servo hydraulic machine (Schenk 250kN). The specimens are heated by a 2 kW induction facility (100–400 kHz). Solid specimens with 11 mm diameters are used. Gauge lengths are mechanically polished down to 1 μm . Temperature is measured by three flattened thermocouples pulled by springs on the gauge. Total strain is monitored by a high-temperature extensometer. The temperature and strain amplitude ranges are from 200°C to 700°C and from 0.4% to 1.2%, respectively. Most of the experiments are performed under 1Hz frequency, but 0.004Hz and 0.05Hz are also tested.

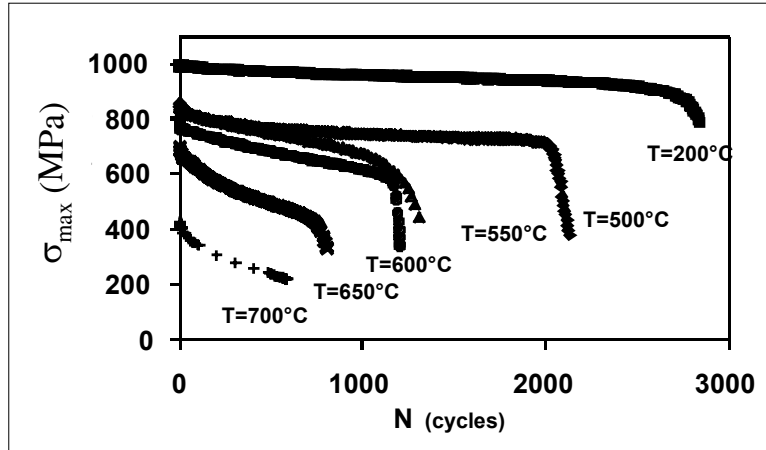
The fatigue striations observed on the fracture surface are used for evaluation of crack propagation rate.

3 Results and discussions

3.1 Cyclic behaviour

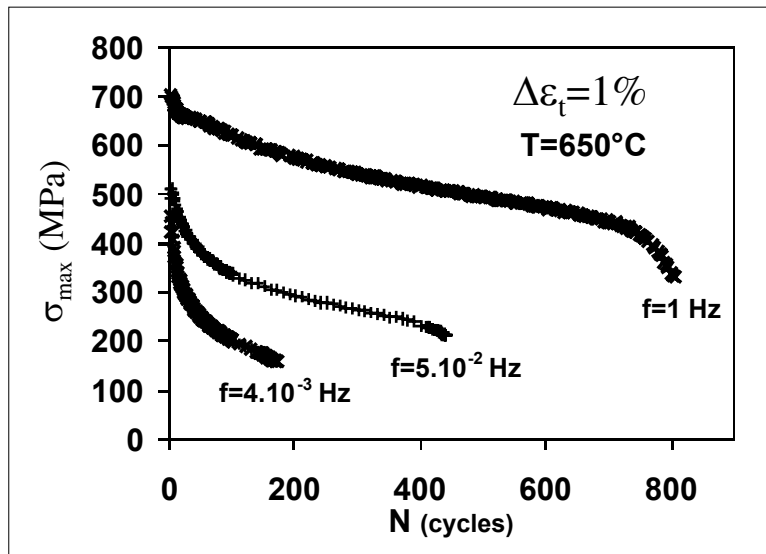
Both nitrated and virgin steels soften under LCF conditions. The softening is observed under isothermal LCF, thermomechanical (Delagnes *et al.*, 1999) and thermal fatigue (Miquel, 1998) for virgin steel. Two softening regimes and a third regime corresponding to the macro crack propagation are reported (Delagnes *et al.*, 1999). The softening is explained by material accommodation owing to microstructure evolutions (dislocation density and arrangement). Such accommodations are reported on different tempered martensitic and ferrite-martensitic duplex steels (Rousseau *et al.*, 1975; Filacchioni *et al.*, 2000). The effect of the temperature (from 200°C to 700°C) on cyclic softening is reported in Figure 1 as the reduction of the stress amplitude versus cycles.

Figure 1 Cyclic softening of nitrided steel as a temperature function



The cyclic softening increases when the frequency decreases (example, 650°C and total strain amplitude of 1%) (see Figure 2).

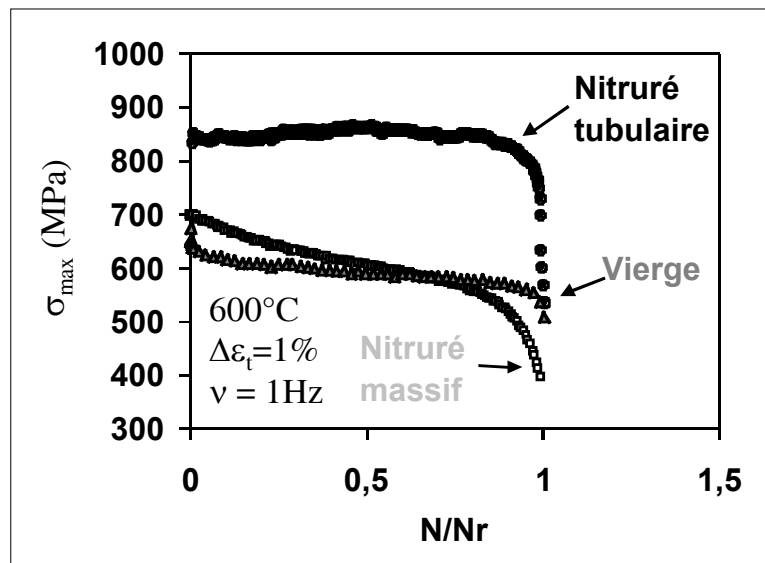
Figure 2 Cyclic softening of nitrided steel as a frequency function



The cyclic softening is similar in virgin and nitrided steels when solid specimens are used. In fact, the nitrided surface supporting the applied load is small in comparison to the virgin part. Therefore the macroscopic (or global) behaviour is not drastically altered in comparison to virgin steel. A very similar softening trend is observed on the virgin tubular specimen in comparison to the solid specimens. On the contrary, in a nitrided steel tubular specimen with an approximately 40% to 50% of the total wall covered by the nitrided layer, cyclic softening is no longer similar to the solid specimen

(see Figure 3). Under 1 Hz frequency, no softening is observed, while under 0.004 Hz, softening is observed. This experimental evidence, shows the drastic modification that the nitrated layer can exert on the local (microscopic) behaviour and softening of the steel. When the frequency is low, the virgin steel presents a higher inelastic behaviour and thus the softening is seen in tubular specimen.

Figure 3 Comparison between virgin and nitrated massive specimen and nitrated tubular specimen



3.2 Crack initiation modelling

Based on cross-section observations, the crack initiation is modelled considering fatigue oxidation interactions. The crack initiation mechanisms of nitrated steel are different depending on whether temperature is higher or lower than 550°C.

Beyond 550°C, cracks are decorated by stratification of the oxide layer (Daffos *et al.*, 2002) as shown in Figure 4. Such oxidation aspect was already reported for low chromium steel contents (Sehitoglu, 1990). The critical length of the crack is given by the following equation:

$$a_c = \delta_{oxide}^{nitrated} \cdot \frac{t}{t_c}$$

with

$$t = \frac{N}{\nu}$$

where N is the number of cycles and ν is the frequency of the cycle.

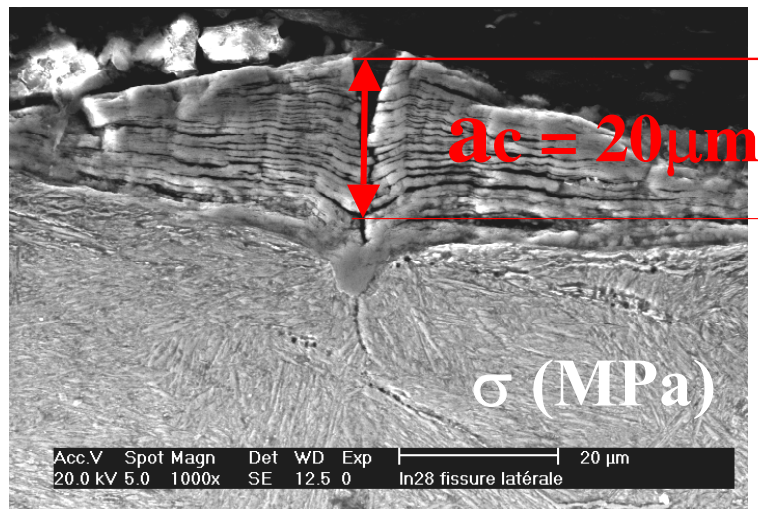
By this way, the number of cycles to initiate a wedged micro crack of about 20 μm depth is estimated:

$$N_a = \frac{a_c \cdot \nu}{\delta_{oxide}^{nitrided}} \cdot t_c$$

with

$$\delta_{oxide}^{nitrided} = K \exp\left(\frac{-Q}{RT}\right) \cdot \sqrt{t_c}$$

Figure 4 SEM image of stratified micro crack



The proposed model allows the number of cycle N_i to be estimated to initiate a wedged defect or micro crack of about 20 μm depth. It uses the oxidation kinetic constants (K and Q), the critical time t_c required to break an oxide layer and the test frequency ν in the following expression:

$$N_i = \frac{a_c \cdot \nu}{K \exp\left(\frac{-Q}{RT}\right)} \sqrt{t_c}$$

Nitrided layer cracks are fulfilled with the stratified oxide layers and crack lips are decorated with Cr-depleted zone. Such oxidation/fatigue interaction and the relative material alteration are frequently reported in the literature (Remy, 1996). Figure 5 shows the affected zone near the crack with oxygen. The relation between the thickness of this oxidized zone and the crack length was used for calculating the number of cycles required for a crack to propagate through the nitrided layer (Daffos, 2004). A constant mean fatigue crack propagation rate of about 2.8 μm per cycle was estimated in nitrided layer ranging from 20 μm to 300 μm (temperature beyond 550°C). No such oxidation mechanism was observed in virgin steel cracking.

Figure 5 X-ray element map of oxygen of affected zone near crack edge



Note: The dark area contains oxygen atoms, the white zone corresponds to nickel plating.

For temperature lower than 550 °C, it is supposed that cracks are initiated through the nitrated layer at the first cycle.

3.3 Crack propagation modelling

Beyond 550 °C, the fatigue striation is observed both in nitrated and virgin steel. Assuming that one striation corresponds to one cycle (Laird, 1967; Oudin, 2001), the fatigue striation spacing accounting allows estimating the crack propagation rate. A Paris law relating the crack propagation rate to the stress intensity factor (Paris and Erdogan, 1963) could be used (Figure 6):

$$\frac{da}{dN} = C_{glob} \cdot (\Delta K)^m$$

where C_{glob} was divided into three corrective factors.

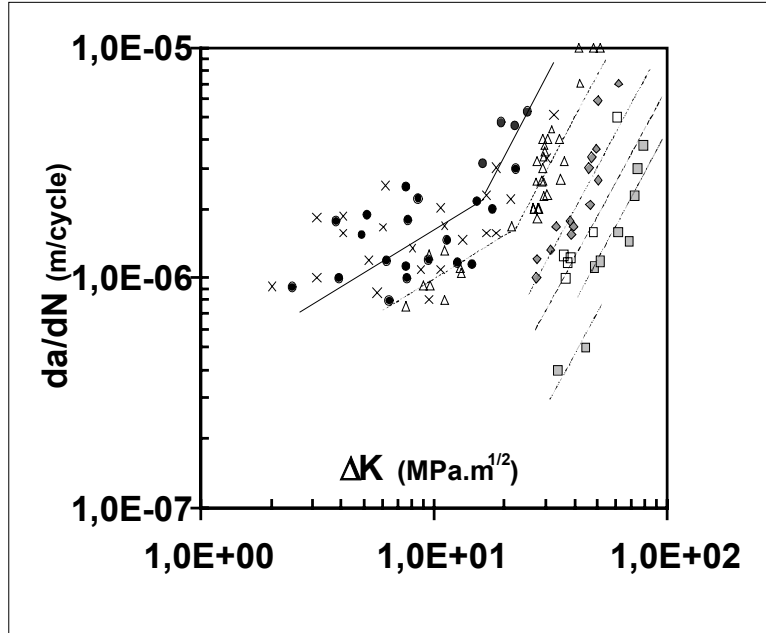
The oxidation and frequency effects are introduced via two first additional corrective factors C_v (frequency) and C_{oxi} (oxidation constant) as:

$$C_v = \alpha \left(\frac{1}{\nu} \right)^\beta$$

and

$$C_{oxi} = K \exp\left(\frac{-Q}{RT}\right).$$

Figure 6 Variation of cracks propagation rates as the function of stress intensity factor for our isothermal LCF test (Paris-type law)



The third factor, C_{mech} is added for taking into consideration the crack opening time in a fatigue cycle, which depends upon the loading (isothermal, or non-isothermal) and loading ratio (Daffos *et al.*, 2002):

$$C_{mech} = \frac{\varepsilon_{p_{min}}}{\varepsilon_{p_{max}}} \cdot f_{OpenTime}$$

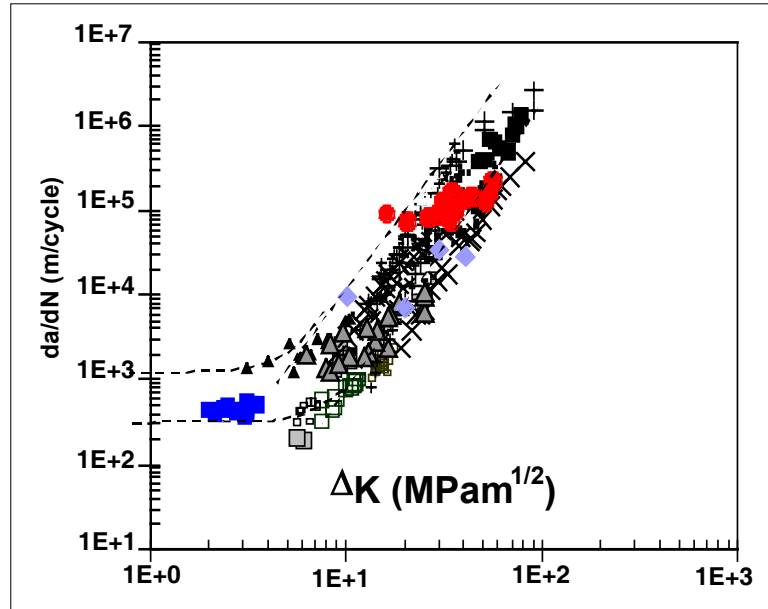
$f_{OpenTime}$ is estimated by a video microscope recording during the fatigue test.

This modified Paris law can rationalise all isothermal LCF and TMF results obtained in these investigations and those reported in the literature (Figure 7):

$$\frac{da}{dN} = \alpha \left(\frac{1}{\nu} \right)^\beta \cdot C_{oxi} \cdot \frac{\varepsilon_{p_{min}}}{\varepsilon_{p_{max}}} \cdot f_{OpenTime} (\Delta K)^m$$

It should be emphasised that depending upon the virgin or nitrided steel, the appropriate oxidation constant C_{oxi} is used. Under test conditions examined, the striation accounting for short cracks (crack length lower than 100 μm) is difficult. Therefore, for crack depth ranging between 20 μm to 1000 μm , it is assumed that the crack propagation cycle, N_p , can be estimated by this modified Paris law.

Figure 7 Modified Paris law applied on all LCF tests presented in Figure 6



For temperature lower than 550 °C, no drastic oxidation effect could be revealed and therefore, the Paris-type law is replaced with a modified Tomkins law (Tomkins, 1968):

$$\frac{da}{dN} = 0,51 \cdot \Delta \varepsilon_p \left[\sec \left(\frac{\pi \sigma}{2 \sigma_0} \right) - 1 \right] \cdot a.$$

This crack propagation model uses the Tomkins' expression based on the amplitude of plastic deformation (Daffos *et al.*, 2002). We have used the following expression for both nitrated and virgin steel:

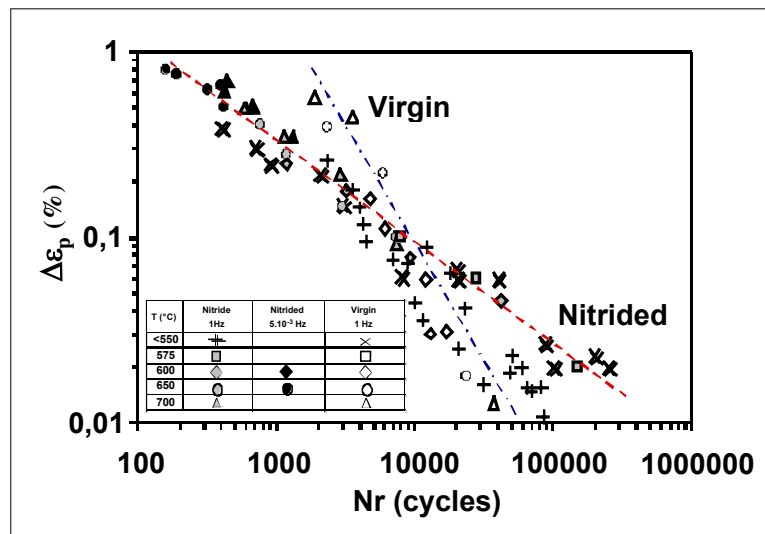
$$\frac{da}{dN} = 0,51 \cdot \Delta \varepsilon_p \left[\sec \left(\frac{\pi \sigma - \sigma_i}{2 \sigma_0} \right) - 1 \right] \cdot a$$

where σ_0 is the yield stress of the virgin steel and σ_i is an internal (or residual) stress, which is nil for virgin steel but not for nitrated steel. In fact, the nitrated layer is under internal residual stresses induced during the nitrating process (Barrallier, 1992). The internal stress is added to external stress, which leads to alteration of the crack propagation rate. The residual stress, σ_i is measured from the surface towards the bulk using an X-ray diffraction method from room temperature to 550°C by successive electro polishing of the nitrated layer (Daffos *et al.*, 2002). The residual stress in the nitrated layer is not constant and changes from the free surface towards the virgin/nitride interface. For simplification, a mean stress value of about 250 MPa is used in the modified Tomkins' expression.

3.4 Fatigue life and prediction

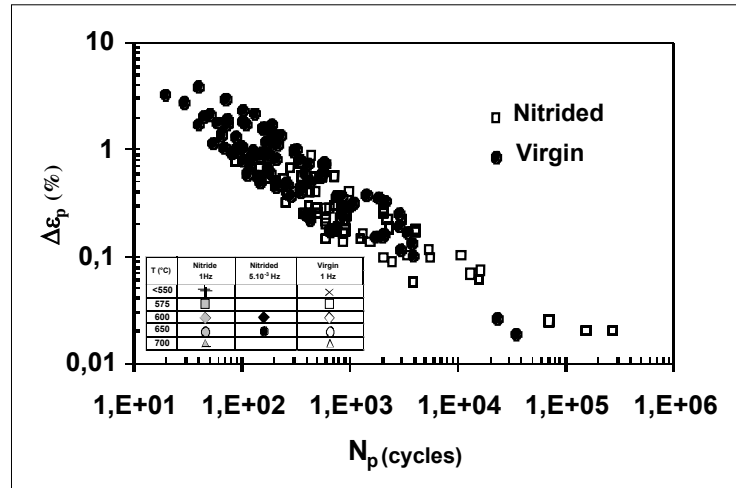
The drastic effect of the temperature on fatigue life as a function of the total strain amplitude or as maximum stress has been discussed in Daffos *et al.* (2002). Based on such fatigue life criterion, a temperature dependency is clearly observed on fatigue life curves. By increasing the temperature, the LCF life decreases under a prescribed loading. It seems that the behaviour of both nitrided and virgin steel decreases drastically for temperatures higher than 550°C. In the same manner, when the test frequency is reduced, the life decreases drastically. In fact, as stress per cycle decreases, the part of the inelastic strain in the total strain is enhanced and so the LCF decreases. Therefore the fatigue life becomes frequency-dependent (Figure 2). Figure 8 presents the fatigue life curve as a function of the inelastic strain range for both virgin and nitrided conditions (at half-life). As can be observed, such fatigue life criterion is not temperature-dependent. In addition, the frequency effect also disappears. Therefore the Manson-Coffin plastic strain-based law $\Delta\varepsilon_p = C.(Nr)^{-\alpha}$ becomes a relevant criterion for isothermal LCF life prediction for temperatures ranging from room temperature to about 700°C and the strain rate reported here. Figure 8 points out another important evidence that beyond a critical inelastic strain range, the isothermal LCF life of nitrided steels is lower than that of virgin steel.

Figure 8 Plastic strain amplitude versus fatigue life in isothermal conditions



This Manson-Coffin law was applied to predict the isothermal fatigue life of X38CrMoV5 (AISI H11) examined in this investigation and also that reported in literature. As can be expected, isothermal LCF life was fairly well predicted. However, when applied to predict the TMF life, the Manson-Coffin models present shortcomings for both nitrided and virgin steels. Figure 9 shows the relation between plastic strain amplitude and the number of propagation sample (as calculated in the previous section) is identical for nitrided and virgin steels.

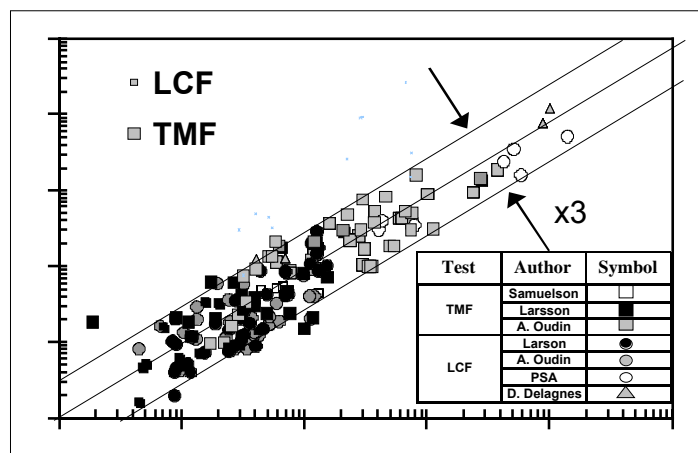
Figure 9 Relation between fatigue life and inelastic strain range measured at half life



So the difference between the LCF life of both steels is due to their crack initiation and mechanisms. The fatigue life, $N_r (= N_i + N_p)$, covers the crack initiation (N_i) and crack propagation (N_p). N_p can be estimated from the modified Paris law for temperatures beyond 550°C for both virgin and nitrided steels. It should be emphasised that, in nitrided steel, a constant propagation rate is considered for crack length ranging between 20 μm to 300 μm . The crack initiation life (N_i) is therefore calculated by $N_i = N_f - N_p$.

For temperature lower than 550°C, it is assumed that the total fatigue life is spent in propagation and cracks initiate from the first cycle ($N_i = 1$). Figure 10 shows a comparison between the experimental and the calculated fatigue life for all experiences examined here and those reported from literature (in the range of factor 3) (Oudin, 2001; Samuelson, 2001).

Figure 10 Comparison between the predicted fatigue life and the experimental results obtained from this work and literature



Sources: Oudin (2001) and Samuelson (2001)

4 Conclusion

Isothermal and thermal-mechanical LCF fatigue life of a tempered martensitic steel, X38CrMoV5 (47 HRC), are investigated under various experiments involving virgin and nitrided conditions.

The role of the oxidation on crack initiation and propagation is examined. It is shown that oxidation plays an important role on the fatigue life of X38CrMoV5 both in nitrided and virgin conditions. It is observed that the nitriding treatment can modify the crack initiation mechanism.

Inelastic strain range is one criterion (Manson-Coffin's law) for isothermal LCF fatigue life that is temperature- (20°C to 700°C) and frequency-independent. Two Manson-Coffin's laws were found for nitrided and virgin steel from 20°C to 700°C. Such laws are relevant for isothermal LCF fatigue life prediction. It is observed that beyond a critical inelastic strain range, the isothermal LCF life of nitrided steels is lower than that of virgin steel. It is observed that isothermal LCF Manson-Coffin's law presents shortcomings for prediction of the thermomechanical fatigue life (fatigue under temperature transition conditions).

The modified Paris and Tomkin's laws are introduced for fatigue life prediction for nitrided and virgin steels by considering environment effect, frequency dependence and the mechanical loading pattern.

The fatigue life models proposed can predict isothermal LCF and TMF experiments in the range of factor 3.

Acknowledgements

This work has been partly funded by the French Ministry of Industry (SIMULFORGE Program). The authors also wish to acknowledge all partners, in particular CETIM, for their support.

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