



## High temperature creep–fatigue–oxidation interactions in 9–12%Cr martensitic steels

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### A B S T R A C T

Detailed observations of fractured specimens of 9–12%Cr martensitic steels subjected to creep–fatigue loadings at 823 K were carried out. Observations revealed that oxidation phenomena strongly influence the creep–fatigue lifetime whereas no creep damage (cavities) can be observed in the present loading conditions. Two main interaction mechanisms between creep, fatigue and oxidation damage were highlighted. These two damage mechanisms correspond to two distinct domains of loading (expressed in terms of total strain range and holding period duration). Based on the identified mechanisms a creep–fatigue lifetime prediction model is proposed. The crack initiation is approached by the Tanaka and Mura model, whereas the crack propagation phase is accounted by the Tomkins formulation.

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### 1. Introduction

Martensitic steels of the 9–12%Cr family are widely used in the energy industry and were selected as candidate materials for structural components of future fusion reactors [1,2]. Typical in-service conditions require operating temperatures between 673 and 873 K, which means that the creep behaviour of these steels is of primary interest. In addition, some components are anticipated to operate in a cyclic mode, leading to complex time-dependencies of temperature, stress and strain in materials. Therefore, in design procedures, fatigue and creep–fatigue data are required. Furthermore, to meet the need for very long in-service lifetime of components (with very long hold times ~one month) reliable cyclic lifetime models are necessary, since complete tests with such long holding periods cannot, of course, be carried out in laboratory. To make these extrapolations safer and more reliable a detailed understanding of the damage and interaction mechanisms is required.

Both damage observations and a theoretical study of oxide layers fracture mechanisms allow qualitative explanations for recorded fatigue lifetimes to be proposed. Indeed, two main creep–fatigue–oxidation interactions are put forward, depending on both the applied fatigue strain and the holding period duration [3,4]. Finally a modeling of creep–fatigue lifetime is described and compared to experimental data. This predictive model is based on the Tanaka and Mura [5] initiation model and on the Tomkins [6]

propagation law. Here only a short account of the results is given, but more details can be found [3,4,11].

### 2. Material and experimental procedures

The material under study is a modified 9%Cr–1%Mo steel which was subjected to pure-fatigue (PF), relaxation-fatigue (RF) and creep–fatigue (CF) tests carried out in air and in vacuum at 823 K. A detailed description of both the material and the experimental procedures can be found elsewhere [3,4].

### 3. Mechanical test results

Holding periods are found to decrease the fatigue lifetime. Creep–fatigue lifetimes with tensile holding periods are found to obey the general Manson–Coffin law (Fig. 1), except for the highest applied strains ( $\Delta\varepsilon_{fat}$ ) where CF lifetimes are systematically shorter than those predicted by the Manson–Coffin curve. These results suggest that the CF lifetime is mainly ruled by the (visco)plastic strain range ( $\Delta\varepsilon_{vp}$ ) applied.

Surprisingly enough, for low applied strains and short holding periods, compressive holds are more deleterious than tensile ones in air, as shown in Fig. 2. This result has already been reported in the literature [7–9] and is said to be linked to oxidation phenomena. This influence of oxidation is confirmed by the fact that, in vacuum, the difference between tensile and compressive holds does not seem to exist (Fig. 3). These tests also showed that, pure-fatigue lifetimes in vacuum are much longer than in air.

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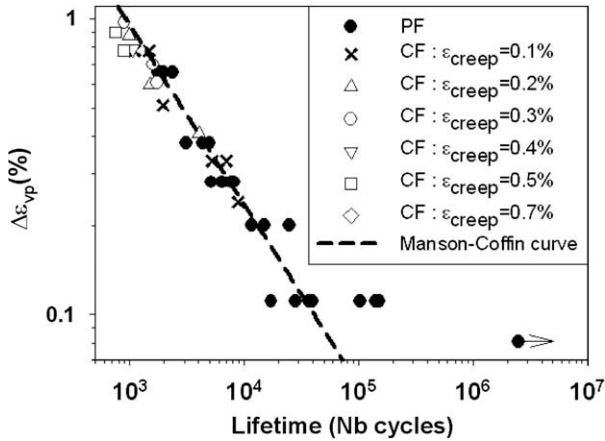


Fig. 1. Pure-fatigue and creep-fatigue (with tensile holding periods) lifetimes expressed in terms of the applied viscoplastic strain range ( $T = 823$  K, air,  $\epsilon_{creep}$  is the creep strain applied during the holding period).

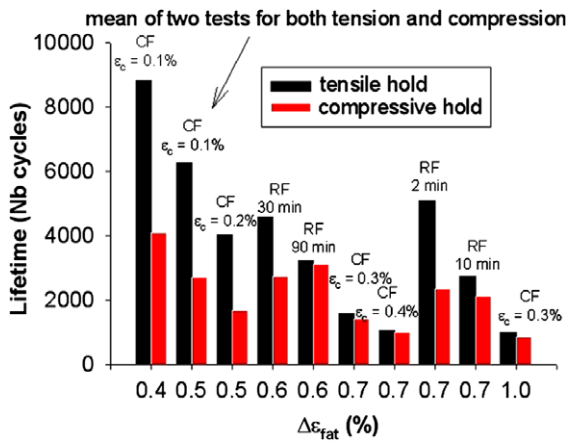


Fig. 2. Comparison of the lifetimes resulting from tensile and compressive holding periods (CF: creep-fatigue, RF: relaxation-fatigue, and  $\epsilon_c: \epsilon_{creep}$ ,  $T = 550$  °C, air).

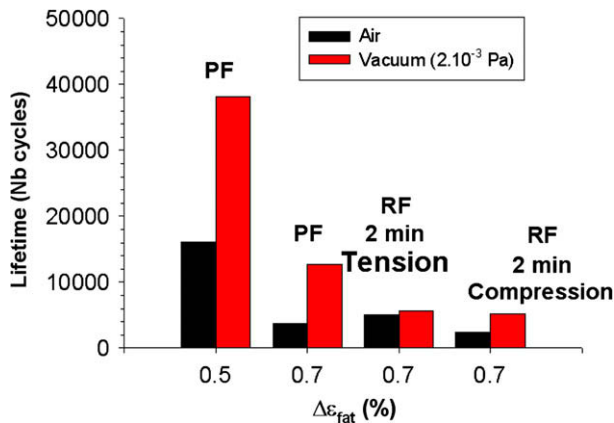


Fig. 3. Lifetimes obtained in air and in vacuum for PF (pure-fatigue) and RF (relaxation-fatigue) loadings ( $T = 823$  K).

4. Observations

Detailed observations of the surfaces, fracture surfaces and polished cross-sections of specimens subjected to pure-fatigue, creep-fatigue and relaxation-fatigue tests were carried out. In all cases the final fracture is due to the initiation and propagation of one

or several fatigue cracks propagating transgranularly. No usual creep damage (cavitation) was observed (the holding duration remained, in all tests, smaller than 1 h per cycle). Two possible types of damage were identified, all tested samples corresponding to one of them (see Fig. 4).

Type 1: the final fracture is due to the propagation of a unique macroscopic crack. This crack is highly bifurcated and branched suggesting a strong influence of the microstructure (crystallographic orientation, grain boundaries, slip bands, etc.) on the crack propagation. The crack initiation proceeds by usual fatigue intrusion/extrusion phenomena (although this cannot be observed in air, due to the formation of an oxide layer, such surface reliefs were observed on samples tested in vacuum).

Type 2: numerous cracks initiated and propagated and the final fracture is due to the coalescence of several macroscopic cracks. Cracks propagated in a straight line and are widely opened. An internal penetration of oxygen is found around the crack tips. The crack initiation is due to the brittle fracture of the oxide layer. Repeated crackings of the oxide layer led to a very thick oxide scale and a high density of cracks. This type of damage is the most severe since the crack initiation can occur as soon as the first cycle, whereas in type 1 many cycles are required to create the necessary surface relief leading to crack initiation.

These two types of damage correspond to distinct domains of loadings as presented in Fig. 5 for tensile holding periods. The illustrative frontier drawn in Fig. 5 highlights the fact that there is a critical strain necessary to break the oxide layer and then to lead to a type 2 damage. The same frontier exists for compressive holding periods but corresponds to a lower critical strain. A simple mechanical analysis [4] shows that, with a tensile holding period, the oxide layer is mainly loaded in compression, whereas for a compressive hold time the oxide scale is loaded in tension. Both finite element calculations and analytical equations [4,10] show that it is easier to break an oxide layer in tension than in compression, which explains the results of Figs. 2 and 3.

5. Lifetime prediction model

Based on the damage mechanisms described above, a lifetime prediction model is proposed. The creep-fatigue lifetime ( $N_f$ ) is divided into an initiation ( $N_i$ ) and a propagation ( $N_p$ ) part:

$$N_f = N_i + N_p$$

For type 1 damage,  $N_i$  is calculated through the Tanaka and Mura [5] model:

$$N_i = \frac{\alpha}{d \Delta \epsilon_{vp}^2}$$

where  $\alpha$  is a temperature dependent constant,  $d$  is the grain size and  $\Delta \epsilon_{vp}$  is the viscoplastic strain range applied per cycle.

For type 2 damage, crack initiation is considered to occur at the first cycle because of the brittle fracture of the oxide layer.

For both types of damage, the crack propagation is modeled thanks to the Tomkins [6] formulation:

$$\frac{da}{dN} = \frac{\pi^2 \Delta \epsilon_{vp} \Delta \sigma^2}{8 \times (2\bar{T})^2} \times a \left( 1 + \frac{\pi^2}{8} \left( \frac{\Delta \sigma}{2\bar{T}} \right)^2 \right)$$

With  $a$  the crack length,  $\Delta \sigma$  the stress range applied at  $N_f/2$  and  $\bar{T}$  standing for the residual tensile strength of the material after a fatigue loading. And  $N_p = \int_{a_0}^{a_c} \frac{da}{da/dN}$  where  $a_0$  is the crack length at crack initiation (arbitrarily set to  $50 \mu m \sim$  former austenitic grain size) and  $a_c$  is taken equal to the sample radius.

Coalescence is also roughly modeled by considering the propagation of five cracks for type 2 damage.

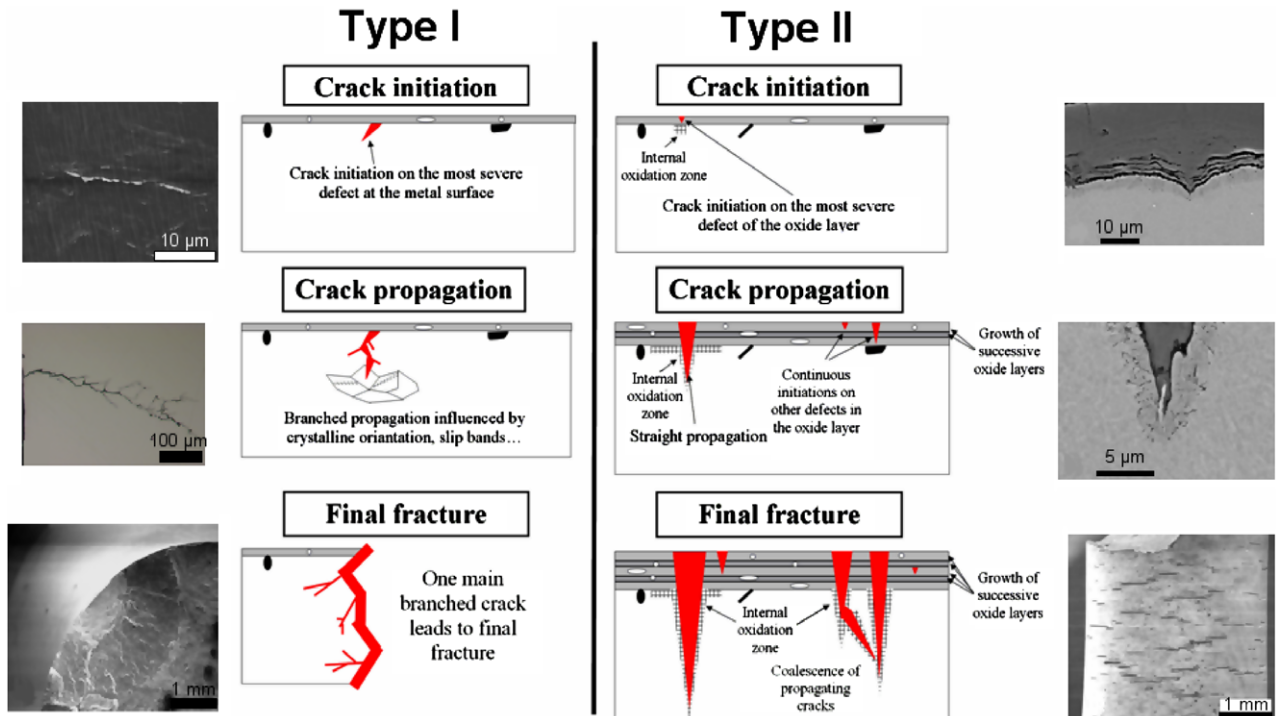


Fig. 4. Schemes of the two types of damage identified after detailed observations.

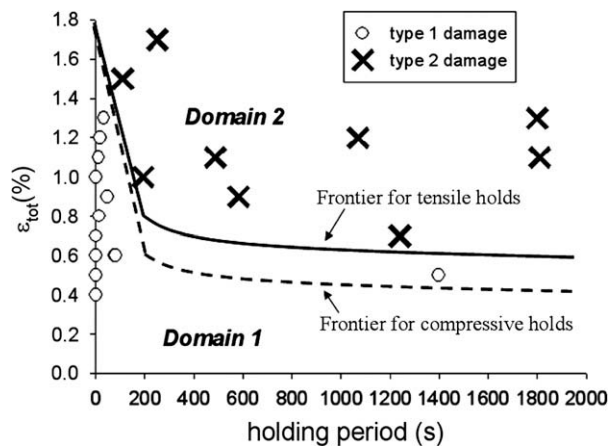


Fig. 5. Type of damage (1 or 2) for CF tests carried out with a tensile holding period and located in a  $[\epsilon_{\text{tot}} (= \Delta\epsilon_{\text{fat}} + \epsilon_{\text{creep}}), \text{hold time}]$  plane. The full line illustrates the frontier between the two domains of loadings for tensile holding periods, whereas the dashed line presents the same frontier, but observed on specimens subjected to CF tests with a compressive hold.

These model parameters are identified on crack propagation tests carried out under low-cycle fatigue conditions reported in previous work [11]. In addition, only one experimental lifetime value is necessary to fit the model's parameters (the experimental lifetime used is measured at  $\Delta\epsilon_{\text{fat}} = 0.4\%$ ).

The predicted lifetimes in pure-fatigue are compared to their experimental counterparts in Fig. 6 (air environment). For  $\Delta\epsilon_{\text{fat}} \geq 0.4\%$  the results are in excellent agreement with the experiments. For lower strains, the material is not fully plastic anymore, therefore the assumptions of the Tomkins model are not valid. Fig. 7 compares the model predictions in creep-fatigue with the experimental data. It turns out that all predictions fall into the range  $[N_{\text{exp}}/2, 2N_{\text{exp}}]$  which is usually considered as a reasonable error band in creep-fatigue.

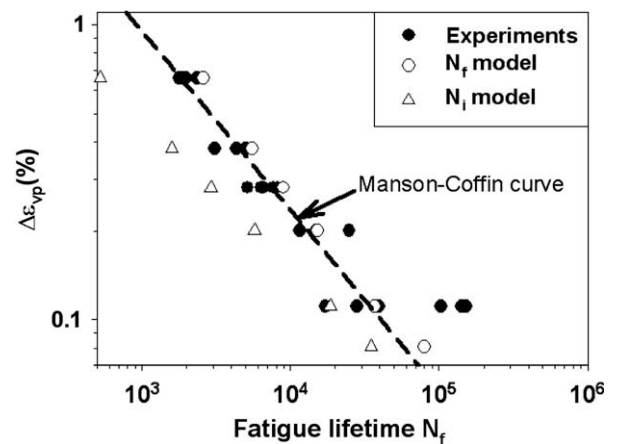
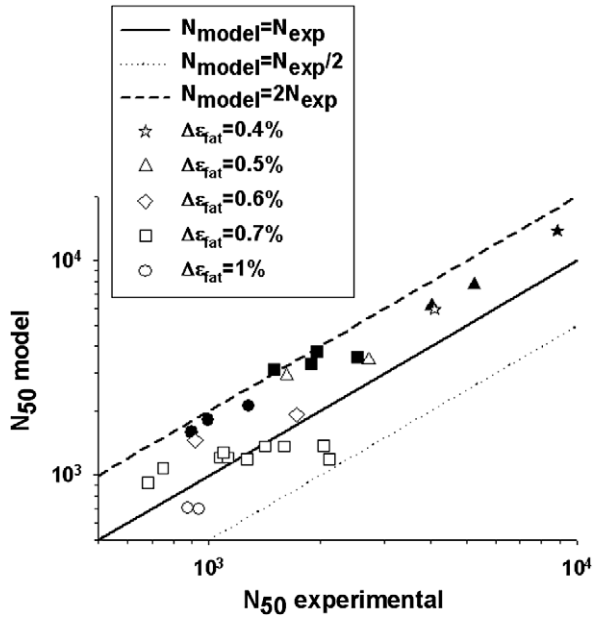


Fig. 6. Comparison between experimental and predicted PF lifetimes. The Manson-Coffin law fitted on the experimental lifetimes is shown in dashed line. The number of cycles to crack initiation  $N_i$  is also reported. The values of the plastic deformation are measured at  $N_f/2$ .

## 6. Conclusions

Pure-fatigue, creep-fatigue and relaxation-fatigue tests were carried out at 823 K. The following conclusions can be drawn:

- Pure-fatigue lifetimes and most of the creep-fatigue (with tensile holding periods) lifetimes are found to obey the Manson-Coffin law.
- The lifetime data highlighted the fact that compressive holding periods are more deleterious than tensile holding periods for low applied strains in air.
- No usual creep damage (cavities) can be observed.
- Detailed observations of the samples showed the primary influence of oxidation phenomena of the damage mechanisms, as confirmed by tests carried out in vacuum.



**Fig. 7.** Comparison of experimental and predicted CF lifetimes, considering the propagation of 5 cracks for type 2 damage. The values of  $\Delta\varepsilon_{vp}$  and  $\Delta\sigma$  are measured at  $N_f/2$ . The empty symbols correspond to a type 2 damage whereas the filled symbols correspond to a type 1 damage. Various creep strains were applied for each  $\Delta\varepsilon_{fat}$  value.

- Two main damage mechanisms were identified corresponding to distinct domains of loading.
- A lifetime prediction model is proposed that accounts for crack initiation and crack propagation stages. Both in pure-fatigue and in creep-fatigue the predictions are in quite good agreement with the experiments.

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