Creep-fatigue-oxidation interactions in 9-12%Cr steels

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Abstract Pure fatigue, relaxation-fatigue and creep-fatigue lifetimes obtained at 550°C under various atmospheres (air, vacuum) on a modified 9%Cr-1%Mo martensitic steel are presented. These experimental results highlight the lifetime reduction due to holding periods (either in creep or stress-relaxation). Contrary to other materials, compressive holding periods are more deleterious than tensile holding periods. The observations carried out on polished cross-sections and on the fracture surfaces revealed that the final fracture is due to the transgranular propagation of cracks. No evidence of intergranular cavity due to creep holds was found. Based on these detailed observations and on the theoretical study of the behaviour of the oxide layer grown in air, two mechanisms of interaction between creep, fatigue and oxidation damage are proposed. These two mechanisms apply for distinct loading ranges.

1 INTRODUCTION

The martensitic steels of the 9-12%Cr family are used for many applications in the energy industry [1-3]. The modified 9Cr-1Mo steel is a candidate for several components of the generation IV nuclear reactors. The repeated start- and stop-operations during service lead to loadings of creep-fatigue type, with very long holding periods (typically one month). As complete tests with such long holding periods cannot, of course, be carried out in laboratory, the actual in-service mechanical behaviour must be extrapolated from the results of short term mechanical tests. Several studies have already been carried out mainly between 538°C and 600°C, both in pure fatigue [4] and stress relaxation-fatigue conditions [5-7]. In addition to usual interactions between creep and fatigue, these studies suggested that environment must be taken into account in the damage accumulation calculation [5,7-9]. In order to make these extrapolations safer and more comprehensive, the present work is focussed on the understanding of the physical mechanisms responsible for damage and failure. This article presents the results of pure fatigue, relaxation-fatigue and true creep-fatigue mechanical tests with a specific emphasis on the cracking phenomena. The lifetimes obtained at 550°C in air, in vacuum and in helium are firstly presented. Secondly a series of detailed observations of the specimens surfaces, fracture surfaces and polished cross-sections are presented and compared. These observations lead to the distinction between two main mechanisms of interaction between creep, fatigue and oxidation damage. The ranges of loadings (in terms of strain and holding periods) for which these interaction mechanisms apply are then presented.

2 MATERIAL AND EXPERIMENTAL PROCEDURES

The experiments were conducted on a P91 steel produced by Usinor (Arcelor, France). The chemical composition is given in table I. The steel plate was austenitized at 1050°C for 30 min, quenched and tempered at 780°C for 1h. The usual tensile properties at 550°C are given in table II. The as-received material presents a very fine tempered martensitic microstructure.

Low Cycle Fatigue (LCF) tests were conducted in air on MAYES ESM100 servo-mechanical machines with resistance furnace heating. Temperature along the gauge length of the specimen was controlled and the precision remained better than ±2°C. The cylindrical specimens are characterized by a 16mm gauge length and a 8mm diameter with a shoulder radius of 16mm. They were finished by fine turning to an average roughness value of 0.8µm. The axial strain was measured with a capacitive extensometer directly attached on the calibrated part. The calibrated extensometer gauge length is equal to 10mm. The accuracy of this device is better than 0.5µm that allows to conduct LCF tests with a strain range as small as 0.1%. The LCF tests were controlled in terms of total strain, which was measured with this extensometer. The cyclic strain rate was equal to $2.10^{-3}s^{-1}$ for all tests. Three types of cyclic tests were carried out: pure fatigue (PF) tests, creep-fatigue (CF) tests (for which the stress is held constant during holding periods) and stress relaxation-fatigue (RF) tests (for which the strain is held constant during holding periods). The main advantage of CF tests compared to RF tests lays in the fact that they enable a higher viscoplastic strain to be applied during the holding period for the same test duration. Figure 1 illustrates the corresponding shapes of hysteresis loops. These holding periods were controlled either in terms of time (e.g. a 10 minutes holding period is applied) or in terms of strain (e.g. the stress is held constant until a 0.5% creep strain is reached) depending on the test. The hold times can be applied either in tension or in compression.

PF and RF tests were also carried out in vacuum ($P=2.10^{-5}$mbar on a MTS hydrolic machine).

3 MECHANICAL TESTS RESULTS

The lifetimes (N_{50} is the number of cycles necessary to lead to a 50% decrease of the stress range) of P91 at 550°C tested in air are presented in figure 2 for both PF and CF (with a tensile holding period). Plotting these lifetimes in terms of the total strain range applied $\Delta\varepsilon_{fat}$ (figure 2.a) highlights the fact that, the higher the creep strain applied, the shorter the lifetime. However when plotting these same data in terms of the (visco)plastic strain applied per cycle $\Delta\varepsilon_{vp}$, a usual Manson-Coffin law seems able to describe all results (both PF and CF lifetimes), at least in the range $0.1\% < \Delta\varepsilon_{vp} < 1\%$. This suggests that the effect of holding periods is comparable to a simple increase of the fatigue strain applied: no evidence of an additionnal damage mechanism can be found.

A more surprising result arises when the effect of tensile and compressive holding periods is compared. Figure 3 compares the fatigue lifetime measured for compressive and tensile holding periods. For low fatigue strain ranges ($\Delta\varepsilon_{fat} \leq 0.5\%$) and limited applied creep strain ($\varepsilon_{creep} \leq 0.2\%$) the fatigue lifetime of compressive CF tests is always shorter than half of the tensile CF lifetime. For higher $\Delta\varepsilon_{fat}$ or ε_{creep} no significant difference exists anymore. For RF tests, the compressive holding period also leads to shorter fatigue lifetime for $\Delta\varepsilon_{fat} = 0.6\%$ and $t_h = 30$min and for $\Delta\varepsilon_{fat} = 0.7\%$ and $t_h = 10$min. Nevertheless this difference does not hold anymore for a longer holding period ($\Delta\varepsilon_{fat} = 0.6\%$ and $t_h = 90$min). These results show that, under certain conditions (more specifically low strain ranges and "short" holding periods), compressive holding periods are more deleterious than tensile holding periods, whereas, for higher strain ranges, the difference between tensile and compressive holds is not significant.

In order to explain the more deleterious effect of compressive holding periods, two main hypothesis can be put forward. When applying a compressive hold time, a positive mean stress is created (whereas the mean stress is negative for tensile holding periods). Positive mean stresses are known to reduce the cyclic lifetime [8,9], however, in the present case this explanation can only partly account for the differences observed between tensile and compressive holding periods [6,7,11,15]. Indeed, even though in CF tests the mean stress can be as large as 90MPa [17-18] , in RF tests the mean stresses are much smaller.

The second possible explanation consists in the interactions between mechanics and oxidation [6-16]. The influence of oxidation is highlighted by the results obtained in vacuum (figure 4). PF lifetimes under vacuum are much longer than in air. In RF tests, no lifetime difference between tensile and compressive holdings can be found in vacuum.

4 OBSERVATIONS

In order to investigate further the damage mechanisms responsible for the experimental lifetimes obtained, a series of detailed observations were carried out in optical microscopy and SEM. Finally, two main types of damage were observed, all the tested samples correspond to one of them. In all cases the damage consists in the propagation of transgranular cracks, no evidence of intergranular cavities (usually corresponding to creep damage) was found.

The first type of damage is presented in figure 5, it is characterized by a very low density of highly branched cracks, a thin (1-2µm) and undammaged oxide layer and a final fracture due to a single transgranular fatigue crack.

The second type of damage is presented in figure 6, it is characterized by a very hich density of straight cracks, a thicker (up to 25µm) and damaged oxide layer with the existence of an internal penetration of oxigen beneath the oxide layer, and a final fracture due to the coalescence of many cracks.

5 DISCUSSION

Figure 7 presents the two scenarios of interaction between creep, fatigue and oxidation damage drawn from the previous observations.

5.1 Crack initiation mechanisms.

Type 1: In this domain, no clear evidence of environmentally assisted crack initiation is found. Therefore, crack initiation may occur in a rather usual way, either on surface inclusions, on fatigue extrusions, or due to intergranular processes.

Type 2: Numerous evidences of an environmentally assisted crack initiation are found. The high strain amplitude leads to the cracking of the oxide layer on defect or porosity. These cracks of the oxide layer induce stress concentration at their tip in the material. Therefore, it is easier for these cracks to propagate inside the material from these high-stress regions, than for a new crack to initiate in the bulk material somewhere else. Experimental evidences (numerous cracks observed are suspected to initiate in the oxide layer) such as literature references [7,11] confirm that in this domain, cracks initiate in the oxide layer. The repeated crackings of the oxide layer lead to the multilayered and thick oxide morphology mentioned above. This mechanism is self-sustained, since the thicker the oxide is, the poorer its mechanical properties are.

5.2 Crack propagation mechanisms.

Type 1: Branched and narrow cracks are observed. Additionally a thin oxide layer covers the whole crack lips up to the crack tip. These observations suggest that the cracks may propagate preferentially along microstructurally "easy-propagation" paths, such as slip bands or highly misoriented boundaries. The thin oxide layer observed along the crack lips is sufficient to increase slip irreversibilty and to prevent crack closure. Such environmentally assisted crack propagation would therefore lead to a reduced fatigue lifetime compared to tests carried out in vacuum.

Type 2: Cracks are found to propagate straightly and to be filled with oxide. Moreover, evidence of internal oxidation was found at crack tips. The straight crack paths may thus come from the fact that the internal oxidation (due to oxide cracking as explained above) damages the microstructural boundaries ahead of the crack tip. Therefore, even though slip bands could still exist, they are no longer the easiest propagation paths. The cracks will propagate along the damaged grain or lath boundaries with no reason to deviate from a simple straight path. However, as the cracks are completely filled with oxide and widely opened, it is not clear, whether the propagation rate is increased or decreased. Indeed, in an extreme case, the crack could even be considered as healed if it is completely filled with oxide and if the applied strain is not high enough to crack it.

5.3 Domains of loading

The second type of damage is obviously the most severe since the crack initiation can take place immediately (oxide layer cracking) whereas several cycles are necessary to initiate a crack due to usual initiation mechanisms. These two types of damage also correspond to two distinct loading ranges as shown in figure 8. In figure 8.a the tests carried out with a tensile holding period are located in a (hold time, $\varepsilon_{tot}=\Delta\varepsilon_{fat}+\varepsilon_{creep}$) plane. It appears that the type 2 damage occurs only when a critical ε_{tot} value is reached, this value decreasing when the hold time increases. The same distinction can be made for tests carried out with a compressive holding period, except that the frontier between the two domains is lowered. This means that it is easier (a lower applied strain is necessary) to crack the oxide layer when the hold time is in compression.

This surprising result is schematically explained in figure 9. During a CF cycle, the longest part of the cycle consists in the holding period. Therefore the oxide layer mainly grows during this hold time. As the oxide layer grows on the surface of the specimen, it is free of stress (if the internal stresses coming from the growth process are neglected). When the hold time is in tension (compression), as soon as it ends, the oxide layer is thus loaded in compression (tension) as illustrated in figure 9. To put it in a nutshell, compressive holding periods lead to oxide scales loaded in tension and conversely.

A literature review of the fracture mechanisms of oxide layers such as finite element calculations were carried out [17-18] and proved that it is easier to break oxide layers in tension than in compression. This explains why the frontier between the two damage mechanisms is lower for compressive holding periods.

6 CONCLUSIONS

Numerous low cycle fatigue tests (pure fatigue, creep-fatigue and relaxation-fatigue tests) were carried out at 550°C on a modified 9%Cr-1%Mo martensitic steel. The obtained fatigue lifetimes confirm the detrimental effect of tensile holding periods. The compressive holding periods were found to be more deleterious than tensile holding periods. Many observations of the tested samples led to the following conclusions:

- A usual Manson-Coffin law allows the PF, CF and RF lifetimes to be correctly described in the range 0.1%< $\Delta\varepsilon_{vp}$ <1%.
- No creep cavity or other type of creep damage were evidenced. Therefore the holding periods do not lead to an additionnal creep damage.
- Crack propagation is mainly transgranular for all testing conditions.
- No clear influence of the fatigue strain range was found on the cracking behaviour in pure fatigue tests.
- Environment plays a major role both in initiation and propagation of cracks.
- Two main domains of cracking behaviour were identified. They depend on both the total applied strain and the holding period duration. For each domain a creep-fatigue-oxidation interaction mechanism is proposed.
- The more severe lifetime reduction due to compressive holding period is related to the fact that the oxide layer is more easily fractured for this loadings.

7 REFERENCES

1. R.W. Swindeman, M.L. Santella, P.J. Maziasz, B.W. Roberts, and K. Coleman. *Pressure Vessels and Piping*, 81, 2004, 507-512.
2. P.J. Ennis, A. Zielinska-Lipiec, and A. Czyrska-Filemonowicz. Parsons 2000 avanced materials for 21st century turbines and power plant. Proceedings of the fifth international Charles Parsons turbine conference, 2000.
3. S.R. Holdsworth. *Materials at High Temperature*, 18, 2001, 261-265.
4. A. Nagesha, M. Valsan, R. Kannan, K. Bhanu Sankara Rao, and S.L. Mannan. International Journal of Fatigue, 24, 2002, 1285-1293.
5. B.G. Gieseke, C.R. Brinkman, and P.J. Maziasz. In microstructures and mechanical properties of aging material, TMS The Minerals, Metals & Materials Society, 1993.
6. K. Taguchi, M. Ueta, K. Douzaki, M. Sukekawa, H. Koto, and Y. Asada. High temperature service and time-dependent failure, ASME, PVP conference, 1993.
7. S. Kim and J.R. Weertman. Metallurgical Transactions A, 19A, 1988, 999-1007.
8. K. Aoto, R. Komine, F. Ueno, H. Kawasaki, and Y. Wada. Nuclear Engineering & Design, 153, 1994, 97-110.
9. T. Sugiura, A. Ishikawa, T. Nakamura, and Y. Asada. Nuclear Engineering & Design, 153, 1994, 87-95.
10. B.A. Kschinka and J.F. Stubbins. Materials Science & Engineering, A110, 1989, 89-102,.
11. R. L. Hecht. Mechanisms operating during high-temperature fatigue with hold periods in two chromium ferritic steels. PhD thesis, Northwestern University, 1992.

12. S.L. Mannan and M. Valsan. International Journal of Mechanical Sciences, 48, 2006, 160-175.
13. K. D. Challenger, A.K. Miller, and C.R. Brinkman. Journal of Engineering Materials and Technology, 103, 1981, 7-14.
14. R.A. Hecht and J.R.Weertman. Metallurgical and Materials Transactions, 29A, 1998, 2137-2145.
15. R.L. Hecht and J.R. Weertman. Metallurgical Transactions, 24, 1993, 327-333.
16. M. Yaguchi, Y. Okamoto, T. Nakamura, and Y. Asada. JSME International journal, 38:220_225, 1995.
17. B. Fournier, M. Sauzay, C. Caës, M. Noblecourt, M. Mottot, A. Bougault, V. Rabeau, A. Pineau. International Journal of Fatigue, in Press.
18. B. Fournier, M. Sauzay, C. Caës, M. Noblecourt, M. Mottot, A. Bougault, V. Rabeau, A. Pineau. International Journal of Fatigue, in Press.

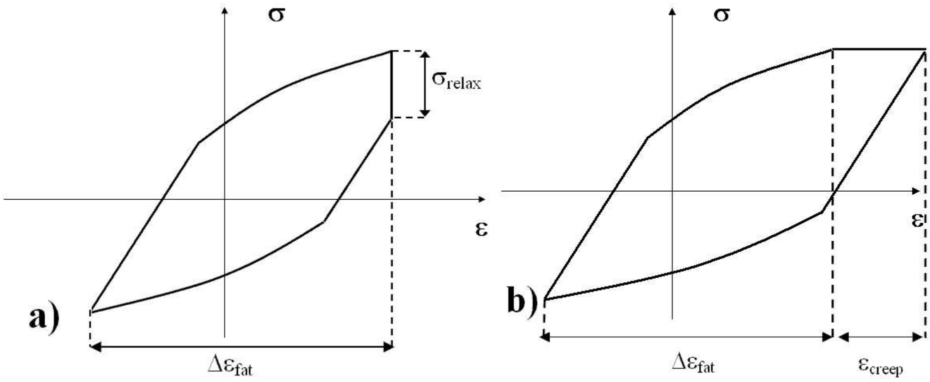

Figure 1. Scheme of the hysteresis loop of a) a relaxation-fatigue test and b) a creep-fatigue test.

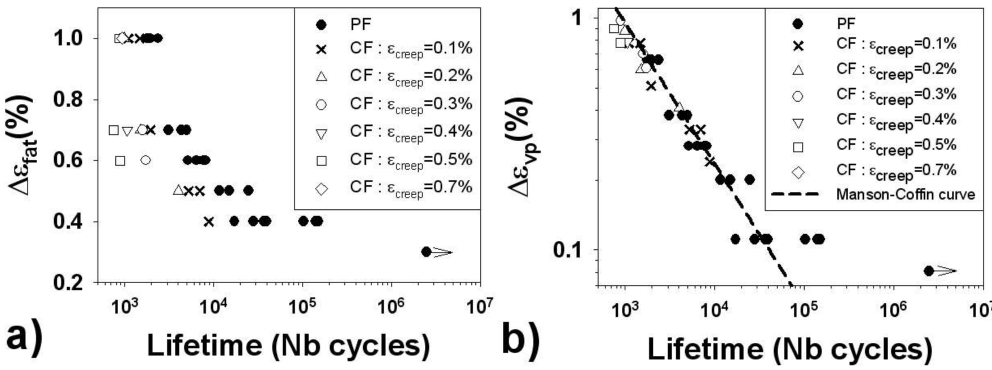

Figure 2. Pure fatigue and creep-fatigue (with tensile holding periods) lifetimes expressed in terms of a) the total strain range and b) the viscoplastic strain range applied (T=550°C, air).

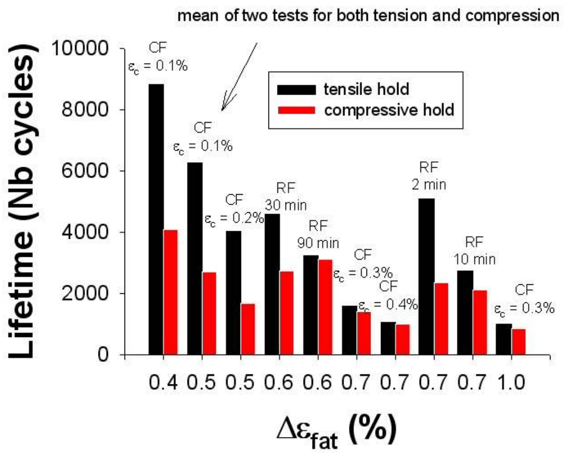

Figure 3. Comparison of the lifetimes resulting from tensile and compressive holding periods (T=550°C, air).

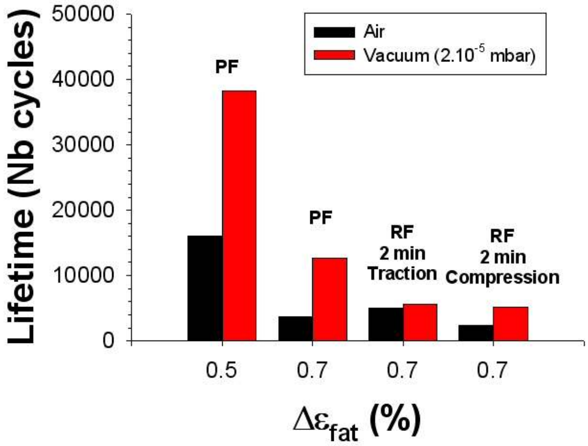

Figure 4 - Lifetime obtained in air and in vacuum for PF and RF loadings (T=550°C).

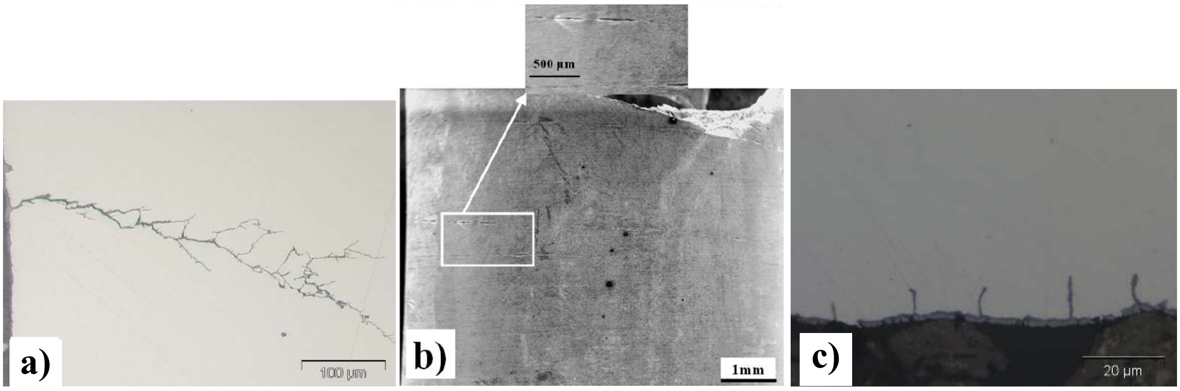

Figure 5 - Observations a) of a branched crack on a polished cross section, b) of the sample surface and c) of the oxide layer. The sample observed was tested in air at 550°C with a CF loading ($\Delta\varepsilon_t$ = 0.4%, ε_{creep}=0.1% in tension).

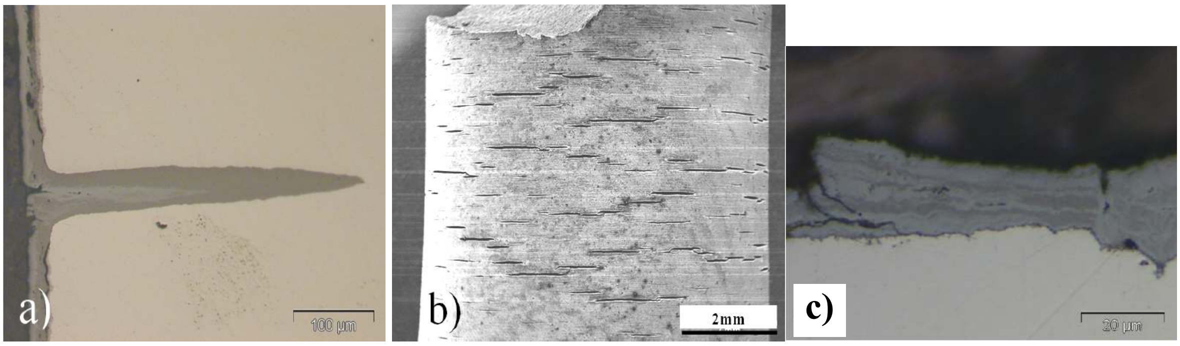

Figure 6 - Observations a) of a branched crack on a polished cross section, b) of the sample surface and c) of the oxide layer. The sample observed was tested in air at 550°C with a CF loading ($\Delta\varepsilon_t$ = 0.4%, ε_{creep}=0.1% in compression).

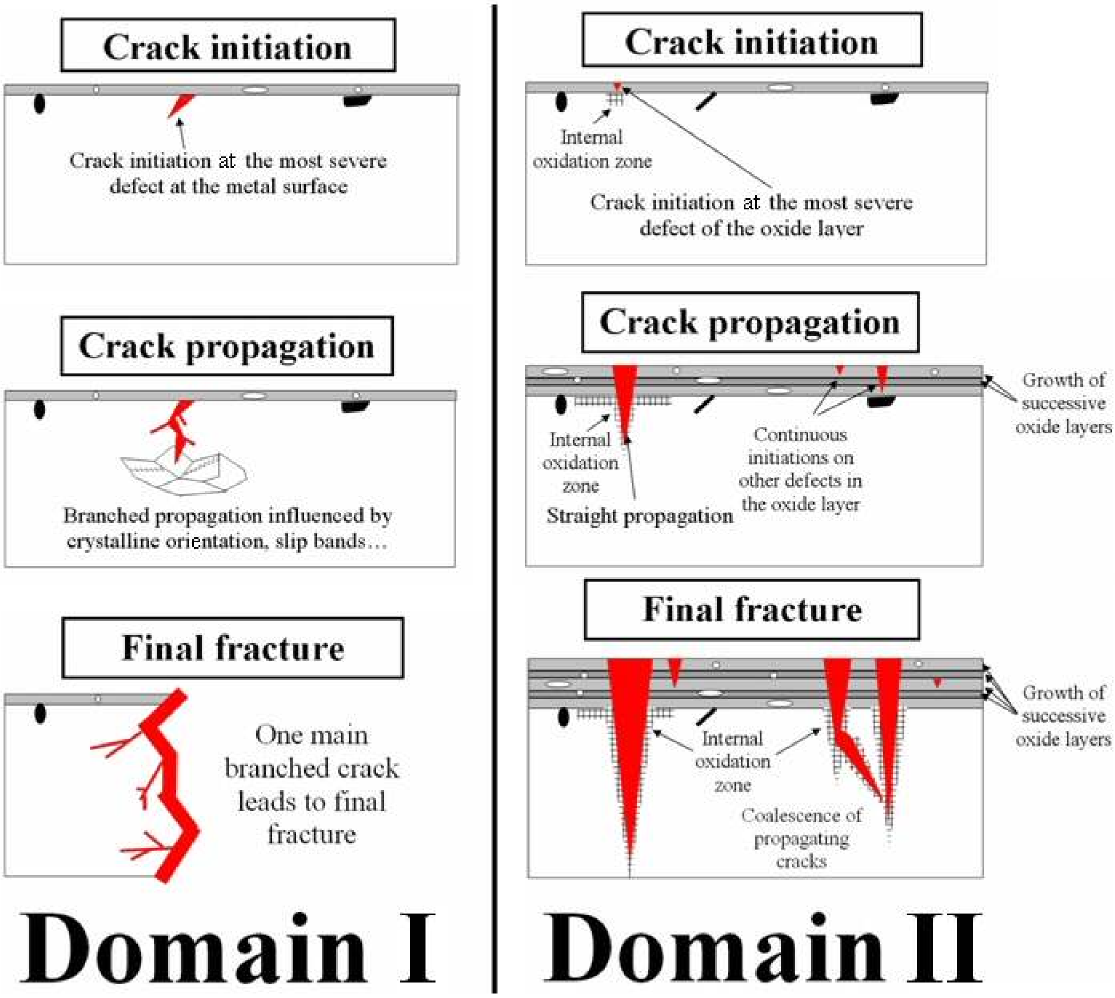

Figure 7 - Schemes of the two types of interactions between creep, fatigue and oxidation damage.

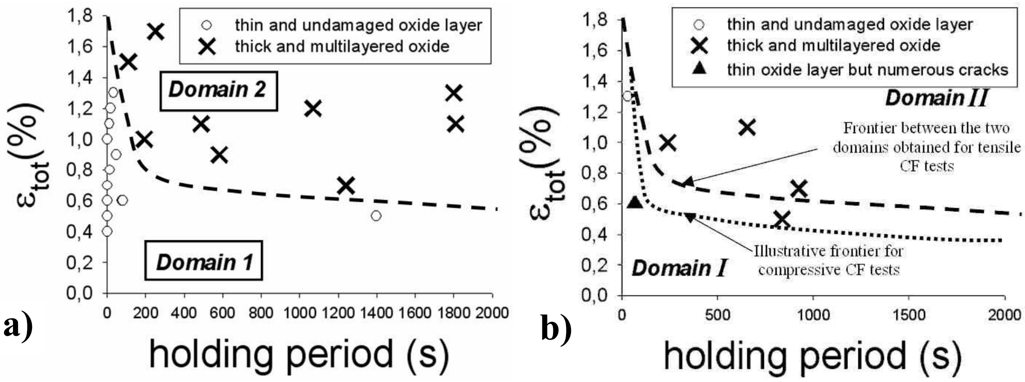

Figure 8 - Distinction between two domains of distinct damage mechanisms in terms of hold time and $\varepsilon_{tot}=\Delta\varepsilon_{fat}+\varepsilon_{creep}$ for a) tensile and b) compressive holding periods.

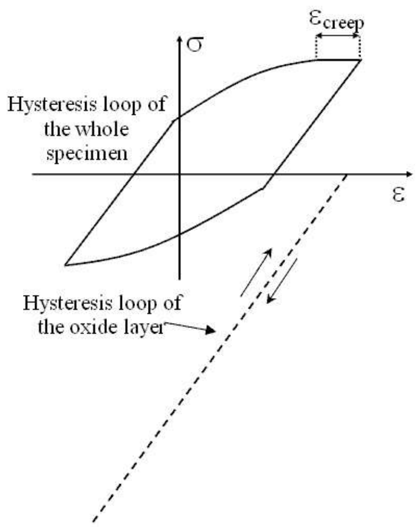

Figure 9 - Scheme of the actual stress state of the oxide layer during a CF test with a tensile holding period.

Table I. Chemical composition of the P91 steel tested in the present study.

Element	C	N	Cr	Mo	Mn	Si	Nb	V
Wt(%)	0.088	0.043	8.776	0.915	0.354	0.329	0.078	0.191

Table II - Quasistatic mechanical properties of the P91 steel under study at 550°C.

E (GPa)	$R_{p0.2}$ (MPa)	R_m (MPa)	Reduction of area (%)	Ultimate elongation (%)
163	329	381	85	33