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***A synthesis report for multi-scale models for
cyclic softening of tempered martensitic steels
including experimental observations***

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Summary

It has been known for a long time that cyclic loading applied to F/M steels at high temperature leads to a large cycle by cycle softening due to combined plasticity and thermal recovering mechanisms. This sub-project aims to provide original experimental data investigating the influence of long term hold times which are expected during in-service conditions. Additionally, macroscopic and physically-based modellings have been elaborated in order to predict both softening and lifetime. Finally, a strong interaction between experiments and modelling has been developed.

First of all, the cyclic test results confirm the effects of strain amplitude and hold time. They can be partly rationalized by using the cumulative viscoplastic strain instead of the number of cycle when considering the evolution of the peak stress. This partial rationalizing can be explained by the effect of viscoplastic slip on the recovering mechanisms such as dislocation- dislocation and low-angle boundary dislocation annihilations. Time effects should play a role too in accordance with the observed effect of temperature. Very large relaxations are observed and the stress level is divided by a factor three to four after one month which corresponds to the expected in-service hold times. A considerable amount of viscoplastic strain is then added to the pure cyclic one, which has strong consequences at low cyclic amplitude. Even after one month, relaxations are not terminated and the stress levels are not saturated. The higher the strain rate, the quicker the relaxation because of the rapid relaxation of strain rate dependent viscous stress. Increasing temperature leads to quicker relaxation too. These effects can be qualitatively rationalized by considering the partitioning of the stress into internal stress and viscous stress. The effect of hold time on softening is found to be saturated for a duration of one hour at high and medium strain amplitudes. Nevertheless, at the much lower amplitude applied during the project, the saturation of the hold time effect requires a much longer duration. Sequential tests including cycles with long hold times and pure fatigue cycles show that the effect of one long term relaxation vanishes quite quickly, that is after only a few ten pure fatigue cycles. Finally, negative mean stresses are measured due to tension hold times inducing an asymmetry of the cycles with a tension viscoplastic strain amplitude higher than the compression one. The inverse occurs with compression hold times which may lead to decrease in lifetime. The mean stresses are rather low at high strain amplitude but much higher at low amplitude due to the ratio between the pure fatigue and relaxation-induced viscoplastic strains. Once more, low strain amplitudes are characteristic of in-service conditions.

Three different modelling approaches have been used. First, the engineering approach based on well-chosen analytical formulae and parameter adjustment by inverse identification provides predictions in reasonable agreement with numerous experimental data. The second approach, based on thermodynamics and macroscopic hardening, flow and damage constitutive laws, is able to provide predictions in good agreement with fatigue and fatigue-relaxation softening data. Nevertheless, its validation out of its identification data base is required concerning typical in-service conditions (low strain amplitude and hold times of several weeks). The third approach is based on polycrystal homogenization and crystal viscoplasticity constitutive laws, accounting for annihilation of dislocations and low-angle boundaries. Various strain rate jump tests carried out at 550 and 600°C permit the inverse identification of the lonely two adjusted parameters, namely the crystal viscoplasticity activation energy and volume at the F/M block scale. The activation energy is close to the bulk self-diffusion

coefficient. The high temperature large strain tensile behavior is then well described. And strain amplitude, strain rate and temperature on cyclic softening are qualitatively well reproduced considering the whole range of strain amplitudes and temperatures considered in the MATTER and MATISSE projects.

The prediction of the softening at very low strain amplitudes and for very long hold times requires the introduction of dedicated terms such as long term recovering by dislocation climb and the heterogeneity in the dislocation blockage induced by the randomness of the obstacle microstructures (MX particles, solid solution clusters, debris). Many original experimental data obtained in conditions close to the in-service ones have been provided by the project and in the next future, the predictions from the different modelling should be compared to the whole set of experimental results. In the future, TEM observations are of interest too for understanding the long term evolutions and validating the microscopic predictions of the physically-based modelling. Finally further research works are highlighted, for instance very low strain amplitude fatigue, fatigue-relaxation at low strain amplitude and long hold time and finally low stress creep strain rate regime.

Approval

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1 Introduction

During the last three decades, numerous experimental studies highlight the large cycle-by-cycle softening observed in all tempered ferritic-martensitic steels (Fig. 1) (see the recent review [1]). The triggering effect of temperature, strain amplitude, strain rate and hold time were largely investigated (see the recent review [2]). The experimental findings concerning the evolution of stress amplitudes with the number of cycles measured during total strain-controlled tests can be summarized that way:

- the higher the temperature, the larger the cyclic softening;
- the lower the strain amplitude, the larger the cyclic softening;
- the lower the strain rate, the larger, the cyclic softening (Fig. 2);
- the longer the hold time, the larger the cyclic softening (Fig. 3). A hold time at the positive peak stress induces the appearance of a negative mean stress and the contrary occurs for a hold time positioned after the negative peak stress. Such mean stress appearance is due to the additional viscoplastic strain added during the hold time which should be recovered at the next peak stress of opposite sign (strain controlled testing and cyclic loop asymmetry).

For all four parameters, both an effect of higher viscoplastic strain amplitude and a recovering effect are expected.

It should be noticed that in Fig. 3, the cumulative viscoplastic strain is used to follow the evolution of the stress amplitude. That allows the incorporation of the additional viscoplastic strain reached during the creep / relaxation hold time and the easier study of the saturation with respect to hold time.

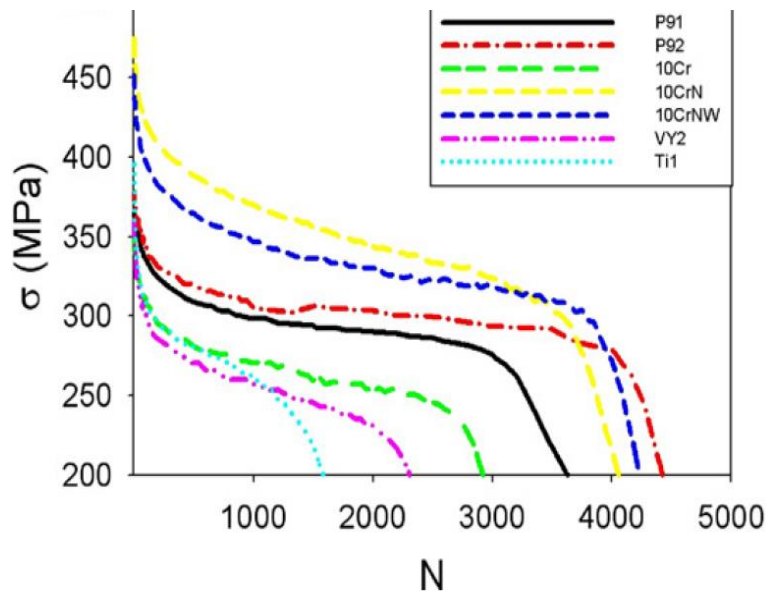


Fig. 1. Experimental evolutions of the stress amplitude with respect to the number of cycles. Symmetric pure cyclic tests. Strain-controlled testing, strain amplitude: 0.35%, strain rate: $2 \cdot 10^{-3}$ /s, 823K [2].

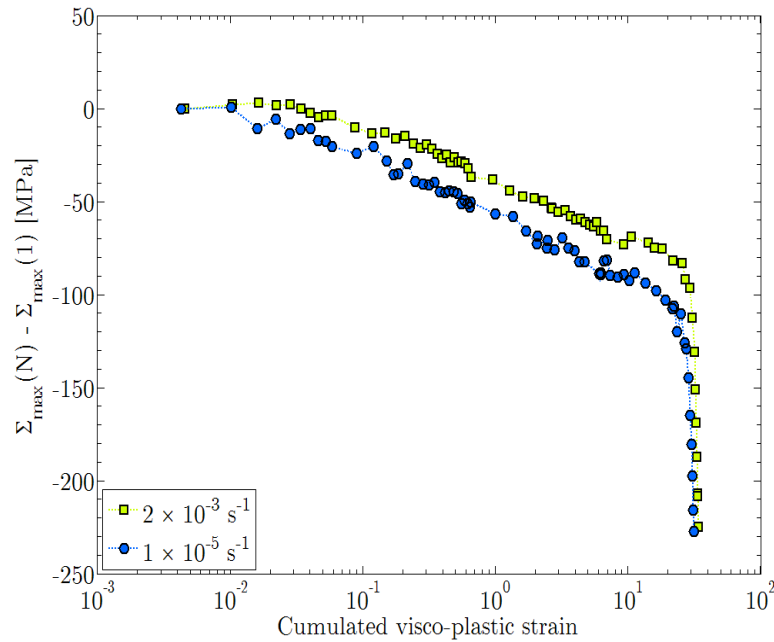


Fig. 2 Strain rate effect on cyclic softening. Grade 92 F/M steel, strain amplitude: 0.35%, strain rate: $2 \cdot 10^{-3}/\text{s}$ and $10^{-5}/\text{s}$, 823K [3].

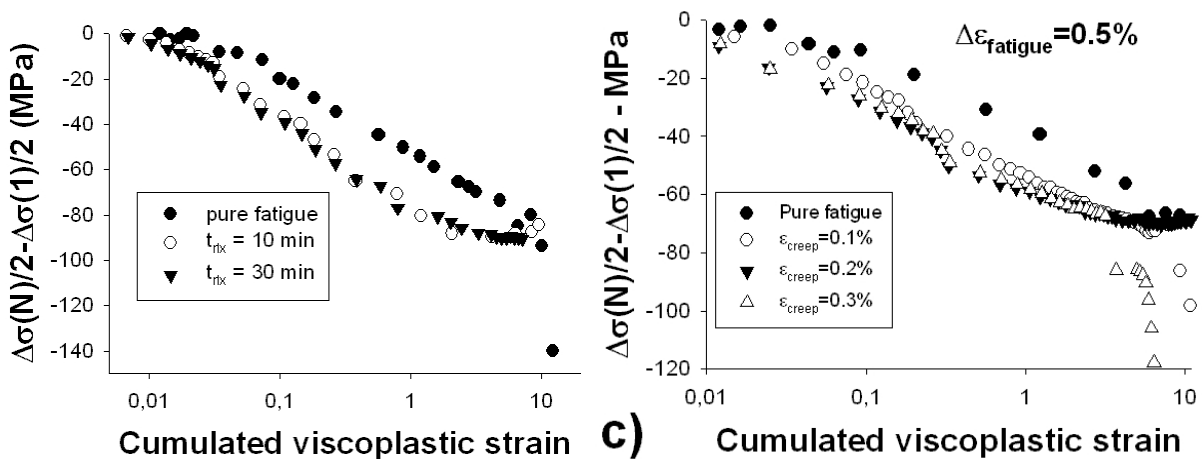


Fig. 3 Hold time effect: a) relaxation hold time (strain amplitude: 0.35%, positive peak stress hold time) and b) relaxation hold time (strain amplitude: 0.25%, positive peak stress hold time). Grade 91, strain rate: $2 \cdot 10^{-3}/\text{s}$ 823K [4].

The macroscopic behavior evolution can be qualitatively explained by microstructure evolution during cycling. The initial microstructure of martensitic steels is characterized by a high free dislocation density and a low subgrain size due to numerous low-angle boundaries (LABs) partitioning the martensite blocks in many subgrains (Fig. 4 a)). This microstructure explains the high initial yield stresses characterizing this class of materials. Numerous TEM observations highlight the large decrease in the free dislocation density during cycling and the annihilation of many LABs leading to a strong increase in subgrain size (Fig. 4 b)). Hold time and strain rate effects can be explained by the larger microstructure evolutions observed if a hold time or a low strain rate is applied (Fig. 4 c)) [1].

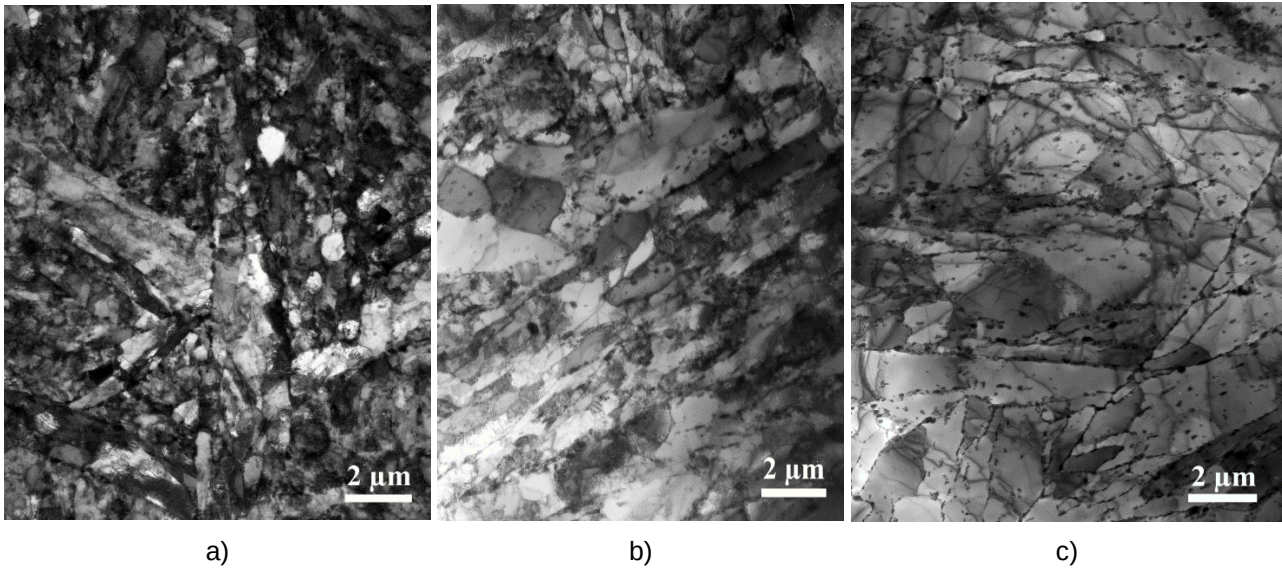


Fig. 4 : observed microstructures, a) before testing, b) after pure cyclic testing using a standard strain rate (0.35%, $2 \cdot 10^{-3}/s$, 823K, 4500 cycles) and c) after slow strain rate fatigue testing (0.35%, $10^{-5}/s$, 823K, 400 cycles). TEM observations [3].

In service conditions are now to be characterized by the followings:

- low strain amplitudes;
- long hold times. The order of magnitude is one month.

Therefore, additional experimental data and modellings are required to account for both low strain amplitude and long hold time

A few long term relaxation tests have been carried out in the past. They clearly showed that at 823-873K, the relaxation has not ended after even two months (Figs. 5 and 6). That holds even for low strain amplitude (Fig. 5). Design codes may underestimate the decrease in stress during relaxation and then the additional viscoplastic strain provided by long term relaxation.

Even if, a master curve could be plotted for long term duration using the cumulative viscoplastic strain, which still remains to be proved, long relaxations should be incorporated in the design rules.

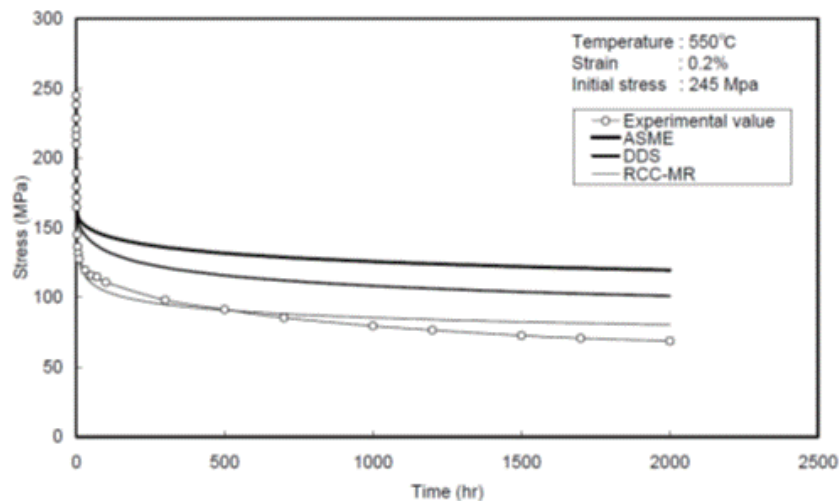


Fig. 5: relaxation curve in grade 91 F/M steel measured at JAEA (Japan) [5]. Comparison with the predictions of various design curves [5].

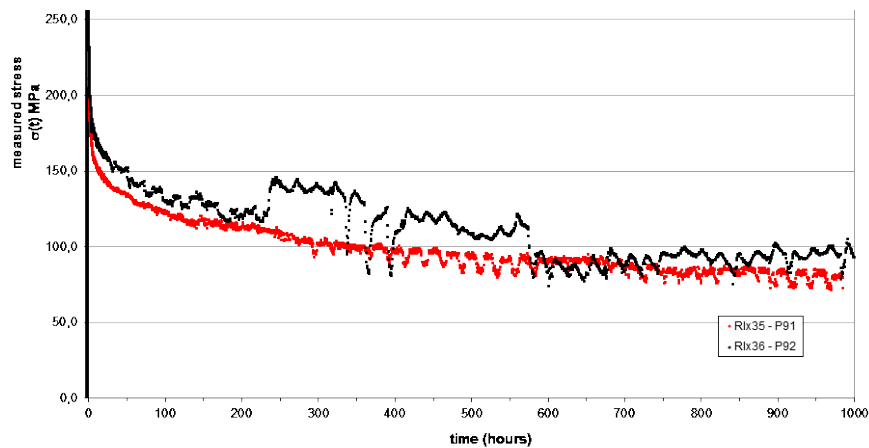


Fig. 6: relaxation curves measured in grade 91 and grade 92 F/Ms measured at CEA (0.35%, $2 \cdot 10^{-3}$ /s, 823K, one month) [3].

From our knowledge, experimental data and modelling concerning the saturation with respect to long hold time durations are missing.

This sub-project aims to provide new experimental data investigating the influence of long term hold times which are expected during in-service conditions. Additionally, macroscopic and physically-based modellings have been developed in order to predict both softening and lifetime. Finally, a strong interaction between experiments and modelling has been developed.

First of all, the new cyclic test results confirm the effects of strain amplitude and hold time (KIT). Then the results of dedicated tests including hold times of a few days applied at each cycle are provided by VTT and JRC. At both institutes, other specific tests including long hold times such as sequential tests are carried out.

CVR carried out fatigue relaxation with intermediate hold times and small strain amplitude allowing us to get a whole picture of hold time effects. Finally, PSI focuses on the experimental study of viscosity effects providing data for adjusting some of the model viscosity parameters.

Three different modelling approaches are then proposed. First, an engineering approach based on well-chosen analytical formulae and parameter adjustment by inverse identification (JRC). The second approach is based on thermodynamics and macroscopic hardening, flow and damage constitutive laws (KIT). The last approach is based on the physical mechanisms involved at the dislocation scale (glide and climb at high temperature) (CEA). The models used experimental data for material parameter and experimental validation. Many interactions between the different involved laboratories allow a rather rich validation in terms of experimental data.

Based on both experimental and modelling results, hold time effects on softening and lifetime are discussed. And further research is proposed, focusing mainly on low strain amplitudes which correspond to in-service conditions.

Final conclusions summarize the different results obtained in the framework of the Matisse project.

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- [5] Asayama and Y. Tachibana. Collect Available Creep-Fatigue Data and Study Existing Creep-Fatigue Evaluation Procedures for Grade 91 and Hastelloy XR. Japan Atomic Energy Agency, 2007.

2 Experimental results

The material used at JRC and VTT is the 60 mm thick plate (heat nr. 20057) from ArcelorMittal. The plate was tested in "as received" product state and was submitted to the following heat treatment: 1060°C – 4Hrs + Water Quenching + 760°C – 3hrs and 20min – Air Cooling. This plate was produced in full accordance with RCC-MR requirements (STR RM 2432).

Another material was used by KIT and PSI during the Matisse project. The USINOR P91 heat-treated material is a 30 mm plate (heat nr. 25240002) delivered by CEA. It material was used during the MATTER project. It was submitted to a heat treatment consisting of austenitization at 1323K during 30 min followed by quenching and tempering at 1053K for 1 h.

The chemical compositions of the two grade 91 steels are given in Table 1 and their tensile properties in Table 2.

Elements	Min/Max	Plate 20057	Plate 25240002
C	0.08/0.12	0.12	0.086
Mn	0.30/0.60	0.41	0.363
Si	0.20/0.50	0.24	0.324
Ni	<=0.20	0.1	0.149
Cr	8.00/9.50	8.32	8.91
Mo	0.85/1.05	1.02	0.917
Cu	<=0.10	0.05	0.068
Al	<=0.040	0.006	0.018
S	<=0.0050	0.001	0.001
P	<=0.0200	0.0091	0.017
V	0.180/0.250	0.235	0.198
Nb	0.060/0.100	0.084	0.080
N	0.030/0.070	0.041	0.0411

Table 1. Chemical compositions in % of the P91 steels tested and assessed in MATTER.

Identif.	Temp	R _{p02}	R _m
60 mm	RT	510	680
30 mm	RT	615	750
60 mm	600°C	260	322
30 mm	600°C	418	

Table 2. Tensile properties of the 60 mm thick P91 plate and the 30 mm thick plate.

2.1 Long hold time – Part I (Rami Pohja, VTT)

Six uniaxial low cycle fatigue tests (LCF) with cycling and hold periods in strain control, but hold periods of 24 to 72 h imposed only during particular cycles of the test, were conducted by VTT within the MatISSE project. The specimens for these tests were manufactured from a 60 mm thick plate of P91 steel, which had undergone austenitizing at 1060°C for 4 h, water quenching, tempering at 760°C for 3 h 20 min and air cooling. The chemical composition of the P91 steel used for the low cycle fatigue tests at VTT within the MatISSE project is presented in Table 1. Three of these tests ($T = 600^{\circ}\text{C}$, $\Delta\varepsilon = 0.5\text{-}0.9\%$, $t_h = 24\text{ h}$) included hold periods in tension for the first 20 cycles, followed by cycling to failure without hold periods. The other three tests ($T = 600^{\circ}\text{C}$, $\Delta\varepsilon = 0.5\text{-}0.9\%$, $t_h = 72\text{ h}$) included 4 hold periods imposed during 4 cycles corresponding to: $\sigma_N/\sigma_{1st} = 1, 0.9, 0.8$ and 0.75 , where σ_{1st} is the peak stress of the first cycle and σ_N is the peak stress of the N^{th} cycle. Other than those four relaxation periods, these tests were cycled to failure without hold periods. The locations

of the hold periods were selected so that the 2 first hold periods (occurring when $\sigma_N/\sigma_{1st} = 1$ and $\sigma_N/\sigma_{1st} = 0.9$) were at the early stage of the tests, the third hold period ($\sigma_N/\sigma_{1st} = 0.8$) was near the mid-life cycle and the fourth hold period ($\sigma_N/\sigma_{1st} = 0.75$) was near the end of the tests. The total strain ratio of $R = -1$ was used for all tests, which means that the peak strain is equal on tensile and compressive side.

The lifetimes measured from these six tests were compared to low cycle fatigue test results for P91 steel without hold period. Fig. 1 shows the number of cycles to failure of the six tests and the average curve provided by the MATTER project (LCF tests at 600°C without hold period). Based on the obtained results, the 24 to 72 h hold periods in tension occurring four times during the test did not have significant effect on the number of cycles to failure. The tests with four 72 h relaxation periods imposed during the selected four cycles exhibited slightly longer cyclic lives than tests with 20 relaxation periods (24 h) imposed at the beginning of the tests. However, the difference was not significant. It can be concluded that the test data are close the average LCF curve. This is not surprising because the tests included only few relaxation periods and during the relaxation periods the stress decreased rapidly to a relatively low level.

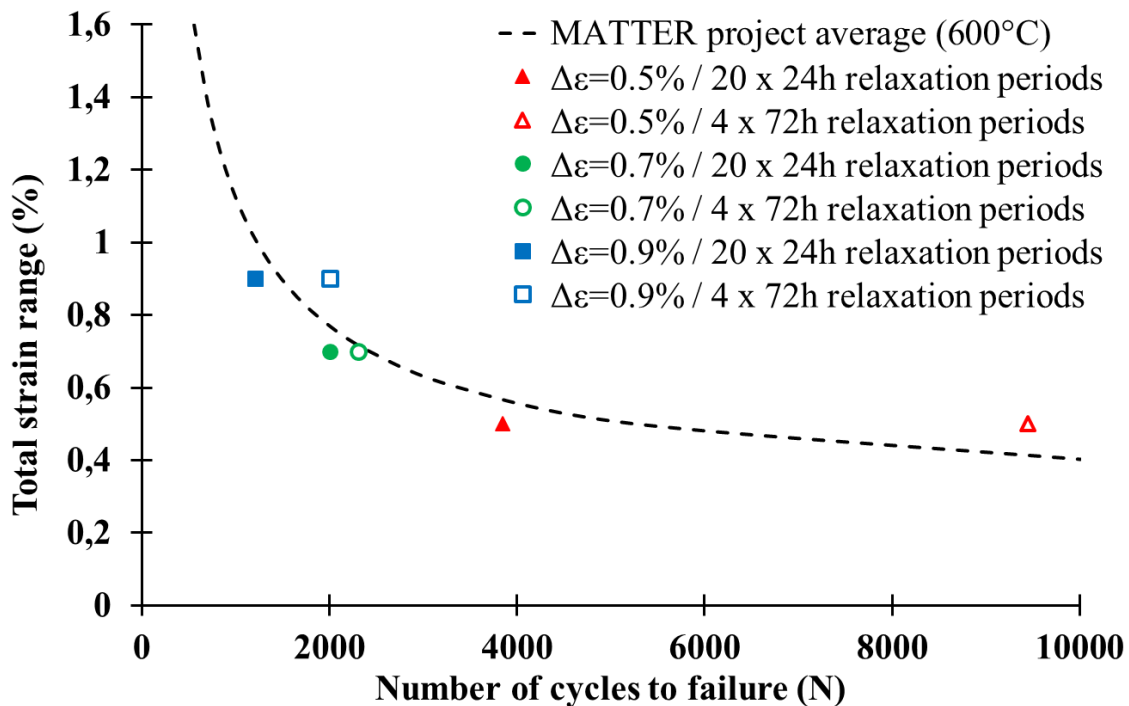


Fig. 7 The cyclic lives provided by the VTT low cycle fatigue tests including only four relaxation periods in tension and the average curve for low cycle fatigue tests without hold periods at 600°C from the MATTER project for P91 steel.

Fig. 8 shows the peak stresses on the tensile and compressive side in the LCF tests at 600°C with 20 relaxation periods (24 h) in tension at the beginning of the tests. When a relaxation period of 24 h on the tensile side was applied, the peak stress on the compressive side increased significantly, which can be clearly seen in Fig. 8. When the tests were continued without the relaxation periods, the peak stresses on the tensile and compressive sides saturated relatively quickly to an equal level.

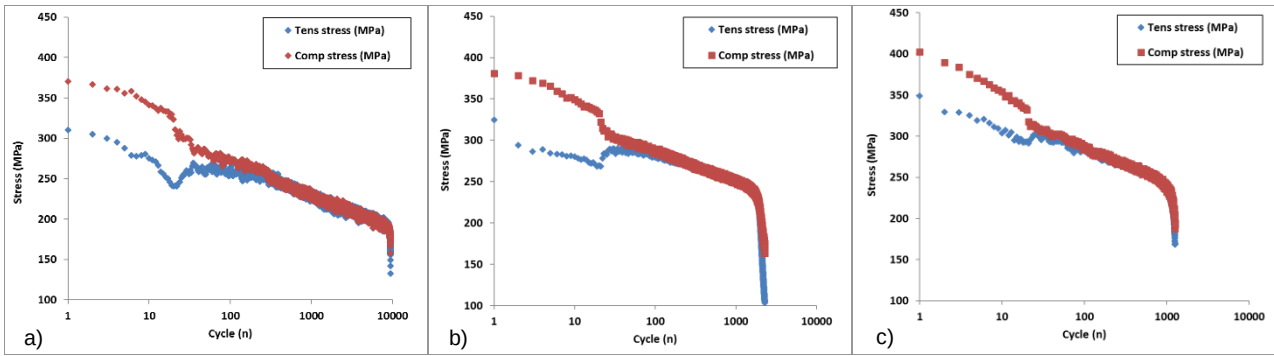


Fig. 8 The peak stresses on the tensile and compressive side in the LCF tests at 600°C with 20 relaxation periods (24 h) in tension at the beginning of the tests with the strain range of a) $\Delta\epsilon = 0.5\%$, b) $\Delta\epsilon = 0.7\%$ and c) $\Delta\epsilon = 0.9\%$.

Fig. 9 shows the relaxed stresses (the peak stress at the beginning of the hold period minus the stress at the end of the hold period) of 20 first cycles from tests with hold periods of 24 h and total strain ranges from 0.5% to 0.9%. It could be deduced from Fig. 9 that the amount of relaxed stress was larger for the largest total strain range (0.9%), but for the strain ranges of 0.5 and 0.7% the difference was not significant. It was also observed that the amount of relaxed stress decreased rapidly after the first few cycles. However, the tests results presented in [1] suggested that the amount of relaxed stress seemed to saturate with the normalized N/N_f . It was almost constant in the 0.1-0.8 range of the LCF tests with tensile hold periods (strain control testing).

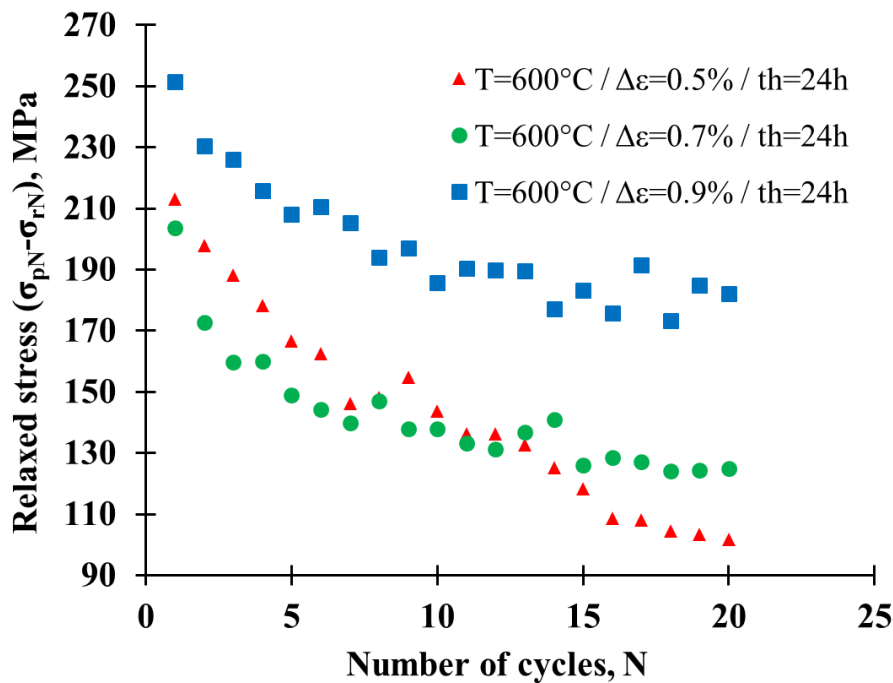


Fig. 9 The relaxed stresses (the peak stress at the beginning of the hold period minus the stress at the end of the hold period) measured during the 20 first cycles from LCF tests with hold periods of 24 h and total strain ranges from 0.5% to 0.9%.

Fig. 10 shows the tensile and compressive peak stresses and stress amplitude ($\Delta\sigma/2$) of LCF tests with 4 relaxation periods (72 h) in tension in the locations; $\sigma_N/\sigma_{1st} = 1, 0.9, 0.8$ and 0.75 , where σ_{1st} was the peak stress of the first cycle and σ_N was the peak stress of the N^{th} cycle. These tests also showed, that when a relaxation period of 72 h on the tensile side was applied, the peak stress on the compressive side increased significantly, followed by a relatively quick saturation of the peak stresses on the tensile and compressive side to an equal level.

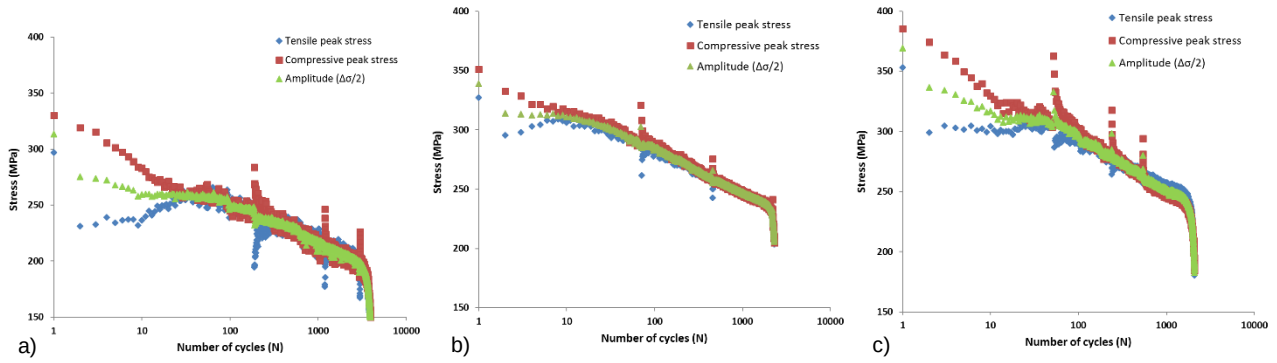


Fig. 10 The tensile and compressive peak stresses and stress amplitude ($\Delta\sigma/2$) of LCF tests with 4 relaxation periods (72 h) in tension in the locations; $\sigma_N/\sigma_{1st} = 1, 0.9, 0.8$ and 0.75 , where σ_{1st} is the peak stress of the first cycle and σ_N is the peak stress of the N^{th} cycle with $T = 600^\circ\text{C}$ and $\Delta\epsilon = 0.5\%$ in a), $T = 600^\circ\text{C}$ and $\Delta\epsilon = 0.7\%$ in b) and $T = 600^\circ\text{C}$ and $\Delta\epsilon = 0.9\%$ in c).

Figs. 11-13 show the tensile stresses as a function of time in the LCF tests during the 72 h relaxation periods, the stresses normalized by the peak stresses as function of time and the amount of the relaxed stress for each relaxation period. These results suggested that the amount of relaxed stress was larger in the as-received material, but decreased when the material is cyclically softened, exhibiting somewhat saturated amounts of relaxed stresses for the cyclic life ratio (N/N_i) in the 0.1-0.8 range, at least in the range of the imposed parameters ($T = 600^\circ\text{C}$, $\Delta\epsilon = 0.5\text{-}0.9\%$, $t_h = 24 - 72$ h).

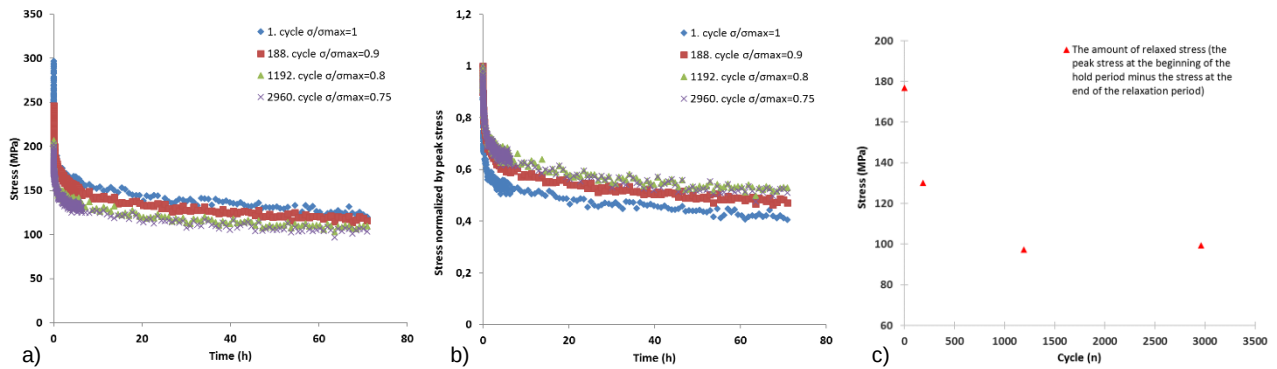


Fig. 11 a) The tensile stresses as function of time in the LCF test with $T = 600^\circ\text{C}$ and $\Delta\epsilon = 0.5\%$ during the 72 h relaxation periods; b) the stresses normalized by the peak stresses as function of time and c) the amount of the relaxed stress for each relaxation period.

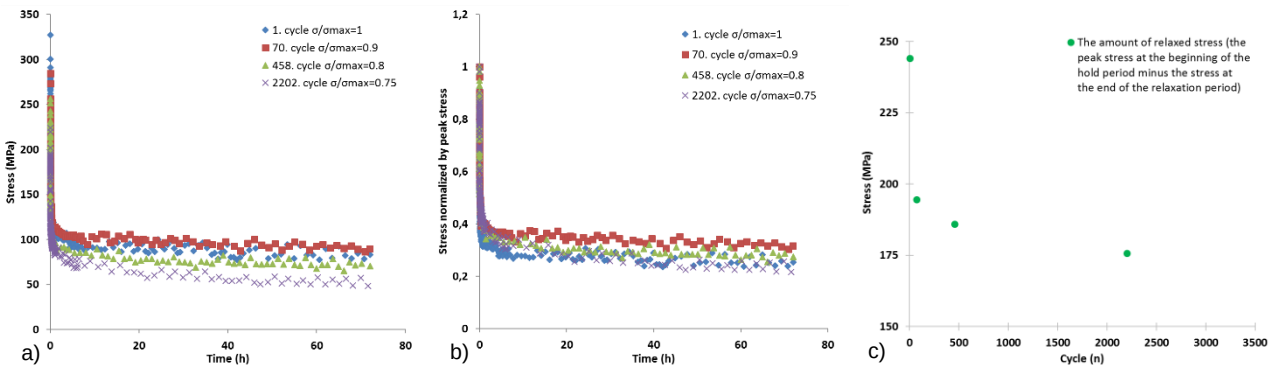


Fig. 12 a) The tensile stresses as function of time in the LCF test with $T = 600^\circ\text{C}$ and $\Delta\epsilon = 0.7\%$ during the 72 h relaxation periods; b) the stresses normalized by the peak stresses as function of time and c) the amount of the relaxed stress for each relaxation period.

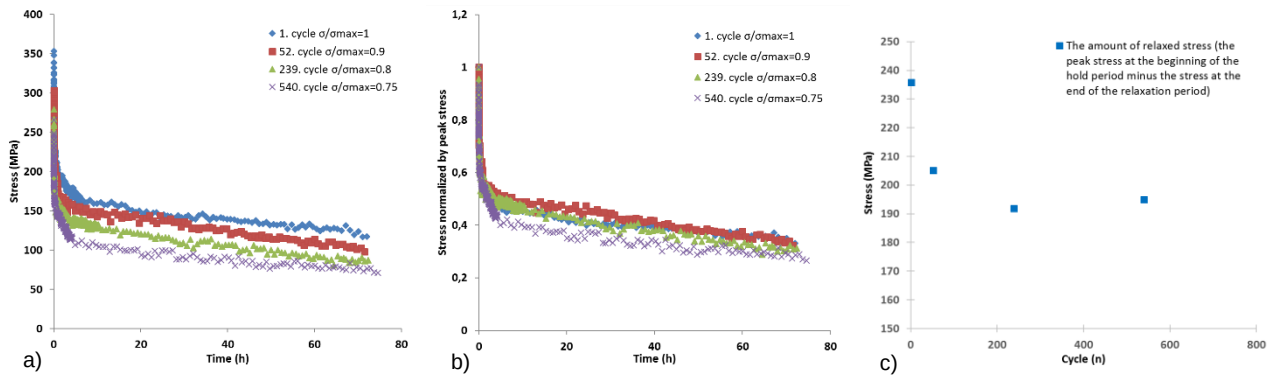


Fig. 13 a) The tensile stresses as function of time in the LCF test with $T = 600^{\circ}\text{C}$ and $\Delta\epsilon = 0.9\%$ during the 72 h relaxation periods; b) the stresses normalized by the peak stresses as function of time and c) the amount of the relaxed stress for each relaxation period.

Six LCF tests ($T = 600^{\circ}\text{C}$, $\Delta\epsilon = 0.5\text{-}0.9\%$) including a few relaxation periods in tension (24 - 72 h) during particular cycles of the tests were performed by VTT in the framework of the MatISSE project. These tests indicated, that when an intermediate relaxation period of 24 - 72 h on the tensile side was applied, the peak stress on the compressive side increased significantly, followed by a relatively quick saturation of the peak stresses on the tensile and compressive side to an equal level, when cycling was continued without relaxation periods. Regarding the fatigue life, the tests results suggested that few (intermediate) relaxation periods in tension in the LCF tests did not significantly decrease the cyclic life of P91 steel specimens. Regarding the stress relaxation, the stress decreased quickly to a relatively low level during the intermediate relaxation periods of 24 - 72 h in tension. The amount of relaxed stress was found to be larger in the as-received material, but to decrease when the material was cyclically softened, exhibiting somewhat saturated amounts of relaxed stresses for the cyclic life part (N/N_f) 0.1-0.8, at least for the range of applied parameters ($T = 600^{\circ}\text{C}$, $\Delta\epsilon = 0.5\text{-}0.9\%$, $t_h = 24 - 72$ h).

To increase our understanding on the obtained results, microstructural investigations are needed as a part of future work. Additionally, more tests are needed to confirm these results and also to study the effect of different parameter values (T , $\Delta\epsilon$, t_h) on the cyclic life, stress relaxation and softening behaviour of P91 steel.

2.2 Long hold time – Part II (Stefan Holström, JRC)

Modified test types were included in the MATISSE test program with the main objective of determining the softening response and relaxation behavior of P91 as a result of combined creep and fatigue. The JRC test conditions are given in Table 3.

Test type	Total strain ranges $\Delta\epsilon$	Temperatures	Hold time	Hold position
LCF/FR	0.5%, 0.7%	550, 600°C	up to 72 h holds	intermediate locations in softening curve, in tension (see Table 5)

Table 3. JRC test conditions and strain ranges for cyclic softening tests.

At JRC both LCF and fatigue-relaxation (FR) tests were performed. The tests include low cycle fatigue tests in strain control as shown in Fig. 14. These tests are used as reference for studying the material softening behavior. The fatigue-relaxation tests as the ones performed at VTT and CVREZ are LCF tests with intermediate relaxation periods, i.e. the hold times (t_h) introduced at specified cycles. The locations of the relaxation in relation to cycles to failure for LCF tests (N/N_f) are given in Table 5. The strain rate of the cyclic part of the test (LCF and FR) was $0.1\%/s$. All hold times were introduced at the tensile maximum (peak) location of the $\pm\Delta\epsilon/2$ cycle as shown in Fig. 15 and Fig. 16.

The changes in softening rates for the specific strain, location and hold time are compared to the LCF softening rates.

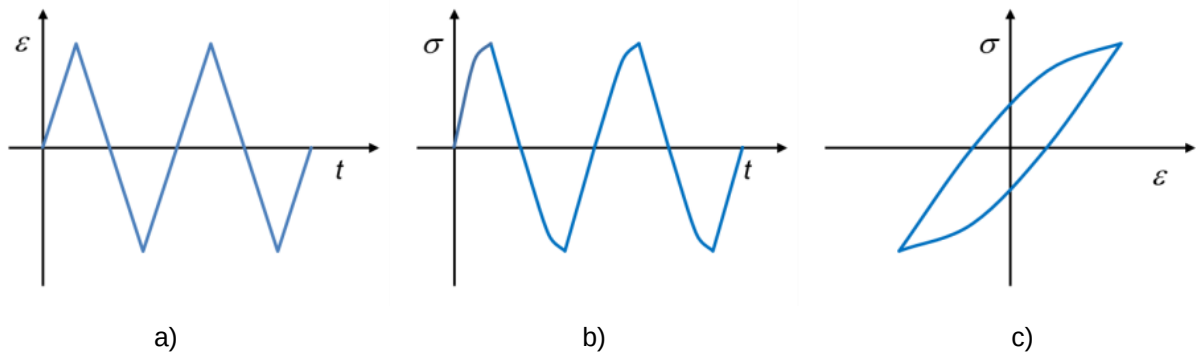


Fig. 14 The strain controlled low cycle fatigue (LCF) test with $R=-1$, a) strain-time plot, b) stress-time plot, c) stress-strain plot.

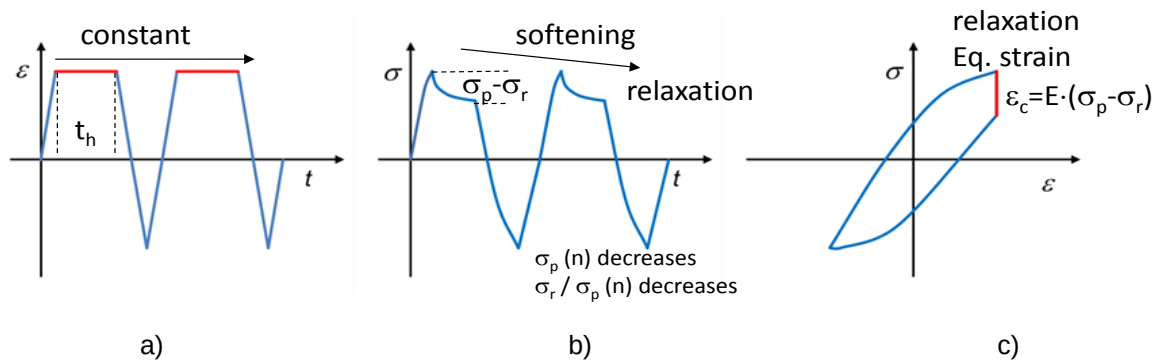


Fig. 15 The strain controlled fatigue-relaxation test with $R=-1$, hold in tension; a) strain-time plot, b) stress-time plot, c) stress-strain plot.

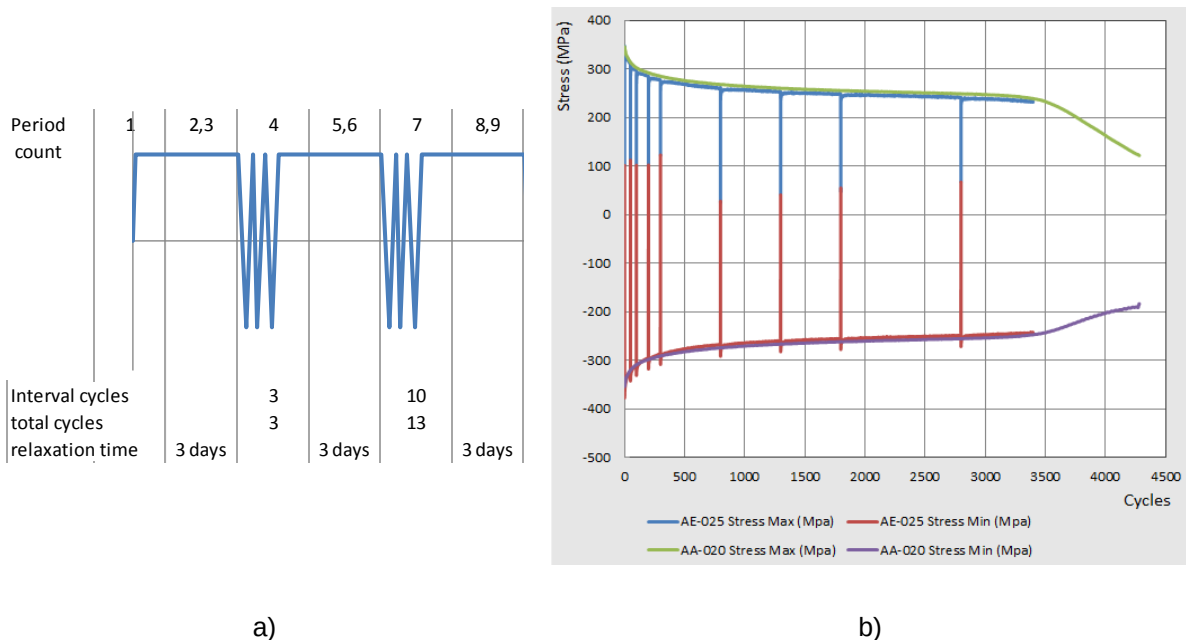


Fig. 16 a) Description of the testing sequence for hold time in specific cycles, b) softening curve of a test at $\Delta\epsilon=0.7\%$ / 550°C with 9 relaxation sequences.

2.2.1 Relaxation and FR tests with long hold times at specified cycles

In Table 4, the JRC relaxations tests data on virgin material (first loading) are provided. The longest relaxation is 948 hours in length. The peak stress σ_{p0} , and relaxed stress σ_{r0} for the tests at 0.25% and 0.35% strain amplitudes (550°C) are given at specified instances of time (0.01, 0.1, 0.3, 1, 3, 12, 24 and 72 hours of relaxation) to simplify the model fitting for the full data set. Note that the fluctuations in the load measurement, most probably caused by laboratory room temperature fluctuations, can potentially have had an impact on the measured relaxed stresses as observed in Fig. 17.

In Table 5, the relaxation data at specified cycle, given as a ratio of LCF cycles to failure (n/N_0) are provided. The peak stress at a specified cycle is σ_{pn} , the corresponding relaxed stress at the same cycle is σ_{rn} . Note the test AQ-006 (600°C, $\Delta\epsilon/2=0.25\%$) has relaxations in every cycle whereas the other 600°C, $\Delta\epsilon/2=0.25\%$ test has only a few relaxation periods at intermediate cycles, i.e. in different locations of the softening curve.

Specimen identifier	Tensile strain $\Delta\epsilon/2$	Cycle (n)	Temp. (°C)	σ_{p0} , (MPa) ¹⁾	σ_{r0} , (MPa)	Ratio: σ_{r0}/σ_{p0}	Hold time (h)				
AE-009	+0.35%	$\frac{1}{4}$	550	338	239	0.704	0.01				
					181	0.537	0.1				
					163	0.482	0.3				
					146	0.432	1				
					132	0.392	3				
					117	0.346	12				
					110	0.324	24				
					$\Delta\sigma=231$ $\Delta\sigma=\sigma_{p0}-\sigma_{r0}(t=72h)$				107	0.317	72
									77	0.228	948h, 39.5 days
AE-025	+0.35%	$\frac{1}{4}$	550	347	244	0.703	0.01				
					194	0.560	0.1				
					170	0.489	0.3				
					148	0.427	1				
					137	0.396	3				
					131	0.379	12				
					125	0.360	24				
					$\Delta\sigma=230$				117	0.337	72
AQ-001	+0.35%	$\frac{1}{4}$	600	308	192	0.623	0.01				
					144	0.468	0.1				
					125	0.406	0.3				
					111	0.361	1				
					108	0.351	3				
					96	0.312	12				
					81	0.263	24				
					$\Delta\sigma=228$				(84)	(0.260)	(72)

AQ-004	+0.35%	¼	600	300	182	0.607	0.01
					128	0.428	0.1
					113	0.377	0.3
					99	0.331	1
					95	0.317	3
					87	0.291	12
					78	0.260	24
$\Delta\sigma=235$					65	0.217	72
AE-024	+0.25%	¼	600	281	178	0.633	0.01
					128	0.456	0.1
					114	0.406	0.3
					100	0.356	1
					87	0.310	3
					87	0.310	12
					80	0.285	24
$\Delta\sigma=204$					77	0.274	72
AQ-005	+0.25%	¼	600	277	180	0.650	0.01
					137	0.525	0.1
					123	0.471	0.3
					108	0.414	1
					100	0.383	3
					92	0.352	12
					84	0.272	24
$\Delta\sigma=193$					80	0.306	72
AQ-006	+0.25%	¼	600	247	163	0.660	0.01
					120	0.486	0.1
					106	0.429	0.3
					98	0.397	1
					99	0.401	3
					94	0.381	12
					91	0.368	24
$\Delta\sigma=155$					(92)	(0.296)	(72)

Table 4. Virgin material peak stress σ_{p0} and relaxed stress σ_{r0} at specified strain range, temperature and hold time. Note that a value in parenthesis indicates an uncertain measurement due to a fluctuating load signal.

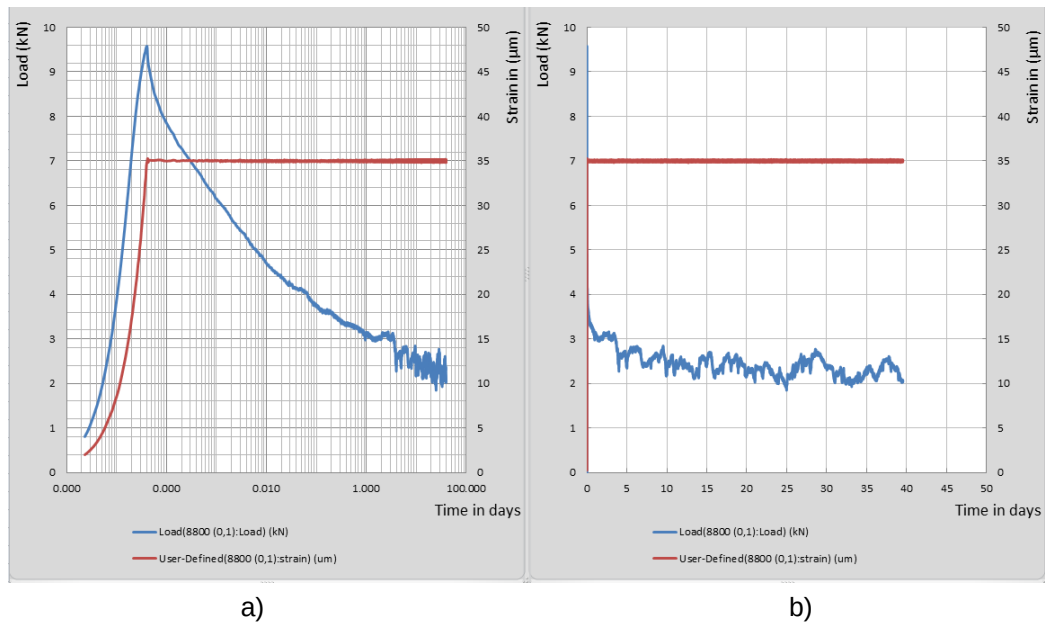


Fig. 17 Relaxation curve (load, kN ,blue line) for the longest virgin material relaxation performed at 550°C and at a constant strain of 0.35% (displacement, μm , red line) plotted with respect to a) logarithmic and b) linear scale. Note the difficulties in acquiring a stable signal in the load raw data. The signal fluctuations can be correlated with the changes in laboratory ambient temperature.

Specimen identifier	Tensile strain $\Delta\epsilon/2$	Cycle (n/N_{f0})	Temp. (°C)	σ_{pn} , (MPa) ¹⁾	σ_{rn} , (MPa) ²⁾	Ratio: σ_{rn}/σ_{pn}	Hold time (h)
AE-025	+0.35%	2800/4400	550	202	151	0.749	0.01
$n/N_{f0} \geq 0.5$					131	0.650	0.1
					121	0.600	0.3
					110	0.546	1
					100	0.496	3
					93	0.461	12
					92	0.456	24
					(68)	(0.337)	(72)
AQ-001	+0.35%	4/3010	600	264	180	0.682	0.01
$n/N_{f0} \sim 0.001$					135	0.512	0.1
					117	0.443	0.3
					104	0.394	1
					100	0.379	3
					93	0.352	12
					81	0.307	24
					67	0.254	72

AQ-001	+0.35%	14/3010	600	256	180	0.703	0.01
n/N _{f0} ~0.005					138	0.539	0.1
					120	0.469	0.3
					106	0.414	1
					99	0.387	3
					93	0.363	12
					90	0.351	24
					(74)	(0.289)	72
AQ-001	+0.35%	143/3010	600	234	167	0.714	0.01
n/N _{f0} ~0.05					131	0.560	0.1
					116	0.496	0.3
					101	0.432	1
					94	0.402	3
					81	0.346	12
					74	0.316	(24)
					71	0.304	72
AQ-001	+0.35%	443/3010	600	210	158	0.753	0.01
n/N _{f0} ~0.15					123	0.586	0.1
					110	0.524	0.3
					98	0.467	1
					89	0.424	3
					77	0.367	12
					59	0.281	24
					(76)	(0.362)	(72)
AQ-001	+0.35%	1443/3010	600	221	96	0.435	0.01
n/N _{f0} ~0.5					79	0.358	0.1
Initiation of crack suspected					72	0.326	0.3
					65	0.294	1
					66	0.299	3
					62	0.281	12
					61	0.276	24
					64	0.290	(72)
AE-024	+0.25%	3921/6640	600	185	138	0.747	0.01
n/N _{f0} ~0.6					111	0.601	0.1
					99	0.536	0.3
					88	0.477	1
					77	0.417	3
					68	0.368	12
					66	0.357	24
					(48)	(0.260)	72

AQ-006	+0.25%	2/6640	600	213	148	0.695	0.01
n/N _{f0} <0.001					119	0.559	0.1
					107	0.502	0.3
					99	0.465	1
					91	0.427	3
					75	0.352	12
					58	0.272	24
					57	0.268	72
AQ-006	+0.25%	3/6640	600	205	142	0.693	0.01
n/N _{f0} <0.001					115	0.561	0.1
					105	0.512	0.3
					97	0.473	1
					89	0.434	3
					78	0.380	12
					(85)	(0.415)	24
					(84)	(0.410)	72
AQ-006	+0.25%	10/6640	600	193	140	0.725	0.01
n/N _{f0} ~0.001					116	0.601	0.1
					107	0.554	0.3
					96	0.497	1
					93	0.482	3
					75	0.389	12
					(92)	(0.477)	24
					69	0.358	72
AQ-006	+0.25%	11/6640	600	223	162	0.725	0.01
n/N _{f0} ~0.001					132	0.591	0.1
					119	0.533	0.3
					108	0.483	1
					99	0.443	3
					96	0.430	12
					88	0.394	24
					76	0.340	72

Table 5. Peak σ_{pn} , relaxed stress σ_{rn} at different n/N_f for tests with $\Delta\epsilon=0.7\%$ (550°C), $\Delta\epsilon=0.7\%$ (600°C) and $\Delta\epsilon=0.5\%$ (600°C). In the case of tests AQ-006, the relaxations are imposed for every cycle. For all other tests the relaxation is imposed at specified cycles (n/N_{f0}). The cycles to failure for LCF (N_{f0}) were obtained from the MATTER project and calculate from a Manson-Coffin relation.

The relaxation behaviour of the virgin material is shown in Fig. 18. It can be observed that at 600°C the stress relaxes below 100 MPa in a few hours whereas at 550°C it takes up to 100 h. The apparent strain rate at 100 MPa for the $\Delta\epsilon/2=0.25\%$ / 600°C test is $\sim 3.0 \cdot 10^{-5}$ 1/h (calculated from relaxation rate and a Young's modulus of $E=156250$ MPa), about one order of magnitude higher than the creep strain rate achieved in a tensile creep test at the same stress /temperature level (600°C / 100 MPa). A creep strain of 0.25% is attained in 350 h at this stress and temperature and after 16150 hours (1.85 years) the strain is about 1% as shown in Fig. 19. The uniaxial (minimum) creep rate at this point in time is $3.6 \cdot 10^{-7}$ 1/h. It is clear that the uniaxial creep strain rates and the relaxation rates are not comparable. The relaxation response includes initial plasticity and recovery.

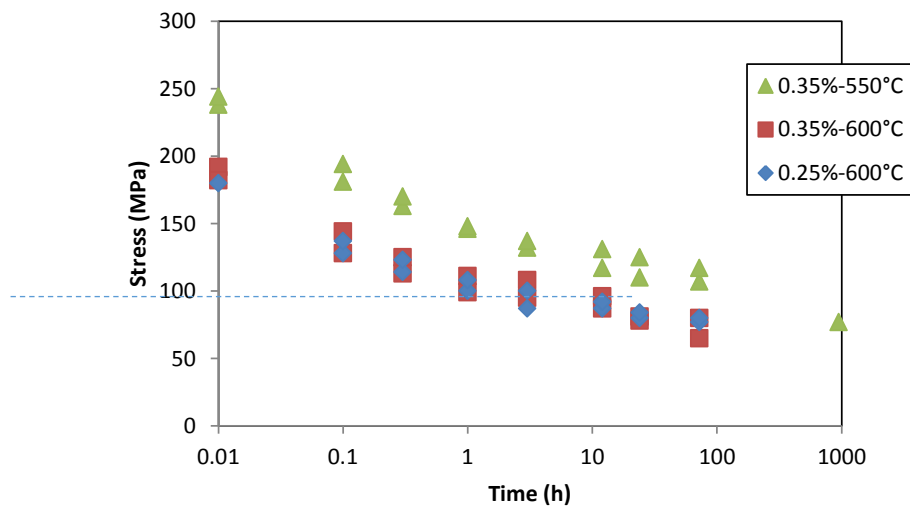


Fig. 18 Relaxation curves for 72 h hold times for virgin material tested at 0.25 and 0.35% tensile strain at 600°C and 550°C.

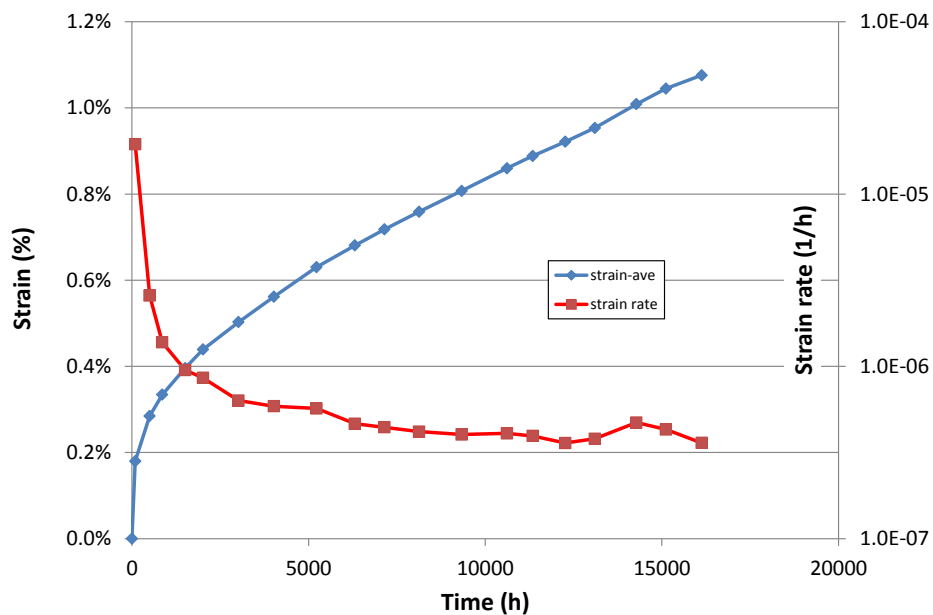


Fig. 19 Tensile creep strain curve for test at 600°C/100MPa. The test is still running after 16150 h and has reached about 1.1% of creep strain and the minimum creep strain rate is likely to have been reached.

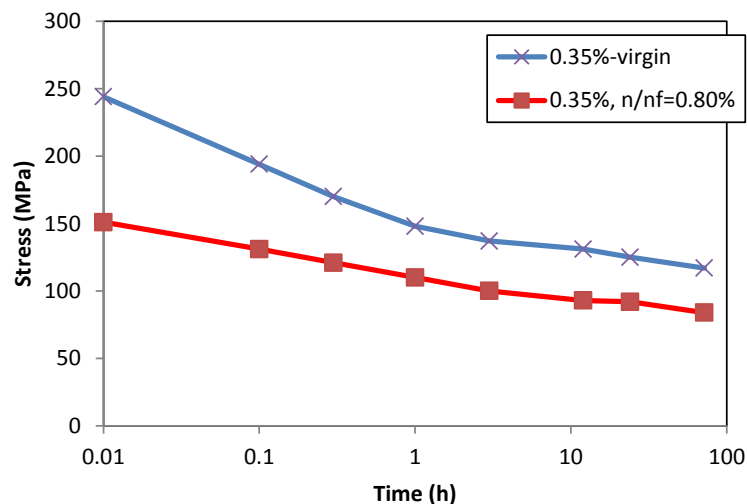


Fig. 20 Relaxation curves for virgin and softened material for $\Delta\epsilon/2 = 0.35\%$ at 550°C . A relaxation hold in virgin state compared to a hold after n cycles, $n/N_{f0}=80\%$.

2.2.2 Test in support of the modelling by CEA

JRC conducted some uniaxial tensile test with varying strain rates to support the modelling by CEA. For calibrating the strain rate effect on the peak stress three tests were conducted at 600°C . One with the standard (ISO 6892-2) strain rate for determining proof stress ($0.3\%/min$), one at the faster strain rate of $7.5\%/min$ used for attaining the tensile strength and a third test at a very slow rate of $0.05\%/min$ where the stress-strain response is expected to be affected by creep. The tests are shown in Fig. 21.

For calibrating the relaxation behaviour three additional relaxation tests were conducted. Two at a strain rate of $7.5\text{mm}/min$ and one at the slowest rate of $0.05\%/min$. The initial stress-strain response of the tests is shown in Fig. 22 and the following relaxation in Fig. 23.

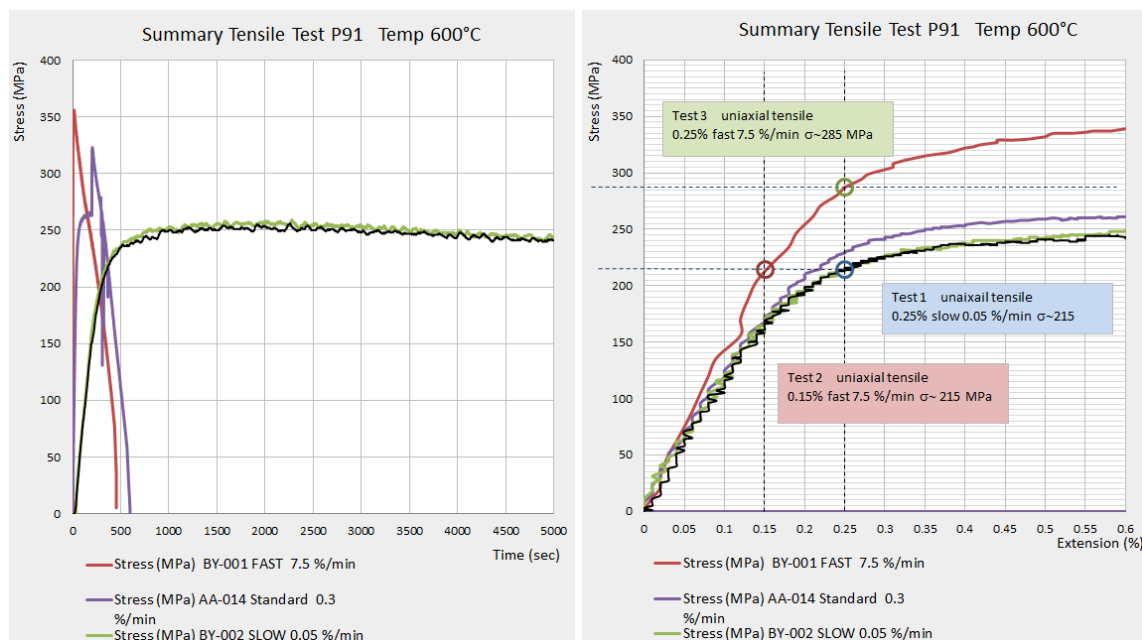


Fig. 21 Stress-strain response of variable strain rate tests at 600°C .

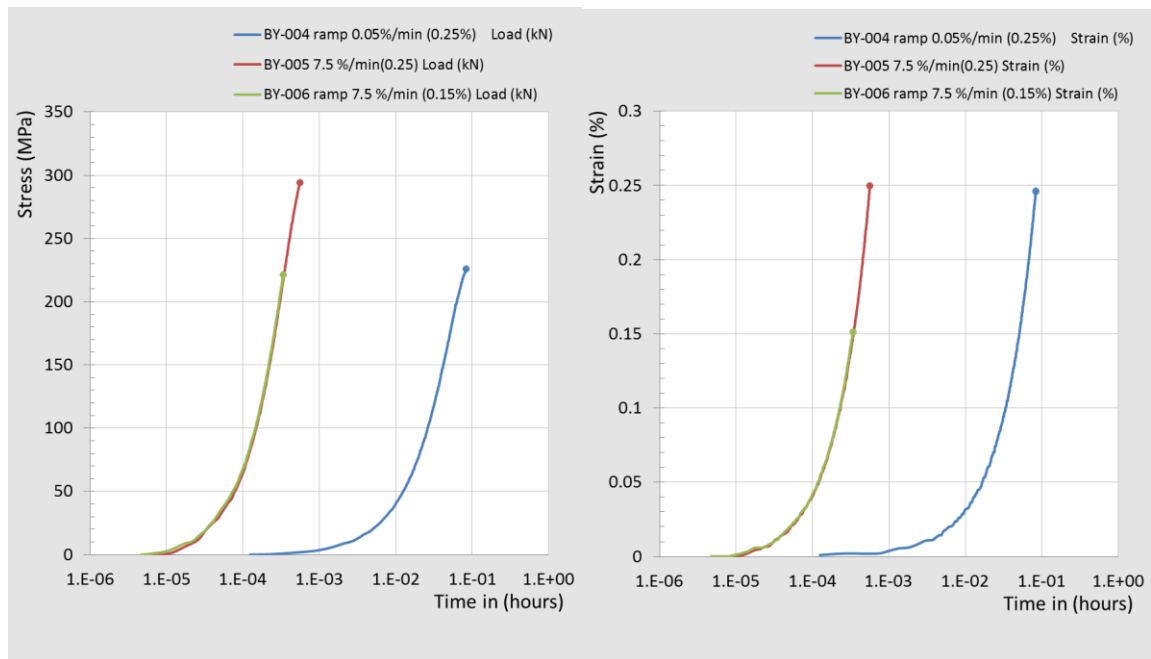


Fig. 22 Stress-strain response of the relaxation tests (600°C) before relaxation hold time.

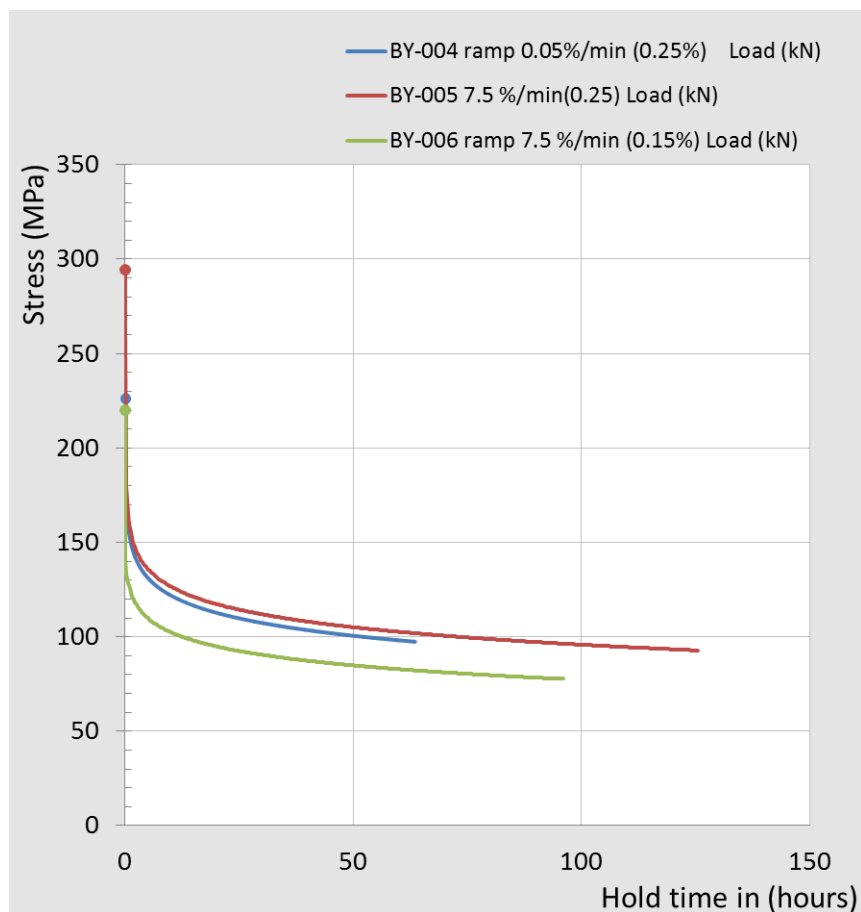


Fig. 23 Stress-relaxation curves from the variable strain rate tests at 600°C.

It can be seen that the relaxed stress after 50 h of relaxation was only marginally affected by the loading strain rate for the tests strained to 0.25% even though the peak stresses were considerably different, i.e. relaxed stresses of 105 and 100 MPa when the peak stresses were 294 MPa and 226 MPa. The relaxation ratios for these tests are $\sigma_p/\sigma_r=0.44$ and 0.36 respectively.

For the test conducted to 0.15% at the higher loading strain rate the relaxed stress after 50 h is considerably lower than for the slow 0.25% test with about the same peak stress at start, i.e. the relaxed stress was 85 MPa when the initial peak stress was 221 MPa. The relaxation ratio is $\sigma_p/\sigma_r=0.38$.

After 14 cycles with $\Delta\epsilon/2 = 0.35\%$ at 600°C the peak stress is approximately the same as for $\Delta\epsilon/2 = 0.25\%$ virgin material (3.6 seconds after relaxation has started) (Fig. 24). The relaxation curves are almost identical. The relaxation accounts for an increase in effective strain range of about 0.7% in tension.

The BY-005 test (7.5%/min) strained to 0.15% (virgin material) is relaxing much slower than the sample cycled at $\pm 0.25\%$ and softened to about the same peak stress (AE-024) (Fig. 25). The stresses of the curves coincide at a relaxation time of 3.6 seconds. The virgin material relaxes (σ_p/σ_r) 63% and the cyclically softened material by 68%. The difference is not so large.

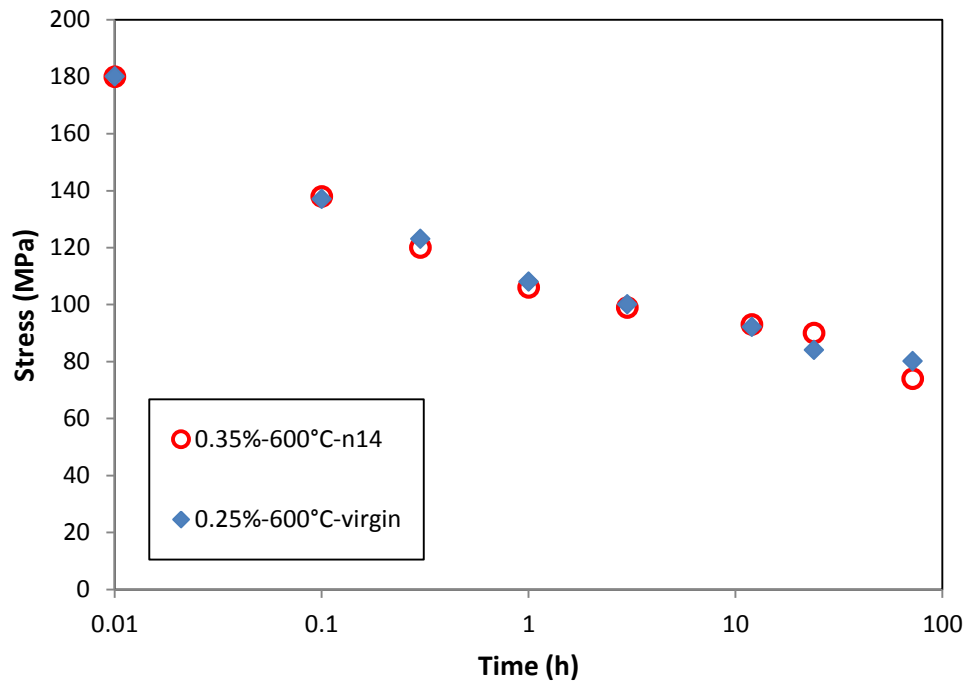


Fig. 24. Relaxation curves starting from the same peak stress.

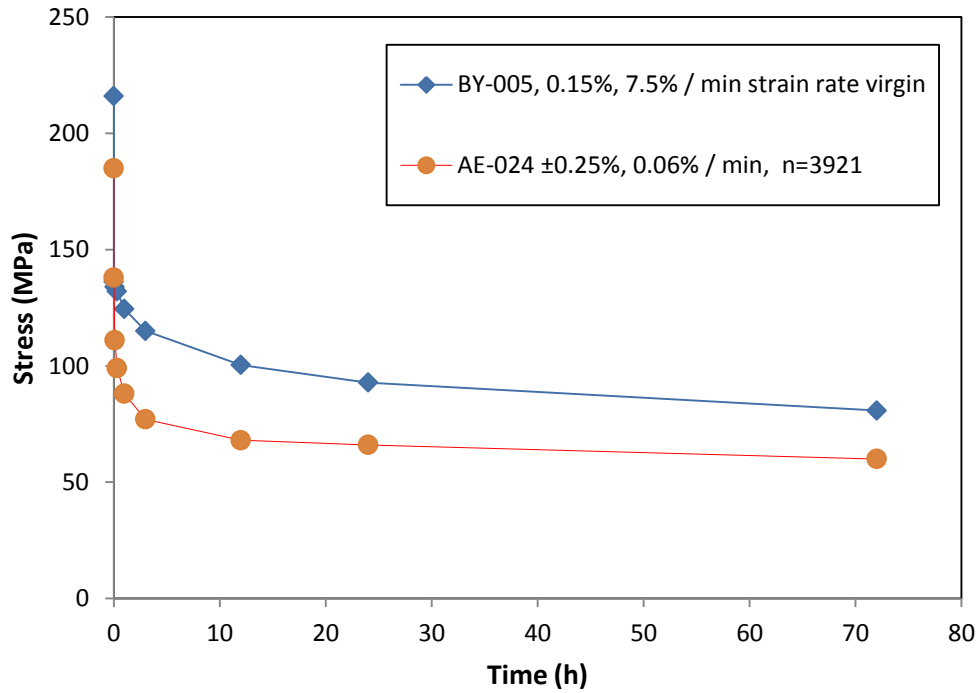


Fig. 25. Relaxation curves starting from the same peak stress and then diverging the one from the other.

2.3 Measurement of activation volume and energy (Jia Chao Chen, Ph. Spätig, PSI)

As part of effort on understanding the deformation and fracture in ferritic/martensitic steels, the present task is aimed to develop models which will provide a basic understanding and prediction of cyclic softening in tempered martensitic steels. Among various deformation mechanisms, dislocation motion is the most important one. The activation parameters of dislocations can be related to the rate controlling processes which is essential in developing prediction model. Therefore the measurements of basic material properties e.g. activation volume and energy for dislocation movement is the topic of this task.

2.3.1 Principles

The plastic deformation is mainly carried by dislocation movement when the load applied on the crystalline materials exceeds the flow stress which is determined by the resistance to dislocation motion. Various obstacles to dislocation motion exist in materials. Generally, work hardening and softening could result from the changes of the obstacles or the mobile dislocation density during plastic deformation. In the framework of thermal activation theory, there are two processes, thermal and athermal, for dislocations to overcome barriers. It depends on the kind of barrier. Long range interaction due to large barriers leads to athermal component (τ_i), varying slowly with the dislocation position on its glide plane and weakly dependent on temperature (T) through the shear modulus (μ). The short-range barrier leads to thermal component τ_e which depend on the plastic strain rate ($\dot{\gamma}_p$) and temperature. Since both types of obstacles are exist in the materials, the measured total shear stress (τ) is given by [1]

$$\tau = \tau_e(\dot{\gamma}_p, T) + \tau_i(\gamma_p) \quad (1)$$

If assuming that a single dominant activation mechanism is operative, then the plastic shear strain rate $\dot{\gamma}_p$ can be described by [2]

$$\dot{\gamma}_p = \frac{\rho_m}{l} \nu b S \exp\left(-\frac{\Delta G(\tau_e)}{kT}\right) \quad (2)$$

Where ρ_m is mobile dislocation density; l is the obstacle spacing; ν is Debye frequency factor including an entropy term; b is the magnitude of the Burgers vector; S is the area swept by a dislocation per event of activation process; τ_e is the thermal component of resolved shear stress; k and T are Boltzmann's constant and temperature, respectively. The activation energy ΔG is given by

$$\Delta G = \Delta g - V_a \tau_e \quad (3)$$

Where Δg is the change in Gibbs free energy due to localized atomic displacement during activation (it is also the activation energy for $\tau_e=0$); V_a is referred to as the activation volume. The activation volume of the thermal process is given by

$$V_a = -\frac{\partial \Delta G}{\partial \tau} \Big|_T \quad (4)$$

Assuming the pre-exponential factor $\dot{\gamma}_{p0} = \frac{\rho_m}{l} \nu b S$ in equation (2) does not vary significantly during a strain rate jump, the activation energy can be described as

$$V_a = kT \left(\frac{\partial \ln(\dot{\gamma}_p / \dot{\gamma}_{p0})}{\partial \tau} \right) \cong kT \left(\frac{\partial \ln \dot{\gamma}_p}{\partial \tau} \right) \quad (5)$$

Thus, V_a can be obtained by experimentally accessible relations between $\dot{\gamma}_p$ and τ as

$$V_a \cong kT \left(\frac{\Delta \ln \dot{\gamma}_p}{\Delta \tau} \right) \quad (6)$$

Eq. (6) can be used to estimation of activation volume (V_a) by strain rate jump experimental.

According to the definition of activation energy, i. e. Eq. (4), the activation energy can be obtained by integration of the activation volume with respect to stress [3]

$$\Delta G = \int_{\tau}^{\tau_p} V_a d\tau \quad (7)$$

Where τ_p is the Peierls effective stress at 0 K. Since experimental data are only available above certain temperature, Eq. (7) could be separated into two stress regions. One is the region covered by experiment, i.e from largest measured stress, τ_m at lowest test temperature to lower measured stress τ at test temperature and another is from τ_p at 0 K to τ_m at lowest measured temperature. Thus the equation can be rewritten as

$$\Delta G = \int_{\tau_m}^{\tau_p} V_a d\tau + \int_{\tau}^{\tau_m} V_a d\tau = \delta G + \int_{\tau}^{\tau_m} V_a d\tau \quad (8)$$

At a specific $\dot{\gamma}_p$, from Eq. (2), one obtain

$$\Delta G = kT \ln \left(\dot{\gamma}_{p0} / \dot{\gamma}_p \right) = \alpha kT \quad (9)$$

Therefore, estimation of activation energy become to determine the coefficient of α . To do so, one firstly integrate from τ_m to τ yields the $(\Delta G - \delta G)$ at measured temperatures related to those stresses τ . Then linear fitting the curve of $(\Delta G - \delta G)$ and temperature obtain the parameter of α . The activation energy could be calculated based on Eq. (9) as a function of temperature and stress. This method is valid below room temperature since it is demonstrated that low temperature plastic deformation obeys Peierls mechanism [4].

At elevated temperature, due to stress and temperature dependence of activation energies, only apparent activation energy can be calculated. The well accepted steady state creep equation is

$$\dot{\epsilon} = A\sigma^n \exp\left(-\frac{Q}{kT}\right) \quad (10)$$

Where A and Q are pre-exponential constant and apparent activation energy, respectively. Denoting $m=1/n$, Eq. (10) could be modified as [5]

$$\sigma = \frac{1}{A^m} \dot{\epsilon}^m \exp\left(\frac{mQ}{kT}\right) \quad (11)$$

The stress strain rate sensitivity m could be calculated by

$$m = \left. \frac{\partial \ln \sigma}{\partial \ln \dot{\epsilon}} \right|_T \quad (12)$$

Eq. (11) and (12) could be used to calculate the apparent activation energy in tensile test with various strain rates. The apparent activation energy Q defined as $\left(\frac{k \partial \ln \sigma}{m \partial (1/T)}\right)$. The slope of the curve displaying $\ln \sigma$ in function of $1/T$ at a constant of $\dot{\epsilon}$ provides a value of mQ/k .

2.3.2 Experiments

The investigated material is P91-CEA in the form of plate with a thickness of 30 mm, being supplied by Arcelor Mittal (heat no. 81243) in the normalized and tempered condition. Its detailed description and chemical composition can be found in the former chapter. Two types of samples were machined for present strain rate jump tensile test. The round dog-bone sample has a gauge size of 3 mm in diameter and 18 mm in length and the mini round dog-bone sample has a gauge size of 1.5 mm in diameter and 8 mm in length. The loading axial of dog-bone specimen are parallel to rolling direction. The tensile strain rate jump tests were performed on an RMC 100 Schenk machine in air at temperature from 123 K to 473 K with a nominal strain rate of 8×10^{-6} at the beginning ($\dot{\epsilon}_1$) and jumped upward to 2.5×10^{-4} ($\dot{\epsilon}_2$) at the yield stress (0.2% plastic strain). In order to investigate the effect of plastic strain on the stress strain rate sensitivity, the strain rate was jumped again downward to 8×10^{-6} ($\dot{\epsilon}_3$) at plastic strain of 2%. After strain-stress curve reaching to steady state curve, then the strain rate jumped upward again to 2.5×10^{-4} ($\dot{\epsilon}_4$) and the tensile test continued up to failure. Stress strain rate sensitivity at first (from $\dot{\epsilon}_1$ to $\dot{\epsilon}_2$) and second (from $\dot{\epsilon}_2$ to $\dot{\epsilon}_3$) jump is denoted as m_1 and m_2 , respectively. Because the temperature chamber equipped in RMC 100 Schenk machine is limited at 473 K, therefore the home made tensile test machine for miniaturized sample was employed to perform strain rate jump tensile tests in vacuum under pressure of around 10^{-3} Pa from 473 to 873 K. To improve the data quality, other strain rate jump steps from nominal 1×10^{-6} to 3×10^{-5} has been performed additionally at 773, 823 and 873 K. The data acquisition counting rate is one measurement per second in all tests. The shear stress is calculated from the measured uniaxial tensile stress σ by dividing Taylor's factor M , $\tau = \sigma/M$ where M is about 3 for bcc structures.

2.3.3 Results

The representative stress-strain curves of uniaxial tensile tests with strain rate jump are showed in Figs. 26-30. Obviously, 4 different types of the curves could be identified in the temperature regions of below 293 K, 373-523 K, 523-723 K, and above 723 K. The smooth stress-strain curves are characterized by a fast transition during strain rate jump below 293 K (see Fig. 26). The stress promptly responses to the strain rate jump and

show positive strain rate sensitivity. In the temperature between 373 K and 523 K, the stress-strain curves are smooth but the stress in transition period during strain rate jump shows relaxations (see Fig. 27). The stress leaps up immediately when strain rate jump upward and then follows a relaxation (stress decrease gradually). In the case of strain rate downward jump, the stress drop immediately then follows a recovery (stress increase gradually). The stress may even show negative strain rate sensitivity after stress reached to the steady state value. In the temperature region of from 523 to 723 K, the stress-strain curves show a serrated shape indicating the typical dynamic strain aging behavior (see Fig. 28). The stress relaxation behavior after strain rate jumping remains.

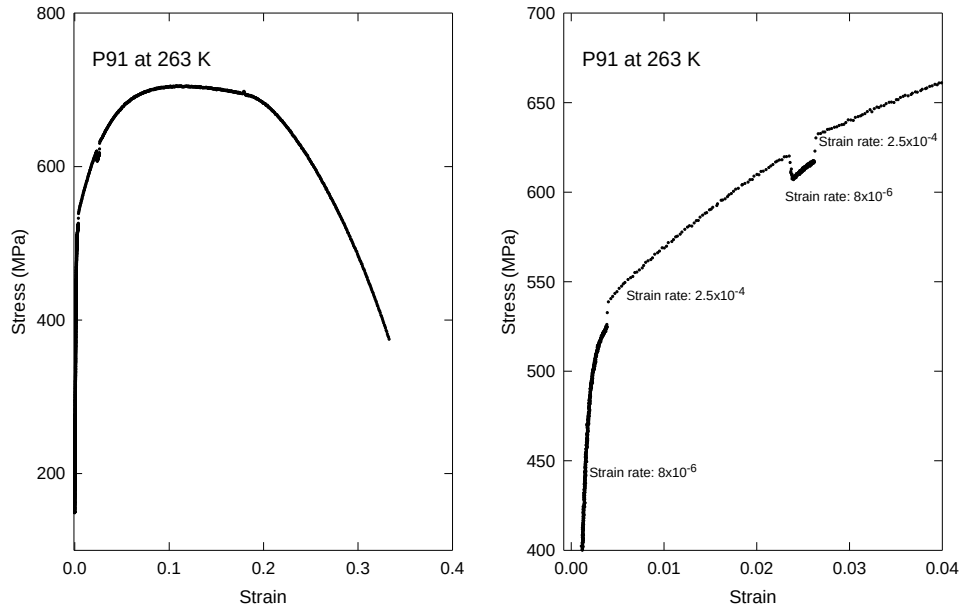


Fig. 26: Stress vs strain curves of strain rate jump tensile test at 263K. Left and right plots show complete tensile curve and detailed steps after strain jumps, respectively.

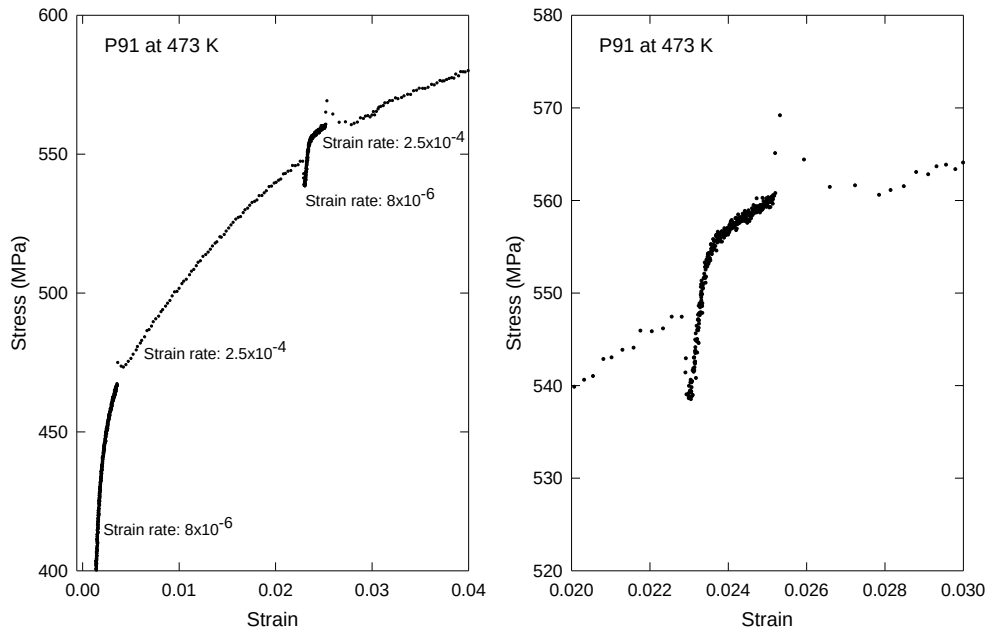


Fig. 27: Stress vs strain curves of strain rate jump tensile test at 473K. The left shows steps after strain jump. The right plots an enlarged second and third strain jumps. The stress evolution shows clear relaxations after jumps.

The stress-strain curve becomes smooth again at temperature above 773 K (see Fig. 29). The transition period after strain rate jump takes longer than at low temperature region. The relaxation after strain rate jump is not remarkable at this temperature region.

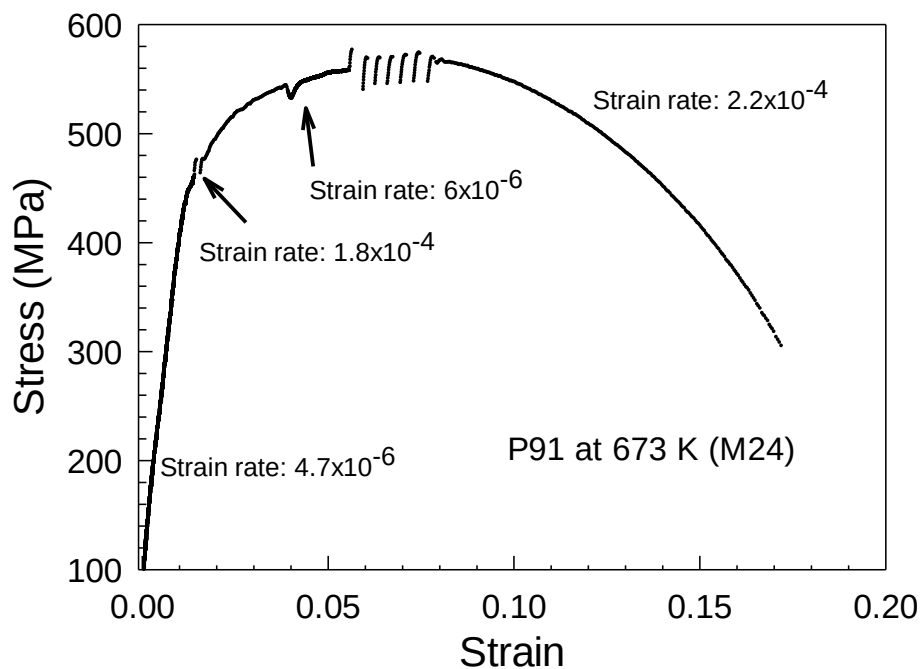


Fig. 28: Stress vs strain curves of strain rate jump tensile test at 673K. The curve shows a serrated shape indicating a typical dynamic strain aging behavior.

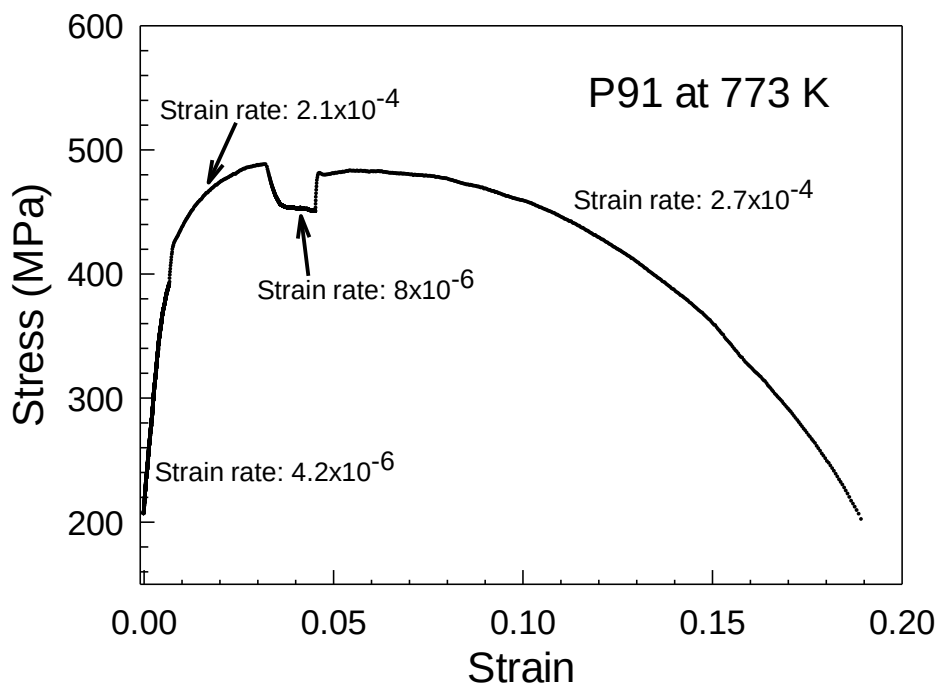


Fig. 29: Stress vs strain curves of strain rate jump tensile test at 773K.

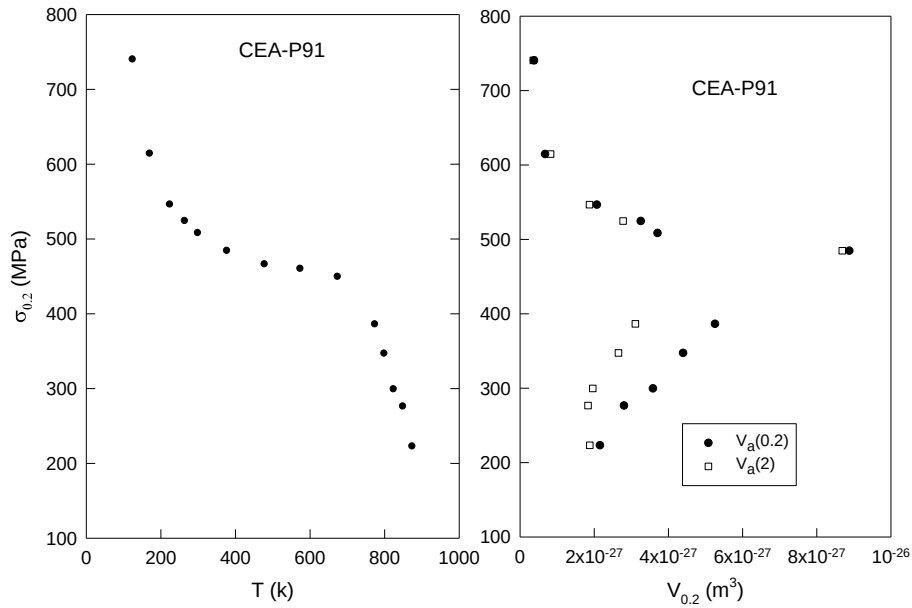


Fig. 30: Temperature dependence of yield stress and activation volume of CEA-P91

The temperature dependence of the yield stress, $\sigma_{0.2}$ defined at 0.2% plastic deformation and activation volumes measured by strain rate jumps (Eq. 5) at 0.2% [$V_a(0.2)$] and 2% [$V_a(2)$] plastic deformation are presented in Fig. 30. The dependence of stress with temperature shows regions named as low (<300 K), intermediate (300 K – 700 K) and high temperature (>700 K) regions. In low and high temperature of regions, the flow stress decreases with temperature increasing. The deformation should be therefore governed by thermal activation process. The measured activation volume of P91 is similar as reported in [4] for F82H and 9Cr-1Mo-VNb model alloy. The V_a value is relatively small at high stress, i.e. at the low temperatures. The V_a value increases rapidly with temperature close to the intermediate region, which is consistent with the dislocation motion governed by athermal process. The stress strain rate sensitivity close to zero or even negative, results in a very large V_a value. At temperature above 773 K, the activation energy decreases with temperature increasing. The remarkable difference between $V_a(0.2)$ and $V_a(2)$ demonstrated that the stress strain sensitivity depends more strongly on plastic strain in the high temperature region. As described in the section named as “Principle”, the task to measure the activation energy at low temperature region consist in determining the coefficient α in Eq. (9), which could be done by using a linear fitting of the ($\Delta G - \delta G$) evolution (Eq. 8) as a function of temperature. This is schematically shown in Fig. 31. As expected, the temperature dependence of ΔG is linear. The δG and α are determined as 0.31 ± 0.04 eV and 36.8 ± 2.3 , respectively.

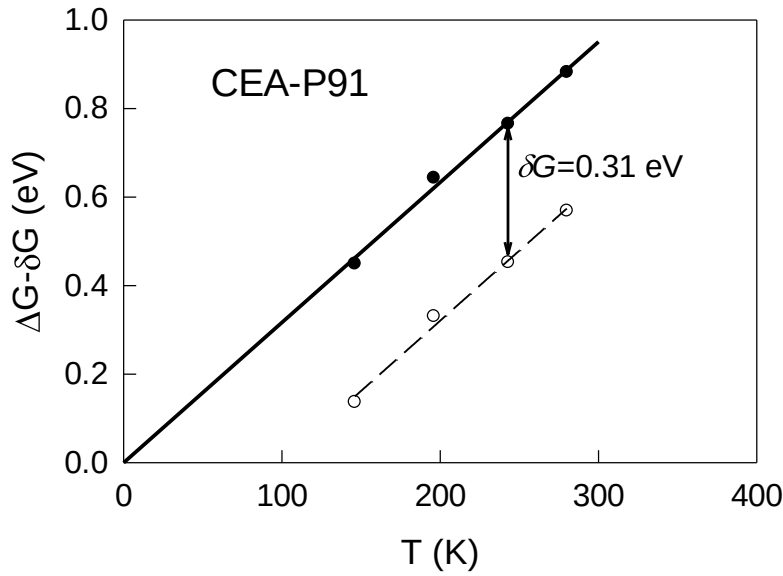


Fig. 31 Schematic picture showing the determination of ΔG and δG after integration of the activation volume. Open symbols indicating the integration of V_a from τ to τ_m as a function of the temperature. The δG value is obtained by requiring that $\Delta G=0$ at 0 K. A linear fitting of measured points gives coefficient α of 36.8.*

In the high temperature region, the approach for determining the apparent activation energy is based on Eq. (11) and (12). The temperature dependence of stress at the two specific strain rates is presented in Fig. 32. For comparison, the plot includes the low temperature data too. Two branches separated at around 673-773 K show clearly two completely different dislocation motion mechanisms. Since an assumption of constant microstructure in steady state creep regime is required, the apparent activation energy is calculated by using the two sets of data of σ_2 . The apparent activation energy (Q), deduced from the temperature dependence of the stress in the low temperature regime ($Q = 0.43$ at 146 K and $=0.76$ at 243 K) agrees exactly with ΔG estimated by Eq. (9). The calculated Q values obtained by applying Eq. (11) and Eq. (12) at high temperature are listed in Table 6. It can be seen that activation energy Q decrease with increasing the temperature from 6.18 eV at 773 K to 2.41 eV at 873 K which are mainly due to the changes of stress strain rate sensitivity. The exponent m increases with increasing the temperature from 0.024 at 773 K to 0.070 at 873 K. These results are comparable with the ones published in ref. [6] where the exponent m measured by creep test changed approximately linearly from 0.059 at 823 K to 0.10 at 923 K. However, the apparent energy measured in [6] showed reverse trends as a function of stress. The present measured Q values decreases with decreasing stress (increasing temperature). The result matrix (Table 6) summarizes the present measurements for thermal activation parameters of dislocation movement.

Table 1: Result matrix of strain rate jumping tensile tests

Sample ID	T(K)	$\dot{\epsilon}_1$	$\dot{\epsilon}_2$	$\dot{\epsilon}_3$	$\sigma_{0.2}$ (MPa)	$\Delta\sigma_{0.2}$ (MPa)	m1	σ_2 (MPa)	$\Delta\sigma_2$ (MPa)	m2	$V_{0.2}$ (m ³)	V_2 (m ³)	ΔG (eV)
P91-005	123	7.10E-6	2.54E-4	9.20E-6	740.6	48.6	0.0178	828.0	47.6	0.0178	3.75e-28	3.55e-28	0.39
P91-004	169	7.56E-6	2.45E-4	9.03E-6	614.7	36.2	0.0165	699.2	28.1	0.0124	6.72e-28	8.22e-28	0.54
P91-003	223	8.50E-6	2.40E-4	9.00E-6	546.7	14.9	8.05e-3	679.2	16.2*	7.35e-3	2.07e-27	1.87e-27	0.71
P91-002	263	6.48E-6	2.48E-4	9.00E-6	524.7	12.2	6.31e-3	620.0	13.0	6.39e-3	3.25e-27	2.78e-27	0.83
P91-001	298	7.56E-6	2.39E-4		508.6	11.5	6.47e-3	-	-	-	3.71e-27		0.94
P91-006	376	7.80E-6	2.46E-4	7.90E-6	484.9	6.05	3.59e-3	570.8	6.16	3.16e-3	8.88e-27	8.69e-27	
P91-007	477	6.30E-6	2.38E-4	7.30E-6	466.8	0	0	547.3	-6.1	-	+Inf	-	
P91-M23	573	4.00e-6	1.68e-4	6.50e-6	460.8	0	0	511.0	-	-	-	-	
P91-M24	673	4.65e-6	1.83e-4	6.14e-6	450.1	0	0	538.0	-	-	-	-	
P91-M12S	773	1.20E-6	2.60E-5	3.37E-6	379.0	20.2	0.0169	451.3	23.9	0.0266	4.87e-27	2.74e-27	6.18\$
P91-M15	773	4.20E-6	2.13E-4	8.00E-6	386.5	23.9	0.0153	483.0	33.8	0.0221	5.26e-27	3.11e-27	7.44\$
P91-M18	798	2.66E-6	2.52E-4	9.47E-6	347.4	34.2	0.0206	441.2	40.9	0.0296	4.40e-27	2.65e-27	6.21\$
P91-M19	823	3.16E-6	2.28E-4	8.79E-6	299.7	40.7	0.0298	381.1	56.6	0.0494	3.58e-27	1.96e-27	3.59\$
P91-M13S	823	1.40E-6	3.36E-5	3.90E-6	302.0	30.1	0.0299	371.1	40.1	0.0531	3.60e-27	1.83e-27	3.34\$
P91-M20	848	2.86E-6	2.23E-4	8.14E-6	276.7	54.6	0.0413	355.6	63.5	0.0594	2.80e-27	1.83e-27	3.09\$
P91-M21	873	3.34E-6	2.43E-4	9.03E-6	223.3	72.0	0.0652	318.8	63.3	0.0672	2.16e-27	1.88e-27	2.62\$
P91-M14S	873	2.00E-6	2.97E-5	3.80E-6	213.0	42.5	0.0674	277.7	38.7	0.0730	2.29e-27	1.92e-27	2.41\$

*: at plastic strain of 3.6%

\$: measured at plastic strain of 2%

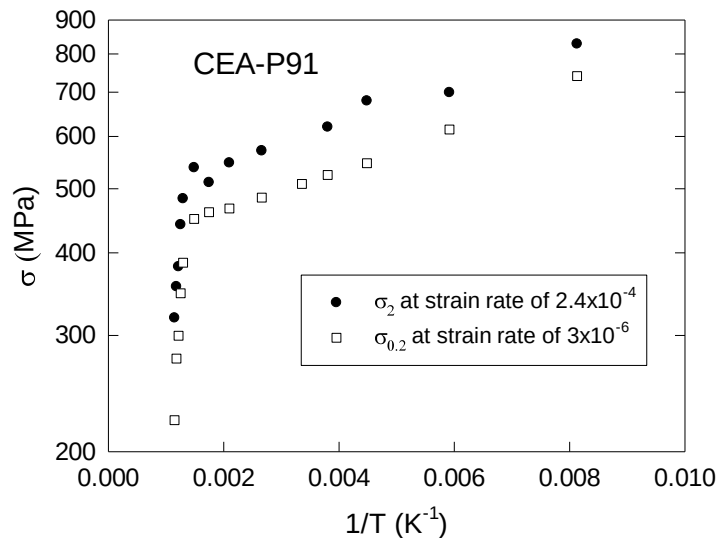


Fig. 32 Stresses as a function of temperature at given strain rates. The slopes of $\ln(\sigma)$ against $1/T$ at high temperature region give a measurement of apparent activation energy for creep deformation.

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2.4 CVR (Jaromir Janousek)

A series of tests related to cyclic softening problems in tempered martensitic steels were carried out. Two scientific articles were published as part of the MatISSE project [2, 3]. The test machines were delivered in the middle of the project and the new Research Centre Rez (CVR) in Pilsen (CZE) laboratories were established. It was then necessary to prepare the machines from trial operation to real operation. The frequent forced power blackouts related to the building of the new workplaces caused a lot of troubles.

The supporting tests focused on strain controlled low cycle fatigue tests with different tension hold times. The cutting plan of the P91 test material block (150x150x60 mm) is shown in Fig. 33 together with the shape and dimensions of the creep-fatigue specimen. The arrow on the block indicates the rolling direction. The chemical composition is defined in Table 7. Chemical composition was also analysed by glow discharge spectroscopy.

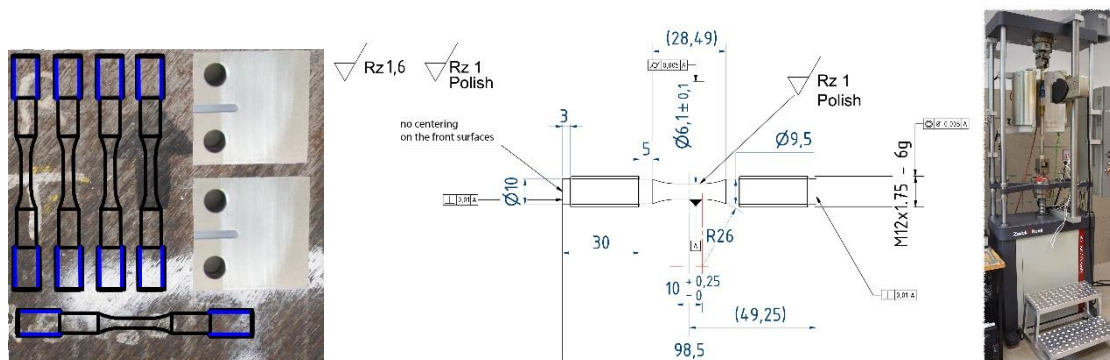


Fig. 33. Cutting plan of block P91 (left), the shape and dimensions of specimen (centre), and testing machine Kappa SS-CF (right).

Elements	C	Mn	Si	Ni	Cr	Mo	Cu	Al	S	P
unit [%]	0.12	0.41	0.24	0.10	8.32	1.02	0.05	0.006	0.001	0.009
Elements	V	Nb	N	As	Sb	Ti	B	W	Zr	O
unit [%]	0.235	0.084	0.041	0.005	0.001	0.002	0.0009	0.001	0.001	15 ppm

Table 7 Chemical composition of the test material

		C	Si	Mn	S	P	Cu	Ni	Cr	Mo	Ti	Nb
P91	% wt	0.097	0.202	0.347	< 0.001	0.007	0.062	< 0.1	7.6	0.885	0.014	0.061
	SD	0.002	0.003	0.003	-----	0.001	0.0005	-----	0.1	0.007	0.0005	0.001

Table 8 Chemical composition analysed by glow discharge spectroscopy.

Tests were performed on a Kappa SS-CF electromechanical creep testing machine which offers a wide range of applications, including creep-fatigue tests through-zero (see Fig. 33). Load capacity is up to 50 kN and speed range is from 1 $\mu\text{m/h}$ to 100 mm/min. The machine is equipped with a 3-zone furnace up to 1200°C and a non-contact high resolution video extensometer.

The type of tests according asymmetry was symmetrical reversed i.e. strain ratio $R = -1$. The intended test matrix corresponds to 6 creep fatigue tests with a tensile hold time applied at every cycle. The first 3 tests used a 12 hours hold time for strain range $\Delta\epsilon = 0.9\%$, 0.7% and 0.5% (i.e. $\epsilon_a = 0.45\%$, 0.35% and 0.25%) and were stopped after 50 cycles. Others with 1 hour hold time for the same strain ranges were planned for stopping after 1000 cycles, max. 1500 cycles or after failure location. The cycle shape had a triangle course and the temperature for all tests was 600°C. The strain rate in cycling was 6 % per minute.

The first experiment was for strain range $\Delta\epsilon = 0.5\%$ (i.e. $\epsilon_a = 0.25\%$) with 1 hour tensile hold time and it was interrupted in the 73rd cycle due to bad tuning (P I values), and the testing system became unstable. The results of this test are shown in Fig. 34.

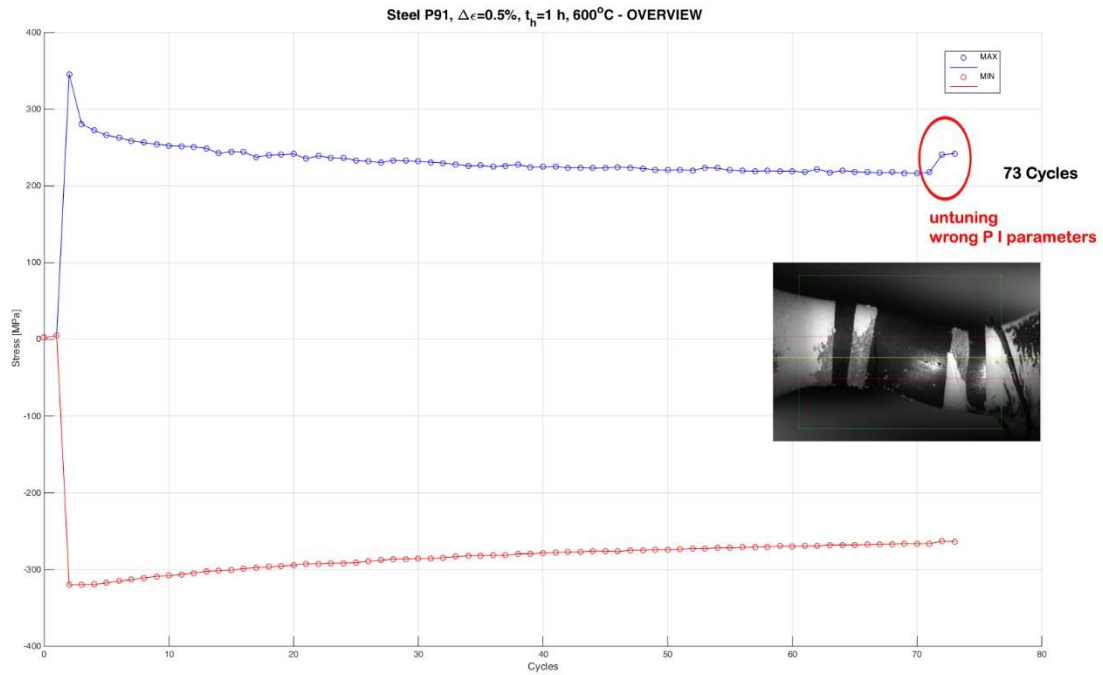


Fig. 34. Test for strain range $\Delta\epsilon = 0.5\%$ (i.e. $\epsilon_a = 0.25\%$), $t_h = 1$ hour, 600°C .

The second experiment used a strain range $\Delta\epsilon = 0.9\%$ (i.e. $\epsilon_a = 0.45\%$) with 1 hour tensile hold time and it was interrupted in 387th cycle due to cracking of the mark line for the video-extensometer. The test results are shown in Fig. 35.

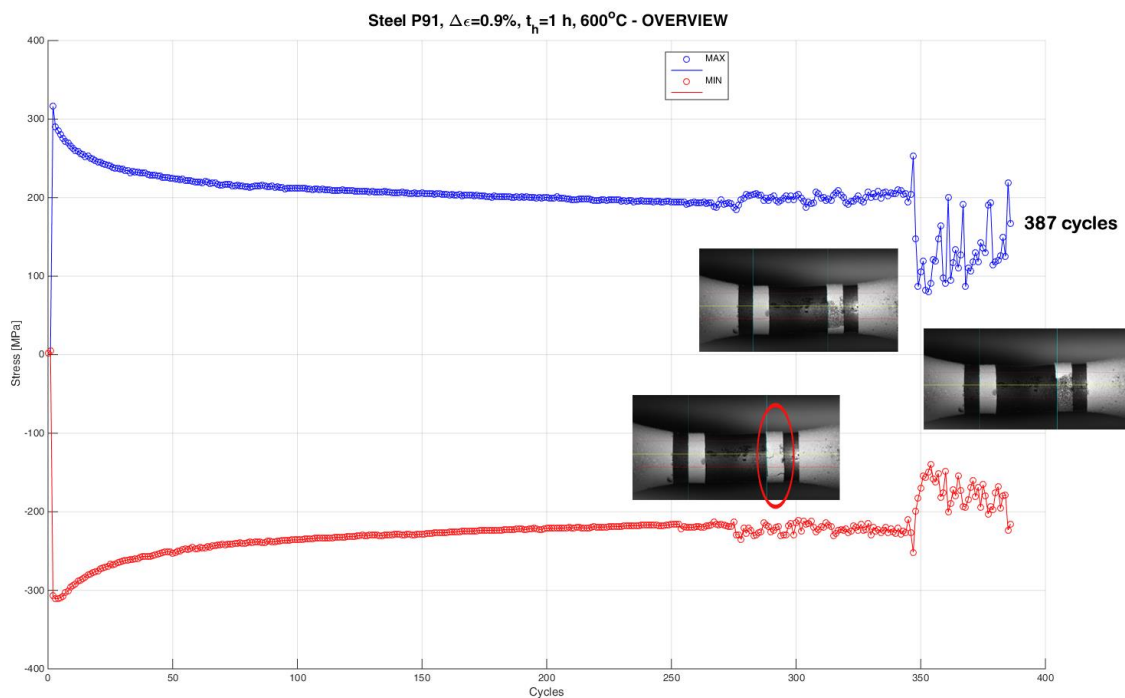


Fig. 35. Test for strain range $\Delta\epsilon = 0.9\%$ (i.e. $\epsilon_a = 0.45\%$), $t_h = 1$ hour, 600°C .

The third experiment was carried out for strain range $\Delta\epsilon = 0.7\%$ (i.e. $\epsilon_a = 0.35\%$) with 1 hour tensile hold time. and it was interrupted in 1670th cycle due to a power blackout. The test results are shown in Fig. 36.

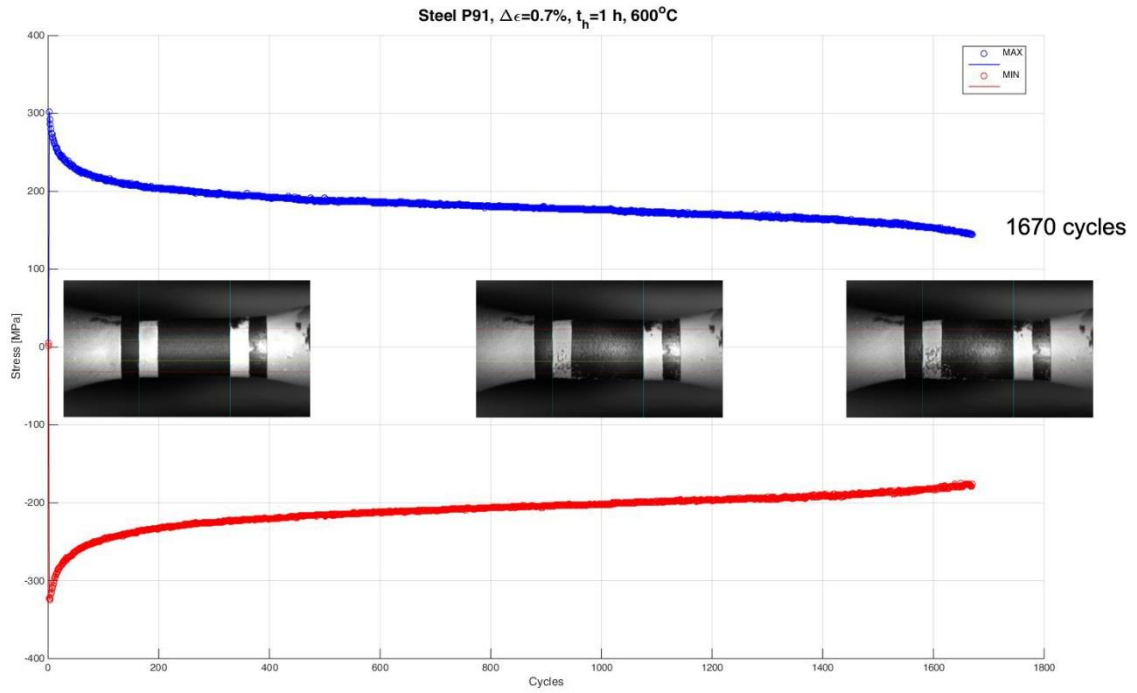


Fig. 36. Test for strain range $\Delta\epsilon = 0.7\%$ (i.e. $\epsilon_a = 0.35\%$), $t_h = 1$ hour, 600°C .

If these test are compared, it is evident that the curve for the lowest amplitude shows the lowest stress values for the compression process. The comparison is shown in Fig. 37.

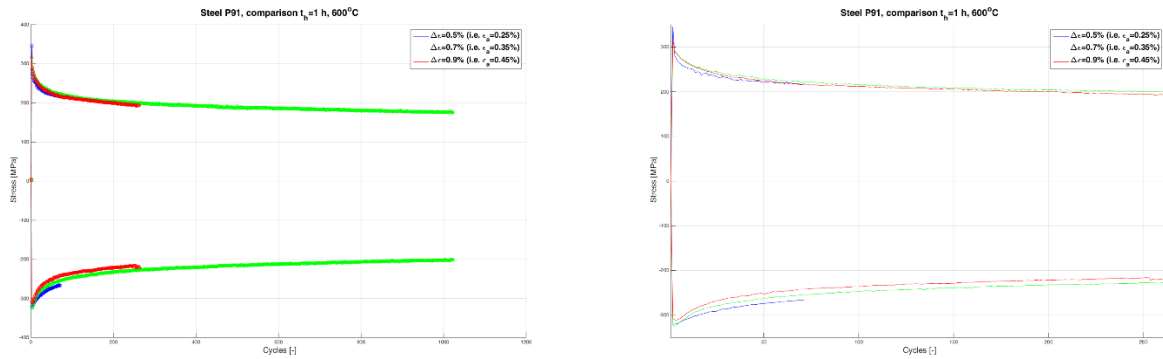


Fig. 37 Comparison of tests with tensile hold time $t_h = 1$ hour in every cycle, 600°C .

In the case of tensile hold time 12 hours in every cycle, the goal was to reach around 50 cycles. This was achieved for each of the 3 tests. The first test for strain range $\Delta\epsilon = 0.9\%$ (i.e. $\epsilon_a = 0.45\%$) was interrupted at the 42nd cycle due to a power blackout and stopped in the 53rd cycle. The test results are shown in Fig. 38.

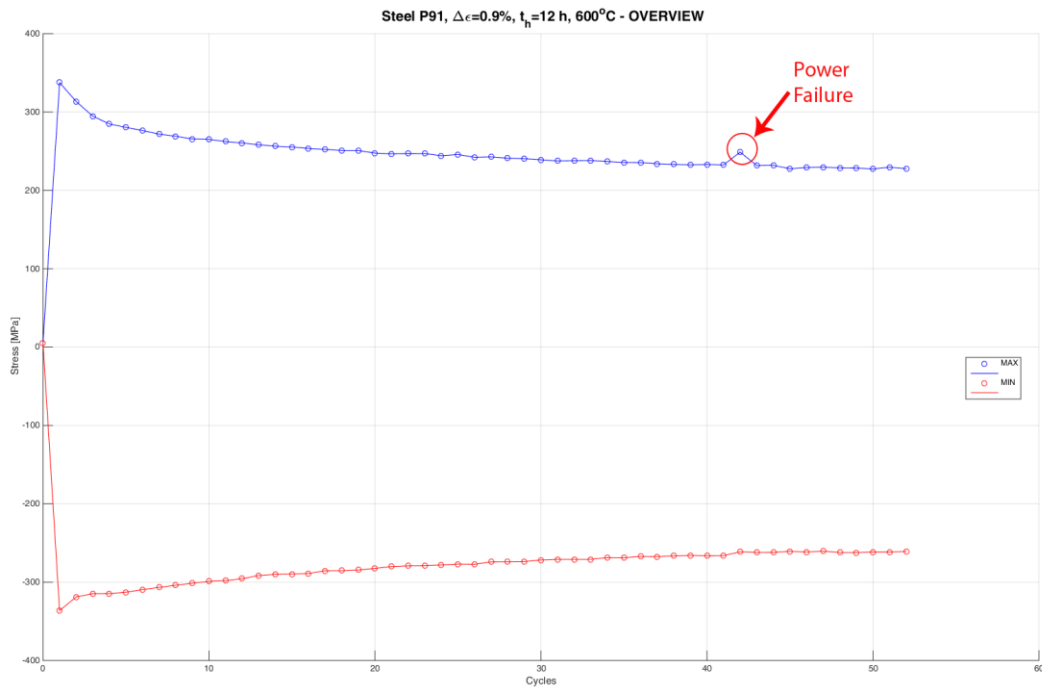


Fig. 38. Test for strain range $\Delta\epsilon = 0.9\%$ (i.e. $\epsilon_a = 0.45\%$), with tensile hold time $t_h = 12$ hours applied at each cycle, 600°C.

The second test for strain range $\Delta\epsilon = 0.7\%$ (i.e. $\epsilon_a = 0.35\%$) was interrupted at the 24th cycle due to a software error (Microsoft Visual C++ Runtime Library) and stopped at the 51st cycle. The test results are shown in Fig. 39.

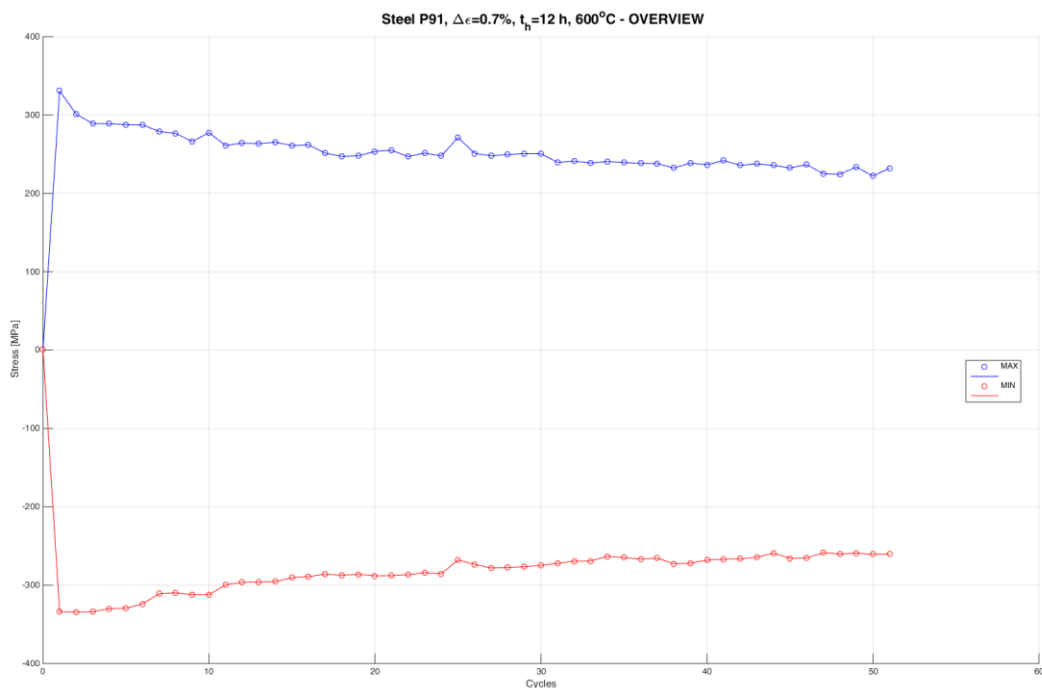


Fig. 39. Test for strain range $\Delta\epsilon = 0.7\%$ (i.e. $\epsilon_a = 0.35\%$), with tensile hold time $t_h = 12$ hour in every cycle, 600°C.

Last but not least, the test for strain range $\Delta\epsilon = 0.5\%$ (i.e. $\epsilon_a = 0.25\%$) was stopped at the 28th cycle. The test results are shown in Fig. 40. This test is still running.

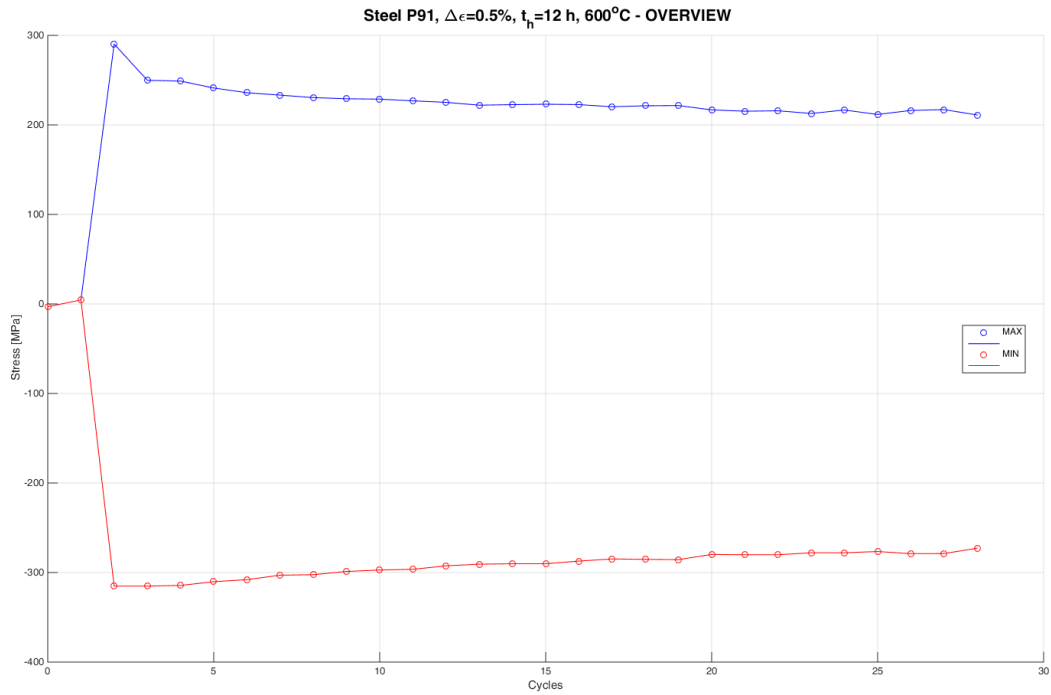


Fig. 40. Test for strain range $\Delta\epsilon = 0.5\%$ (i.e. $\epsilon_a = 0.25\%$), with a tensile hold time $t_h = 12$ hour at every cycle, 600°C .

The comparison is shown in Fig. 41. The first maximum stress corresponds to each strain amplitude. It is worthy of notice that the curve for strain deformation 0.25% is markedly lower than the other two. The minima of the curves also have a similar trend.

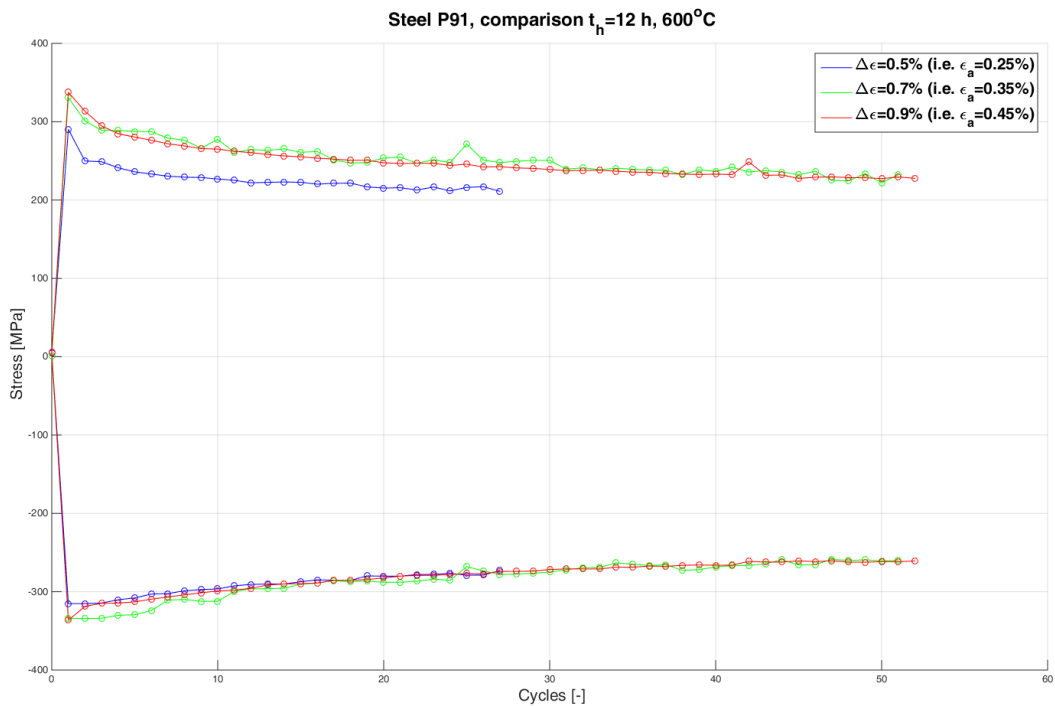


Fig. 41. Comparison of tests with tensile hold time $t_h = 12$ hours at every cycle, 600°C .

A servo-hydraulic machine with load capacity 100 kN was also used. This testing machine is more difficult to prepare and use for testing. There were several complications, such as cooling of adapters and aggregates,

incorrect evaluation of fracture, load impulses during oil pressure change, problems with data storage, misalignment, galling of threads or an achievement of smoother process of test. The last problem was solved by signal filtration, extensometer modification, better P I tuning and removing of software errors. Fig. 42 shows the setup of the servo-hydraulic machines and how the hysteresis loop looked before and after the changes.

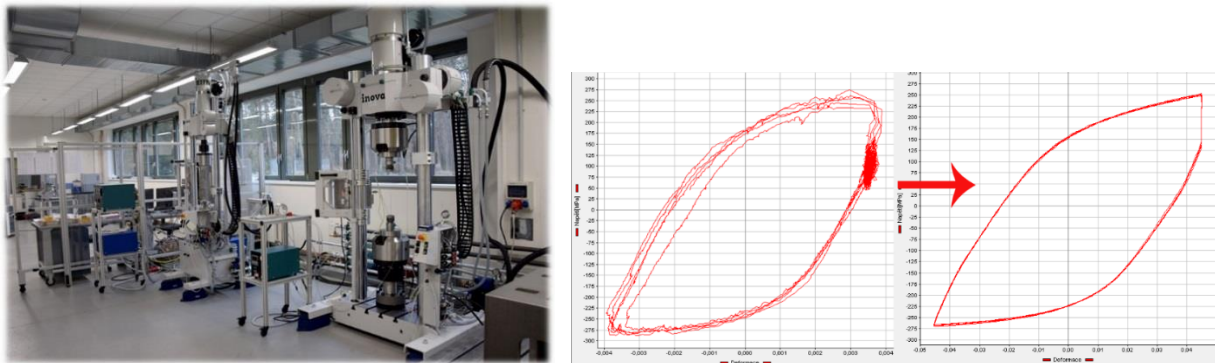


Fig. 42 The set of servohydraulic machines and appearance of hysteresis loops before and after the changes.

Unfortunately for optimization a lot of specimens (4 pieces) were used. The first successful test was performed for strain range $\Delta\varepsilon = 0.9\%$ (i.e. $\varepsilon_a = 0.45\%$) with 1 hour tensile hold time. The test was stopped after 95 cycles due to fracture (see Fig. 43).

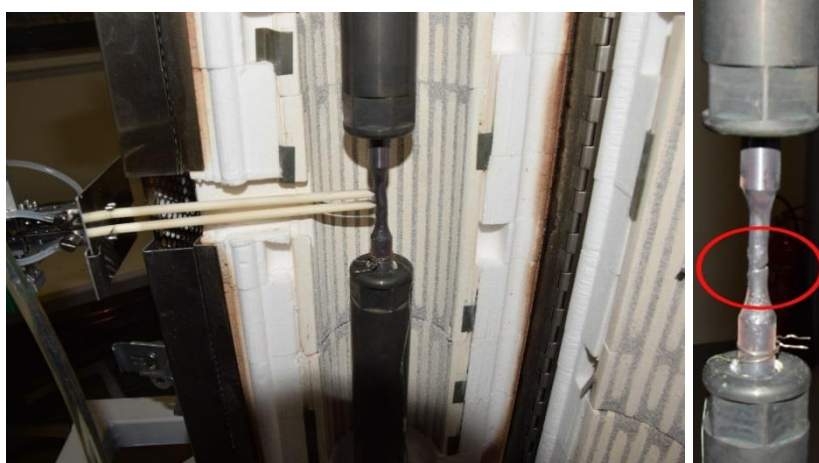


Fig. 43. The view inside the furnace and the final fracture.

If the results from both machines are compared, we observe that the maximum values from the servo-hydraulic machine have higher absolute values than from the electromechanical testing machine. This comparison is shown in Fig. 44.

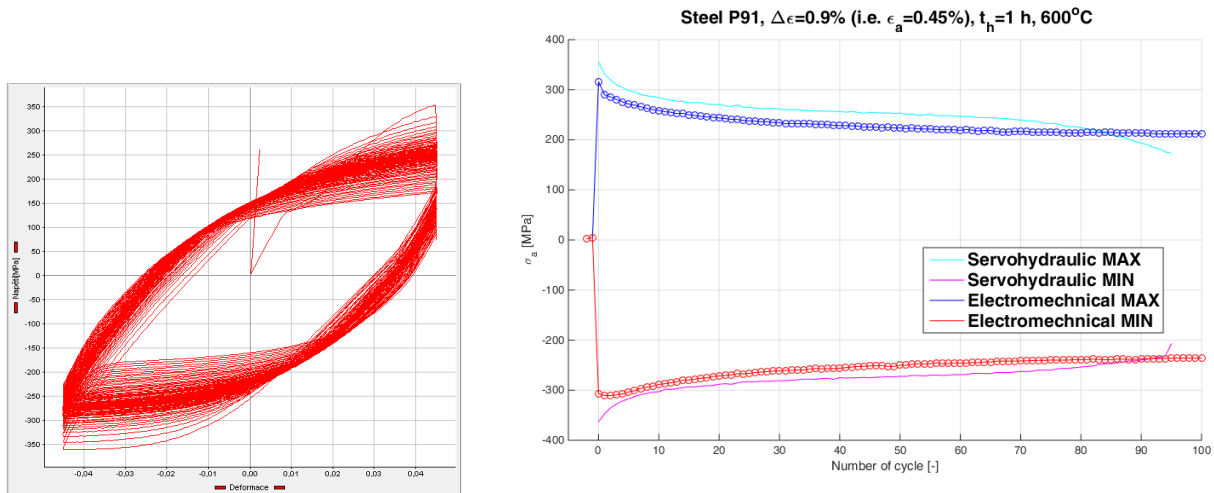


Fig. 44. Test performed on servo-hydraulic machine for strain range $\Delta\epsilon = 0.9\%$ (i.e. $\epsilon_a = 0.45\%$), $t_h = 1$ hour, 600°C – the changes of the hysteresis loop and comparison of results with electromechanical testing machine.

Another test with a hold time of 1 hour was performed for strain range $\Delta\epsilon = 0.5\%$ (i.e. $\epsilon_a = 0.25\%$). It had several stops due to a software error and the test finished because of fracture after 592 cycles. The comparison with the electromechanical machine results is depicted in Fig. 45. There are enough cycles for sufficient comparison with the electromechanical test machine. However, the start maximum values seem lower for the servo-hydraulic machine, then the values become closer and end at the same stress level.

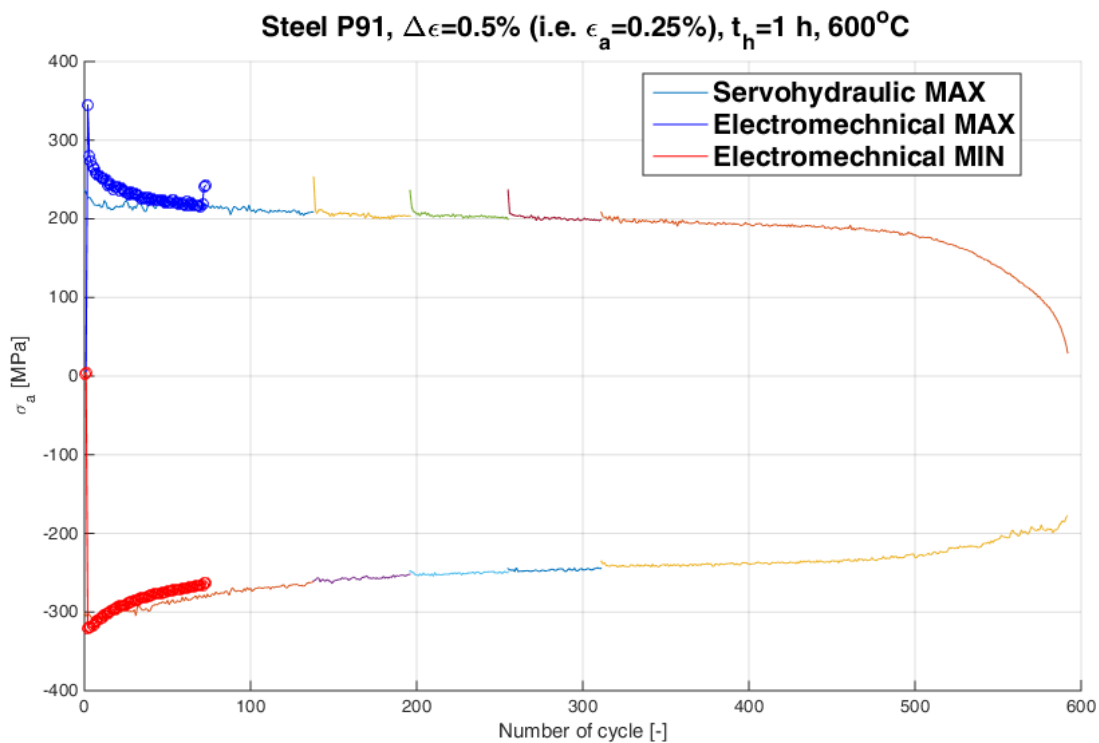


Fig. 45. Comparison of test performed on servo-hydraulic machine for strain range $\Delta\epsilon = 0.5\%$ (i.e. $\epsilon_a = 0.25\%$), $t_h = 1$ hour, 600°C with electromechanical testing machine results.

Only one successful test for tensile hold time 12 hours in every cycle was performed on the servo-hydraulic machine for strain range $\Delta\epsilon = 0.5\%$ (i.e. $\epsilon_a = 0.25\%$). The test was interrupted in the 98th cycle due to a power blackout. The test results are shown in Fig. 46 together with results from the electromechanical machine for the same parameters.

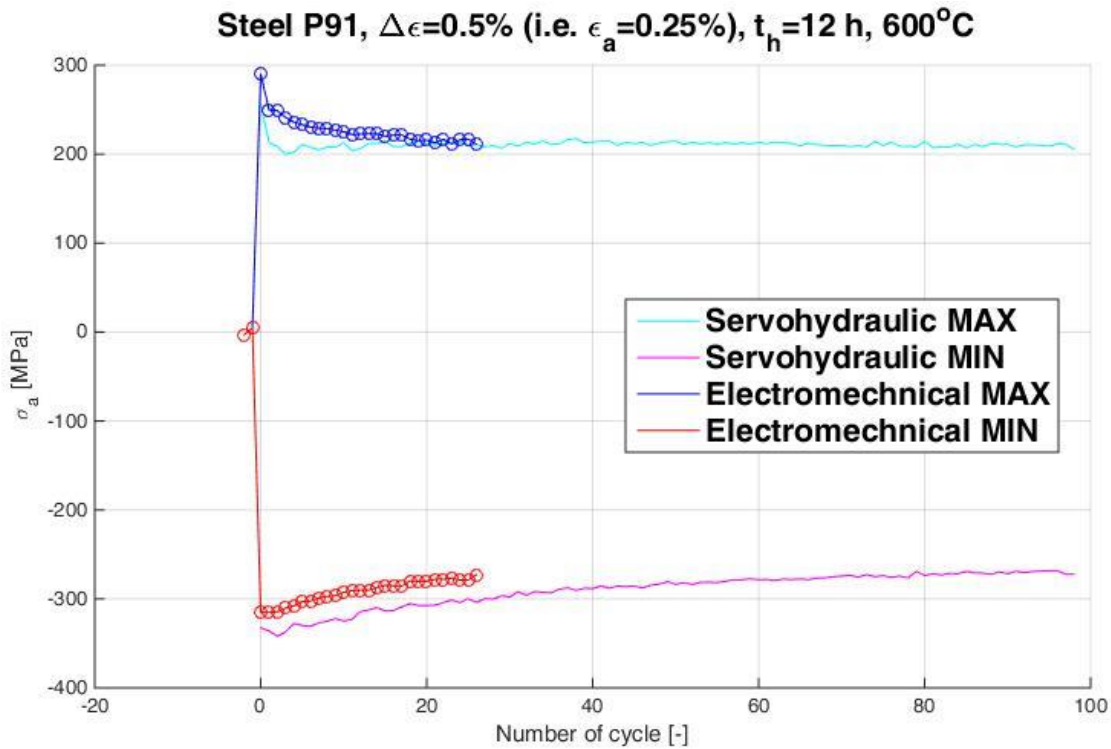


Fig. 46. Comparison of test performed on servo-hydraulic machine for strain range $\Delta\epsilon = 0.5\%$ (i.e. $\epsilon_a = 0.25\%$), $t_h = 12$ hours, 600°C with electromechanical testing machine results.

2.5 KIT (Ulrich Führer)

To support modelling efforts, a broad test series was conducted to investigate especially the influence of hold times on cyclic softening of ferritic-martensitic steels. The test series was performed on the modified 9Cr1Mo steel P91 which is the most characterized, well established representative of this type of steels. This test campaign consisted of two parts: In the first step, tensile, creep and strain-controlled low cycle fatigue tests at 550°C were planned to support parameter determination of the model. In the second step, low cycle fatigue tests with hold time under tension, compression and combined tension and compression in every cycle were planned to support further model improvement [1]. Because initial test data from low cycle fatigue tests was found adequate for model parameter determination, it was agreed to replace tensile and creep tests of part 1 by additional low cycle fatigue tests with hold time in part 2.

All tests were carried out on a Schenck 160kN servohydraulic test machine equipped with an Instron 8800 digital control unit. Radiant heating and Type S (Pt10Rh-Pt) thermocouples attached to the sample surface outside of the probed gage length were used. A calibration sample equipped with 5 thermocouples along the gage length showed a temperature gradient of 5°C (min 549°C , max 554°C) across the gage section fulfilling the requirements of ASTM E2714-13 [2]. Sample diameter was $8.8\text{mm} \pm 0.01\text{mm}$ and probed gage length was 20mm. The diameter of each sample was measured before the test and the actual diameter was used for stress calculations. Samples were precision-ground to a maximum roughness of $R_t=1\mu\text{m}$.

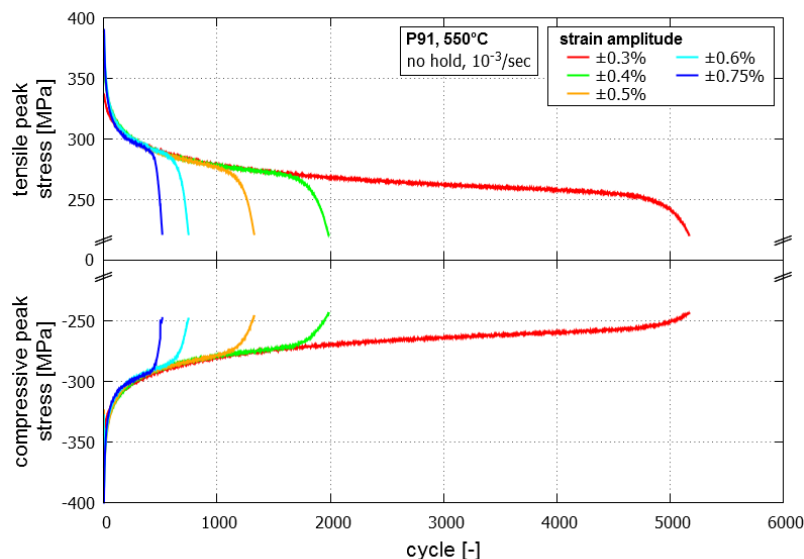


Fig. 47. Results of low cycle fatigue tests without hold time.

Fig. 47 summarizes results of low cycle fatigue tests without hold time at 550°C. All tests were conducted under strain control using a triangular waveform and a strain rate of $10^{-3}/s$ (0.1%/s). Strain amplitudes of 0.3% up to 0.75% (corresponding to total strain ranges of 0.6% to 1.5%) were considered. All tests show symmetric peak stresses and considerable cyclic softening without saturation with a drop in peak stresses of ~25% between the first cycle and the half-life cycle. These tests were used as a baseline for part 2 of this test series.

Strain range	Strain rate	Hold time position	Hold time duration
$\pm 0.75\%$	$10^{-3}/s$	Tension	1min, 10min, 1h, 3h
		Compression	1min, 10min, 1h, 3h*
		Tens. + Comp.	1min, 10min, 1h, 3h*
$\pm 0.5\%$	$10^{-3}/s$	Tension	1min, 10min
		Compression	1min, 10min, 1h
		Tens. + Comp.	1min, 10min
$\pm 0.3\%$	$10^{-3}/s$	Tension	1min, 10min*, 1h*
		Compression	1min, 10min
		Tens. + Comp.	1min, 5min, 10min

Table 10. Overview of low cycle fatigue tests with hold time.

All tests performed in part 2 of this test series are listed in appendix 0. Tests marked with * were continued without hold time (pure cycling) after the linear stage of cyclic softening was reached. In total, around 7000h of successful testing was achieved in this test series. References to detailed results of all tests are listed in appendix.

Concerning the influence of hold time on cyclic softening, main observations will be presented in the following paragraph. Although only selected test results are shown here, similar behavior was observed in all tests. First, the influence of hold time position is illustrated in Fig. 48.

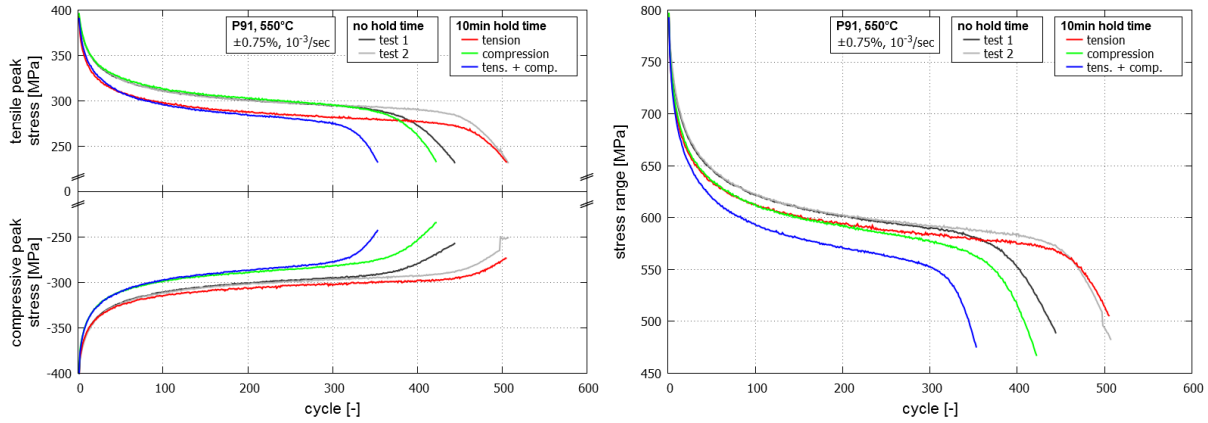


Fig. 48. Influence of hold time position on cyclic softening regarding peak stresses (left) and stress range (right).

The baseline test without hold time was repeated to judge reproducibility of cyclic softening. Both tests showed identical results in softening and expected scatter in lifetime. Comparing the softening response of tests with hold time and tests without hold time, the influence of hold time position is apparent: tensile peak stresses are reduced due to tensile hold time whereas compressive peak stresses are reduced due to compressive hold time. Combined hold times under tension and compression lead to lower stresses under tension as well as compression. Notably, this leads to a further reduced stress range compared to single hold times which show a similar stress range for both tensile and compressive hold time up to the point of macro crack initiation (see Fig. 48 (right)).

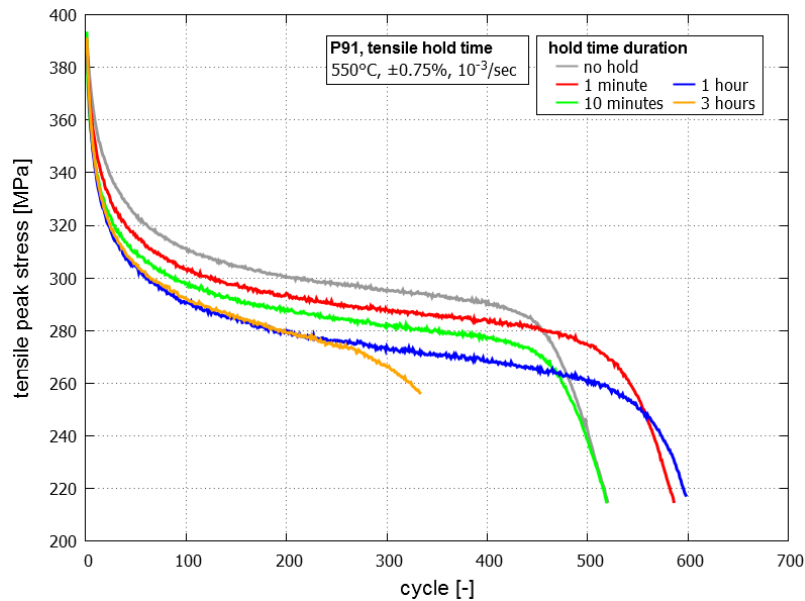


Fig. 49. Influence of hold time duration on cyclic softening.

The influence of hold time duration on softening is shown in Fig. 49. For hold times up to 1 hour, longer hold times lead to additional decrease in peak stresses, and therefore to an additional increase in cyclic softening. For further increase in hold time duration, no additional decrease of peak stresses was observed. Interestingly, increasing hold time from 1 hour to 3 hours significantly reduced number of cycles to failure by almost a factor of 2 whereas tensile hold times up to 1 hour show a slight increase in lifetime. Compressive peak stresses are similar to those of tests without hold time.

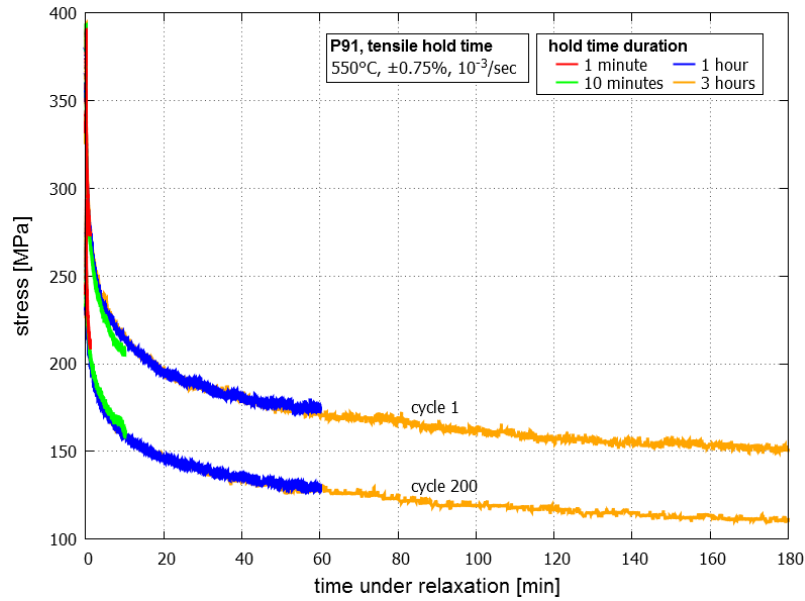


Fig. 50. Comparison of relaxation behavior of the 1st and 200th cycle for different hold time durations.

Comparison of the relaxation during hold time for the 1st and 200th cycle (Fig. 50) shows that after an initial period of a few minutes the stresses during relaxation are very similar for all time durations although tests with longer hold times up to 1 hour show a lower peak stress in the beginning. This is due to a faster initial relaxation in tests with short hold times. Here, another result of cyclic softening can also be seen: Although the material relaxes to lower stresses in the softened state, the amount of relaxed stress (that is, the difference between peak stress and the stress at the end of the hold time) is significantly lower in the softened state. In the first cycle, the material relaxes by about 240 MPa during 3 hours whereas after 200 cycles, the amount of stress relaxation after 3 hours is only 170 MPa.

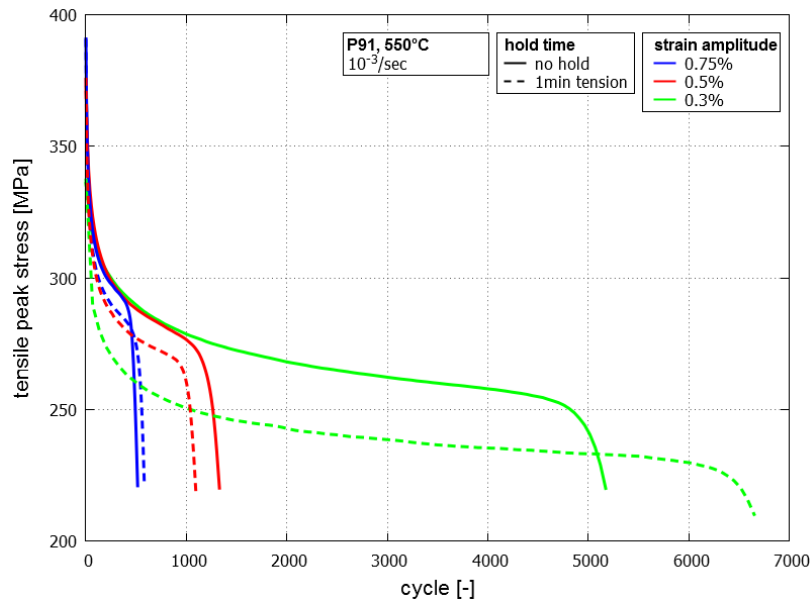


Fig. 51. Influence on cyclic softening and lifetime of 1min tensile for different strain amplitudes.

In Fig. 51, softening curves of same hold time duration and position are compared for different total strain amplitudes. The effect of hold times on cyclic softening is significantly more pronounced for smaller strain amplitudes. Due to this observation, originally planned tests on strain rate dependence of softening were replaced by further tests at low strain amplitudes in agreement with project partners. This dependence on strain amplitude was also found for cycles to failure (being defined as a 10% load drop compared to the linear region of softening). For 0.3% strain amplitude, the number of cycles to failure increased by more than 1500 cycles, which equals to about 30%. In contrast, for larger strain amplitudes and comparatively short tensile hold times, either a modest increase or decrease in lifetime was observed.

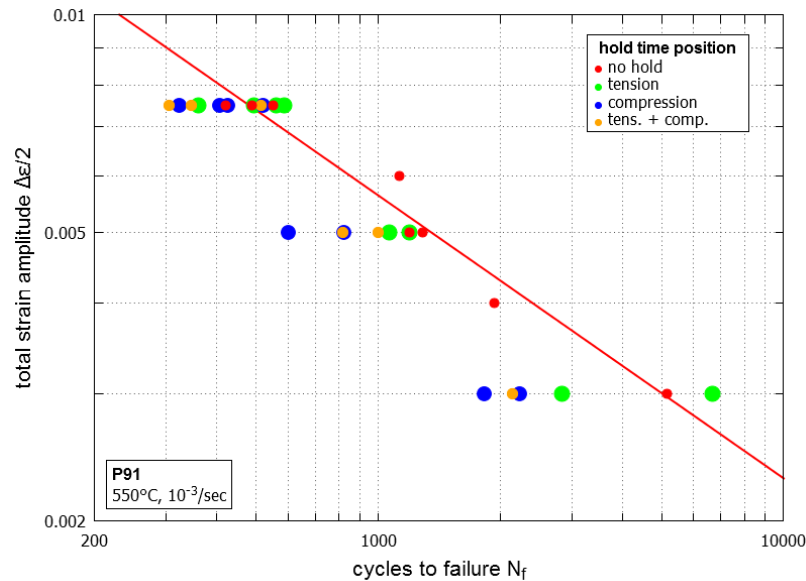


Fig. 52. Cycles to failure for tests with and without hold time.

Cycles to failure are summarized in Fig. 52 for all tests. Tests without hold time follow a linear relation in the log-log plot. A slight improvement in lifetime was noted for tests with short tensile hold times, whereas tests including compressive hold time show reduced lifetime. Similarly to its effect on cyclic softening, the effect of compressive hold time on lifetime is more pronounced in tests with small strain amplitudes. In this test series, this is most pronounced for 10min compressive hold time at strain amplitude of $\pm 0.3\%$, which resulted in a reduction of cycles to failure by 65%. Consequently, accelerated testing at high strain amplitudes underestimates the influence of hold times on cyclic behavior of ferritic-martensitic steels.

Additionally, further tests with multi-step loading were carried out to guide model development. Results of these tests are shown in Fig. 53. In this test series, the sample was loaded at $\pm 0.3\%$ strain amplitude and 1min compressive hold time for 1000 cycles first. This was followed by 1000 cycles without hold time. The next 1000 cycles included 1min of tensile hold time, followed by cycling without hold time up to failure. The test was also repeated with switched hold time positions, that is with 1min tensile hold time in the first step and 1min compressive hold time in the third step.

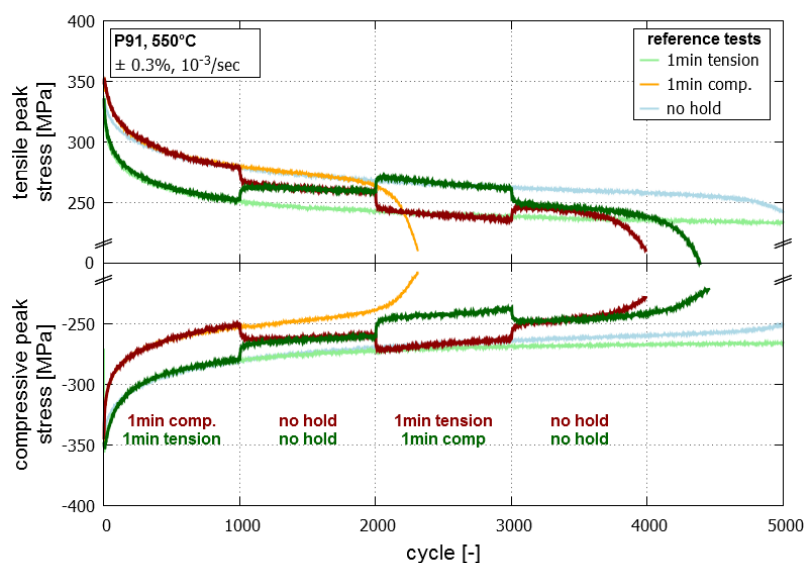


Fig. 53. Results of tests with multi-step loading.

After hold time is removed from the loading path, a transient stage (lasting for about 10 cycles) is followed by symmetrical peak stresses. Stresses in this second stage are lower than in a test without hold time from the beginning, with stress range similar to the last cycles with hold time. This indicates additional isotropic softening due to hold time. This additional softening is overlaid by a kinematical shift of hysteresis loops during hold time under tension or compression only. This shift happens again after hold time is reintroduced, with strain range again being equal to last cycles without hold time. After 3000 cycles, hold time is excluded again and difference in peak stresses between test without hold time from the beginning and test with intermittent hold time increased again, indicating further additional isotropic softening due to tensile hold period. Stresses during pure-cyclic steps do not depend on position of previous hold time, with tensile and compressive hold time of same duration leading to the same stress level during following steps without hold time.

Concluding, additional reduction of peak stresses under tension only for tensile hold times (or compression for compressive hold times, respectively) is a result of additional isotropic softening, which is still present during subsequent continuous cycling and an overlaid kinematic shift of stresses during hold-time phases arising from the asymmetric loading with hold time (That is, for tensile hold time, the amount of inelastic deformation going from tension to compression is larger than going from compression to tension).

Interestingly, both tests showed similar lifetime independent of hold time order. Starting with (detrimental) compressive hold time did not lead to reduced lifetime compared to the test starting with tensile hold time. Further, as shown in Table 11, linear lifetime behavior regarding hold time influence is observed in this test with a sum of life fractions of almost unity.

	Cycles to failure (N_f)	Cycles, test 1 (N_1)	N_1/N_f	Cycles, test 2 (N_2)	N_2/N_f
no hold	5151	1982	0.38478	2333	0.45292
1min tens.	6689	1000	0.14950	1000	0.14950
1min comp.	2233	1000	0.44783	1000	0.44783
$\Sigma N/N_f$			0.98211		1.05025

Table 11: Calculated life fractions of steps with multi-step loading

References

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3 Modelling

Three different modelling approaches are then proposed. First, an engineering approach based on well-chosen analytical formulae and parameter adjustment by inverse identification (JRC). The second approach is based on thermodynamics and macroscopic hardening, flow and damage constitutive laws (KIT). The last approach is based on the physical mechanisms involved at the dislocation scale (glide and climb at high temperature) (CEA). The models used experimental data for material parameter and experimental validation. Many interactions between the different involved laboratories allow a rather rich validation in terms of experimental data.

3.1 JRC (Stefan Holmström)

The target of the JRC modelling was to supporting the determination of creep-fatigue damage as shown in the flow diagramme in Fig. 54. The modelling performed by JRC for the MATISSE project covers the stages I-IV.

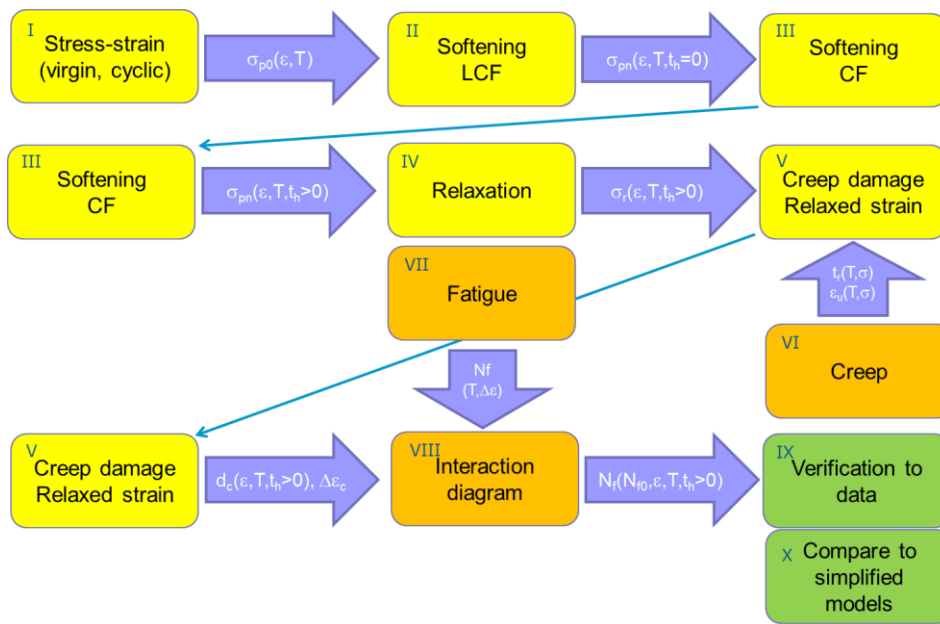


Fig. 54. The models needed for determining creep-fatigue damage in a softening steel. The models VI-VIII labeled in orange can be stand-alone models (or design rule defined) whereas the models labeled in yellow are closely interlinked and need to be based on the same material batch. The green stages (IX and X) are the model verification stages future projects. The blue arrows indicate the main output of the specified model.

3.1.1 Models for LCF and CF softening

It can be shown that the softening rate of LCF tests as a function of normalized cycles is well presented by Eq. 1. The chosen softening (decay) function is inspired from the Manson-Halford equation for creep-fatigue cyclic life [16].

$$\frac{\sigma_{pn-LCF}}{\sigma_{p0}} = A_1 + \frac{A_2}{A_3 + \frac{N}{N_{f0}}} \quad (3.3.1)$$

The cyclic LCF peak stress (MPa) at cycle n is then simply $\sigma_{pn-LCF} = f(n/N_f) \cdot \sigma_{p0}(\Delta\epsilon, T)$, where σ_{p0} is the virgin material peak stress ($1/4$ cycle) at the specified strain and temperature. The parameter A_1 is describing the lower bound of softening and the parameters A_2 and A_3 influence the rate of softening. The function is optimized

in the n/N_{f0} range 0-80%, where n is the cycle number and N_{f0} is the number of cycles to failure in a LCF test at the specified strain and temperature. The initial parameter values, acquired for the softening model using data for both the MATTER materials, are given in Table 11 and the fit to the measured LCF softening curves is shown in Fig. 55.

Parameters	LCF softening (Eq.3.3.1)
A_1	0.70170
A_2	0.01276
A_3	0.04441

Table 11. Initial fitting values for the LCF softening model (Eq.1).

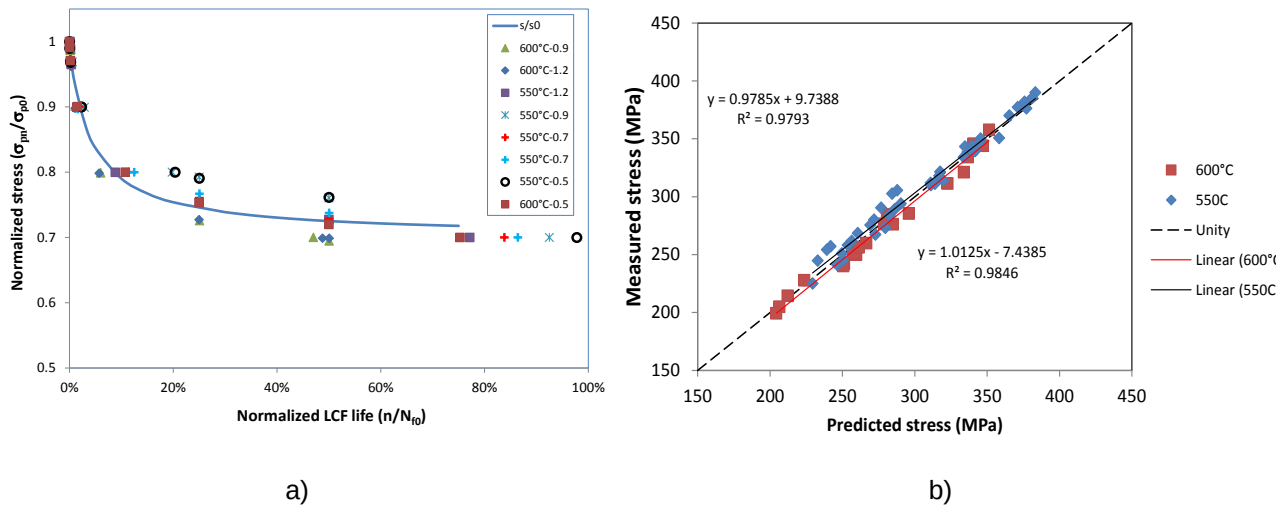


Fig. 55. a) model (blue line) for LCF softened material at 550 and 600°C, b) predicted versus measured peak stresses at specified n/N_f .

It can also be shown that when hold times (t_h) are introduced the softening rate is increased. The effect of hold time is intuitively (at least partly) explained by an increased plastic strain range caused by the relaxing stress where elastic strain is converted into plastic strain.

In the case of CF tests with hold times the softening rate can be corrected by introducing two correction factors as given in Equation 3.3.2 where $P_1 = f(t_h)$ and $P_2 = f(\Delta\epsilon)$. The chosen functions for the t_h and $\Delta\epsilon$ dependence are given in Equations 3.3.3 and 3.3.4. The corresponding initial fitting parameters are given in Table 12.

	Parameters	
CF softening, t_h correction (Eq.3.3.3)	$B_1=0.94$	$B_2=-0.02$
CF softening, $\Delta\epsilon$ correction (Eq.3.3.4)	$C_1=1.025$	$C_2=-0.067$

Table 12. Initial fitting values for the CF softening model (Eq. 3 and 4).

The final form of the correction factors P_1 and P_2 will need to be further optimized for application in a wider range of strains and hold times.

$$\frac{\sigma_{pn}}{\sigma_{p0}} = P_1 \cdot P_2 \frac{\sigma_{pn-LCF}}{\sigma_{p0}} \quad (3.3.2)$$

$$P_1(t_h) = B_1 + B_2 \cdot \log(t_h) \quad (3.3.3)$$

$$P_2(\Delta\varepsilon) = C_1 + C_2 \cdot \frac{1}{\Delta\varepsilon} \quad (3.3.4)$$

With these equations in place the hold time and strain range dependent peak stress in an arbitrary location of the softening curve (0-80%) can now be predicted as $\sigma_{pn} = f(\Delta\varepsilon) \cdot f(t_h) \cdot f(N/N_f) \cdot \sigma_{p0}$.

To test the model on data that has not been a part of the fitting data set the model was used to predict the relaxation behavior of two additional tests. The predicted versus measured peak stress at the beginning and in the middle of the cyclic life is shown in Fig. 56 a for the KIT test with a hold time of 3h and cycled at a strain range of $\Delta\varepsilon/2=0.75\%$. Note that the data is presented as a function of normalized (LCF) cyclic endurance. In Fig. 56 b, the initial 70 cycles of a REZ test with a hold time of 1h cycled at a strain range of $\Delta\varepsilon/2=0.25\%$ presented as a function of cycles. The model prediction for the high strain range test seems to be good throughout the softening curve. For the low strain range test the measured softening rate is faster than the predicted one, especially in the initial cycles. However, even for this test the predicted rate of softening (slope) seems to match.

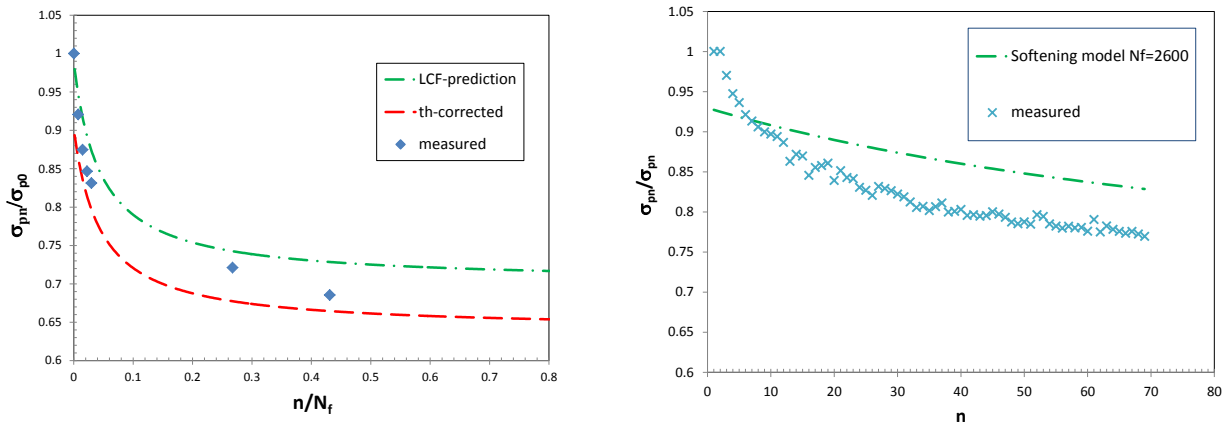


Fig. 56 Measured and predicted peak stresses for CF tests with a) 3h hold, $\Delta\varepsilon=1.5\%$, 550°C and b) 1h hold, $\Delta\varepsilon=0.5\%$, 600°C. Note that if the normalization is done by true cyclic endurance then the hold time corrections should not be applied.

3.1.2 Model for relaxation

The relaxation curve from an arbitrary location in the softening curve can be fitted to a Wilshire model (WE) [17] modified for use with relaxation data. The model was chosen since it has been applied successfully in a European Creep Collaborative Committee round-robin on long term (static) relaxation modelling [18]. The WE stress-time behavior during the hold period is given in Eq. 5;

$$\frac{\sigma_{rn}}{\sigma_{ref}} = \exp\left(-k(t_{rel} \cdot \exp(\frac{-Q}{R \cdot T}))^u\right) \quad (3.3.5)$$

where t_{rel} is the relaxed time (h), u and k are fitting factors and Q is the activation energy (J/mol) optimized on the relaxation data and R is the gas constant (8.314 J/molK). The normalizing reference stress σ_{ref} (MPa) is based on the ultimate tensile strength R_m at temperature T (Kelvin). The $\sigma_{ref}(\Delta\varepsilon, t_h) = R_m(T)$ for the virgin material and no hold time and a strain range of 0.7%. The reference stress is corrected as given in Equation 3.3.6 for both virgin and cyclically softened material. Note that two separate models may be needed when more data is available.

$$\sigma_{ref} = \frac{R_m(T)}{P_0 \cdot P_1} \cdot P_2 \quad (3.3.6)$$

The parameter P_0 is the LCF softening ratio calculated from Eq.3.3.1 and P_1 is the parameter giving the effect of hold time as in Eq.3.3.3 and P_2 as in Eq.3.3.4.

By rearranging Eq. 3.3.5 the time to acquire a specified level of relaxation can be calculated as given in Eq 3.3.7. And the relaxed stress as a function of peak stress, hold time and temperature as given in Eq. 3.3.8.

$$t_{rel} = \left(\frac{\ln(\frac{\sigma_{rn}}{\sigma_{ref}})}{-k} \right)^{1/u} \cdot \exp\left(\frac{Q}{R \cdot T}\right) \quad (3.3.7)$$

$$\sigma_{rn} = \exp\left(\ln(\sigma_{ref}) - k(t_{rel} \cdot \exp(\frac{-Q}{R \cdot T}))^u\right) \quad (3.3.8)$$

Note that since the WE relaxation model is divided into two stress regions and the u and k parameters have to be chosen accordingly.

The initial fitting parameters for the relaxation model are given in Table 13 and the resulting predicted versus measured relaxed stresses are presented in Fig. 58 for the JRC data.

	Parameters	$\sigma/\sigma_{ref} \geq 0.35$	$\sigma/\sigma_{ref} < 0.35$
All data	k	55.554	0.1478
	u	6.9512	0.07088

Table 13. Initial fitting values for the WE relaxation model (Eq. 5). $Q = 180$ kJ/mol.

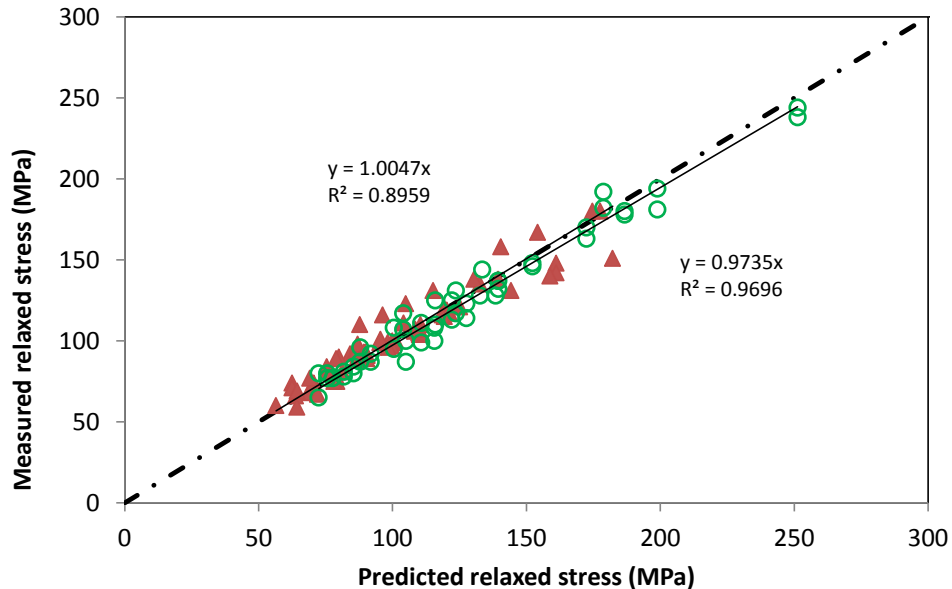


Fig. 57. Modelled and measured relaxed stress at specified time for virgin (green open) and cyclically softened (red triangle) material.

To test the relaxation model on data that has not been a part of the fitting data set the model was applied on two relaxation curves from literature. In Fig. 58, the relaxation test by Takahashi [12] is plotted against the above described model. In Fig. 59, a test curve from a JAEA report [11] is compared to the corresponding predicted curve. The JAEA curve fits well if the reference stress is increased by 15%. This difference can be

directly related to differences in peak stress since the applied model is based on rather low strength material heats. The fit for the Takahashi case is matching the measured behavior if the reference stress is increased by 5%.

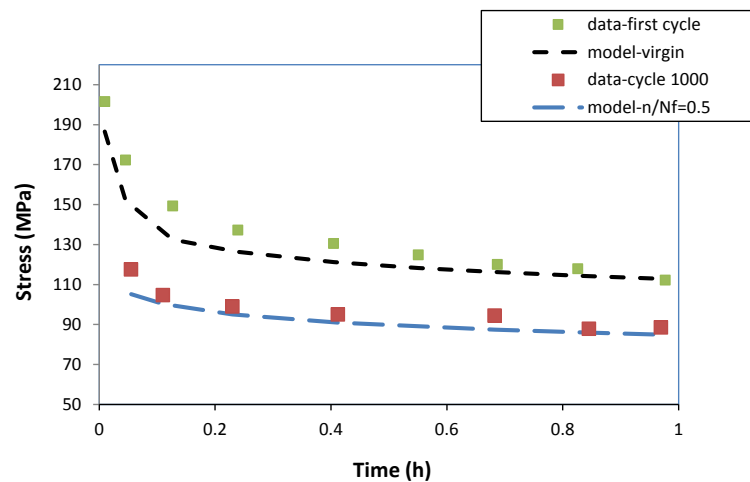


Fig. 58. Measured and predicted relaxed stress of test performed by Takahashi [12], 600°C, $\Delta\epsilon=0.5\%$, virgin and $n=1000$ cycles.

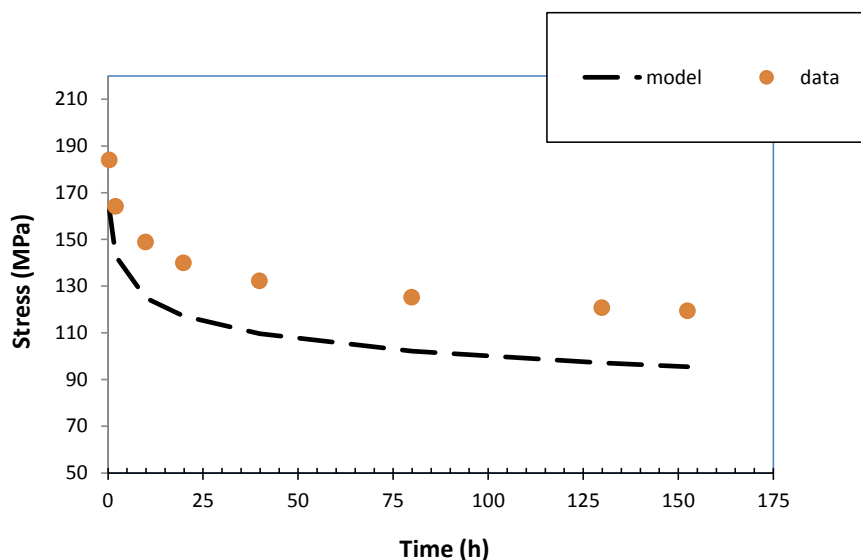


Fig. 59. Measured and predicted relaxed stress of a test presented in JAEA report [11], 550°C, $\Delta\epsilon=0.6\%$, virgin material.

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3.2 KIT (Ulrich Führer)

In this approach, an existing coupled deformation and damage model based on continuum mechanics was extended to include observed influences of hold time on cyclic softening and lifetime. This model by Aktaa and Schmitt [1] is based on the Chaboche model [2] **Erreur ! Source du renvoi introuvable.** and was specifically developed for ferritic-martensitic steels. Good performance of the model was shown before for low cycle fatigue loadings without hold time, but description of softening behavior with hold time required further improvement. A summary of the isothermal, uniaxial formulation of the model is given in the following section with a multiaxial formulation given in the appendix.

This model belongs to the unified deformation models and describes viscoplasticity without separation in time-independent plasticity and time-dependent creep. Consequently, the total mechanical strain rate is divided into an elastic and an inelastic part:

$$\dot{\varepsilon} = \dot{\varepsilon}_{el} + \dot{\varepsilon}_{in}$$

The elastic strain is calculated using Hook's law with direct coupling to the damage model with σ , E and D denoting stress, Young's modulus and Damage, respectively:

$$\varepsilon_{el} = \frac{\sigma}{E(1 - D)}$$

Based on the von Mises yield criterion, a flow rule is formulated for calculation of inelastic strain rate. Inelastic deformation is a function of stress as well as internal state variables describing kinematic hardening (Ω) and isotropic softening (ψ):

$$\dot{\varepsilon}_{in} = \left\langle \frac{|\Sigma| - k}{Z} \right\rangle^n \text{sgn}(\Sigma) \text{ with } \Sigma = \frac{\sigma}{\psi(1 - D)} - \Omega$$

k represents initial yield strength of the material and brackets $\langle * \rangle$ operate as $\langle x \rangle = (x + |x|)/2$. Accordingly, no inelastic strain is calculated for stress states inside the elastic domain.

Kinematic hardening describes shifting of the yield surface in stress space and primarily defines the shape of hysteresis loops. On the other hand, isotropic softening results (among others) in contraction of the yield surface and constitutes progressive diminution of stresses due to cyclic softening.

Non-linear kinematic following the Armstrong-Frederick hardening rule [3] hardening is included. To account for mean stress induced by hold time, a second kinematic hardening term is added. Good performance is achieved allowing only static recovery, but no dynamic recovery for the added term. The following evolution equations were applied for kinematic hardening:

$$\Omega = \Omega_1 + \Omega_2$$

$$\dot{\Omega}_1 = H_1 \dot{\varepsilon}_{in} - C_1 \Omega_1 |\dot{\varepsilon}_{in}| - R_1 |\Omega_1|^{m_1-1} \Omega_1$$

$$\dot{\Omega}_2 = H_2 \dot{\varepsilon}_{in} - R_2 |\Omega_2|^{m_2-1} \Omega_2$$

For an improved representation of the influence of hold time on cyclic softening, the isotropic softening variable Ψ is subdivided into three parts with

$$\psi = \psi_1 + \psi_2 + \psi_3 \text{ with } \psi_1(t=0) = 0, \psi_2(t=0) = 1 \text{ and } \psi_3(t=0) = 0$$

Each part of isotropic softening is described by a separate evolution equation:

$$\dot{\psi}_1 = -h_1 |\dot{\varepsilon}_{in}|$$

$$\dot{\psi}_2 = c(\psi_s - \psi_2) |\dot{\varepsilon}_{in}|$$

$$\dot{\psi}_3 = -r_\psi |\psi_3 - \psi_r|^{m_\psi-1} (\psi_3 - \psi_r) + h_3 |\dot{\varepsilon}_{in}|$$

$$\text{with } \psi_s = 1 - \psi_{s,\infty} \left(1 - \exp \left(-c_s \left(\max_{-\infty < \tau < t} \varepsilon_{in}(\tau) - \min_{-\infty < \tau < t} \varepsilon_{in}(\tau) \right) \right) \right)$$

In comparison to the original model, the description of softening due to hold time was improved by decoupling of deformation-dependent and time-dependent terms into ψ_2 and ψ_3 . Addition of linear hardening in ψ_3 ensures negligible influence of ψ_3 on cyclic softening under pure cyclic conditions without hold time as terms for time-dependent softening and linear hardening balance each other at small values under these conditions. Additionally, higher inelastic strain range in tests with hold time under both tension and compression in

comparison to tests with hold time under tension or under compression only is considered in an adjusted calculation of ψ_s .

In a last step, damage was modeled using a simplified version of the ISRM model [4] as in the original modelling approach. Like the deformation model, this model was developed within the framework of continuum damage mechanics and allows direct coupling of deformation and damage models. An internal state variable D is introduced with a value of 0 representing virgin material and 1 for totally damaged material. The change of damage is described by an additional evolution equation:

$$\dot{D} = \left(\frac{\sigma}{A} \right)^r |\varepsilon_{in}| (1 - D)^{-\kappa}$$

Therefore, no additional damage is assumed under compression as well as under purely elastic deformation. Compared to damage models used in similar approaches recently [5], this model does not require direct linking to a damage threshold.

Material parameters $k, Z, n, H_1, C_1, R_1, m_1, H_2, R_2, m_2, h_1, c, r_\psi, \psi_r, m_\psi, h_3, \psi_{s,\infty}, c_s, A, r$ and κ as well as Young's modulus E are temperature dependent and have to be determined by fitting of the model response to experimental results. Using obtained data presented in section 2.5, the parameter set shown in Table 14 was calculated. A χ^2 -type error function describing the deviation of calculated material behavior from experimental results was minimized using the MINUIT optimization code developed at CERN [6][7].

Parameter	Value	Parameter	Value	Parameter	Value
E [Mpa]	156250	H ₁ [Mpa]	74696	h ₁	2.6061 x10 ⁻³
k [Mpa]	162.85	C ₁	563.29	c	2.2734
Z [Mpa s ^{1/n}]	220.61	R ₁ [Mpa ^{1-m₁} s ⁻¹]	2.5571 x10 ⁻⁴	r _ψ [s ⁻¹]	2.6126 x10 ⁻³
n	9	m ₁	2.0302	ψ _r	4.1365 x10 ⁻²
A [Mpa s ^{1/r}]	737.99	H ₂ [Mpa]	2013.2	m _ψ	2.85
r	4.9918	R ₂ [Mpa ^{1-m₂} s ⁻¹]	2.7699 x10 ⁻⁴	h ₃	4.2602 x10 ⁻³
κ	25	m ₂	0.10485	ψ _{s,∞}	0.22368
				c _s	2288.6

Table 14. Model Parameters calculated for P91 at 550°C.

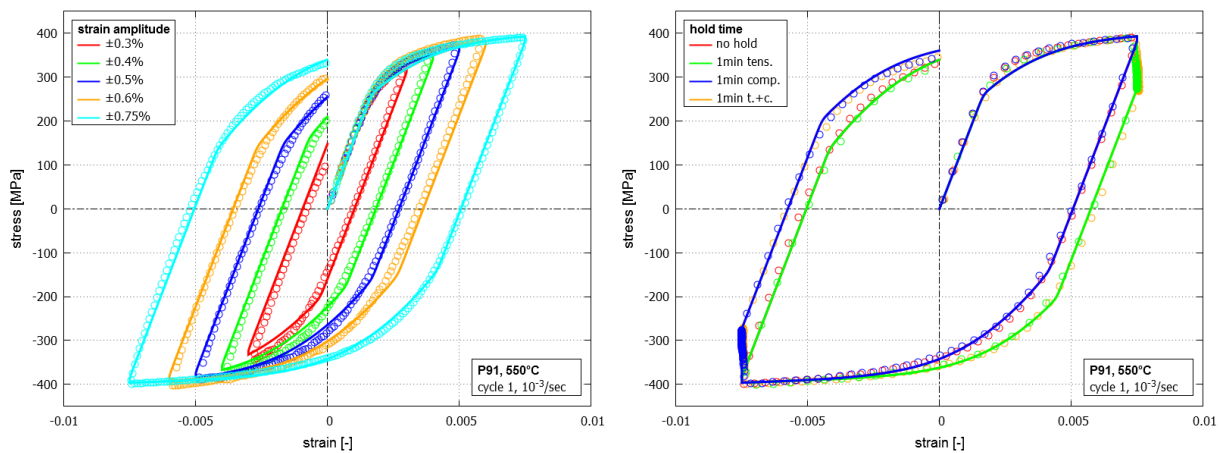


Fig. 60. Comparison of experimental and calculated hysteresis loops for first cycle, left: without hold time, right: with hold time.

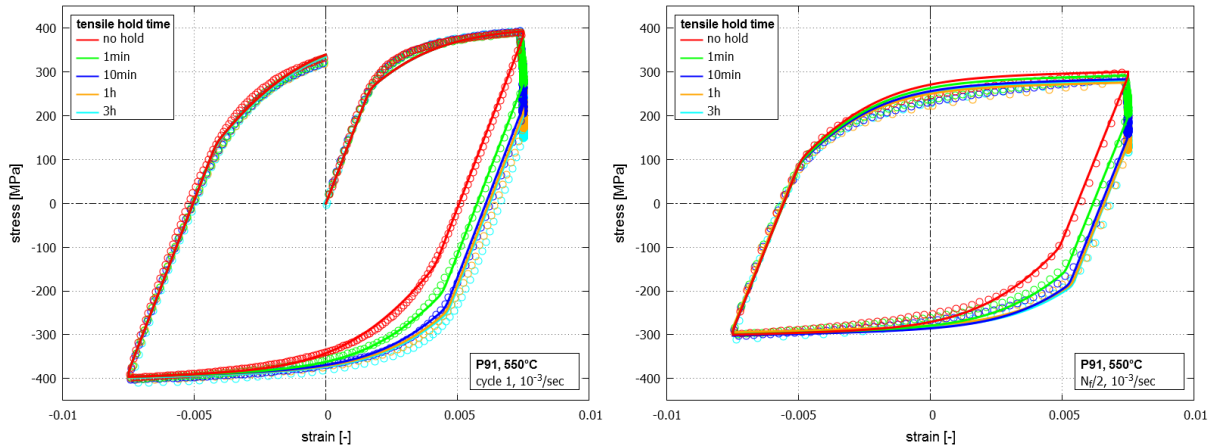


Fig. 61. Comparison of experimental and calculated hysteresis loops for different hold durations, left: 1st cycle, right: half-life cycle.

Fig. 60 compares experimental and simulation results of first cycles without hold time (left) and with hold time at different positions (right). Circles represent experimental data and continuous lines simulation results. Hysteresis loops are well described by the modified model. Note overlapping of calculated hysteresis loops with loops without hold time as well as with combined hold time under tension and compression covered by loops with hold time under tension or under compression only. Similarly, different hold time durations are well captured as shown in Fig. 61 for the first cycle as well as for the half-life cycle with slight underestimation of relaxation for hold times longer than 1 hour. Agreement of experimental results and calculations of the softened material is similar to the first cycle showing good representation of the softening effect under fatigue and creep-fatigue loadings. Underestimation of relaxation during long hold times is smaller in the softened state than in the first cycle. Notably, the reduced amount of stress relaxation for the softened material observed experimentally is also recognized by the model.

Regarding the performance of the model to describe observed softening behavior, Fig. 62 compares experimentally measured and calculated softening response for 1 hour of tensile hold time at strain amplitude of $\pm 0.75\%$. The amount of softening as well as the asymmetry of peak stresses (with tensile peak stresses lower than compressive peak stresses due to tensile hold time) is well described by the modified model.

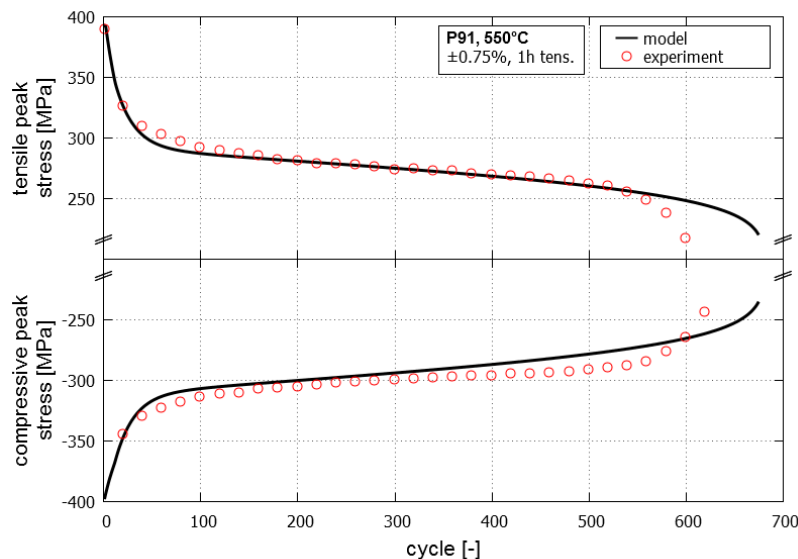


Fig. 62. Comparison of experimental and calculated softening response for 1 hour of tensile hold time.

Calculated softening response for different hold time positions is well matching the experimental results (see Fig. 63 (left) and Fig. 48 for comparison). The modified deformation model predicts reduced tensile stresses due to tensile hold time, reduced compressive stresses due to compressive hold time and a reduction of

stresses under tension as well as under compression due to combined hold times under tension and compression. Calculated additional softening due to hold time is of similar amount as observed experimentally.

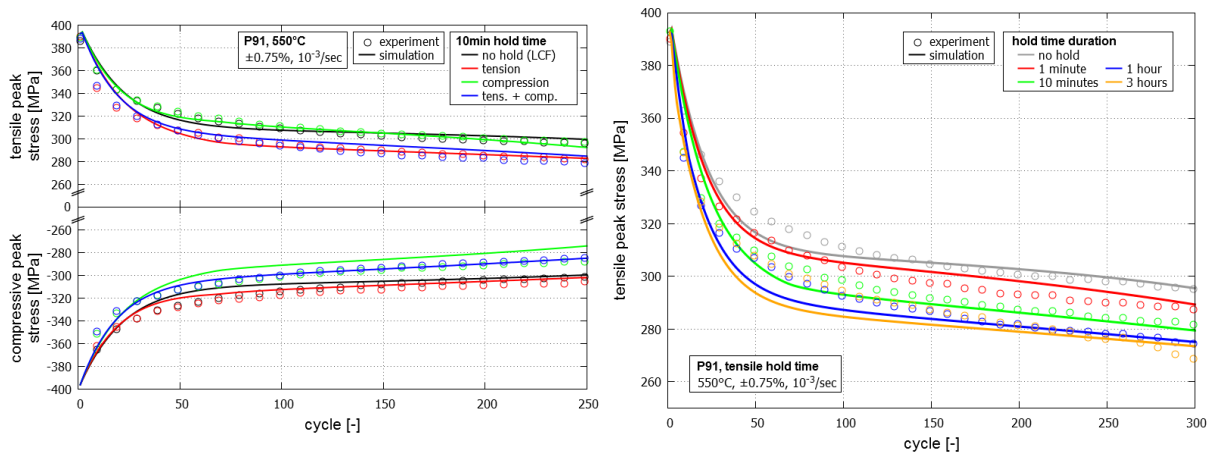


Fig. 63. Calculated influence of hold time position (left) and duration (right) on cyclic softening.

Similarly, calculated influence of hold time duration on cyclic softening correlates well with experimental results (see Fig. 63 (right) and Fig. 49 for comparison). Minimal further reduction in peak stresses for hold times longer than 1 hour is reproduced by the modified model consistent with experimental observations.

In summary, the modified deformation model manages to capture all major experimental observations regarding the influences of hold time on softening behavior satisfactorily. As accurate prediction of the deformation behavior is required for successful application of the damage model, this should enable appropriate lifetime calculation.

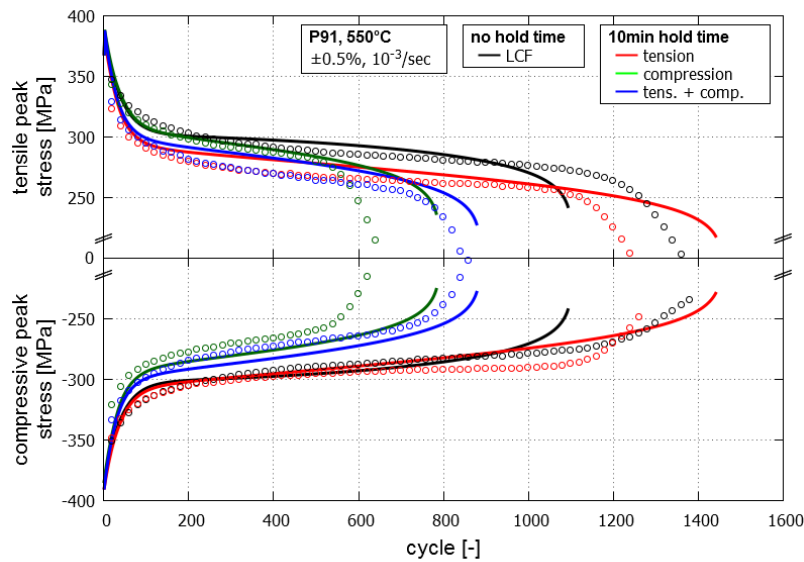


Fig. 64. Comparison of experimental and calculated hold time influence on lifetime.

In accordance to the 10% load drop criterion used for experimental determination of the number of cycles to failure, a damage value of $D=0.1$ was used as an end-of-life criterion for model calculations. In Fig. 64, measured and calculated softening curves and lifetimes are compared. No data at this strain amplitude was used for model calibration. Additionally to adequate softening prediction, the model captures the major influence of hold time on lifetime for ferritic-martensitic steels: Significantly shorter lifetimes are calculated for tests including compressive hold time. Besides, a larger number of cycles to failure was predicted for tensile hold time as it was observed in some tests.

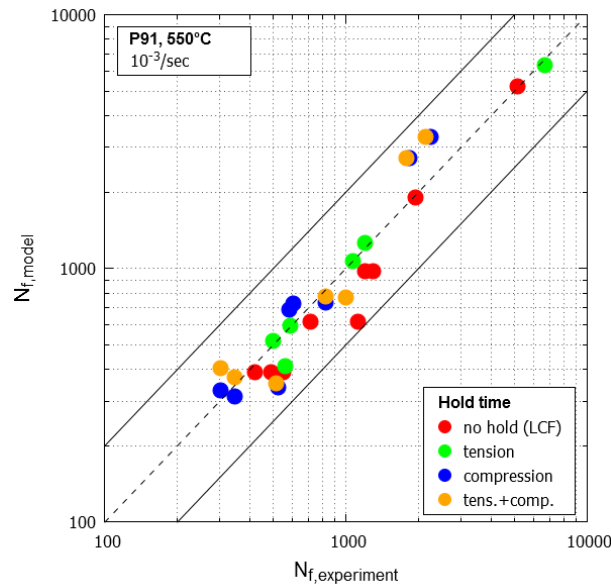


Fig. 65. Comparison of measured and calculated lifetimes for all tests conducted in this test series.

Calculated and measured lifetimes are compared in Fig. 65. All data points lie within a range of factor two, notably the largest discrepancy between measured and calculated lifetime was observed for a repeated test without hold time at $\pm 0.6\%$ strain amplitude. The first repetition of this test, however, is well described by the model indicating that a factor of 2 is a reasonable criterion for model performance considering also the scatter of experimental lifetime data.

In summary, the modified deformation model allows satisfactory description of the softening behavior of ferritic-martensitic steels under cyclic loadings with and without hold time. Based on this, the damage model allows capturing the detrimental effect of compressive hold time and gives good agreement between experimentally observed and calculated lifetimes.

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3.3 CEA (Maxime Sauzay, Diogo Gonçalves, P.-F. Giroux)

During the last three decades, numerous experimental studies highlight the large cycle-by-cycle softening observed in all tempered martensitic steels. The triggering effect of temperature, strain amplitude, strain rate and hold time were largely investigated. The macroscopic behavior evolution can be qualitatively explained by microstructure evolution during cycling. The initial microstructure of martensitic steels is characterized by a high free dislocation density and a low sub-grain size due to numerous low-angle boundaries (LABs), partitioning the martensite blocks in many subgrains. This microstructure explains the high initial yield stresses characteristic for this class of materials. Numerous TEM observations highlight the large decrease in the free dislocation density during cycling and the annihilation of many LABs leading to a strong increase in subgrain size. From our knowledge, no micromechanical modelling of these mechanisms has been proposed yet, with a full implementation in polycrystal simulation of cyclic deformation and careful comparison of the predictions at both macroscopic and microscopic scales with experimental results and observations. A detailed description of the modelling assumptions is provided in [1-3]. Only a summary is exposed in the following.

3.3.1 Assumptions

In each martensite block, twenty four easy slip systems are accounted for (BCC structure, temperature range 20°C-650°C). The constitutive laws are first based on a classical viscoplastic flow law using both activation volumes and energy. It is based on the Taylor law (dislocation-dislocation interactions) in conjunction with a kinematic stress acting on each slip system which mimics the tension line effect induced by the free dislocation bowing out along LABs which average spacing is the subgrain size. Finally, accounting for all slip systems and LABs, three populations of dislocations are accounted for: edge (mobile dislocations), screw (mobile) dislocations and LAB edge/screw dislocations. Each density obeys a specific evolution law, accounting for production (bowing-out), annihilation (climb, cross-slip) and a dedicated term describing the annihilation between mobile dislocations gliding toward LAB dislocations leading to annihilation between free and LAB dislocations (knitting-in / knitting out mechanism proposed by Caillard and Martin [4] and later by Eggeler and co-workers in the case of F/M steels [5]).

The input parameters are:

- our measured values of initial density of free dislocations and sub-grain size (TEM) [6,7] , misorientations carried out by the LABs located inside each block (HR-EBSD) and texture (EBSD) [7];
- the activation volume and energy which are adjusted for allowing the simulated tensile curves to reproduce accurately the effect of strain rate jumps.

More informations are now provided to allow a more detailed understanding on the quantitative point of view. A model for the disappearance of sub-boundaries under the effect of cyclic or monotonic viscoplastic strain was proposed based on the previous observations. According to TEM observations in-situ or dynamic calculations of dislocations on the stability of stressed sub-boundaries [8], indicate that sub-boundaries of tempered martensitic steels are pinned by precipitates and their displacements are limited. Therefore, apart from sub-boundary dislocations, mobile dislocations appear to play an essential role in softening processes. According to the Read and Shockley model [9], low-angle boundaries may be represented as networks of one, two or three family(ies) of dislocations. Figure 66 a shows an example of a symmetric tilt boundary (i.e. formed from identical edge dislocations). Annihilation between mobile dislocations and sub-boundary dislocations occurs leading to a reduction in the density of dislocations in sub-boundaries and possibly the disappearance of some of them. The subgrain size is correspondingly increased. Calculations of changes to densities or disorientations of a sub-boundary formed by a family of edge or screw dislocations are described for example in [6]. The formal definition is similar to that used by Differt and Essmann [10] and is based on continuous dislocation densities, athermal annihilation distances of two parallel edge (screw) dislocations with opposite signs, y_e (y_s) [1-3] (Figure 66 a, for edges) and the corresponding annihilation volumes. In the case of a single active system causing slip of edge dislocations with a sign opposite to dislocations of the symmetric tilt sub-boundary (Figure 66 a), the rate of change of this sub-boundary disorientation angle, θ_e , is proportional to the viscoplastic slip rate induced by mobile edge dislocations, $\dot{\gamma}_e^{vp}$, and is given by:

$$\dot{\theta}_e = \frac{y_e}{b} \theta \dot{\gamma}_e^{vp} \quad (1)$$

where b is the length of the Burgers vector. The equation for the change of the torsion boundary disorientation (composed of two types of screw dislocations) is similar in principle but is actually more complex because each screw dislocation can potentially glide on six different systems in the cubic centered structure with two families

of systems [1]. The probability of activation of one or several slip systems in a given block leading to annihilation (i.e. corresponding to dislocations similar to the sub-boundary dislocations) then has to be calculated. This probability depends on the nature (screw or edge) of the dislocations, the number of families of slip systems and the number of systems activated per block [1].

An infinite wall of parallel edge dislocations does not induce a long range field. The magnitudes of short range repulsive and attractive zones (for LAB parallel dislocations with the same sign) are equivalent close to the sub-boundary ($\sim h$) [11,12]. But if the height of the boundary is finite, $2L$, there is a relatively long range ($\sim L$) repulsive field and also a short range repulsive field ($\sim$ a few inter LAB dislocation spacing, h , with $b \ll h \ll L$) [11,12] (see Figure 66 b). At the scale of a subgrain aggregate, and due to the many sub-boundaries present, it would appear that there is internal stress higher than the macroscopic stress. But shorter range repulsive stresses found on a boundary with a finite height would explain why mobile edge dislocations with opposite signs would be attracted by the sub-boundary while edge dislocations with the same sign would be repelled (see Figure 66 b). Fields of similar stresses were calculated with boundaries formed from one or two families of screw dislocations [13]. Therefore for reasons of simplicity, we have only considered the flow of mobile dislocations with a sign opposite to the sign of the sub-boundary. Therefore we neglected all mechanisms that increase the disorientations and create sub-boundaries (knitting in mechanism).

The variation in free dislocation densities is modelled by different creation / annihilation mechanisms [1-3]. In the simple case of edge dislocations, the variation equation becomes [1-3]:

$$\dot{\rho}_e = \frac{2}{bd} \dot{\gamma}_s^{vp} - 2 \frac{\gamma_e}{b} \rho_e \dot{\gamma}_e^{vp} - \frac{\gamma_e}{b} \frac{1}{bd} \theta_e \dot{\gamma}_e^{vp} \quad (2)$$

where ρ_e , is the density of mobile edge dislocations and $\dot{\gamma}_s^{vp}$ is the viscoplastic slip carried by mobile screw dislocations. The first term in equation (2) simulates the production of dislocations by activation of sources in a subgrain with size d (dependent on the plastic slip rate carried by screw dislocations, assuming square loops). The second term describes annihilation with mobile dislocations with opposite sign. Finally, the last term is representative of the annihilation of mobile dislocations with sub-boundary dislocations, the mechanism at the origin of equation (1). As mentioned in the description of Eq (1), the equation for the variation of the density of screw dislocations is similar in principle but is actually more complex because each screw dislocation can potentially glide along six different systems in the cubic centred structure if accounting for two main families of systems [1-3]. Finally, at high temperature, a climb-induced annihilation term is added to Eq. (2) to account for annihilation between edge dislocations climbing in opposite directions. This term is proportional to the lattice self-diffusion coefficient. It then becomes negligible at room temperature and is more influent as temperature increases.

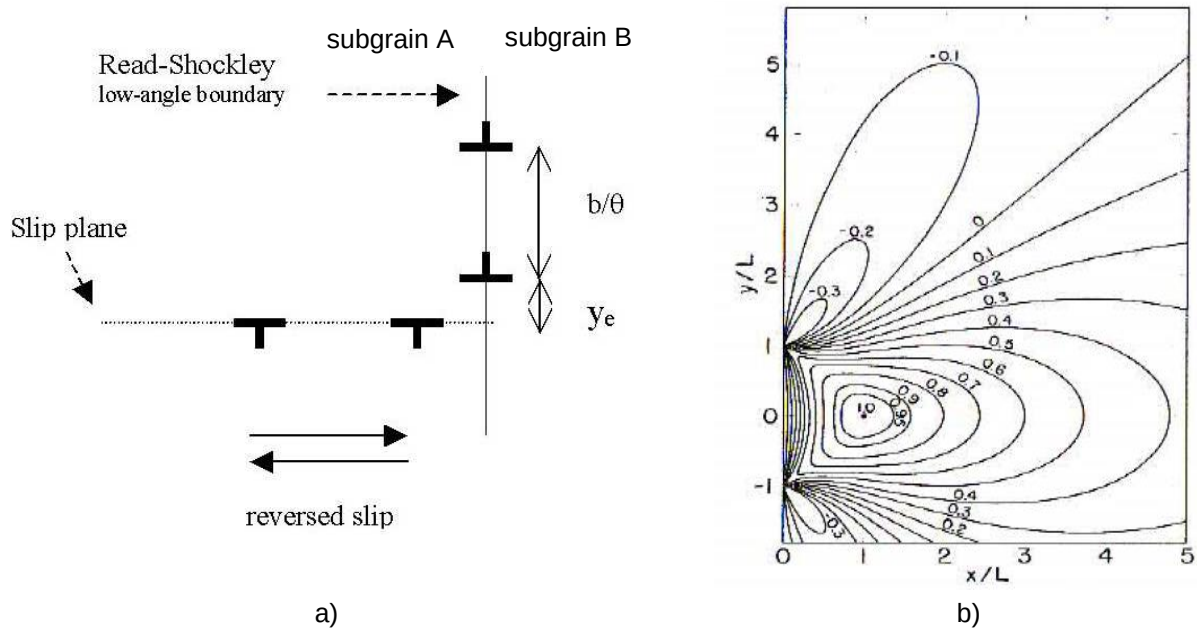


Figure 66. a) Interactions between mobile dislocations and dislocations of low-angle boundaries (symmetric tilt boundaries) (length of the Burgers vector: b ; disorientation on the sub-boundary: θ) [10]; b) isovalues of the shear stress, σ_{xy} , induced by the symmetric bending boundary with finite length $2L$. Edge dislocations have an infinite length along the direction perpendicular to figure [11,12]. Strain unit: $\mu b/2\pi(1-\nu)h$ (elastic shear modulus: μ ; Poisson's ratio: ν ; inter-dislocation distance in the bending boundary : $h=b/\theta$).

Each block of a given crystallographic orientation is assumed to be embedded in a homogenous matrix (Fig. 67), for which mechanical behavior is predicted using a self-consistent assumption. The polycrystal homogenization is carried out using an elastic-viscoplastic transition rule, referred to as Kröner-secant. The mean-field homogenization procedure has been carefully validated through the comparison with numerous crystal viscoplasticity FE computation results [13]. A polycrystalline model based on a mean-field homogenization procedure was proposed [1,3]. It is based on:

- the softening model at the scale of a block based on two mechanisms. The number of sub-boundaries reduces depending on the activation of block slip systems, which causes an increase in mobile dislocation pinning distances due to the growth in subgrain size. Indeed, the Orowan critical stress induced by the line tension reduces due to the increase in the curvature radius. Edge and screw dislocations are treated similarly because the critical (maximum) stress is slightly dependent on the nature of the dislocation (edge or screw) although the line tension coefficient depends much more on the dislocation nature. The dislocation density then reduces subsequent to annihilation between mobile dislocations. The stress required for crossing the dislocation then reduces too. The Taylor law assumes that the corresponding critical stress is proportional to $\sqrt{\rho}$, where ρ is the dislocation density. It is assumed that the two critical shear stresses can be added although also other combinations may be used (summation under the square root).
- a block $\rightarrow$ polycrystal localization law and a homogenisation procedure (for example see [1]). Many different self-consistent models are available such as Kröner, Hill-Hutchinson or Molinari models can be used depending on the level of plastic strain and the influence of viscosity. As the high temperature behaviour of grade 91 steel is elastic-viscoplastic, particularly if we are concerned by fatigue-relaxation. Then the Kröner-secant polycrystal scheme validated by FE computations is used in the following. The SiDoLo software has been modified accordingly. This software is used to implement macroscopic or crystalline viscoplasticity laws and homogenisation models. The crystalline structure can also be modified. The behaviour laws are then integrated using Runge-Kutta type schemes.
- Numerous similar inclusion problems, similar as the one shown in Fig. 67 b, are solved altogether at each loading increment, account for a large number of random crystallographic orientations of the F/M blocks. Accounting for the self-consistence allows the solving of the whole set of equations and the computation of F/M block and macroscopic strains and stresses. Usually, considering $N=100$ random orientations permits us to provide predictions which are stable with respect to the number of blocks and the sets of N random orientations.

A specific subsection is dedicated to the material parameters which number is rather low (about ten). Most of them are directly measured in the as-given material and allow a description of the initial state. Other ones are provided by literature (annihilation distances, self-diffusion coefficients). Finally, as the obstacles may be clusters of solid solution atoms, small intra-subgrain carbo-nitrides, dislocation debris, a direct measurement of the activation volume and energy for dislocation glide seems out of the scope of our study. An inverse identification is then used, based on specific tests carried at PSI including strain rate jump tests.

The next subsection focuses on the prediction of the cyclic macroscopic behaviour which is compared to experimental data provided by KIT.

The last subsection shows the difficulties in predicting long term relaxations (one month) and proposes to account for the heterogeneity in the microstructure induced by a large number of different obstacles resulting in delayed relaxation. That additional and progressive relaxation is carried out by a certain fractions of the dislocations for which glide does not stop as expected when they encounter more strong and numerous obstacles.

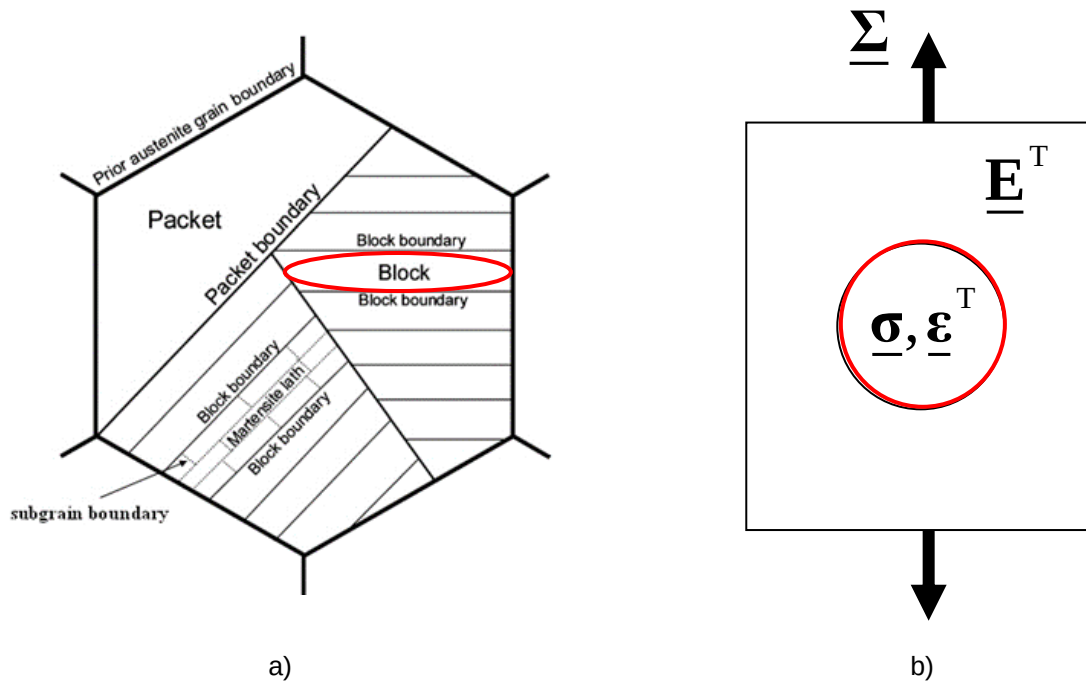


Fig. 67. a) Complex microstructure of F/M steels. The blocks are almost homogenous on the crystal orientation point of view but contain low-angle boundaries and numerous free dislocations; b) One F/M block embedded in a matrix which mimics the behavior of the whole polycrystal.

3.3.2 Material parameters

Tables 15 and 16 provide parameters values corresponding to the material under study. The edge and screw annihilation distances have been measured in face-centred cubic alloys such as austenitic stainless steels in the temperature range of interest (see [1] for details). Parametrical studies reported in [1] showed that the predictions are rather stable with respect to the physical range of variation of the material parameters.

	823K	873K
ν	0.3	0.3
E_y	170 GPa	140 GPa
b	0.254 nm	0.254 nm
γ_e	3.74 nm	3.74 nm
γ_s	50 nm	50 nm

Table 15. Parameters provided by literature (isotropic elasticity coefficients, Burgers vector length, critical edge and screw annihilation distances).

$D=D_{sd} \cdot \exp(-Q_{sd}/RT)$	Reference [15]
D_{sd}	10.1 (m ² .s ⁻¹)
Q_{sd}	254000 J/mol

Table 16. Lattice self-diffusion parameters.



Fig. 68. Microstructure of subgrains inside each ferritic-martensite block (TEM picture: B. Fournier). TEM observations of free dislocations inside blocks [6]. Measurement of subgrain size ($0.37 \pm 0.1 \mu\text{m}$) and free dislocation density ($1.1\text{--}1.6 \times 10^{14} \text{ m}^{-2}$) [7]. As-received state.

The free dislocation density was measured during previous studies carried out on the material of interest (30 mm thick plate) (Fig. 68 a). TEM observations allowed the measurement of the average subgrain size too (Fig. 68 b). The measured values and the error bars are given in Table 18.

	Grade 91 steel (30mm thick plate) [6,7]	Grade 92 steel [1]
Free dislocation density	$1.1\text{--}1.6 \times 10^{14} \text{ m}^{-2}$	$2 \times 10^{14} \text{ m}^{-2}$
Subgrain size	$0.37 \pm 0.1 \mu\text{m}$	$0.43 \pm 0.1 \mu\text{m}$

Table 17. Free dislocation density and average subgrain measured at CEA/SRMA (France) in grade 91 and grade 92 steels.

Finally, the activation energy and volume characterizing the thermally-activated glide of dislocations have been adjusted using inverse identification. As different obstacles may influence the microstructure, such as clusters of solid solution atoms, MX precipitates and dislocation debris, a more straightforward measurement of Q and V seems out of our scope. We focus on the 550-600°C temperature ranges. The two parameters were adjusted in order to provide predicted curves close to the experimental ones, accounting for one strain rate jump test at 550°C and three tensile tests at constant strain rates at 600°C. The comparisons between predictions and experimental data are shown in Figs. 69 and 70.

It should be noted that the activation energy for glide is rather close to the lattice self-diffusion coefficient ($Q_{sd}=2.63\text{eV}$). This may indicate that climb-assisted glide plays a role as thermally-activated process at such high temperatures, as shown by creep experiments carried out on this class of alloys.

Q	V	Q/V	
2.25eV	$110b^3$	199.96 MPa	Grade 91 (30mm thick), this study

Table 18. Activation energy and volume. The adjustment procedure is shown in Figs. 69-70.

The measurement of the temperature dependent activation energy and volume, carried out by PSI, assume that the Taylor assumption holds at the F/M block scale ($M=3$). The activation energy measured at 873K accounts for test results carried out at 823 and 873K. The measured values are 2.6 and 2.4 at 873K. The activation volume ranges between 1.88 and $2.28 \cdot 10^{-28} \text{ m}^3$. Our adjusted value of $V=1.7 \cdot 10^{-28} \text{ m}^3$ is close to the measured values provided by PSI, which depends on the adopted value of the Taylor factor ($M=3$ in the PSI evaluation). Using a rather accurate polycrystal homogenization procedure [13], it is believed that the values evaluated in Table 18 are slightly more accurate than the ones obtained by using a conventional value of the Taylor factor.

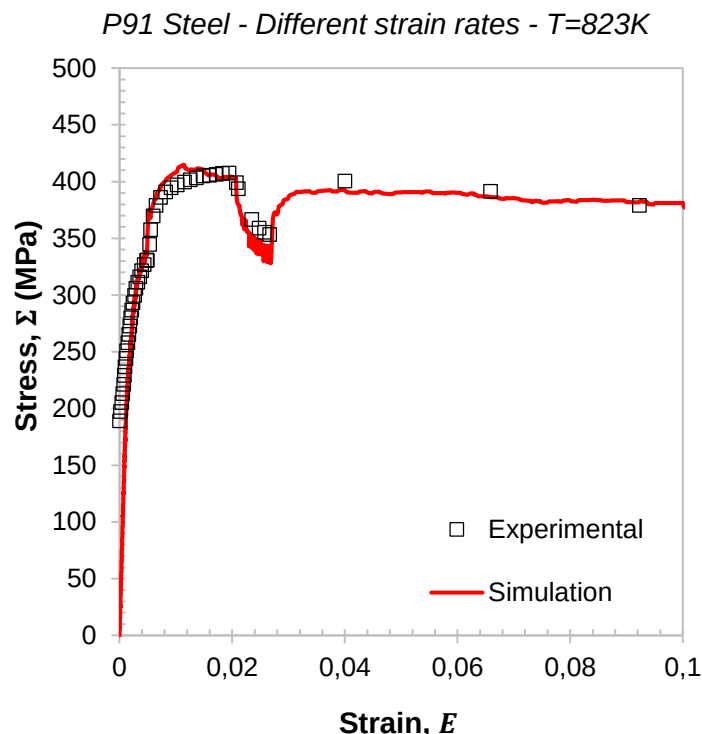


Fig. 69. Comparison between predicted and measured stress depending on strain. Strain jump data: PSI. Grade 91 steel, 823K. Identification of the crystal viscoplasticity parameters, V and Q .

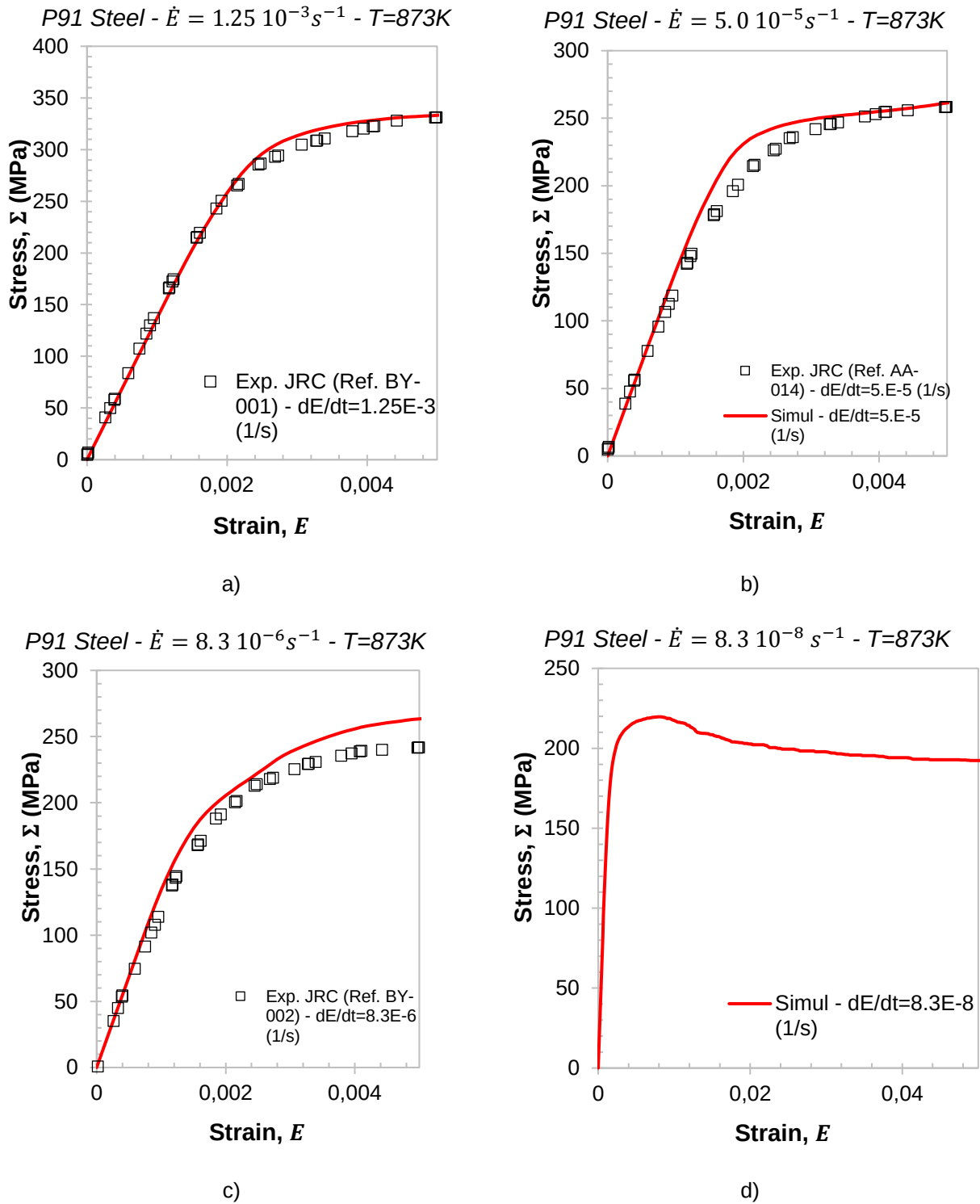


Fig. 70. Comparison between predicted and measured stress depending on strain. Strain jump data: PSI. Grade 91 steel, 823K. Identification of the crystal viscoplasticity parameters, V and Q .

3.3.3 Cyclic loading

The influence of three different cycling loading parameters is now discussed with respect to data provided by the Project and others.

First, the stronger softening with increased strain amplitude is reproduced as shown by the comparison between Fig. 71 a and b. As expected the final quick stress drop induced by fatigue damage is not predicted by the polycrystal model, which account only for viscoplasticity mechanisms.

Second, to discuss temperature effect, computations have been carried out for temperatures of 550 and 600°C. The corresponding experimental data were produced during the MATTER Project. In agreement with experiments, simulations show that the higher the temperature the more pronounced the softening (Fig. 72).

Third, softening is triggered at low strain rate, still due to higher plastic strain and time-recovering. Such trend is reproduced by experiments and simulations (Fig. 73).

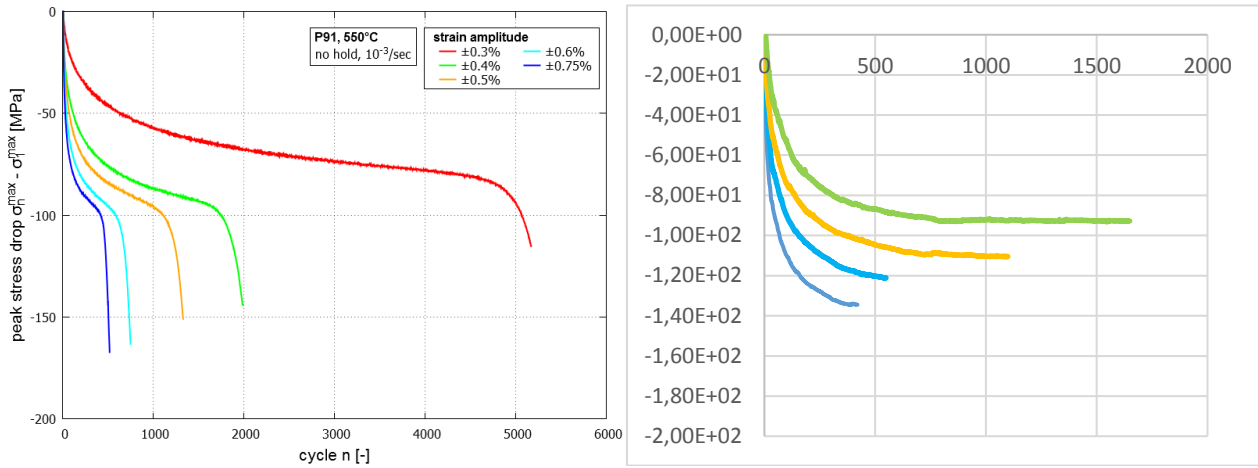


Fig. 71. Comparison between predicted (a) and experimental (b) softening curves plotted with respect to the number of cycles. First half-cycle. Low strain amplitude, 823K. Experimental data provided by KIT.

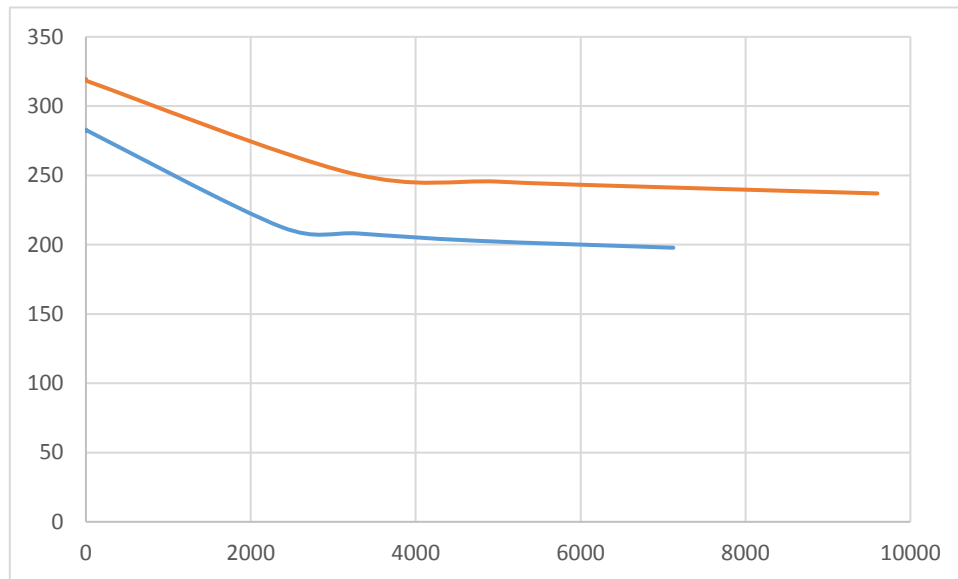


Fig. 72. Measured evolutions of stress amplitude with the number of cycles. Strain amplitude: 0.25%, strain rate: $10^{-3}/\text{s}$ and temperatures: 550°C (orange line) and 600°C (blue line). Data provided by JRC.

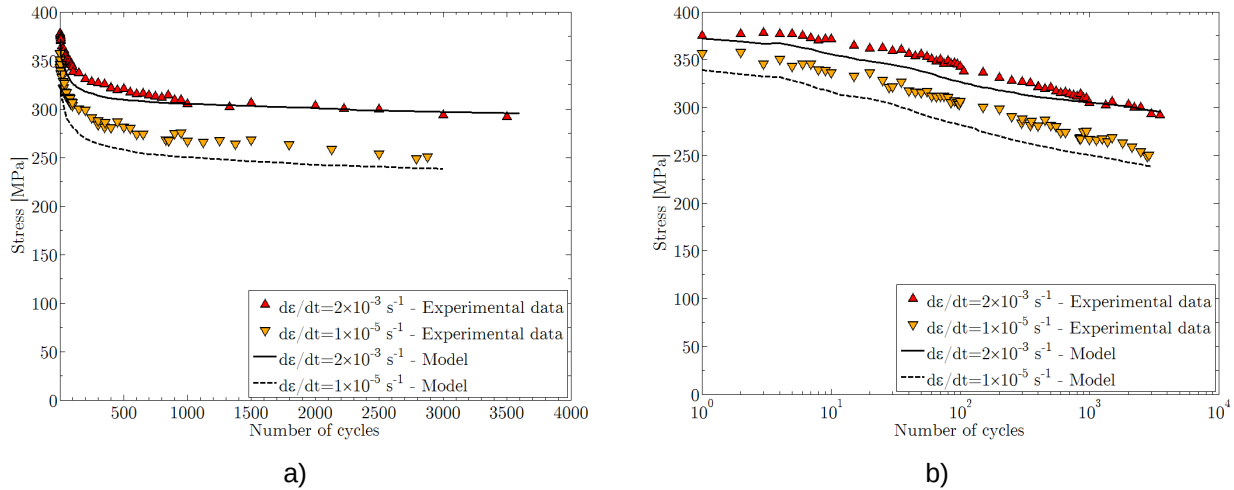


Fig. 73. Variation of predicted and measured stress amplitudes: a) linear scale; b) logarithmic scale (steel grade 92, cyclic strain: $\pm 0.35\%$, strain rate $2 \times 10^{-3} /s$ and $10^{-5} /s$, $T=550^{\circ}C$) [1].

3.3.4 Long term relaxation

During the project, many long term relaxations curves, at 550 and 600°C, were generated with duration from three days up to one month. Additionally, the effect of the peak strain has been investigated. And finally, even the influence of loading ramp strain rate has been experimentally studied. Four institutes participated to these experiments, mainly JRC and VTT, but also, KIT and CVR.

First of all, all the predictions compared to experimental relaxation curves show that short term and long term relaxation should be separated:

- Provided the time is lower than one tenth of hour, the agreement between simulations and experiments remains reasonable (Figs. 74 a and b);
- But if time is higher than one tenth of hour, then relaxation is largely underestimated by the modelling. The stress level usually saturates after a simulated time of about one hour (Fig. 74 b).

The early saturation of the computed stress level compared to experimental data is also observed when predicting long term relaxation of austenitic stainless steels too (600-750°C). At least one important mechanism seems to be missing when modelling the glide of free dislocations at low stress and low strain rate. In the experiments shown in Fig. 74, the strain rate is of the order of $/s$ after one tenth of hour.

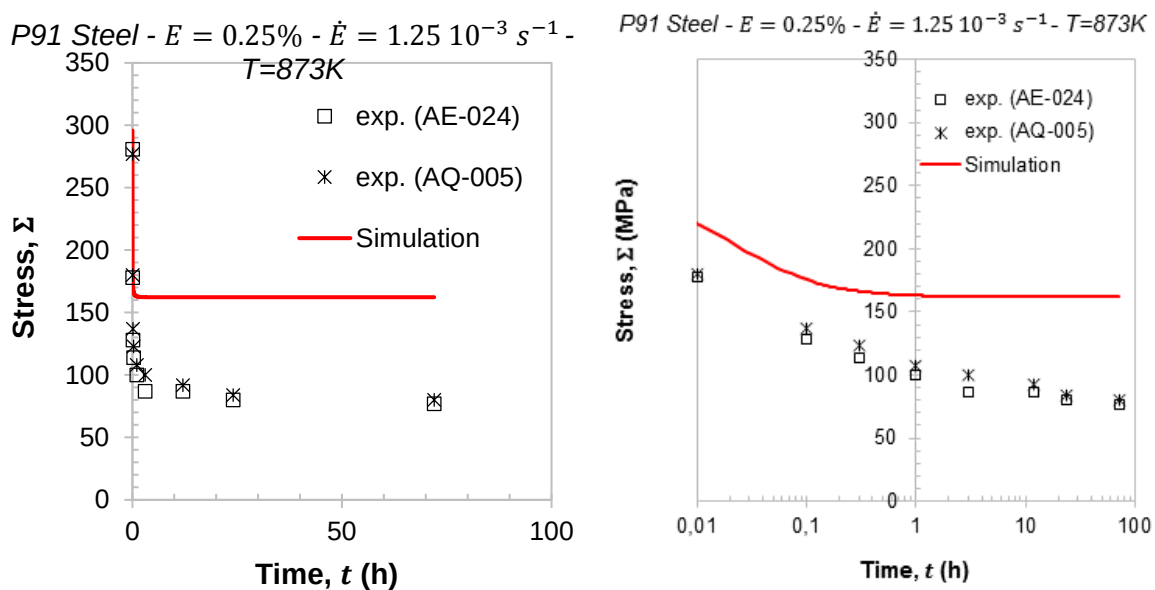


Fig. 74. Predicted and measured relaxation curves. Experimental data provided by JRC. Only one unique population of dislocation gliding through a regular array of forest obstacles is accounted for.

Therefore some essential mechanisms are missing. In particular, some of the dislocations seem to keep on gliding even below the conventional yield stress. Additionally, intergranular stresses are added due to plastic deformation and incompatibilities. These forward and backstresses either hinder or help dislocations keeping on gliding. Such internal stresses are accounted for in the modelling but do not allow a fair description of long term relaxation. Several assumptions have been considered:

- Additional recovery during long term creep. As the material is heat-treated, the temperature is rather low and the relaxation time short, only a very modest recovery is expected and TEM observations do not show any clear evolution for instance during tensile loading at low strain rate [1];
- The effect of the coupling between the critical shear stress (dislocation forest obstacles) and the bowing stress induced by the pinning of gliding dislocations at LAB is investigated too. And using an additive version or a square root version does not affect much relaxation;
- Creep tests proved that at low stress, a specific strain rate regime may be observed (see section 4, recommended further work) [2]. At 600°C, the transition occurs at a tensile stress of 70MPa and for a creep strain rate of about 10^{-11} /s. Such a low stress regime of viscoplastic deformation should then be accounted for after one month at 550°C for instance. Nevertheless, another mechanism should be accounted for as the strain rate reaches about a few 10^{-7} /s during relaxation, as the predicted stresses are much overestimated with respect to measured values (Fig. 74 b). Moreover the transition strain rate is more than 10000 higher than the one observed during low stress creep tests. Clearly some of the dislocations keep on gliding even if the local shear stress is lower than the critical shear stress computed using the Taylor equation, neglecting both bowing stress and viscous stress induced by short range interactions. It should be noticed that accounting for climb deformation would not change much the relaxation predictions because pure climb becomes efficient only at very low stress and may explain the very low stress creep strain rate regime [2]. The question is still open (see 4.3 Recommended further work).
- One last assumption remains to be discussed. As the obstacle distribution is heterogeneous in steels (MX precipitates, clusters of solid solution atoms, forest dislocation), the use of a unique critical shear stress applied to all dislocation is questionable, particularly as stress is low. Above a rather high critical shear stress, for instance evaluated through the Taylor equation, all the dislocations glide along large distances as shown by discrete dislocation dynamics results. And there seems to exist an intermediate domain of shear stress at which some of the dislocations glide through rather loose arrays of obstacles whereas other ones are blocked by stronger local distributions of obstacles. The unique reason of such differences between dislocation behaviour during relaxation could be caused by the locally heterogeneous distribution of obstacles, for instance forest dislocations.

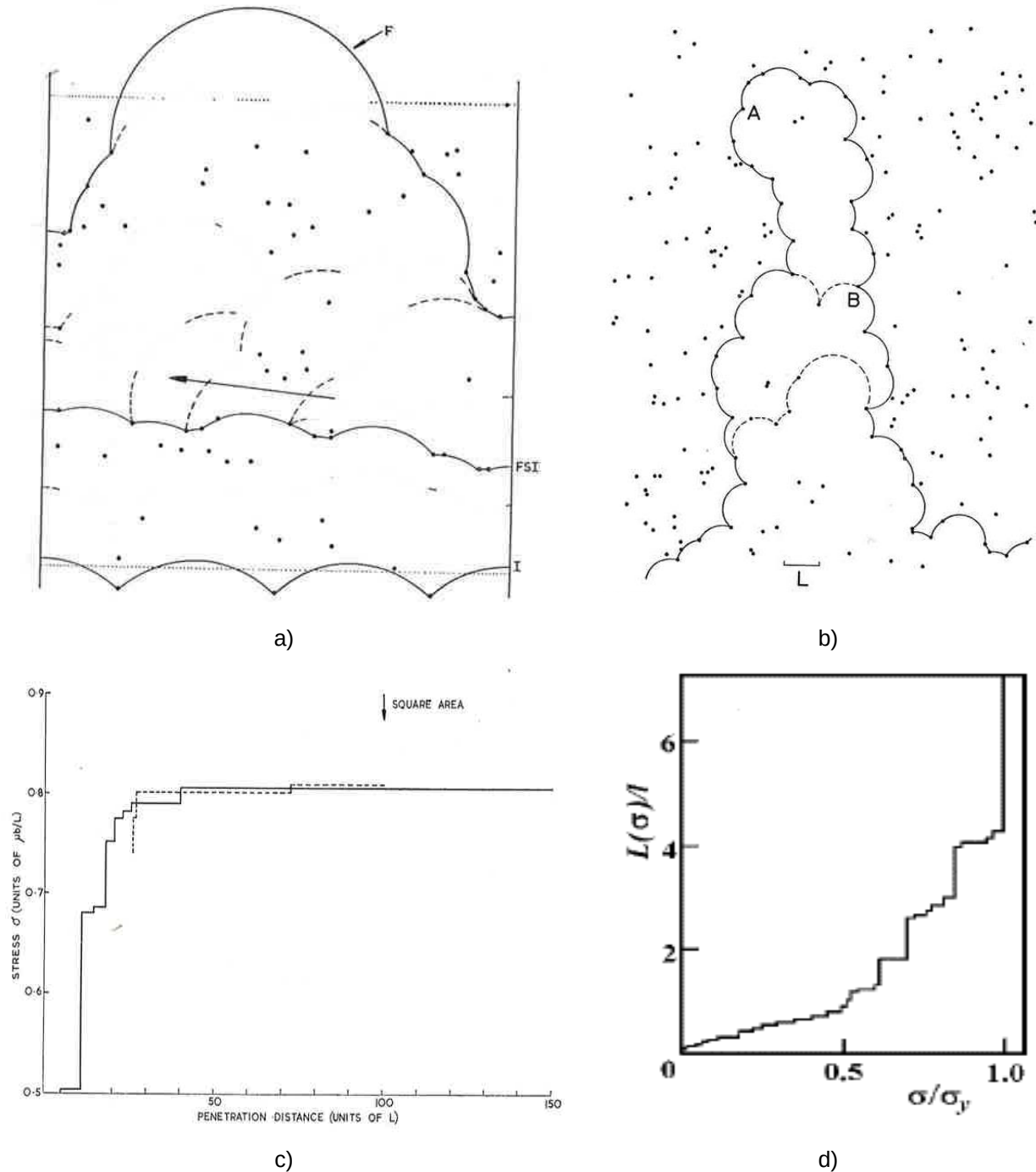


Fig. 75. Examples of glide of one individual dislocation in a heterogeneous 2D array of obstacle of either a) high breaking angle (130°) or b) low breaking angle (10°), or equivalently low and high obstacle force [15]. The average inter-obstacle distance is denoted as L . In Fig. 75 a, the initial position of the dislocation is denoted as 'I', the intermediate one as 'FSI' (final stress increase) and the final one as 'F' (the dislocation is definitively stopped at the applied shear stress; c) Maximum normalized gliding distance (x-axis) with respect to the normalized resolved shear stress (y-axis) (same conditions as Fig. 75 a) with $\sigma_{th}=0.8\mu b/l$) and d) the reversed plot in the same conditions as Fig. 75 b.

Interestingly, a careful examination of the microplasticity regime shows that dislocations may glide at a shear stress level well below the critical stress evaluated by the Taylor equation, σ_{th} . This may indicate that considering different populations of dislocations with different critical shear stresses may be meaningful.

Such dependence of glide distances with respect to the heterogeneous local obstacles configurations has been reported by several authors. They carried out discrete dislocation dynamics computations, account for various densities of obstacles and obstacle force (or inversely breaking angle) (Fig. 75 a and b, for respectively low and high breaking angles, or equivalently high and low obstacle forces). Similar results were found more recently accounting for large ranges of variations of obstacle densities and forces [18], aiming to provide

analytical formulae describing the gliding distance depending on applied stress, obstacle density and obstacle force. These formulae are accounted for in viscoplasticity laws is climb included to by-pass the strongest obstacles configurations (Figs. 75 a and b, dislocation positions 'F' and 'A' respectively). The maximum normalized gliding distance (x-axis) with respect to the normalized resolved shear stress (y-axis) (Fig. 75 c with the same conditions as Fig. 75 a). At an applied shear stress close to $\sigma_{th}=0.8\mu b/l$, then the gliding distance becomes infinite, which allows the usual definition of the critical shear stress, $\sigma_{th}=0.8\mu b/l$, which applies to all local obstacles configurations. Width of the simulation box: 100L (continuous line) and 10000L (dashed lines) [15]. Fig. 75 b corresponds to a shear stress of $0.7\mu b/l$, that is -15% below the critical stress. A similar plot but the inverted x and y axis is shown in Fig. 75 d in the same conditions as Fig. 75 b) [16].

The ultimate goal is to predict low stress creep deformation in metals and alloys at high temperature. Introducing such heterogeneous dislocation viscoplasticity law below the critical shear stress is not straightforward. For the sake of simplicity, and as a first attempt, we simply assumed the followings:

- Dislocations are assumed to be completely arrested when the applied shear stress is below than one-half of the usual critical shear stress, that is $\sigma_{th}/2$ (Figs 75 c and d);
- In each F/M block, for each slip system and for both edge and screw dislocations, the populations are divided into five sub-populations with effective critical shear stresses σ_{yth} , $0.9 \sigma_{th}$, $0.8 \sigma_{th}$, $0.7 \sigma_{th}$ and $0.6 \sigma_{th}$, which is in reasonable agreement with Figs 75 c and d). Considering the crudely linear evolution of the gliding distance between $0.6 \sigma_{th}$ and σ_{th} , assuming that the sub-populations have the same densities seems reasonable as a first attempt.

Such assumptions are easily implemented in the polycrystal code. No additional fitting parameter is required. Nevertheless, introducing $n=5$ populations of dislocations with decreasing critical stress, in agreement with Figs 75 a and b requires a new adjustment of the activation volumen and energy (thermally-activated glide). The same procedure as the one described in subsection 3.3.2 was applied (Figs. 69-70). It leads to updated values of $Q=2.75\text{eV}$ and $100b^3$, instead of $Q=2.25\text{eV}$ et $110b^3$ (one homogenous population of dislocations, Table 18). The activation energy is now closer to the lattice self-diffusion energy ($Q_{sd}=2.63\text{eV}$), which may us suggest that climb-assisted glide may be an important thermally-activated glide mechanisms in F/M steels at 550-600°C.

It should be noted that using a higher number of subpopulations does not affect much the results. Nevertheless, if the results are improved with respect to the ones obtained assuming a unique dislocation population, either gliding freely above the critical stress or not gliding at all below (Figs. 74 a and b), then refinements in the modelling will be introduced as accounting more precisely of the obstacle density and force of interest (dislocation forest) and accounting of some low but not fully negligible dislocations glide below $0.6 \sigma_{th}$. The average gliding distance can be more accurately accounted for as well as its nonlinear evolution with σ/σ_{th} thanks to numerous discrete dislocation dynamics computations allowing a more reliable analytical description of the dependence of the average gliding distance with the normalized shear stress [18].

A first loading case is studied in details using Figs 76-77. The peak stress is rather well predicted by the models even if the computed peak stress is about ten MPa higher than the measured one (Fig. 76 a). The computed and measured evolution of stress with time are qualitatively similar even if the predicted values are about thirty MPa higher than the measured values (Fig. 76 b). It seems that no saturation of the stress level occurs even after a 3 days relaxation which is in agreement with experimental data. The computed and measured evolutions of the drop in stress are even closer than the stress level evolution (Figs. 77 a and b) because of the initial overestimation at the peak strain and a very short term relaxation, which is a bit larger in experiments than in the modelling. For a duration lower than one hour, the measured and predicted stresses are almost linear with $\log(t)$, which is expected from the expression of slip rate with respect to viscous stress (exponential function using the activation volume value). And then, between one and 72 hours, a convex shape is both observed and predicted showing a decrease in the relaxation rate. No relaxation curve is used in the identification procedure, but including the progressive arrest of gliding dislocations induced by the heterogeneity in the obstacle distribution, improves clearly the ability of the modelling to predict LT relaxations. The possible saturation with then is discussed below considering one and two months long relaxations.

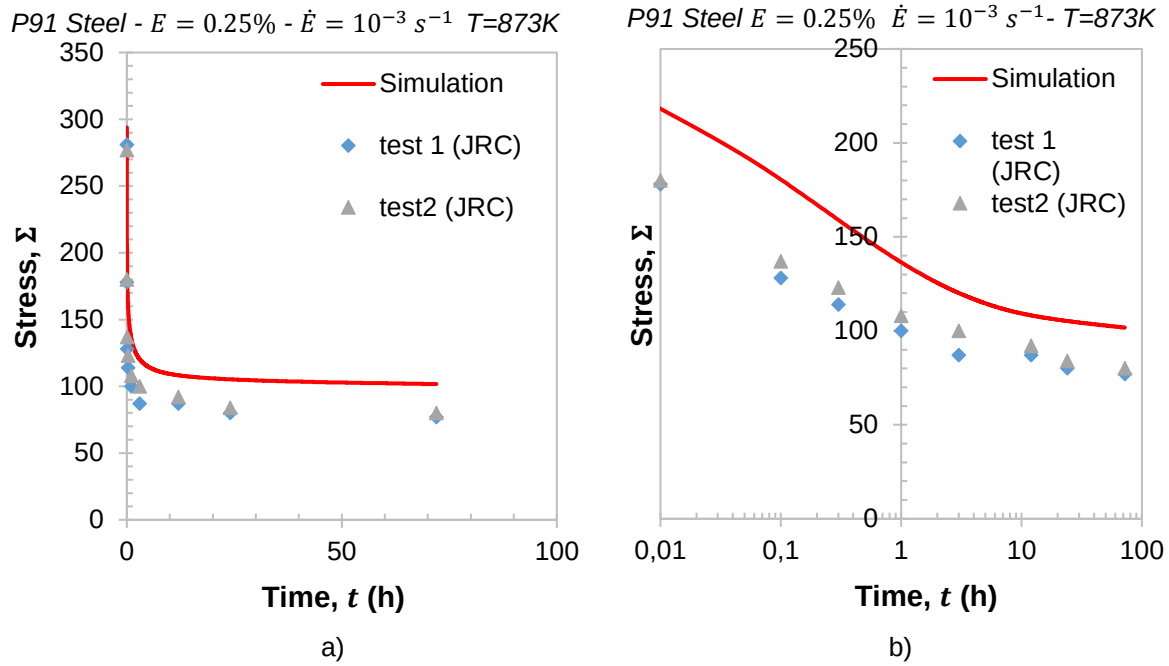


Fig. 76. Comparison between predicted and experimental relaxation curves. First half-cycle. Low strain amplitude, 873K, hold time: 72h (tensile peak). The modelling account for the heterogeneous distribution of the forest of dislocations. a) linear scale and b) log scale.

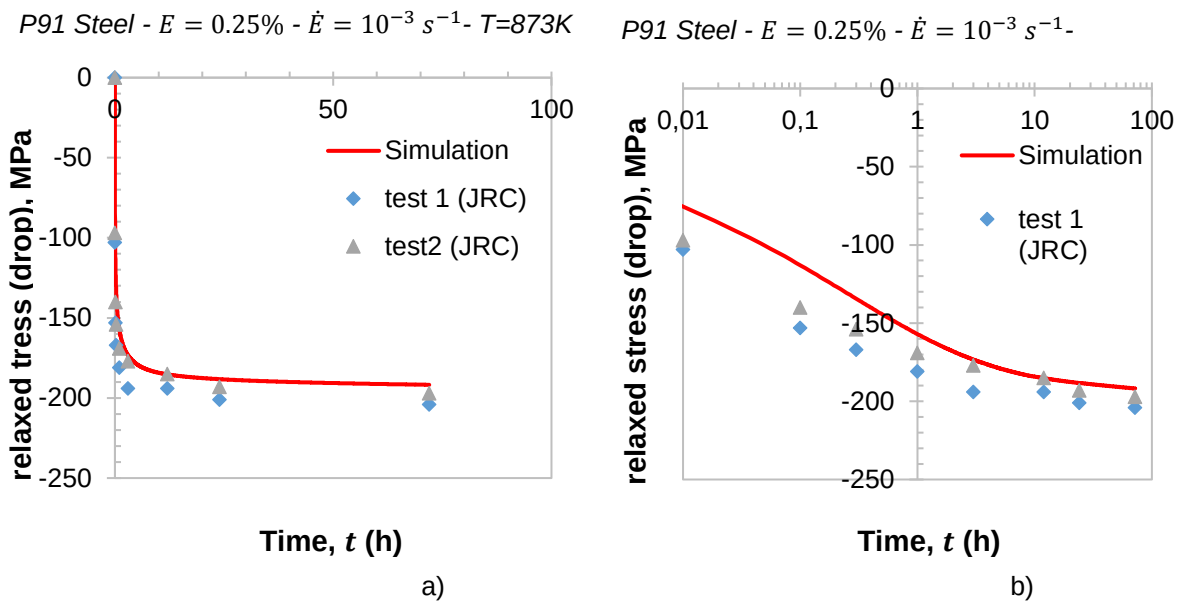


Fig. 77. Comparison between predicted and experimental evolution of the drop in stress during a 72h relaxation. First half-cycle. Low strain amplitude, 873K, hold time: 72h (tensile peak). The modelling account for the heterogeneous distribution of the forest of dislocations. a) linear scale and b) log scale.

The temperature and peak strain effects can be studied considering Fig. 78 a) and Fig 78 b, showing respectively the experimental and predicted relaxation curves.

As expected, for the same peak strain, the experimental and simulated stress curves at 600°C are below their counterpart at 550°C. The temperature drift is surestimated by modelling. Except for that drift, the shapes of the relaxation curves are rather similar at both temperatures which is shown experimentally and theoretically.

At a given temperature, the relaxation curves are very similar from 6min up to 100 hours. Both modelling and experiments show similar trends. Only during the very short term relaxation should differ from each other depending on the peak strain value (0.25 and 0.35%). The shapes of the relaxation curves are fairly well predicted by a drift is once more observed (about +30MPa).

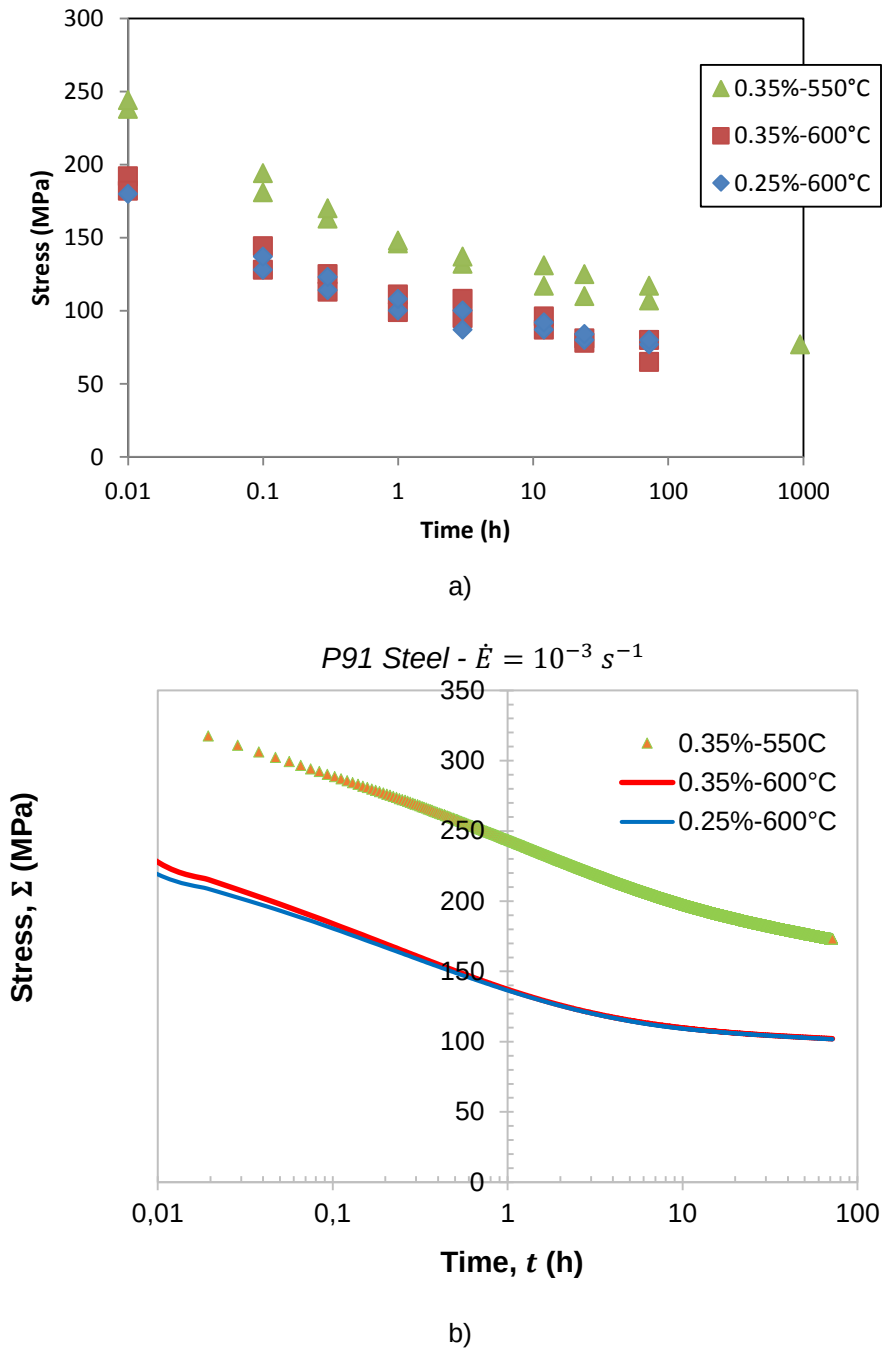
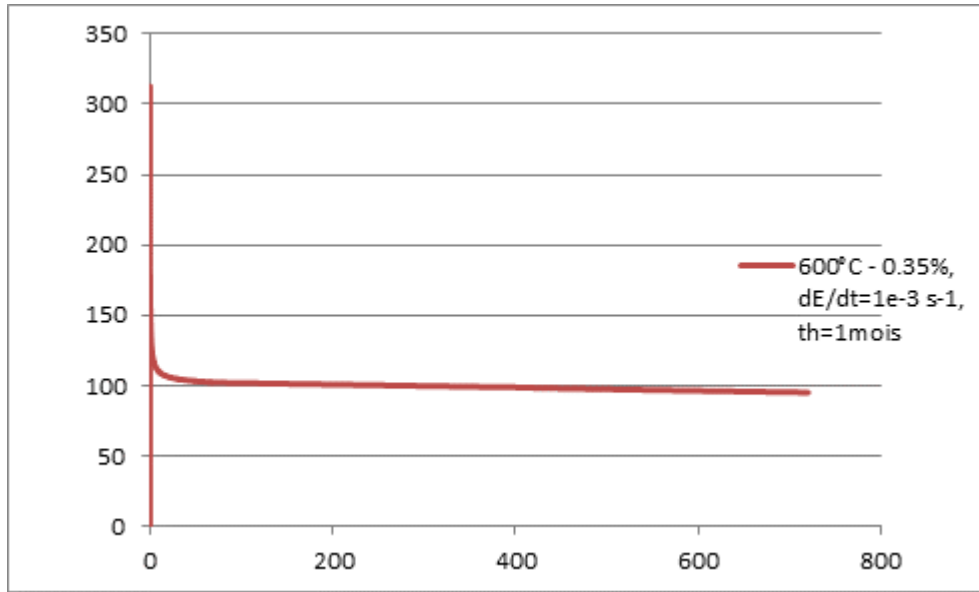
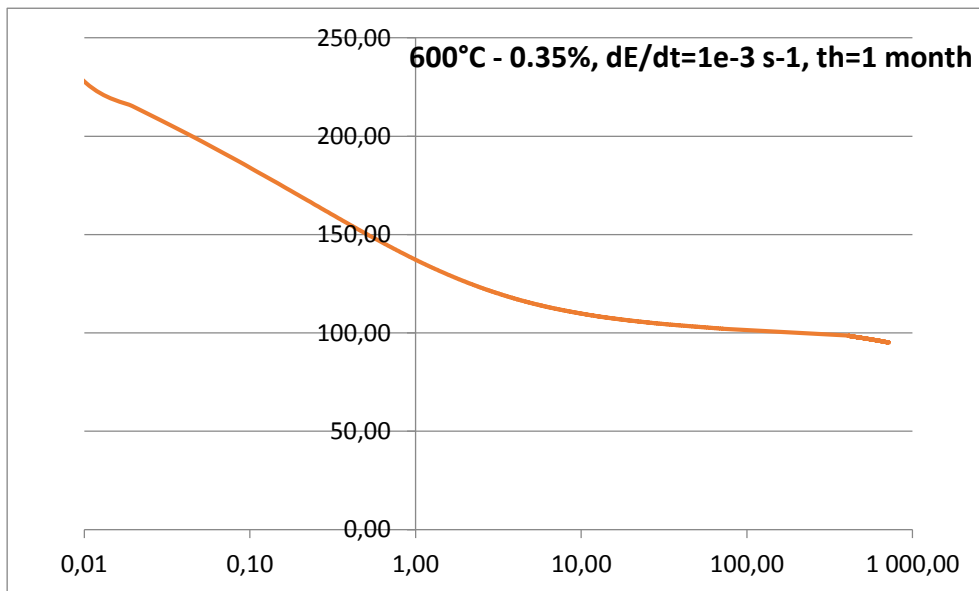


Fig. 78. Comparison between the evolution of stress measured during relaxation obtained by a) experimental measurements (JRC) and b) modelling First. half-cycle. Three couples of strain amplitude and temperature are considered: (0.35%, 550°C), (0.35%, 600°C) and (0.25%, 600°C). The modelling accounts for the heterogeneous distribution of the forest of dislocations.

A long term relaxation was simulated (0.35%, $10^{-3}/\text{s}$, one month, 600°C). The two stage relaxation is once more shown. Interestingly, even after one month, the relaxation is still active and the stress level is not saturated as it seems to be on the experimental point of view (Figs. 5, 6, 78 a). The stress level reached after one month is 90MPa, which is in reasonable agreement with the experimental values, (70-80MPa).



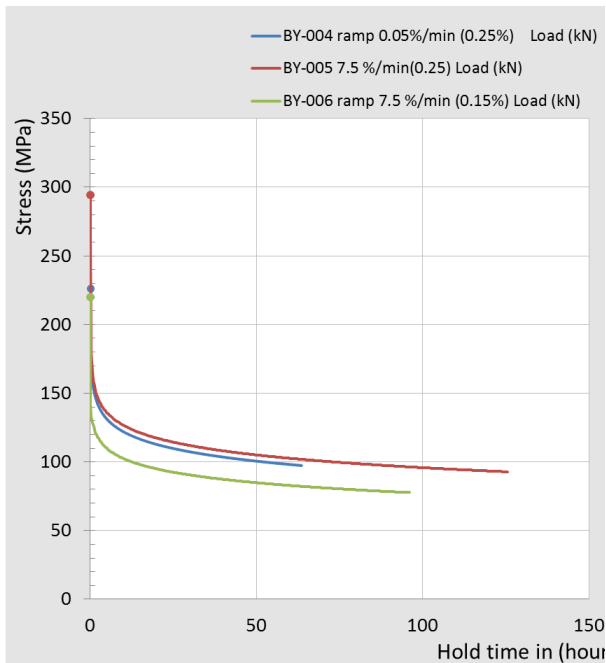
a)



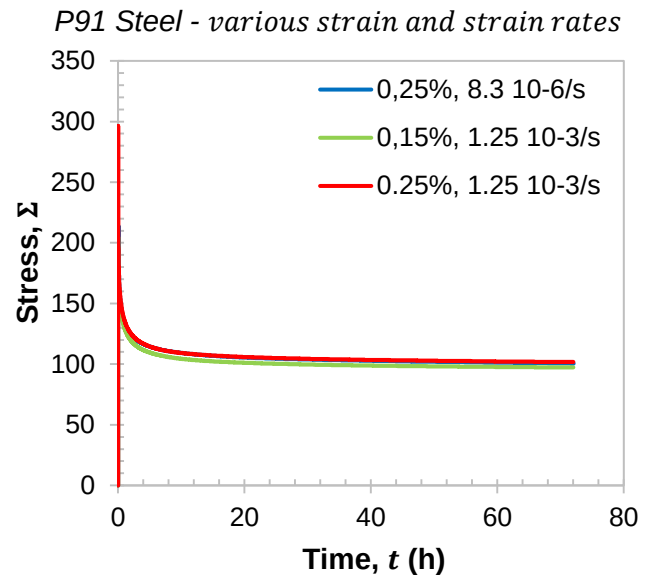
b)

Fig. 79. Long term relaxation curves: stress level (MPa) with respect to a) time t and b) $\log(t)$ (hours).

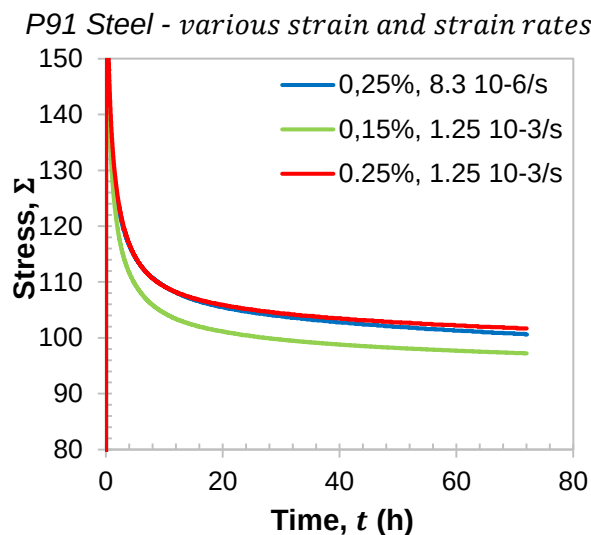
Finally, peak strain and loading ramp strain rate can be investigated both experimentally and theoretically. The experimental curves provided by JRC are plotted in Fig. 80. They are already described in subsection 2.5. Fig a shows that if the peak stress is chosen to be the same, then relaxation is higher if the loading strain rate was higher. That was explained by higher viscous stress because of higher strain rate but lower internal stresses due to lower plastic strain. Interestingly, the polycrystal model accounts for both viscous stresses and internal stresses at the F/M block scale. And on a quantitative point of view, the loading strain rate effect is predicted (Fig. b and c). Nevertheless, the measured drift after 50h is ~ 15 MPa compared to the predicted one, 5 MPa. Both simulations and experiments provided relaxation curves very close to each other, at the highest strain rate irrespective of the strain rate. Only the short term relaxation is affected by the difference in strain rate.



a)



b)



c)

Fig. 80. Influence of peak strain and strain rate on the evolution of the stress level with time: a) experimental data; b) predicted curves and c) zoom applied on the predicted curves.

Three different loading parameters affect relaxation: peak strain, loading strain rate and temperature.

- For the same peak stress but different strain rates, the lower the strain rate, the lower the relaxation (Fig. 80 a). This may be explained in terms of balance between internal stresses and viscous stresses. Modelling predicts such effect even if it underestimates it (Fig. 80 c);
- Changing the strain rate but not the peak stress affects only short term relaxation (<1h) but not long-term relaxation (Fig. a). This may be explained by a quick relaxation of the additional viscous stress and predicted by modeling (Fig. 80 b and c)
- The shapes of the evolution curves of the stress level with time are rather similar whatever the temperature (Fig. 78 a). Of course a drift is observed, which is temperature dependent. Such trends are at least qualitatively predicted.

The modelling accounting for the heterogeneity in the obstacle distributions based on dislocation dynamics computations seems to be able to predict the very large relaxation observed at both 550 and 600°C, when final stress level approaches 80MPa after LT relaxation (three days at 600°C and one month at 550°C).

Introducing the effect of the heterogeneity of the random spatial distribution requires somewhat more accurate description of the glide length dependence with normalized stress. The project provides many additional data showing the effect of softening on relaxation (fatigue-relaxation tests, Figs. 24 and 25). Sequential test data are provided too (Fig. 10). The further comparison between predictions and experimental data will allow us to check the validity domain of the modelling.

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4 Synthesis (Maxime Sauzay)

4.1 What do we learn from the experiments?

A large number of parameters are involved in the study of the cyclic behaviour of F/M steels such as strain amplitude, strain rate, relaxation time and temperature [1-4]. As mentioned in the introduction, they all affect softening. Both plasticity and thermal recovery mechanisms are involved in softening during cycling. In order to rationalize such results the evolution of stress should not only be plotted with respect to the number of cycles, N , but more importantly as function of the cumulative plastic strain, $p(N)$ [5-7]. The cumulative plastic strain allows us to better correlate the measured softening to different parameters (strain amplitude, relaxation time, strain rate). The strain amplitude effect for instance appears smaller if the stress is plotted against the cumulative plastic strain, $p(N)$, than when plotted with respect to the number of cycles, N (Figs. 81 a and b). Similarly, the hold time effect is smaller if we consider the stress evolution with respect to $p(N)$ rather than with N (Figs. 82 a and b). Finally, Giroux showed that the strain rate effect is smaller when referring to $p(N)$ compared to N [6].

It is not surprising that a master curve is better represented using the cumulative plastic strain instead of the number of cycles way because the number of active slip systems is dependent on the plastic amplitude and it very probably affects the probability of annihilation between free dislocations and LAB dislocations (see 3.3). Finally, recovery certainly plays a role in dislocation annihilations, for instance due to climb. That may explain that the hold time influence cannot be fully rationalized by the use of the cumulative plastic strain.

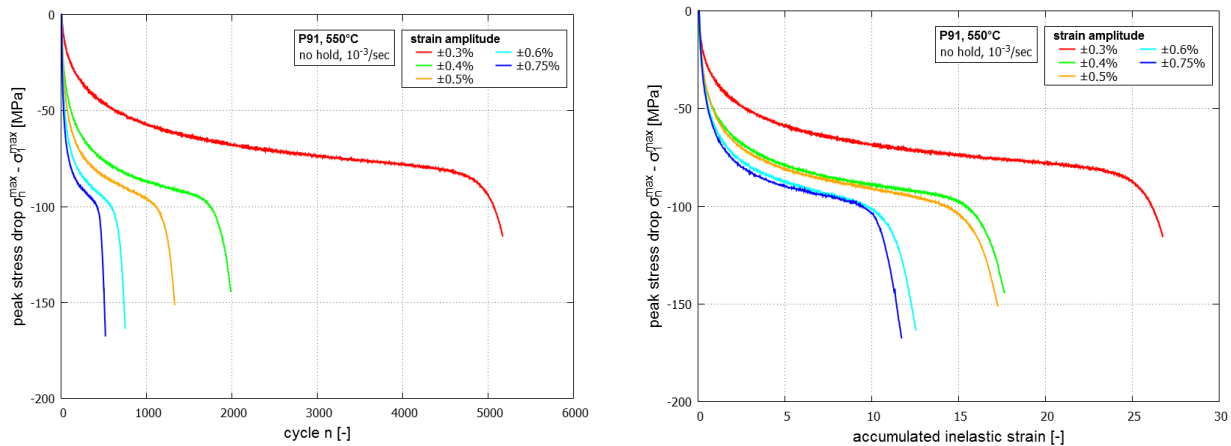


Fig. 81. Softening curves measured for different amplitudes and plotted with respect to a) the number of cycles and b) the cumulative viscoplastic strain, $p(N)$. 823K. Experimental data and plots: KIT.

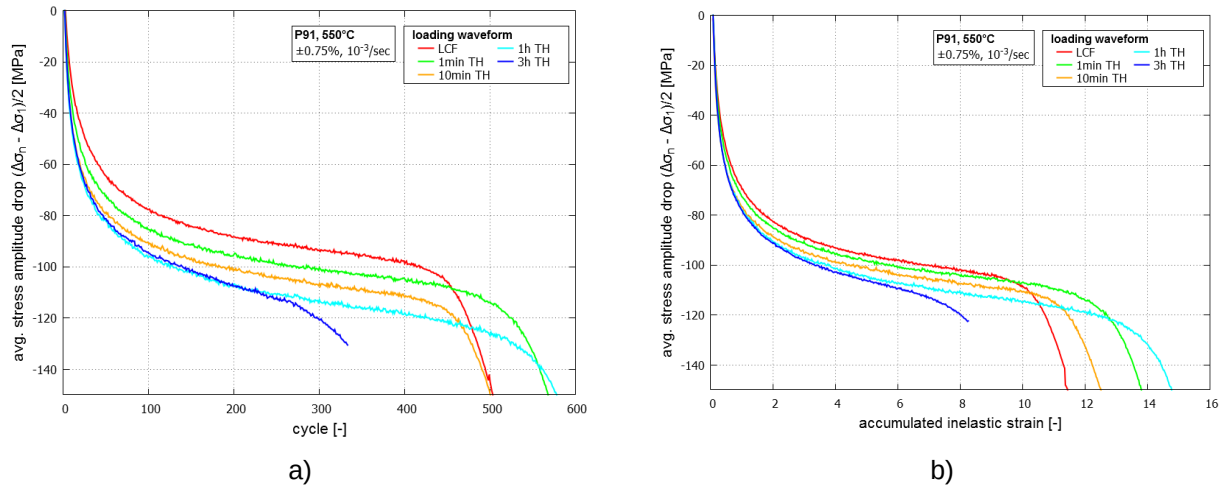


Fig. 82. Softening curves measured for different hold time but the same strain amplitude, and plotted with respect to a) the number of cycles and b) the cumulative viscoplastic strain, p(N). 823K. Experimental data and plots: KIT.

Many experiments carried out in the framework of the MATISSE project show clearly that a significant relaxation occurs; the stress is reduced by a factor of about 4-5 after a 3 days relaxation at 600°C and 1 month at 550°C. This leads to a large additional plastic strain corresponding to the relaxed elastic strain. In case of large strain amplitudes, the additional plastic strain per cycle is much lower than the plastic strain amplitude, which may allow a saturation of the hold time effect on softening for a rather short hold time (about 1h for a total strain amplitude of 0.75%, Figs. 82 a and b). Moreover the lower the strain amplitude, the higher the effect of the additional plastic strain provided by relaxation and the saturation of the hold time effect occurs for higher relaxation time.

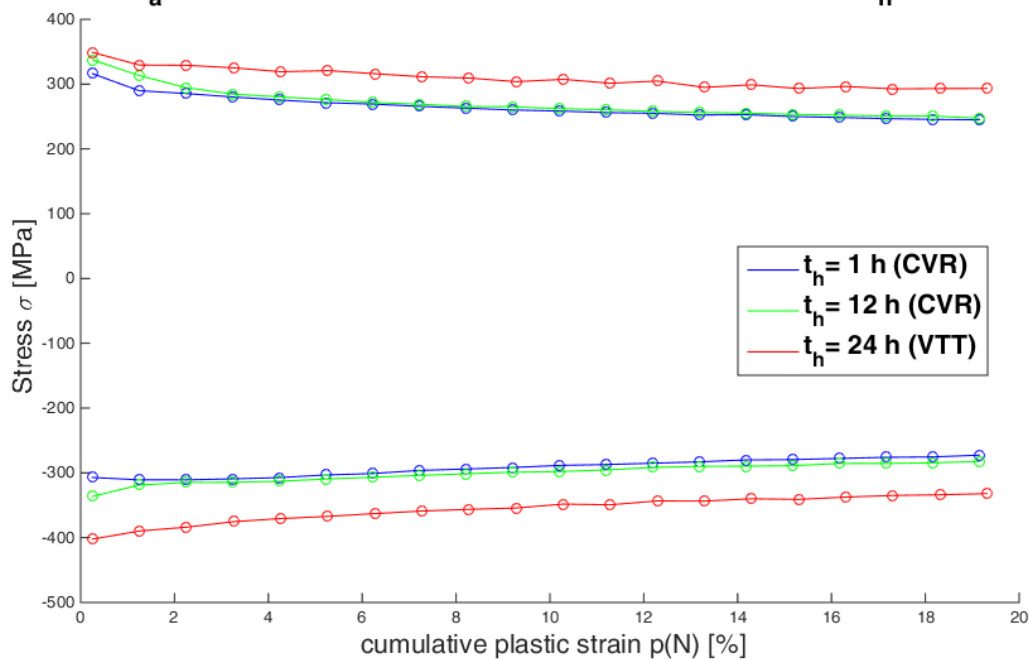
The same trend is observed for the mean stress magnitude induced by relaxation hold time and the subsequent asymmetry of the hysteresis loop.

Therefore, the small amplitude regime corresponds not only to the in-service conditions but also to the strain domain for which the hold time effects are most important. As in-service hold times range from a few days to one month, and a careful examination of the effect of long hold time in case of small amplitude is of high interest.

Plots provided by CVR illustrating the hold time effect on softening are shown in Figs 83 a-c for strain amplitudes 0.45%, 0.35% and 0.25% respectively. If we consider the CVR test results only, it seems that at strain amplitudes of 0.45% and 0.35%, the hold time effect is almost stabilized after 1h. As a hold time of 1h is required at very high strain amplitude, it is expected that one hour is required at intermediate strain amplitude. These results agree with the ones shown in Fig. 3 (saturation after a 30 min hold time at a strain amplitude of 0.35% and 550°C). Nevertheless, at the smallest amplitude, 0.25%, the stresses obtained with hold times of 1 and 12h differ, showing that saturation with respect to hold time has not been reached yet. The stress magnitudes measured by VTT are generally higher than the ones measured at CVR without a clear explanation yet.

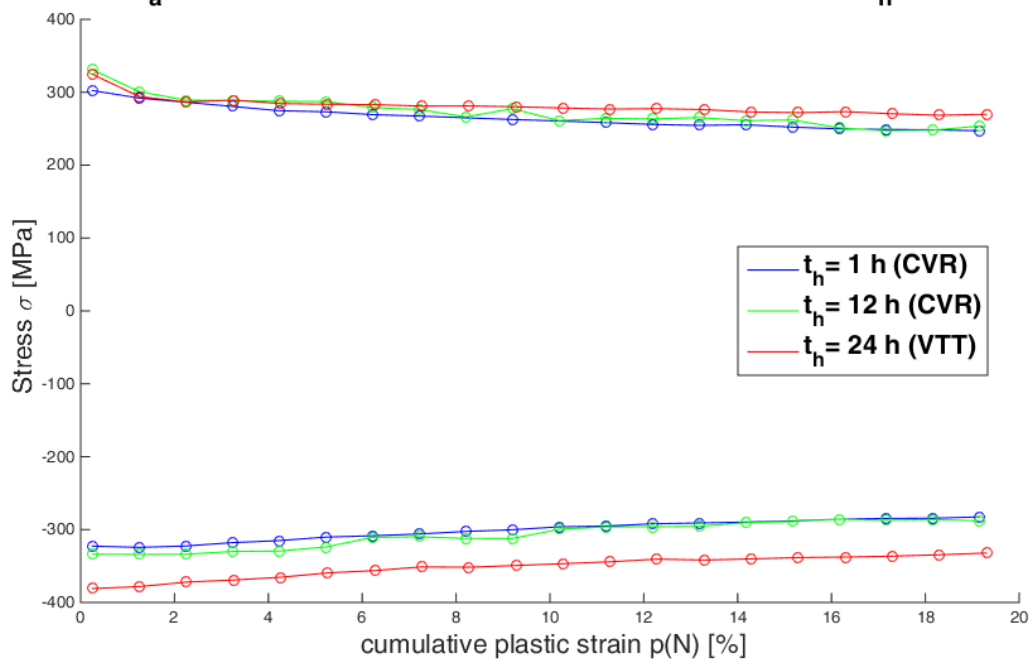
Figs. 83 a-c also show that for a given hold time, the lower the strain amplitude, the higher the magnitude of the mean stress. If we consider the difference in magnitude between the positive and negative peak stress after 20 cycles including 24h hold times, it can be deduced that the lower the strain amplitude, the higher the difference between the two magnitudes, the stronger the cycle asymmetry and the lower the compressive mean stress. In case of hold time imposed at the tensile peak stress, a compressive mean stress is measured (Fig. 83 a-c). This may increase lifetime. Nevertheless, if the hold time is applied at the compressive peak stress, then a tensile mean stress is measured which may decrease lifetime. For a strain amplitude of 0.25% and a hold time of 24h, the mean stress amounts to about -50MPa after 20 cycles, which is far from being negligible.

Steel P91, $\epsilon_a = 0.45\%$, 600°C , cumulative plastic strain $p(N)$ for $t_h = 1, 12$ and 24 h

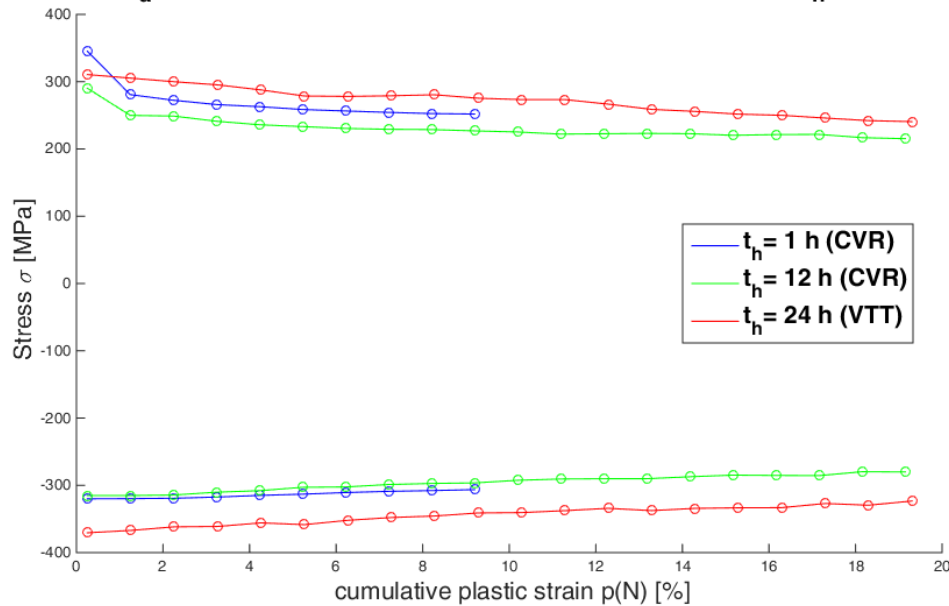


a)

Steel P91, $\epsilon_a = 0.35\%$, 600°C , cumulative plastic strain $p(N)$ for $t_h = 1, 12$ and 24 h



b)

Steel P91, $\epsilon_a = 0.25\%$, 600°C , cumulative plastic strain $p(N)$ for $t_h = 1, 12$ and 24 h

c)

Fig. 83. Evolution of the maximum / minimum stresses for three different hold times: 1h, 12h and 24h. Decreasing strain amplitude: a) 0.45%; b) 0.35% and c) 0.25%. Temperature: 600°C . Thick grade 91 plate (60mm). All tests: twenty first cycles.

Sequential tests carried out in the framework of the project clearly show that the effect of even long hold times vanishes rapidly during further cycling without hold time (Figs. 8 and 10). After a few ten cycles, the softening curves without hold time is recovered and the mean stress vanishes. Only frequent long hold times would therefore affect the softening behaviour.

4.2 How may modelling help to predict the cyclic behaviour in in-service conditions?

The test data and the models presented demonstrate the complexity and challenges related to the accurate prediction of relaxation behavior. However with the relaxation and softening models in place it will now be possible to study the impact of strain range and hold time on different CF damage concepts such as the time life fraction and the ductility exhaustion as well as simplified models.

Since the cyclic material characteristics, such as cyclic softening in this case, clearly have an effect on the relaxation / creep rate during strain holds, it is also clear that the virgin material or the mid-life cycle alone are not necessarily sufficient representatives of the cyclic behavior of the material.

For components in-service the material softening in the form of decreased hardness could be a good indicator for detecting creep or creep-fatigue damage.

The robust prediction of relaxation under conditions with wide range of temperature, strain and very long hold periods during the whole 60 years lifetime of a component may be challenging, even for the most advanced methods.

Three different modelling approaches have been proposed.

- First, the engineering approach based on well-chosen analytical formulae and parameter adjustment by inverse identification provides predictions in reasonable agreement with numerous experimental data (subsection 2.1).
- The second approach, based on thermodynamics and macroscopic hardening, flow and damage constitutive laws, is able to provide predictions in good agreement with fatigue and fatigue-relaxation softening data (section 2.2). Nevertheless, its validation out of its identification database is required concerning typical in-service conditions (low strain amplitude and hold times of several weeks).

- The third approach is based on polycrystal homogenization and crystal viscoplasticity constitutive laws, accounting for annihilation of dislocations and low-angle boundaries. Various strain rate jump tests carried out at 550 and 600°C permit the inverse identification of the lonely two adjusted parameters, namely the crystal viscoplasticity activation energy and volume at the F/M block scale. The activation energy is close to the bulk self-diffusion coefficient. The high temperature large strain tensile behavior is then well described. And the cyclic softening is rather well reproduced and long term relaxation behavior. Strain amplitude, Strain rate, temperature effects are qualitatively reproduced.

Based on the numerous original experimental data, the predictions of these three models need to be compared with cyclic data obtained for various strain amplitude and temperature in the MATTER and MATISSE projects, and published in literature (Fig. 84 a). Sequential test results allow a further validation of the predictions. Finally, long term relaxation curves measured in various loading conditions as well as fatigue-relaxation test results obtained for long hold times should be used in the validation process. If a model can reproduce the different experimental observations then it could be considered as robust and having a very large application domain.

4.3 Recommended further work

As mentioned previously, long term cyclic tests carried out at amplitudes as low as 0.15-0.2% are necessary to validate the feasibility of models to address in- service observations; the hold time effects (saturation only at long hold time, higher mean stress) are most visible at low amplitude and in-service conditions correspond to low strain. The influence of the total applied strain amplitude has been studied in details. In a previous experimental work carried out at CEA, a cyclic test at a very small amplitude of $\pm 0.15\%$ was carried out up to 3.3 million cycles (Fig. 84 a) [6,8]. Only cracks of a few tens of microns were observed on the surface by SEM (Scanning Electron Microscopy). The stress reduction was -55MPa while the stress amplitude during the first cycles was below the conventional yield stress. The stress drops by 22% after 3.3 million cycles. Therefore cyclic softening is very significant even under very low viscoplastic strain amplitudes ($<10^{-4}$). No saturation is observed, which agrees with the results of tests under high vacuum or under sodium which show that softening continues beyond the lifetime in air that is considerably shorter than in more inert environment, particularly in the high-cycle regime [1,6]. As in Astrid, N and Na environment will be used, a prolonged softening is expected.

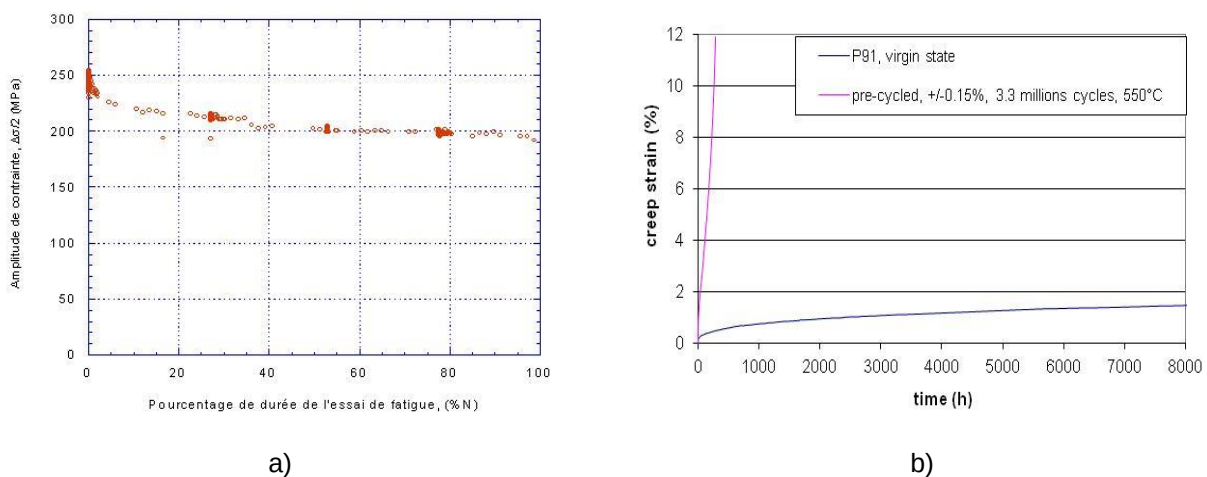


Figure 84. a) Stress variation during cyclic load at very low total strain (grade 91, $\pm 0.15\%$, 550°C, 3.3 million cycles) [5]. b) comparison between measured time – creep strain curves either obtained with the virgin specimen or the pre-cycled specimen. Steel grade 91, 550°C, nominal creep stress: 185 MPa, or the final level reached during the preliminary fatigue test [8].

The consequences of such low strain with prolonged softening may be dramatic as shown by the creep tests carried out on a virgin specimen and the pre-cycled specimen. Fig. 84 b shows that the specimen previously subjected to very low strain cycling has a minimum creep rate that could be 100 times larger than for the virgin specimen. Such high deformation rate can be qualitatively explained by the difference in microstructures between the virgin and the precycled specimens (Fig. 85 a). Generally, creep-fatigue tests showed that the material subjected to softening deforms much quicker than the virgin material. At mid-lifetime, a factor of 100 is found (Fig. 85 b).

Modelling should be able to predict such very strong trend to show that it captures rather well the involved mechanisms.

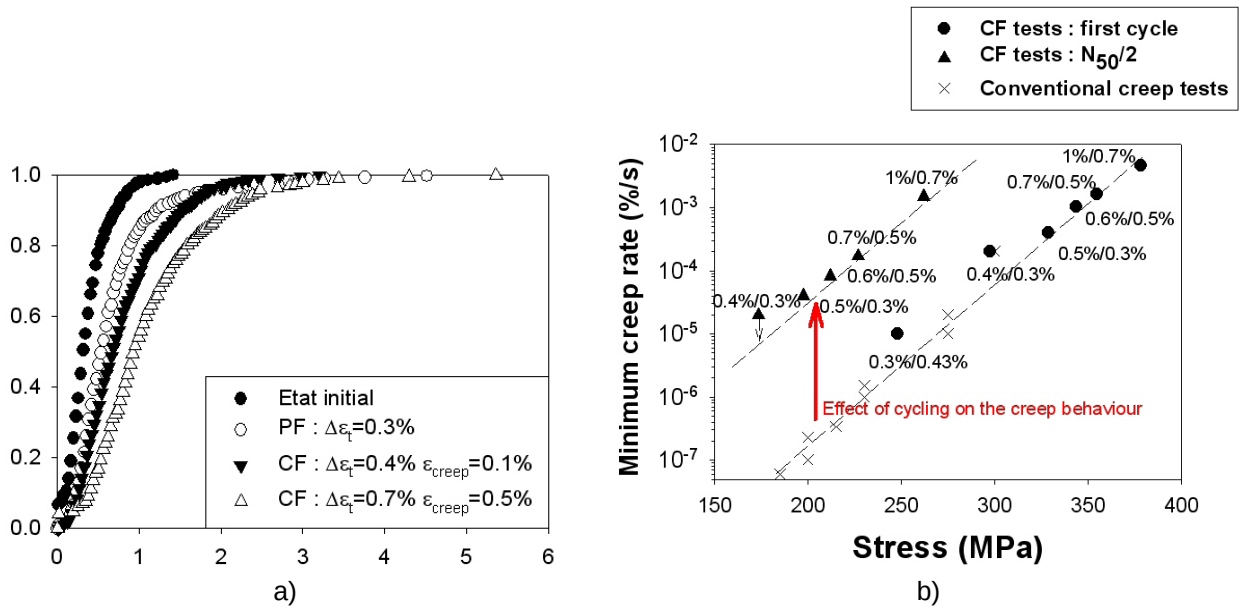


Figure 85- a) cumulative probability of the subgrain size distribution in the initial state and after cyclic testing in various condition [9]. Even at a strain amplitude of 0.15%, the evolution of the subgrain size distribution is clearly observed explained the macroscopic cyclic softening; b) minimum creep strain rate measured either during the 1st cycle or at half-life of fatigue-creep tests with holding time in tension. The applied total strain amplitude and pure creep strain are given for each experimental point. For the sequential test with pre-cycling, a factor 100 is measured too (Fig. 84 b). Grade 91, 550°C.

Finally, as mentioned in sub-section 3.3, a transition between high stress and low stress creep regimes is highlighted by experimental tests (see [6] for a review). For instance, at 600°C, the transition occurs at a stress of about 80MPa and a creep strain rate of a few 10^{-11} /s. Two consequences are expected. If we refer to long-term relaxation (3 days at 600°C and one month at 550°C), the final stress level is typically as low as 70-80MPa. That means that the low stress strain rate regime is reached. Then, strain rates may be much higher than predicted from the extrapolation of the Norton law line for which parameters are usually adjusted in the high strain regime. For instance, at 60MPa, the measured strain rate is ten times lower than the extrapolated value (Fig. 87). Moreover, the intergranular damage depends on cavity nucleation, which is directly related to strain rate in agreement with the well-known Dyson law. Then the usual high strain rates shown in Fig. 87 trigger intergranular damage and reduce long term creep lifetimes as shown in austenitic stainless steels [10]. Experimental creep tests carried out at lower stresses and lower temperatures are needed to fully characterized the low stress creep behaviour and providing data for a safe design.

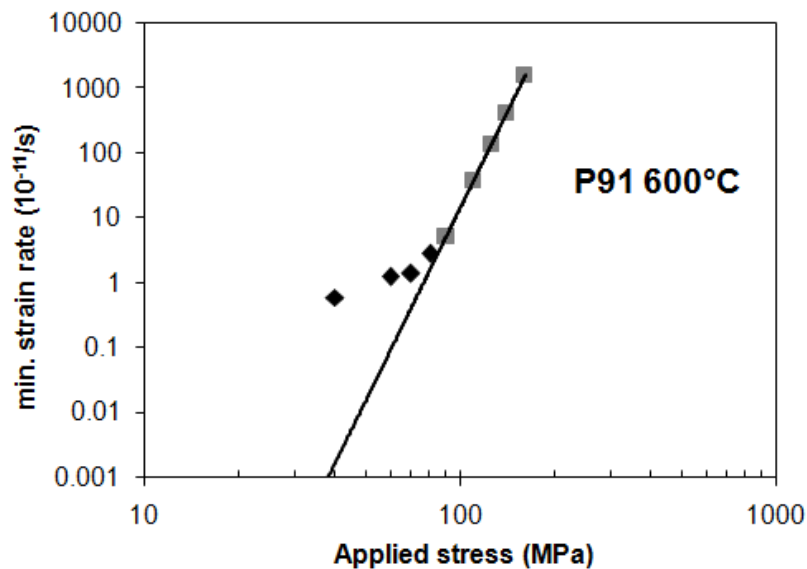


Figure 87: strain rate regime at high stress (≥ 90 MPa) and low stress (< 90 MPa). Steel grade 91, temperature: $T=600^\circ\text{C}$ [6]. Extrapolation of the Norton straight line below the high stress regime used to identify its parameters. The time to failure at a stress of 90 MPa is about 94000 h.

Considering very long hold-times in case of low strain amplitude fatigue-relaxation tests is required too, to study the saturation of hold time effects.

The measurement of microstructural variations in terms of growth in subgrain size and reduction in dislocation density is required to validate the predictions of physically-based modelling at the microscopic scale. Only the validation of the predictions of such modelling at both microscopic and macroscopic scales are required to provide some confidence in the predictions, particularly for low strain and long term relaxation. The predicted kinetics of the subgrain size and dislocation density evolution are in qualitative agreement with experimental measurements on interrupted tests on both steel grade 92 [5] and Eurofer 97 [11]. A very fast drop in dislocation density as a function of the number of cycles is observed [5,11], followed by a slower reduction but that does not saturate. The subgrain size also increases quickly at the beginning of the cyclic loading [5-7,11], but at a slower rate than the dislocation density. A slow but continuous growth occurs after this fast growth. Similar observations of microstructure evolution kinetics have been published concerning creep of tempered martensitic steels (see [6] for a review). The same mechanisms are observed in creep, however with an increased role of diffusion of vacancies and the increase in dislocations (acceleration of annihilation mechanisms) and precipitation (modification to sub-boundary pinning conditions, modification of the effects of solid solution, etc.). It should be noticed that softening and microstructure recovery induced by pure creep has been largely reported in literature [6,12-13] with probably many similarities which what happens during cycling and cycling with hold time.

Proposing new mechanisms such as the heterogeneous plastic slip of dislocations (see 3.3) or introducing a low stress regime of viscoplasticity below the final stresses observed after the longest relaxation times used in the project may allow the upscaling of more phenomenological models. It seems that rather similar mechanisms may be introduced in these models. Nevertheless, the direct relationship with DDD simulations is lost with comparison to physically-based viscoplastic modelling. A very few number of relevant parameters should be chosen and fitted either using experimental data (Fig. 87) or even the predictions of physically-based models.

Interestingly, many concepts discussed for Grade 91 steels are valid for all F/M steels (Fig. 1) [11,14]. They even apply to materials with some initial microstructure features similar to the martensitic steel ones considered here, such as bainitic steels and ultra-fine grain (UFG) materials [15], and showing experimentally the same cycle-by-cycle softening

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5 Conclusions

It has been known for a long time that cyclic loading applied to F/M steels at high temperature leads to a large cycle-by-cycle softening due to combined plasticity and thermal recovering mechanisms. This sub-project aims to provide original experimental data investigating the influence of long term hold times which are expected during in-service conditions. Additionally, macroscopic and physically-based modellings have been elaborated in order to predict both softening and lifetime. Finally, a strong interaction between experiments and modelling has been developed.

First of all, the cyclic test results confirm the effects of strain amplitude and hold time. This can be partly rationalized by using the cumulative viscoplastic strain instead of the number of cycle when considering the evolution of the peak stress. This partial rationalizing can be explained by the effect of viscoplastic slip on the recovering mechanisms such as dislocation- dislocation and low-angle boundary dislocation annihilations. Time effects should play a role too in accordance with the observed effect of temperature. Very large relaxations are observed and the stress level is divided by a factor three to four after one month which corresponds to the expected in-service hold times. A considerable amount of viscoplastic strain is then added to the pure cyclic one, which has strong consequences at low cyclic amplitude. Even after one month, relaxations are not terminated and the stress levels are not saturated. The higher the strain rate, the quicker the relaxation because of the rapid relaxation of strain rate dependent viscous stress. Increasing temperature leads to quicker relaxation too. These effects can be qualitatively rationalized by considering the partitioning of the stress into internal stress and viscous stress. The effect of hold time on softening is found to be saturated for a duration of one hour at high and medium strain amplitudes. Nevertheless, at the much lower amplitude applied during the project, the saturation of the hold time effect requires a much longer duration. Sequential tests including cycles with long hold times and pure fatigue cycles show that the effect of one long term relaxation vanishes quite quickly, that is after only a few ten pure fatigue cycles. Finally, negative mean stresses are measured due to tension hold times inducing an asymmetry of the cycles with a tension viscoplastic strain amplitude higher than the compression one. The inverse occurs with compression hold times which may lead to decrease in lifetime. The mean stresses are rather low at high strain amplitude but much higher at low amplitude due to the ratio between the pure fatigue and relaxation-induced viscoplastic strains. Once more, low strain amplitudes are characteristic of in-service conditions.

Three different modelling approaches have been used. First, the engineering approach based on well-chosen analytical formulae and parameter adjustment by inverse identification provides predictions in reasonable agreement with numerous experimental data. The second approach, based on thermodynamics and macroscopic hardening, flow and damage constitutive laws, is able to provide predictions in good agreement with fatigue and fatigue-relaxation softening data. Nevertheless, its validation out of its identification data base is required concerning typical in-service conditions (low strain amplitude and hold times of several weeks). The third approach is based on polycrystal homogenization and crystal viscoplasticity constitutive laws, accounting for annihilation of dislocations and low-angle boundaries. Various strain rate jump tests carried out at 550 and 600°C permit the inverse identification of the lonely two adjusted parameters, namely the crystal viscoplasticity activation energy and volume at the F/M block scale. The activation energy is close to the bulk self-diffusion coefficient. The high temperature large strain tensile behavior is then well described, and strain amplitude, strain rate and temperature on cyclic softening are qualitatively well reproduced considering the whole range of strain amplitudes and temperatures considered in the MATTER and MATISSE projects.

The prediction of the softening at very low strain amplitudes and for very long hold times requires the introduction of dedicated terms such as long term recovering by dislocation climb and the heterogeneity in the dislocation blockage induced by the randomness of the obstacle microstructures (MX particles, solid solution clusters, debris). Many original experimental data obtained in conditions close to the in-service ones have been provided by the project and in the next future, the predictions from the different modelling should be compared to the whole set of experimental results. In the future, TEM observations are of interest too for understanding the long term evolutions and validating the microscopic predictions of the physically-based modelling. Finally further research works are highlighted, for instance very low strain amplitude fatigue, fatigue-relaxation at low strain amplitude and long hold time and finally low stress creep strain rate regime.

6 Annexes

KIT

Experiments

A list of MatDB references of all tests. TBD.

Modelling

The uniaxial coupled deformation and damage model presented in section 3 can be generalized to the following multiaxial formulation:

$$\begin{aligned}
 \dot{\boldsymbol{\varepsilon}} &= \dot{\boldsymbol{\varepsilon}}_{el} + \dot{\boldsymbol{\varepsilon}}_{in} \\
 \boldsymbol{\varepsilon}_{el} &= ((1-D)\mathbf{E})^{-1} : \boldsymbol{\sigma} \\
 \dot{\boldsymbol{\varepsilon}}_{in} &= \frac{3}{2} \left(\frac{\Sigma_{eq} - k}{Z} \right)^n \frac{\boldsymbol{\Sigma}}{\Sigma_{eq}} \text{ with } \boldsymbol{\Sigma} = \frac{\mathbf{s}}{\Psi(1-D)} - \boldsymbol{\Omega} \text{ and } \Sigma_{eq} = \sqrt{\frac{3}{2} \boldsymbol{\Sigma} : \boldsymbol{\Sigma}} \\
 \boldsymbol{\Omega} &= \boldsymbol{\Omega}_1 + \boldsymbol{\Omega}_2 \\
 \dot{\boldsymbol{\Omega}}_1 &= \frac{2}{3} H_1 \dot{\boldsymbol{\varepsilon}}_{in} - C_1 \boldsymbol{\Omega}_1 \dot{p} - R_1 \Omega_{1,eq}^{m_1-1} \boldsymbol{\Omega}_1 \\
 \dot{\boldsymbol{\Omega}}_2 &= \frac{2}{3} H_2 \dot{\boldsymbol{\varepsilon}}_{in} - R_2 \Omega_{2,eq}^{m_2-1} \boldsymbol{\Omega}_2 \\
 \text{with } \dot{p} &= \sqrt{\frac{2}{3} \dot{\boldsymbol{\varepsilon}}_{in} : \dot{\boldsymbol{\varepsilon}}_{in}} \text{ and } \Omega_{i,eq} = \sqrt{\frac{3}{2} \boldsymbol{\Omega}_i : \boldsymbol{\Omega}_i} \\
 \Psi &= \Psi_1 + \Psi_2 + \Psi_3 \\
 \text{with } \Psi_1(t=0) &= 0, \Psi_2(t=0) = 1 \text{ and } \Psi_3(t=0) = 0 \\
 \dot{\Psi}_1 &= -h_1 \dot{p} \\
 \dot{\Psi}_2 &= c(\Psi_S - \Psi_2) \dot{p} \\
 \dot{\Psi}_3 &= -r_\Psi |\Psi_3 - \Psi_r|^{m_\Psi-1} (\Psi_3 - \Psi_r) + h_3 \dot{p} \\
 \text{with } \Psi_S &= 1 - \Psi_{S,\infty} (1 - \exp(-c_S \Delta \varepsilon_{in,eq})) \\
 \text{and } \Delta \varepsilon_{in,eq} &= \max_{-\infty < \tau < t} \max_{-\infty < \tau_0 < t} \sqrt{\frac{2}{3} (\boldsymbol{\varepsilon}_{in}(\tau) - \boldsymbol{\varepsilon}_{in}(\tau_0)) : (\boldsymbol{\varepsilon}_{in}(\tau) - \boldsymbol{\varepsilon}_{in}(\tau_0))} \\
 \dot{D} &= \left(\frac{X(\boldsymbol{\sigma})}{A} \right)^r \dot{p} (1-D)^{-\kappa} \\
 \text{with } X(\boldsymbol{\sigma}) &= \alpha_1 \sigma_I + \alpha_2 \text{Trace}(\boldsymbol{\sigma}) + (1 - \alpha_1 - \alpha_2) \sigma_{eq} \\
 \text{with } \sigma_{eq} &= \sqrt{\frac{3}{2} \mathbf{s} : \mathbf{s}}
 \end{aligned}$$

$\mathbf{s}$ is the deviatoric part of the stress tensor, $\mathbf{s} = \boldsymbol{\sigma} - \frac{1}{3} \text{Trace}(\boldsymbol{\sigma}) \mathbf{1}$, and σ_I the maximum principal stress. α_1 and α_2 are additional temperature-dependent material parameters that have to be determined using lifetime data from multiaxial creep experiments.