SOFTENING AND RELAXATION OF GR.91 STEEL IN CREEP-FATIGUE CONDITIONS

Jaromír JANOUŠEK [1, 2], Maxime SAUZAY [3], Rami POHJA [4], Stefan HOLMSTRÖM [5]

[1]CVR - Research Centre Řež, Plzeň, Czech Republic, EU, jas@cvrez.cz

[2]ZČU - University of West Bohemia, Faculty of Mechanical Engineering, Department of Material, Pilsen, Czech Republic, EU

[3]CEA - The French Alternative Energies and Atomic Energy Commission, Saclay, France, EU

[4]VTT - Technical Research Centre of Finland Ltd, Espoo, Finland, EU

[5]JRC - Joint Research Centre, Petten, Netherlands, EU

Abstract

The data presented here were obtained as part of the European FP7 MatISSE project to model the softening and relaxation behaviour of Grade 91 steel under creep-fatigue loading because this type of thermal loading is a major degradation mechanism in fast reactors. The supporting tests were focused on strain controlled low cycle fatigue tests with different strain amplitudes (ε_a = 0.45 %, 0.35 % and 0.25 %) and different tension hold times (1 and 12 hours) for temperature 600°C. The results were compared with results for 24 hour hold time from another testing laboratory (VTT). The results were evaluated with respect to the number of cycles, *N*, and also as a function of the cumulative plastic strain, *p(N)*.

Keywords: Creep-Fatigue, softening, relaxation, P91

1. INTRODUCTION

The research within the European FP7 MatISSE project (2014-2017) dealt with the basic understanding and prediction of cyclic softening in tempered martensitic steels such as P91, which is a frequent choice for high temperature/pressure piping and heat exchangers in steam-based power plants [1]. This material together with steel 316L and 316L(N) are also the main candidates for the construction of the planned Myrrha, Astrid, Alfred and Allegro research reactors. The experiments are immensely important for validation of the models especially for the effect of the hold time on cyclic softening and hence on damage behaviour, because thermal creep-fatigue is a major degradation mechanism in fast reactors. Cyclic softening is typically observed in all tempered martensite-ferritic steels [2], bainitic steels [3] and ultrafine-grained (UFG) materials [4]. The problem corresponds with strong creep acceleration after the minimum creep strain rate and can occur without any obvious intergranular damage or necking.

An existing elasto-viscoplasticity model simulates the key mechanisms such as decrease of dislocation density, increase in sub-grain size and recovery phenomena. The model is modified to also incorporate the effect of hold time on the cyclic softening and the supporting experimental programme produces data for calibration and validations of the developed model. The major part of the results from MatISSE project concerning softening have already been presented in [5].

2. EXPERIMENTAL PROGRAMME

A series of tests related to cyclic softening problems in tempered martensitic steels were carried out at the Research Centre Rez (CVR) in Pilsen (CZE) in the Material Testing Laboratories. The supporting tests were focused on strain controlled low cycle fatigue tests with different tension hold times. Long hold times were targeted in particular. **Figure 1** shows how the loading pattern is controlled by strain, a graph of stress response and a typical hysteresis loop where the tension hold time has an impact on the shape as a line. The development of time-dependent inelastic strains (relaxed strain) during these tests was related to softening and the corresponding creep behaviour (strain rates) and creep-fatigue life was directly affected.

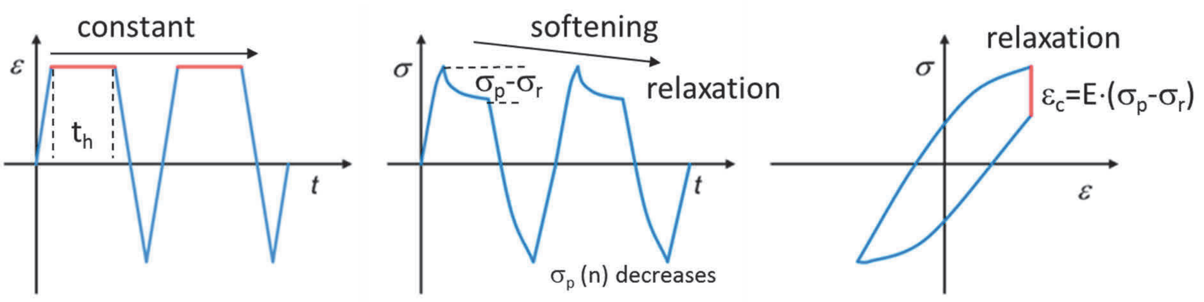

Figure 1 Loading pattern controlled by strain (left), response by stress (middle) and typical hysteresis loop for tension hold time (right)

The cutting plan of the P91 test material block (150 x 150 x 60 mm) is shown in **Figure 2** together with the shape and dimensions of the creep-fatigue specimen. The arrow on the block indicates the rolling direction. The chemical composition is given in **Table 1**. Chemical composition was also analysed by glow discharge spectroscopy (see **Table 2**). Tensile properties were: yield strength at 20 °C ≥ 445 MPa, ultimate tensile strength at 20 °C = [580 - 760] MPa and elongation at 20 °C ≥ 20 %. Thermal state corresponded to 1060 °C - 4Hrs + Water Quenching + 760 °C - 3Hrs20min - Air Cooling.

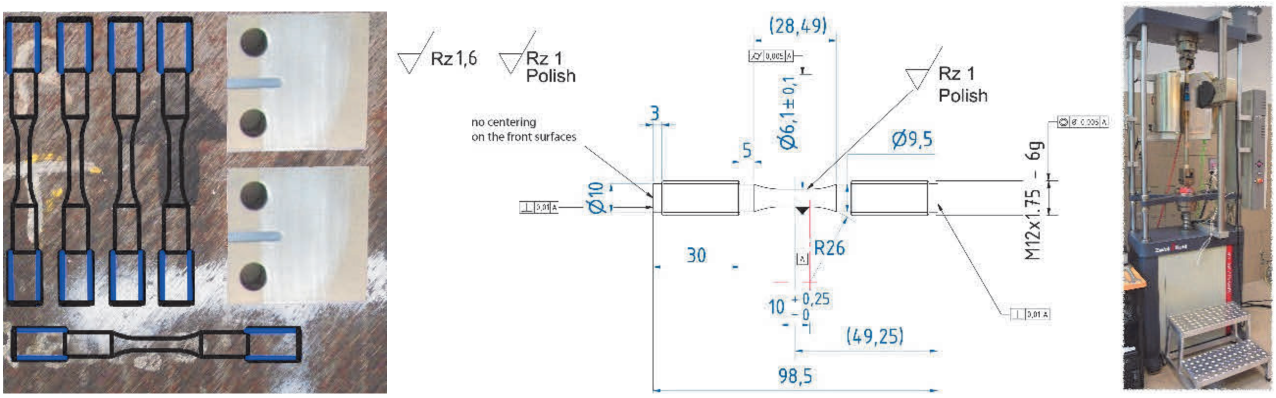

Figure 2 Cutting plan of block P91 (left), the shape and dimensions of specimen (middle), and Kappa SS-CF testing machine (right)

Table 1 Chemical composition of the test material

Elements	C	Mn	Si	Ni	Cr	Mo	Cu	Al	S	P
[wt. %]	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
[wt. %]	0.235	0.084	0.041	0.005	0.001	0.002	0.0009	0.001	0.001	15 ppm

Table 2 Chemical composition analyzed by glow discharge spectroscopy

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

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 **Figure 2**). Load capacity was up to 50 kN and speed range was from 1 μm/h to 100 mm/min. The machine was equipped with a 3-zone furnace up to 1200 °C and a non-contact high precision video extensometer with 50 mm field of view and corresponding resolution 0.25 μm. It is necessary when using this type of non-contact video extensometer for high temperature to create two line marks by airbrush. The aluminium oxide powder had to be suspended in ethanol in a mixing ratio 1 : 4 (Al_2O_3 : ethanol). The type of cycle according to asymmetry was symmetrically reversed i.e. strain ratio R = -1. The cycle shape had a triangular course and the temperature for all tests was 600 °C. The strain rate in cycling was 6 % per minute. The intended test matrix corresponds to 6 creep fatigue tests with a tensile hold time in every cycle. The first 3 tests with 1 hour hold time were performed for strain range $\Delta\varepsilon$ = 0.9 %, 0.7 % and 0.5 % (i.e. ε_a = 0.45 %, 0.35 % and 0.25 %) and each test was designed to be stopped after 1000 cycles or after failure location. Other tests with a 12 hour hold time for the same strain ranges were planned for stopping after 50 cycles.

3. EVALUATION OF RESULTS

The first experiment, for strain range $\Delta\varepsilon$ = 0.5 % (i.e. ε_a = 0.25 %) with 1 hour tensile hold time, was interrupted in the 73rd cycle due to bad tuning (P I values), and the testing system became unstable. The second experiment was for strain range $\Delta\varepsilon$ = 0.9 % (i.e. ε_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 third experiment was for strain range Δ_{ε} = 0.7 % (i.e. ε_a = 0.35 %) with 1 hour tensile hold time, and it was interrupted in 1670th cycle when we found cracking. If these tests 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 **Figure 3**.

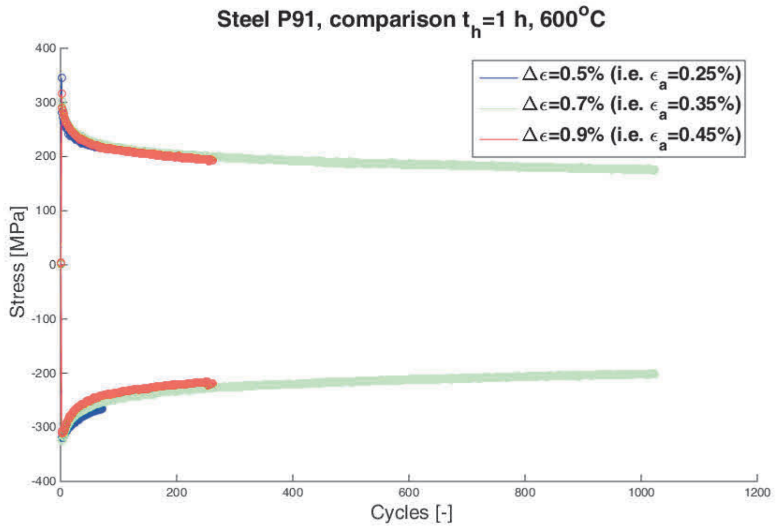

Figure 3 Comparison of maxima and minima for tests with different strain amplitudes 0.25, 0.35 and 0.45 % with tensile hold time t_h = 1 hour in every cycle and for temperature 600 °C

With a tensile hold time of 12 hours in every cycle, the goal was to achieve 50 cycles. This was achieved for all 3 tests. The first test for strain range $\Delta\varepsilon$ = 0.9 % (i.e. ε_a = 0.45 %) was interrupted in the 42nd cycle due to a power blackout and stopped in the 53rd cycle. The second test for strain range $\Delta\varepsilon$ = 0.7 % (i.e. ε_a = 0.35 %) was interrupted in the 24th cycle due to a software error and stopped in the 51st cycle. Last but not least, the test for strain range $\Delta\varepsilon$ = 0.5 % (i.e. ε_a = 0.25 %) was stopped in the 61st cycle. The comparison is shown in **Figure 4**. It was seen that the first maximum stress corresponds with the value of the strain amplitude. It is worth noting 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.

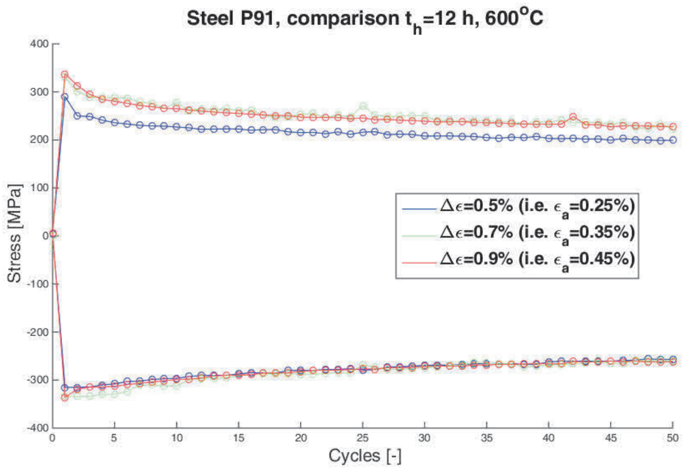

Figure 4 Comparison of maxima and minima for tests with different strain amplitudes 0.25, 0.35 and 0.45 % with tensile hold time t_h = 12 hours in every cycle and for temperature 600 °C

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 [6-9]. 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 a function of the cumulative plastic strain, *p(N)* [10]. The cumulative plastic strain allows better correlation of 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*. Similarly, the hold time effect is smaller if the stress evolution is considered with respect to *p(N)* rather than to *N*. Finally, Giroux showed that the strain rate effect is smaller when referring to *p(N)* compared to *N* [10].

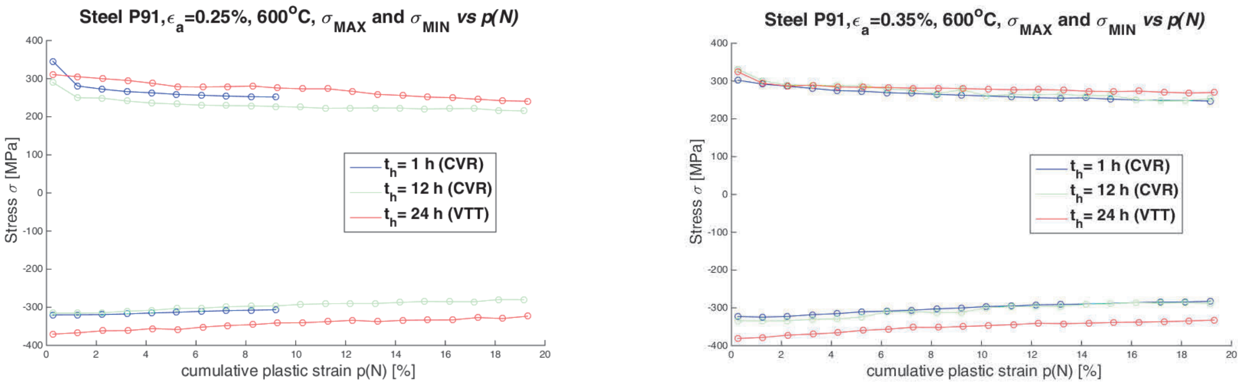

Figure 5 Comparison of maxima and minima for tests with different hold times (1, 12 and 24 hours) for strain amplitude 0.25 % (left) and 0.35 % (right) as a function of cumulative plastic strain *p(N)*

Using this information about cumulative plastic strain, the plots are represented in **Figures 5** and **6** for different amplitudes with different hold times. These curves are compared with VTT results for a 24 hour hold time and illustrate the effect of hold time on softening. If only the CVR test results are considered, it seems that at strain amplitudes of 0.45 % and 0.35 %, the hold time effect stabilizes after 1 hour. As a hold time of 1 hour is required at very high strain amplitude, it is expected that at least 1 hour hold time is required at intermediate strain amplitude. Nevertheless, at the smallest amplitude, 0.25 %, the stresses obtained with hold times of 1 hour and 12 hours differ, showing that saturation with respect to hold time has not yet been reached. The cyclic peak stresses measured by VTT are generally higher than those measured at CVR and a clear explanation has not yet been found. **Figure 6** shows the dependence of cyclic softening. The highest values are for the 24 hour hold time and the lowest for the 1 hour hold time. Generally it can be concluded that the lower the hold time, the lower the value of softening, but this trend was not confirmed for smaller amplitudes.

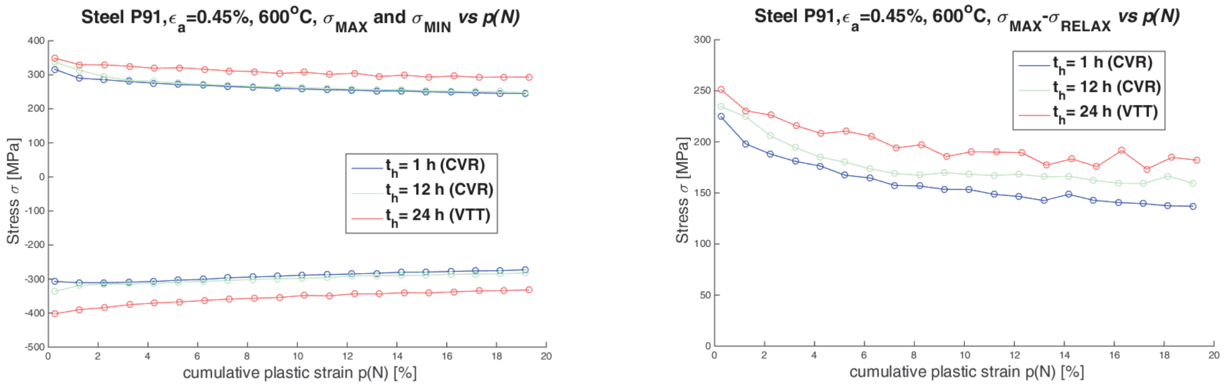

Figure 6 Comparison of maxima and minima for tests with different hold times (1, 12 and 24 hours) for strain amplitude 0.45 % (left) and dependence of evaluated cyclic softening for amplitude 0.45 % (right) as a function of cumulative plastic strain *p(N)*

Figures 5 and **6** show that for a given hold time, the lower the strain amplitude, the higher the magnitude of the stress. If we consider the difference in magnitude between the positive and negative peak stress after 20 cycles including the 24 hour 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. For a hold time imposed at the tensile peak stress, a compressive mean stress is measured. This may increase the lifetime. Nevertheless, if the hold time is applied at the compressive peak stress, then a tensile mean stress is measured which may decrease the lifetime. For strain amplitude of 0.25 % and a hold time of 24 hours, the mean stress amounts to about -50 MPa after 20 cycles which is far from negligible.

4. CONCLUSIONS

Tests focused on cyclic softening were performed on Grade 91 steel as part of the European FP7 project MatISSE project. Thermal state corresponded to 1060°C - 4Hrs + Water Quenching + 760°C - 3Hrs20min - Air Cooling. Chemical composition was analysed by glow discharge spectroscopy which gave a composition Cr 7.6 % by weight and Mo 0.885 % by weight. Tests were symmetrically reversed (R = -1), triangular course, temperature 600 °C and strain rate 6 % per minute. Tensile hold time was 1 hour in every cycle and then 12 hours for strain amplitudes ε_a = 0.45 %, 0.35 % and 0.25 %. The results were evaluated with respect to the number of cycles, *N*, and also as a function of the cumulative plastic strain, *p(N)*. Results were compared with VTT tests for a 24 hour hold time to illustrate the effect of hold time on softening. The highest values of softening are for the 24 hour hold time and the lowest for 1 hour hold time with 0.45 % amplitude. Generally it can be said that the lower the hold time, the lower the value of softening but this trend was not confirmed for smaller amplitudes. Longer hold times will induce an increased apparent strain range. It seems that at strain amplitudes of 0.45 % and 0.35 %, the effect of the hold time stabilizes after 1 hour when looking at the cumulative plastic strain. As a hold time of 1 hour is seemingly required for high strain amplitudes, it is expected that longer hold times are required at intermediate and low strain amplitudes. At the smallest amplitude, 0.25 %, the rate of softening with holding times of 1 hour and 12 hours still differ, indicating that saturation with respect to hold time has not yet been reached.

For a given hold time, the lower the strain amplitude is, the higher the magnitude of the mean stress seems to be. For instance, if we consider the mean stress after 20 cycles for the tests with 24h hold time, it is found that lower strain amplitude leads to stronger cyclic asymmetry and an increase in the compressive mean stress. The compressive mean stress may have a positive impact on the cyclic life.

ACKNOWLEDGEMENTS

This work was financially supported by the Ministry of Education, Youth and Sport Czech Republic - project LQ1603 (Research for SUSEN), project LM2015093 (SUSEN) and within the European FP7 MatISSE project, contract nr. 604862.

REFERENCES

[1] SWINDEMAN, R. W., SANTELLA, M. L., MAZIASZ, P. J., ROBERTS, B. W., COLEMAN, K. Issues in replacing Cr-Mo steels and stainless steels with 9Cr-1Mo-V steel. *International Journal of Pressure Vessels and Piping*, 2004, vol. 81, pp. 507-512.

[2] FOURNIER, B., DALLE, F., SAUZAY, M., LONGOUR, J., SALVI, M., CAËS, C., TOURNIÉ, I., GIROUX, P.-F., KIM, S.-H. Comparison of various 9-12%Cr steels under fatigue and creep-fatigue loadings at high temperature. *Materials Science and Engineering*, 2011, vol. 528, pp. 6934-6945.

[3] AGAMENNONE,R., BLUMA, W., GUPTA, C.,CHAKRAVARTTY, J. K. Evolution of microstructure and deformation resistance in creep of tempered martensitic 9-12%Cr-2%W-5%Co steels. *Acta Material*, 2006, vol. 54, pp. 3003-3014.

[4] HÖPPEL, H. W., KAUTZ, M., XU, C., MURASHKIN, M., LANGDON, T. G., VALIEV R. Z., MUGHRABI, H. An overview: Fatigue behaviour of ultrafine-grained metals and alloys. *International Journal of Fatigue*, 2006, vol. 28, pp. 1001-1008.

[5] HOLMSTRÖM, S., DE HAAN, F., FÜHRER, U., POHJA, R., JANOUSEK, J. Engineering models for softening and relaxation of Gr. 91 steel in creep-fatigue conditions. *International Journal of Structural Integrity*, 2017, vol. 8, pp. 670-682.

[6] KIM, S., WEERTMAN, J. R. Investigation of microstructural changes in a ferritic steel caused by high temperature fatigue. *Metallurgical Transactions A*, 1988, vol. 19, pp. 999-1007.

[7] EGGELER, G. The effect of long-term creep on particle coarsening in tempered martensite ferritic steels. *Acta Metallurgica*, 1989, vol. 37, pp. 3225-3234.

[8] LUKAS, P., KUNZ, L., SKLENICKA, V. Interaction of high cycle fatigue with high temperature creep in two creep-resistant steels. *Materials Science and Engineering: A*, 1990, vol. 129, pp. 249-255.

[9] VASINA, R., LUKAS, P., KUNZ, L., SKLENICKA, V. Interaction of high cycle fatigue and creep in 9%Cr-1%Mo steel at elevated temperature. *Fatigue & Fracture of Engineering Materials & Structures*, 1995, vol. 18, pp. 27-35.

[10] GIROUX, P.-F. PhD Thesis, CEA and Ecole des Mines de Paris, 2013.