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# Confirmation of low stress creep regime in 9% chromium steel by stress change creep experiments

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# Abstract

High sensitive helicoid spring specimen creep technique was modified to allow stress changes during the low stress, low strain rate experiments. The results obtained on the P-91 type modified martensitic 9% Cr steel support the previous observations of the change of the creep deformation mechanism at low stresses. The stress exponent derived from the stress change experiments approaches a value of about 4.5, considerably higher than that derived from the constant stress experiments. On the other hand, it is much lower than the value of 12, reported for the power-law creep regime of the same steel at stresses above 100 MPa. The creep behavior at low stresses is still not fully understood.

The transient stages were observed after each stress change including back flow after the stress decrease. The relaxation periods are relatively long, notably after the stress decrease. The stress redistribution is suggested as a main source of the observed transient effects. © 2004 Elsevier B.V. All rights reserved.

Keywords: Viscous creep; Power-law creep; Creep-resistant steel

# 1. Introduction

Extrapolation procedures are frequently used to obtain long-term creep data from relatively short-term creep experiments. Validity of the extrapolation procedures is limited by possible changes in the microstructure of the material, as well as possible changes in the creep deformation mechanisms. While the long-term microstructural stability of the creep resistant materials is widely studied, the possibility of the change in deformation mechanism is usually neglected.

Transition from the power-law to viscous creep behavior has been recently reported for both martensitic and austenitic creep-resistant steels [1–4] just under conditions corresponding to the industrial applications of the steels, i.e. at 873 K and 100 MPa. The transition stress is considerably higher than that observed in model materials. The "low stress creep regime" should also be taken into account in industrial applications of the creep-resistant steels. Moreover, the results show that the strengthening microstructural elements in the

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steels are much less efficient under low stress creep regime [5].

The creep properties observed in the previous experiments [3,4] could not be simply related to any of the known theories of the viscous creep. There is a large discussion but no general agreement about the low stress creep deformation mechanisms in the literatures [6–9]. The dislocation Harper–Dorn creep, as well as Nabarro–Herring and Coble diffusional creep and the creep mechanism based on the grain boundary sliding are usually considered. The stress change creep experiments were performed to provide broader basis of experimental results for better understanding of the deformation mechanisms.

#### 2. Experimental material and procedures

Pipes from the 9% Cr ferritic–martensitic creep-resistant steel of P-91 type were supplied by the Vitkovice Steel in "ready to use" condition. Chemical composition of the material is shown in Table 1.

Helicoid spring specimen technique [10,11] was used for the creep testing at 873 K and 34–44 MPa. The technique was modified to allow stress changes within the protective

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Table 1 Chemical composition of the P-91 type steel in wt.%

Cr	8.5
Мо	0.88
Si	0.43
Mn	0.40
V	0.23
Ni	0.10
Nb	0.10
С	0.10
Ν	0.045
Al	0.018
Р	0.015
S	0.006
Fe	Balance

atmosphere of purified argon. Coil spacing was measured optically. Helicoid spring specimens were made by machining from the pipe to preserve original microstructure. Specimens were aged by long-term annealing at 923 K for 10 000 h before the creep test. The resulting microstructure is described elsewhere [12]. Since the stress and strain in the helicoid spring are essentially shear ones, they were transformed to the equivalent tensile quantities using a well known relations resulting from the von Mises criterion:  $\sigma = \sqrt{3}\tau$  and  $\varepsilon = \gamma/\sqrt{3}$ , where  $\sigma$  is tensile stress,  $\tau$  is shear stress,  $\varepsilon$  is tensile strain and  $\gamma$  is shear strain.

### 3. Experimental results

The creep curve recorded in the experiment is shown in Fig. 1. Each stress increase is followed by pronounced transient effect resembling primary stage of the constant stress creep curve. In the case of stress decrease, the negative creep

flow followed in all cases. The individual segments of the creep curve are depicted in Fig. 2, where segments are denoted by the sequential number of precedent stress change k. The creep curves of individual segments in Fig. 2 are fitted by the equation derived by Li [13]:

$$\varepsilon = \dot{\varepsilon}_{s} t_{p} \ln \left( 1 + \frac{\dot{\varepsilon}_{i} - \dot{\varepsilon}_{s}}{\dot{\varepsilon}_{s}} \left( 1 - \exp \left( -\frac{t}{t_{p}} \right) \right) \right) + \dot{\varepsilon}_{s} t, \quad (1)$$

where  $\varepsilon$  is the strain,  $\dot{\varepsilon}_i$  the initial strain rate,  $\dot{\varepsilon}_s$  the steady-state strain rate,  $t_p$  the primary stage relaxation period and t the time. The fit is generally good. The "steady-state" or pseudo-secondary creep is described by the parameter  $\dot{\varepsilon}_s$  while the transient creep can be characterized by the parameters  $t_p$ ,  $\dot{\varepsilon}_i$  or by the transient strain  $\varepsilon_t$ , for which the relation  $\varepsilon_t = \dot{\varepsilon}_s t_p \ln(\dot{\varepsilon}_i/\dot{\varepsilon}_s)$  holds. The  $\dot{\varepsilon}_s$  is practically identical to the creep rate derived directly at the end of each segment of the creep curve.

# 4. Discussion

#### 4.1. Steady-state creep rate

Steady-state creep rate dependence on applied stress and the segment sequential number is shown in Fig. 3 and in more detail in Fig. 4, together with the same dependence derived from the constant stress experiments. The constant stress data were taken from [3]. The creep rate of the first segment correspond well with the constant stress results, while in subsequent segments it is lower and lower. Thus, the stress changes cause creep strengthening of the material. This finding is in agreement with the results of Kunz and Lukáš [14] showing that small vibrations superposed on the creep stress can lead to the lower creep rates.



Fig. 1. Creep curve of the stress change experiment at 873 K. Individual segments are marked by the sequential number k.



Fig. 2. Individual segments of the creep curve recorded after stress changes, where k is the sequential number of the preceding stress change.

#### 4.2. Stress exponent

The stress exponent n, defined generally as

$$n = \frac{\Delta \left( \ln \left( \dot{\varepsilon}_{\rm s} \right) \right)}{\Delta \left( \ln \left( \sigma \right) \right)} \tag{2}$$

is considered as a main parameter to identify the creep deformation mechanism. In this case, the value can be estimated from the curve slope in Fig. 3 and is also plotted in Fig. 5. The stress exponent for the stress changes approaches the value of about 4.5, which is much higher than the value of about 1 derived from the constant stress creep experiments for the low stress creep regime, but it is much lower than the value of about 12 reported for the high stress creep regime. The higher value of the stress exponent is caused by the strengthening effect described above. Nevertheless, the results lead to the conclusions, that the simple viscous creep mechanisms are not capable to describe the creep behavior of the tested steel. The description of the creep effect by the power-law and stress exponent derived from the constant stress or constant load is insufficient, since the loading history of the specimen is very important. On the other hand, the results confirm the fact, that the creep behavior under low stresses is strongly different than that under high



Fig. 3. Dependence of the steady-state creep rate on the applied stress, compared to the results of constant stress experiments [4]



Fig. 4. Dependence of the steady-state creep rate on the applied stress, detail of the results of the stress change experiment.

stresses. Extrapolation from the high stress results to lower stresses is not reliable.

#### 4.3. Transient effects

The parameters  $t_p$  and  $\varepsilon_t$  are plotted against the time of the corresponding stress change in Figs. 6 and 7. The transient stage after stress decrease is generally about two times longer than that after stress increase. The transient relaxation time seems to increase with decreasing stress difference. Another unexpected effect is that the total transient strain after the stress decrease is independent of the stress difference, while that for the stress increase is proportional to the stress difference. Since the errors of estimation of the parameters are relatively large and almost comparable to the discussed differences, it is impossible to draw any strong conclusions from the effects. The transient strain should be probably assigned to the stress redistribution [15], which assumes the large stress concentrations around some microstructural elements. The elastic modulus for the steel at 873 K is reported as G = 97.4 GPa [16]. The equivalent elastic deformation corresponding to the applied stress change is then about



Fig. 5. Stress exponent  $n = \Delta(\ln(\hat{\epsilon}_s))/\Delta(\ln(\sigma))$  plotted against the time of the corresponding stress change. The level of n = 4.5 is marked by dotted line.



Fig. 6. Magnitude of the total transient strain  $\varepsilon_t$  plotted against the time of the corresponding stress change.



Fig. 7. Transient relaxation period  $t_p$  plotted against the time of the corresponding stress change.

 $\varepsilon_{\rm e} \approx 4 \times 10^{-5}$ , what makes the above assumption acceptable. Milička and Dobeš [17] measured the internal stress in the same material and found the internal stress  $\sigma_{\rm i}$  is equal to the applied stress  $\sigma$  below  $\sigma = 140$  MPa. The observed large back flow after each stress decrease then can be expected.

# 5. Conclusions

The results of creep experiments on the P-91 type steel at 873 K and stress changes in the range 33–45 MPa confirm the creep behavior being strongly different from that ob-

served at higher stresses. On the other hand, the stress exponent is approaching the value of about 4.5, thus, the simple mechanisms of the viscous creep, proposed previously, are not applicable. The results clearly show that the description of the creep effect based on the constant stress or constant load experiments is insufficient since the loading history has very strong influence on the creep deformation.

Every stress change is connected with the large transient stage. Measured parameters of the transient stages exhibit some unexpected values, like an apparent independence of the transient strain on the stress change magnitude. There is still no explanation for the effect.

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