# VISCOUS CREEP IN METALS AT INTERMEDIATE TEMPERATURES

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Creep at stresses below 100 MPa and temperatures from 600 °C to 750 °C was investigated on two types of creep resistant steels: ferritic-martensitic P-91 type and austenitic AISI-316H type. Evidences were found that the creep process at low stresses is very different from that at high stresses in many features. The most important is that the efficiency of strengthening microstructural elements drops down drastically at low stresses. Deformation mechanism of the low stress creep remains unclear, but it is very sensitive to the fine microstructural changes which cannot be simply detected by optical and electron metallography.

Key words: creep test, heat resistant steel

## 1. Introduction

Though a low stress creep has been known for decades, debate has continued during the last decade in the literature [1–5]. Since the acquisition of necessary experimental data is difficult and usually extremely time consuming, the experimental evidences are rather limited. A most important common characteristic of all the low stress creep mechanisms is a weak dependence of strain rate on applied stress. The steady state creep rate is directly proportional to the applied stress, so the creep behaviour is described as "viscous".

The creep due to diffusional transport of matter between grain boundaries, so called Nabarro-Herring or Coble diffusional creep, is one mechanism considered for the low-stress creep deformation. On the other side, several dislocation mechanisms of viscous Harper-Dorn creep independent of grain boundaries were suggested. Nevertheless, none of the mechanisms is capable to explain fully all observed properties of the low stress creep in various metallic materials [6].

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Undoubtedly, the understanding of flow mechanisms operating under low stresses and intermediate temperatures has an important impact on the engineering practice, that is in design and development of performance and lifetime codes which are used in this practice. The low stress creep data are rare since they require time consuming testing and they are not easy to extrapolate to rupture.

The research of low stress creep of metallic materials using unconventional testing techniques was initiated by prof. Čadek of about twenty years ago and remained probably the most systematic activity in the field [7–10]. This article reviews and summarizes arguments and evidences for the special importance of low stress creep regime in engineering practice, while the underlying micromechanisms remain unclear.

## 2. Experimental materials and procedure

Experimental examination of the low stress creep has been focused recently on the creep resistant steels in order to find its importance for engineering practice. Two types of the creep resistant steels were tested at conditions leading to the low strain rates by the helical spring specimen technique [10]. The ferritic 9% chromium steel (type P-91) was supplied by Vítkovice Steel, and the austenitic type AISI-316H was supplied by Poldi Steel. Both steels were supplied in the form of pipes ready for use in industrial application. The chemical composition is summarized in Table 1.

Table 1. Chemical composition of the experimental materials [wt.%]

Material	$\mathbf{Cr}$	Ni	Mo	Si	Mn	V	Nb	С	Ν	Al	Р	S
P-91	8.5	0.1	0.88	0.43	0.4	0.23	0.1	0.1	0.045	0.018	0.015	0.006
AISI-316H	16.9	12.5	2.45	0.45	1.21			0.07			0.028	0.014

The helical spring specimens were machined from the pipes and used for the creep testing with or without prior heat treatment. In some cases, the experiment was modified to allow the stress changes during creep. The P-91 steel was tested at temperatures from 873 K to 923 K and stresses from 1.5 MPa to 100 MPa. The AISI-316H steel was tested at temperatures between 923 K and 1073 K and stresses from 1.5 MPa to 30 MPa. The creep tests times were between 1100 h and 2500 h for both cases. The grain size measured by the optical metallography is  $\bar{L} \approx 50 \,\mu\text{m}$  for the P-91 steel and  $\bar{L} \approx 100 \,\mu\text{m}$  for the AISI-316H steel, where  $\bar{L}$  is the mean intercept length. The testing procedures were described in more details in previous publications [11, 12].

#### 3. Results and discussion

An example of a typical set of creep curves obtained from low stress creep experiment is in Fig. 1. All creep curves were fitted by an equation derived by Li [13], from which the secondary stage creep rates  $\dot{\varepsilon}_s$  were derived:

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

where  $\varepsilon$  is strain,  $\dot{\varepsilon}_{\rm s}$  is steady state creep rate,  $t_{\rm p}$  is primary stage relaxation time,  $\varepsilon_{\rm p}$  is primary strain and t is time. The fit was good. The testing times were set to fulfill the condition  $t \geq 5t_{\rm p}$  for the accurate estimation of the  $\dot{\varepsilon}_{\rm s}$ .



Fig. 1a. Typical creep curves obtained in low stress creep experiments. Stresses from 3.2 MPa to 8.7 MPa.

Fig. 1b. Creep curves from Fig. 1a plotted as log(strain rate) against strain.

It is important to stress that no change in the microstructure was detected by comparing the TEM examinations before and after the creep tests. This is not surprising with such a low total strains. On the other hand, the primary stage cannot be explained by the dislocation density development, as had been assumed by Li. Nevertheless, the assumption of the equilibrium between first order and second order kinetics works very well.

## 3.1 Low stress creep mechanisms

The permanent question about the low stress creep regime is whether it is caused by some independent creep deformation mechanism (like diffusional creep, for instance) or whether it is the feature of normal "power-law" dislocation mechanism only. A model unifying the low stress and high stress creep mechanisms was



Fig. 2. Stress dependence of secondary stage creep rate  $\dot{\varepsilon}_{s}$  and effective stress  $\sigma^{*}$ . Values of  $\sigma^{*}$  from [15].

Fig. 3. Stress dependence of secondary stage creep rate  $\dot{\varepsilon}_{s}$  fitted by Wu and Sherby equation [14] and by a sum of viscous and power-law equations.

proposed by Wu and Sherby [14] and is based on the internal stress. The direct measurement of the effective stress  $\sigma^* = \sigma - \sigma_i$  by Milička and Dobeš [15] shows relatively good agreement with the transition from "viscous" to "power-law" creep regime, as is shown in Fig. 2. The agreement with the internal stresses models based on dislocation density is also good [6].

On the other side, for our tests, the transition between the creep regimes is relatively sharp and cannot be fitted well by the Wu and Sherby model, while the fit based on the two independent mechanisms is quite good (Fig. 3). The different reaction, to the microstructure evolution, in the two creep regimes provides even stronger argument for two independent deformation mechanisms. If the steel P-91 is annealed for long time, the Z-phase particles grow on the expense of MC particles. The result of the process is the apparent creep weakening under the "power-law" creep regime, while it does not have an apparent effect on the "viscous" creep regime (Fig. 4). Moreover, the creep strengthening microstructural elements in the steels mostly lose their effectivity under the viscous creep regime. This fact is apparent from Fig. 5, where the secondary creep rates are normalized by the creep rate of pure mild iron showing the strengthening effect [16]. Also the temperature dependence expressed by the apparent activation energy is very different in both creep regimes, as is depicted in Fig. 6 [12].

At the moment, it is difficult to make a conclusive decision about the low stress creep mechanism. But, in authors' opinion, the arguments for an independent low stress creep mechanism are stronger.

## 3.2 Stress change response

Stress change experiments were done for the P-91 steel in low stress creep



Fig. 4. Stress dependence of secondary stage creep rate  $\dot{\varepsilon}_s$  for as received steel and steel after ageing at 650 °C for 10 000 hrs.



Fig. 5. Stress dependence of strengthening efficiency factor  $\eta = \dot{\varepsilon}_{s(Fe)} / \dot{\varepsilon}_{s(Steel)}$ .



Fig. 6. Stress dependence of apparent activation energy  $Q_{\rm a}$ .

Fig. 7. Detail of creep curve with stress reduction from 34.9 MPa to 34.1 MPa.

regime [17]. Even very small stress decrease leads to the immediate reverse creep flow (Fig. 7). This result is fully consistent with the result showing that the internal stress equals the applied stress under the low stress creep conditions. The effect of creep strengthening caused by the stress changes is even more important. This leads to lower strain rates in the stress change experiment and to higher stress exponent in comparison with the constant stress experiment. While the constant stress experiment gives the stress exponent  $n \approx 1$ , the stress changes yield the value  $n \approx 5$  (Fig. 8). It is clear, that the term "viscous creep" is misleading, since the material behaviour is far from that of the viscous Newtonian fluid. The dependence  $\dot{\varepsilon}(\sigma, T)$  obtained from the constant stress-constant temperature creep experiments is strongly affected by the microstructure development including the internal stress.

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Fig. 8. Stress dependence of secondary stage creep rate  $\dot{\varepsilon}_{\rm s}$  for constant stress experiments and after stress changes.

The changes in microstructure are not detectable by the TEM observations, since the strain connected to them is very low. Deformation micromechanism shows probably different  $\dot{\varepsilon}(\sigma, T)$  dependence, which opens new space for the micromechanism modelling. It should be also stressed that the simple relation  $\dot{\varepsilon} = A\sigma^n$  with A and n constant cannot be used in the modelling of dynamic creep response of various parts loaded at elevated temperatures. This approach is used frequently and is clearly completely wrong.



Fig. 9. Maps of creep deformation mechanisms. Dotted curves are lines of constant strain rate from  $10^{-13}$  s<sup>-1</sup> to  $10^{-9}$  s<sup>-1</sup>.

#### 3.3 Relevance to the engineering practice

The creep regime maps have been constructed using the creep test results in both "viscous" and "power-law" creep conditions [18]. The maps are shown in Fig. 9. It is clear that the industrial application conditions lay mostly in the "viscous creep" region, close to the transition boundary. These conditions provide relatively the best creep resistance, as apparent from Fig. 5. On the other hand, the laboratory testing conditions lay mostly in the power-law region, so its results have limited validity for the industrial use of the steels. Taking into account the different creep behaviour depending on stress, the extrapolation methods relying mainly on power-law regime used for the evaluation of the creep strength and creep life need revision.

## 4. Conclusions

Recent results of low stress creep experiments can be summarized as follows: – The low stress creep regime is very different from the high stress one in many parameters.

– The underlying micromechanism is still not clear. At variable stress it does not seem to obey the same stress and temperature dependences as those from the constant conditions tests.

- The microstructure development and loading history are very important for the low stress creep strength. Nevertheless, the microstructure changes are "hidden", that is they are too fine to be detected by the standard TEM observations.

- The industrial application loading conditions fall mostly within the low stress creep regime. The codes for creep strength and life should reflect this fact.

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#### REFERENCES

- [1] RUANO, O. A. et al.: Mater. Sci. Eng. A, 165, 1993, p. 133.
- [2] BURTON, B.-REYNOLDS, G. L.: Mater. Sci. Eng. A, 191, 1995, p. 135.
- [3] RUANO, O. A. et al.: Mater. Sci. Eng. A, 211, 1996, p. 66.
- [4] LANGDON, T. G.: Scripta Mater., 35, 1996, p. 733.
- [5] BLUM, W.—MAIER, W.: Phys. Stat. Sol. (a), 171, 1999, p. 467.
- [6] KLOC, L.—FIALA, J.—ČADEK, J.: In: Creep Behavior of Advanced Materials for the 21st Century. Eds.: Mishra, R. S., Mukherjee, A. K., Murty, K. L. Warrendale, PA, USA, TMS 1999, p. 471.
- [7] FIALA, J.—NOVOTNÝ, J.—ČADEK, J.: Mater. Sci. Eng. A, 60, 1983, p. 195.
- [8] FIALA, J.—NOVOTNY, J.—CADEK, J.: Acta Metall., 33, 1985, p. 905.

- [9] FIALA, J.—KLOC, L.—ČADEK, J.: Mater. Sci. Eng. A, 137, 1991, p. 163.
- [10] FIALA, J.—ČADEK, J.: Kovove Mater., 20, 1982, p. 277.
- [11] KLOC, L.—SKLENIČKA, V.: Mater. Sci. Eng. A, 234–236, 1997, p. 962.
- [12] KLOC, L.—SKLENIČKA, V.: Mater. Sci. Eng. A, 319–321, 2001, p. 774.
- [13] LI, J. C. M.: Acta Metall., 11, 1963, p. 1269.
- [14] WU, M. Y.—SHERBY, O. D.: Acta Metall., 32, 1984, p. 1561.
- [15] MILIČKA, K.—DOBEŠ, F.: Engineering Mechanics, 5, 1998, p. 165.
- [16] KLOC, L.—FIALA, J.—SKLENIČKA V.: Chem Listy, Symposia, 96, 2002, p. S212.
- [17] KLOC, L.—SKLENIČKA, V.: Mater. Sci. Eng. A, 387–389C, 2004, p. 633.
- [18] KLOC, L.—SKLENIČKA, V.: In: EUROMAT 2001 Conference Proceedings. Milano, Italy, AIM 2001, art. no. 752.