On Creep Behaviour of Several Metallic Materials at Low Stresses and Elevated Temperatures

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Received 24 February 1999

Viscous creep was observed in many materials at temperatures close to the value of one half of absolute melting temperature at very low creep rates. The results were interpreted as diffusional creep and/or Harper—Dorn creep. Not all of creep mechanisms acting at conditions mentioned above can explain their own primary stage. Nevertheless, the primary creep has been observed in almost all creep experiments. Hence, special primary creep mechanisms must be responsible for primary creep observed.

Many creep results are in very good agreement with the Coble theory, and can support the idea of the diffusional creep operating as an important deformation mechanism under certain conditions. Unfortunately, the theory of diffusional creep seems to be too simplified to describe accurately the diffusional creep processes. In several materials, dependence of the creep rate on grain size corresponds to the Coble mechanism for grain sizes below approximately 100 μm, while large data scattering for coarse grain sizes replaces this dependence. This behaviour used to be interpreted as a transition from the diffusional to the Harper—Dorn creep regime. The structural parameter responsible for large scattering of creep rates at large grain sizes has not been identified yet. This paper discusses the transition from viscous to power-law creep regime for two materials with very different structure.

The most important aspect of any engineering material is not only its chemical composition but also the structure, because material properties are closely related to this feature. To be successful in “material tailoring” one must have a good understanding of this relationship between structure and properties and also must have a good knowledge of degradation mechanisms acting in materials under conditions of engineering practice (*i.e.* temperature, stress, corrosion, *etc.*).

Viscous creep was observed in many materials at low stresses and high or intermediate homologous temperatures. Viscous creep means that the strain rate is directly proportional to the applied stress, *i.e.* the value of the stress exponent is of one. This kind of creep is often interpreted as the Nabarro—Herring [1, 2] or the Coble [3] diffusional creep or the Harper—Dorn dislocation creep [4].

The terms “Nabarro—Herring creep”, “Coble creep” or “Harper—Dorn creep” look similar, but their meaning is different. While the Coble or Nabarro—Herring diffusional creep mean clear physical processes of Newtonian flow, the term Harper—Dorn creep describes the very creep process. This fact is a frequent source of misunderstanding. The term Harper—Dorn creep is valid for any creep mechanism having unity stress exponent and not depending directly on grain size.

At the intermediate temperatures, some limitations should be considered:

Coble diffusional creep predominates over Nabarro—Herring creep in all cases. The Nabarro—Herring creep can be neglected.

The conditions for viscous creep by grain boundary sliding according to *Fukuyo et al.* [5] are not fulfilled.

Strain rates measured are very low. No such effects like denuded zones can be observed.

The Coble diffusional creep is defined as a transport of matter among grain boundaries by grain boundary diffusion. It is described by the Coble equation

$$\dot{\varepsilon} = A_{\mathrm{C}} \frac{D_{\mathrm{B}} \delta_{\mathrm{B}} \omega \sigma}{k T d^3} \qquad (1)$$

where $\dot{\varepsilon}$ is strain rate, A_{C} is a constant depending on grain shape, $D_{\mathrm{B}} \cdot \delta_{\mathrm{B}}$ is a product of grain boundary diffusion coefficient and effective width of grain boundary, ω is atomic volume, σ applied stress, k Boltzmann's constant, T absolute temperature, and

d is grain size. The problem of the equation consists in unclear relation between the model describing parameters A_{C} and d and the measurable parameters describing grain structure of real material. It was shown [6] that deviations up to one order of magnitude in strain rate can be accounted to that reason. In this paper, values of $A_{\mathrm{C}} = 148/\pi$ and of $d = 1.57\overline{L}$ are used ($\overline{L}$ is mean intercept grain size). These values are derived for the spherical grains. Eqn (*1*) contains only such parameters, which can be determined independently. Thus the diffusional creep model is a "hard" model and the calculated rate cannot be fitted on the experimental data.

The Harper—Dorn dislocation creep was observed originally in aluminium and its alloys at very high temperatures close to their melting point. Its features and conditions were summarized by *Yavari*, *Miller*, and *Langdon* [7] and many models were developed to describe it [8]. The loss of grain boundary dependence in creep experiments at intermediate temperatures and low stresses used to be interpreted as a transition from diffusional to Harper—Dorn creep, even if some conditions established for high temperatures were not fulfilled. Most probably, the mechanism of Harper—Dorn creep at intermediate temperatures is different from that at high temperatures. Some new models of Harper—Dorn creep, which are suitable for intermediate temperatures should be mentioned. The model based on internal stress by *Wu* and *Sherby* [9] and the dislocation network model by *Ardell* [10] will be discussed further.

The primary creep was observed in almost all low-stress creep experiments even if the main attention was paid to secondary state mostly. Since some mechanisms assumed to be responsible for creep deformation in secondary stage are not capable to explain the primary creep, special primary creep mechanisms must be taken into account. Better understanding of these mechanisms seems to be important for engineering practice since the overall strain allowed for some components in high temperature technology may be essentially exhausted during the primary creep stage duration.

EXPERIMENTAL

Helicoid spring specimen technique was used for low-stress creep experiments. Creep results obtained on various materials were presented in detail earlier. This study discusses experiments on pure α-Fe [11, 12], pure α-Zr [13], primary CuAl solid solution with 2 mass % Al [14, 15], primary CuAl solid solution with 6.5 mass % Al [16], and primary NiCr solid solution with 15 mass % Cr [17], and type P-91 heat-resistant stainless steel [18, 19].

Different initial structure results from the differences in specimen preparation. Pure metals and solid solutions were firstly cold rolled and drawn to wire, then wound on stainless steel threaded bolt and annealed for various time to obtain different grain size. Resulting recrystallized structure consisted of equiaxed grains with low dislocation density (below 10^{12} m^{-2}). The annealing temperature was in any case at least by 50 K higher than that of the subsequent creep test. On the other hand, we used the P-91 steel in "ready to use state" delivered by the Vítkovice Steel Company. The helicoid specimens were prepared by machining from tube. Their structure is very complex, having grain size $\overline{L} \approx 55$ μm, subgrains and/or martensitic laths dimensions below 1 μm, dislocation density inside subgrains of about 2×10^{14} m^{-2} and various precipitates. All parameters were observed experimentally on the same material in Refs. [20, 21].

RESULTS

Creep curves of various materials exhibit normal primary stage, during which the creep rate decreases and finally approaches the constant secondary value. Typical creep curves for various materials are shown in Fig. 1. The primary and secondary stages of creep are described and analyzed separately.

Primary Creep

From Fig. 1 it is obvious that the creep curve for NiCr is very similar to that of Cu—2 % Al. Except for α-Zr, all creep curves exhibit normal primary stage with decreasing deformation rate.

Pure α-Zr

In most tests of pure α-Zr, the primary stage was not observed at all. In some others it was observed, but in a very small extent. Thus the results of α-Zr are not discussed below.

Pure α-Fe and Solid Solutions

Creep curves of these materials could be well fitted by the McVetty equation [22]

$$\varepsilon = \dot{\varepsilon}_{\mathrm{s}} t + \varepsilon_{\mathrm{p}} \left(1 - \exp\left(\frac{-t}{\tau}\right)\right) \qquad (2)$$

where ε is the creep strain, $\dot{\varepsilon}_{\mathrm{s}}$ is the secondary creep rate, ε_{p} is the total primary strain, and τ is the relaxation time of the primary stage. In this case, initial creep rate is given as $\dot{\varepsilon}_{\mathrm{i}} = \dot{\varepsilon}_{\mathrm{s}} + \varepsilon_{\mathrm{p}}/\tau$. The secondary creep rate under given conditions corresponds to viscous creep, due to the Coble diffusional mechanism and/or due to the Harper—Dorn dislocation creep mechanism [11—17].

Initial to secondary creep rates ratio does not depend markedly on stress (Fig. 2) indicating that the stress exponent of initial creep rate is the same as

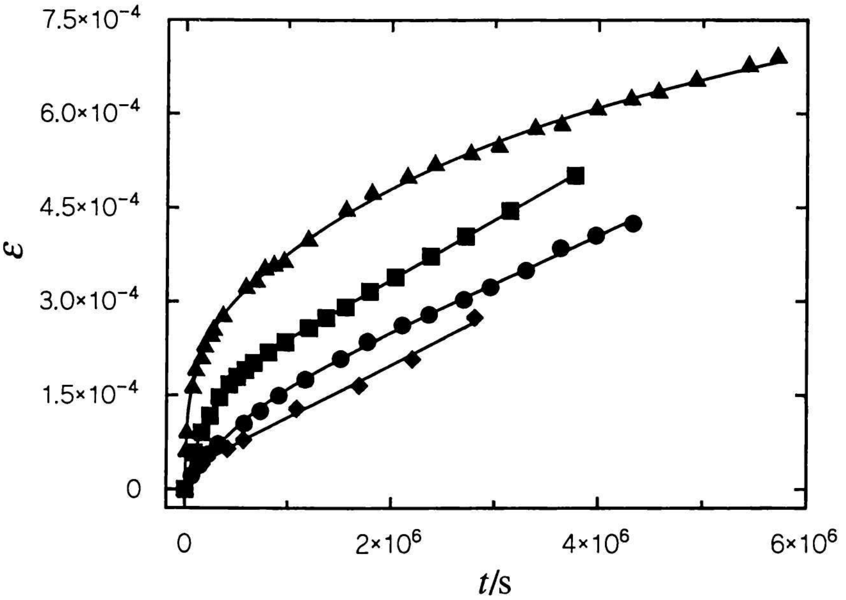

Fig. 1. Typical creep curves for various materials considered in this paper. Testing conditions were selected to obtain average creep rate of about 10^{-10} s^{-1}: 50 MPa, 873 K for P-91 (▲), 2.57 MPa, 1073 K for NiCr (■), 3.34 MPa, 773 K for Cu—2 % Al (●), and 0.86 MPa, 773 K for α-Zr (◆).

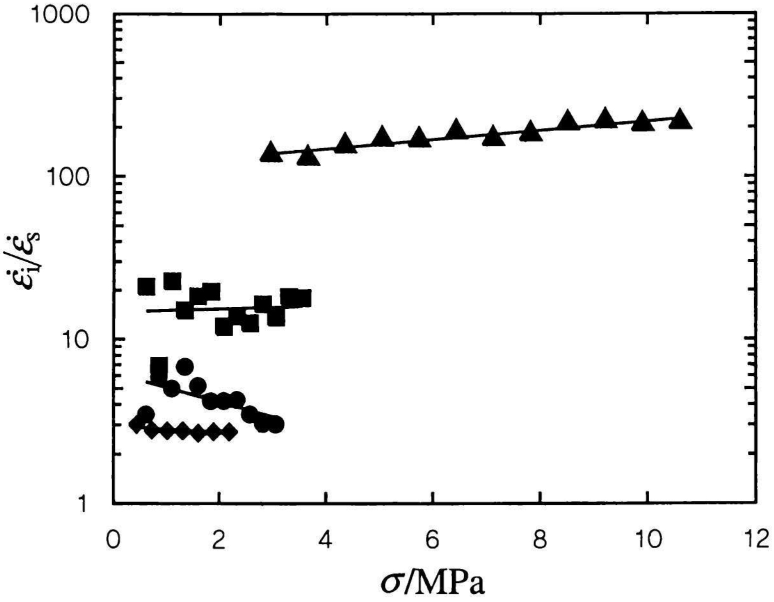

Fig. 2. The $\dot{\varepsilon}_i/\dot{\varepsilon}_s$ ratio dependence on stress: P-91 (▲) at 898 K, NiCr (■) at 1073 K, Cu—2 % Al (●) at 773 K, and α-Fe (◆) at 643 K.

that of secondary creep rate. The $\dot{\varepsilon}_i/\dot{\varepsilon}_s$ ratio dependence on temperature is not conclusive either (Fig. 3). Initial creep rate is then governed by the same rules as the secondary creep rate.

The primary stage duration, characterized by the parameter τ, is independent of stress, as can be seen from Fig. 4. This behaviour indicates that the secondary stage can be reached even in very low-stress experiments.

P-91 Heat-Resistant Steel

Creep curves of P-91 type heat-resistant steel exhibit large primary stage with the different shape compared to previous ones. Curves cannot be fitted by any of the equations used for primary stage having a finite value of initial creep rate. The best but not satisfactory fit is provided by the Li's equation [19]. Creep curves were fitted by spline functions and appropri-

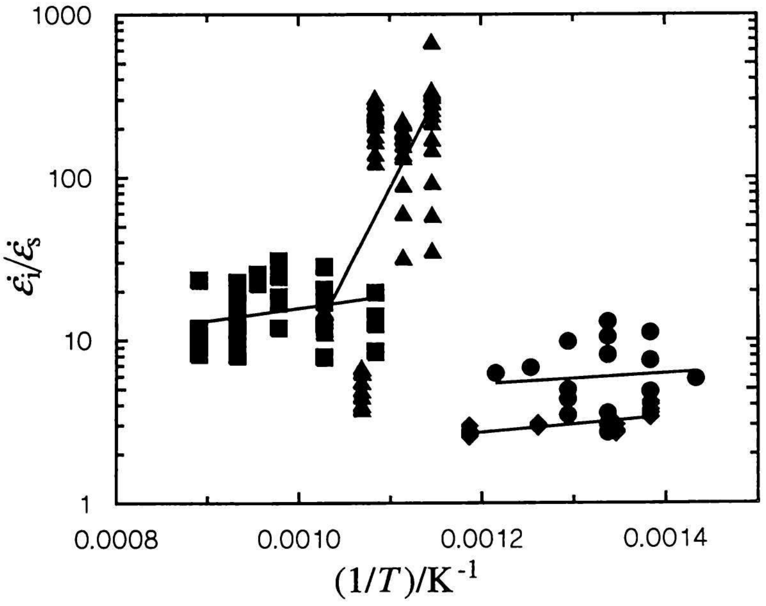

Fig. 3. The $\dot{\varepsilon}_i/\dot{\varepsilon}_s$ ratio dependence on reciprocal temperature. Denotation as in Fig. 2.

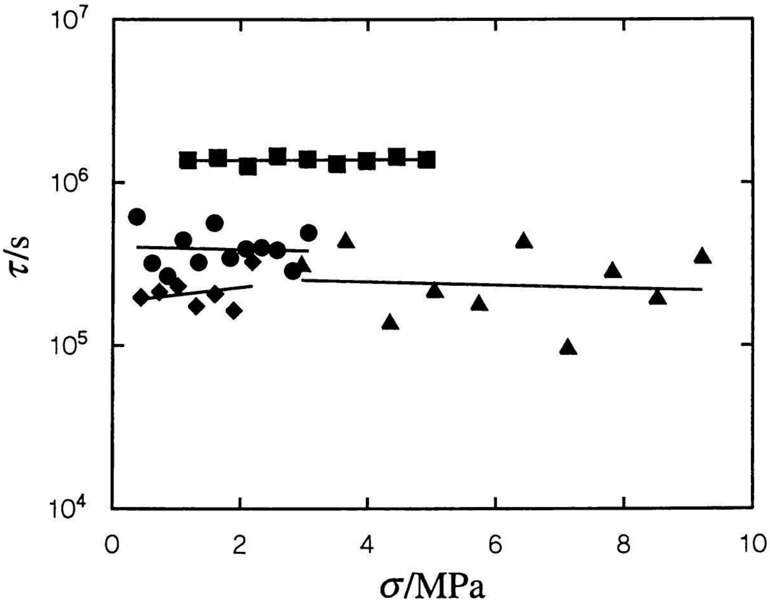

Fig. 4. Primary stage duration dependence on stress: P-91 (▲) at 898 K, NiCr (■) at 1073 K, Cu—2 % Al (●) at 773 K, and α-Fe (◆) at 643 K.

ate parameters $\dot{\varepsilon}_i, \varepsilon_p$, and τ were derived numerically. Unlike the previous case, the parameters are not generally dependent. Along with the different shape of creep curves, the $\dot{\varepsilon}_i/\dot{\varepsilon}_s$ ratio is strongly dependent on temperature and reaches much higher values.

Secondary Creep

The temperature dependence of creep rate can be described by means of the Arrhenius relation $\dot{\varepsilon}_s \propto \exp(-Q_a/RT)$ where Q_a is apparent activation energy and R is universal gas constant. In all cases, Q_a was close or even lower than activation enthalpy of grain boundary diffusion.

The dependence of creep rate on grain size is the most important factor to identify viscous creep mechanism. These dependences are shown in Figs. 5—7 No attempt to change grain size was made with P-91 steel, since in such a complex structure it is impossible to change grain size without changing many other

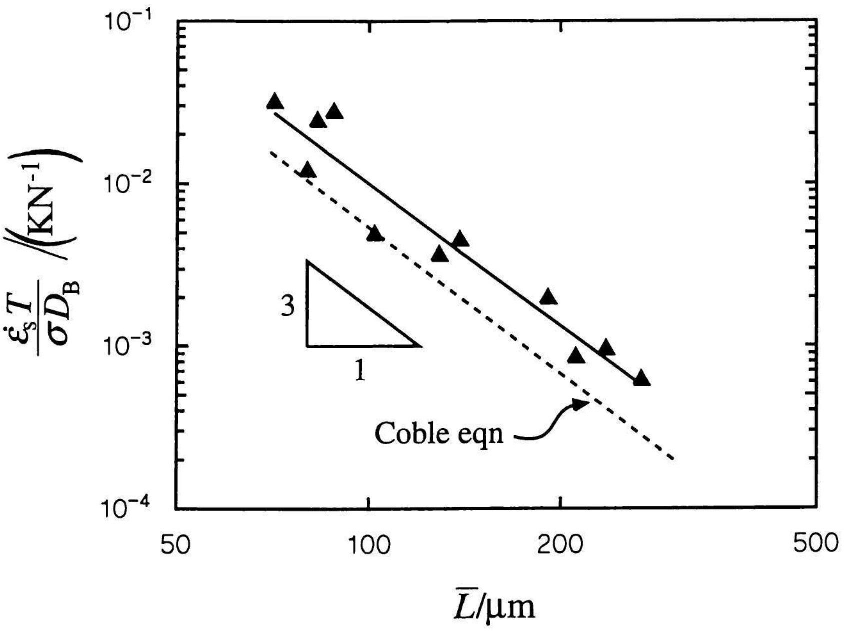

Fig. 5. Dependence of temperature and stress compensated creep rate on grain size for the Cu—6.5 % Al primary solid solution at 773 K. The triangle in the figure represents the line slope of 3 corresponding to the Coble theory (eqn (*1*)).

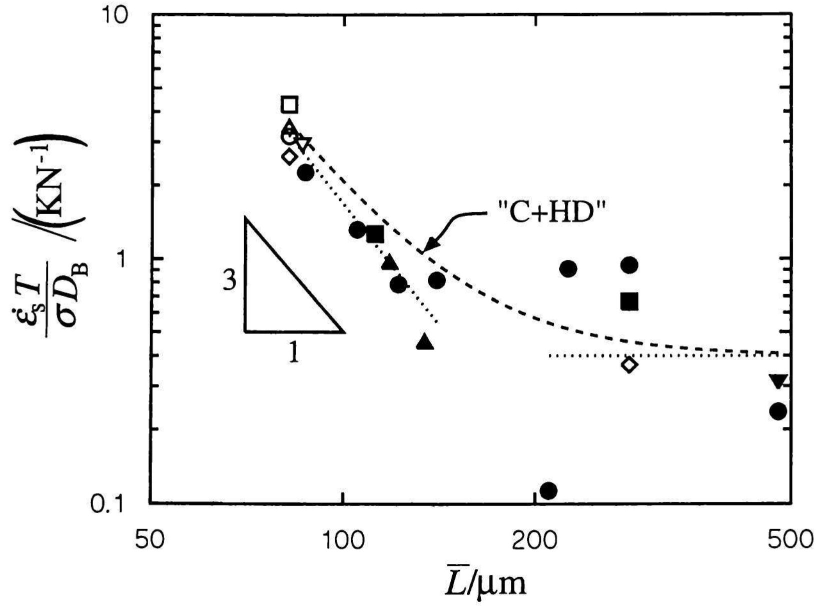

Fig. 6. Dependence of temperature and stress compensated creep rate on grain size for pure α-iron. ○ 723 K, □ 743 K, △ 773 K, ▽ 793 K, ◇ 823 K, ● 843 K, ■ 868 K, ▲ 918 K, ▼ 943 K. The triangle in the figure represents the line slope of 3 corresponding to the Coble theory (eqn (*1*)).

structural parameters. Viscous creep, *i.e.* linear dependence of creep rate on applied stress, was observed for stresses up to approximately 5 MPa for pure metals and solid solutions. For higher stresses the deviation from viscous flow to the stress exponent of about two was observed (Fig. 8). On the contrary, viscous creep in P-91 steel was detected in the wide range of stresses up to approximately 100 MPa at 873 K (Fig. 9).

DISCUSSION

In the present paper, the normal primary creep was observed to take place simultaneously with the Coble diffusional creep and/or the Harper—Dorn creep.

Primary Creep

Initial creep rates depend on testing conditions in

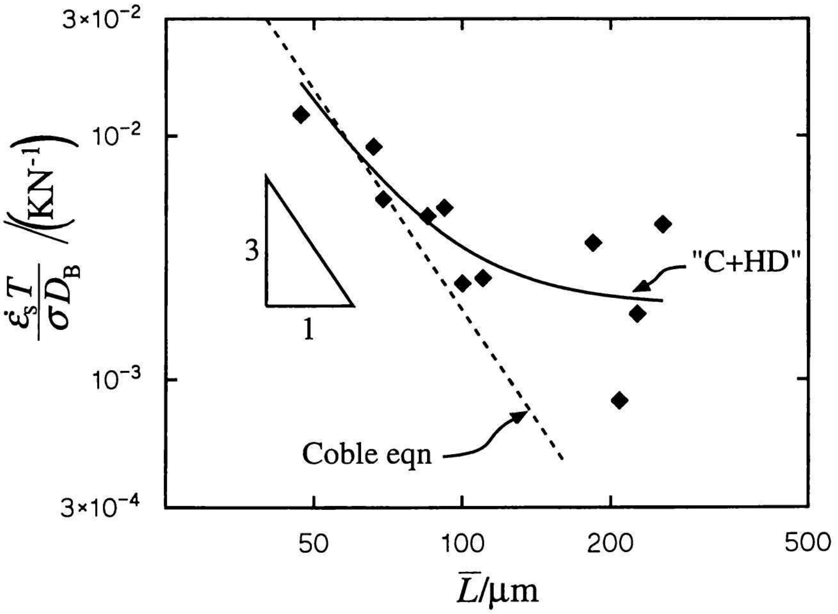

Fig. 7. Dependence of temperature and stress compensated creep rate on grain size for the Ni—15 % Cr primary solid solution. The triangle in the figure represents the line slope of 3 corresponding to the Coble theory (eqn (*1*)).

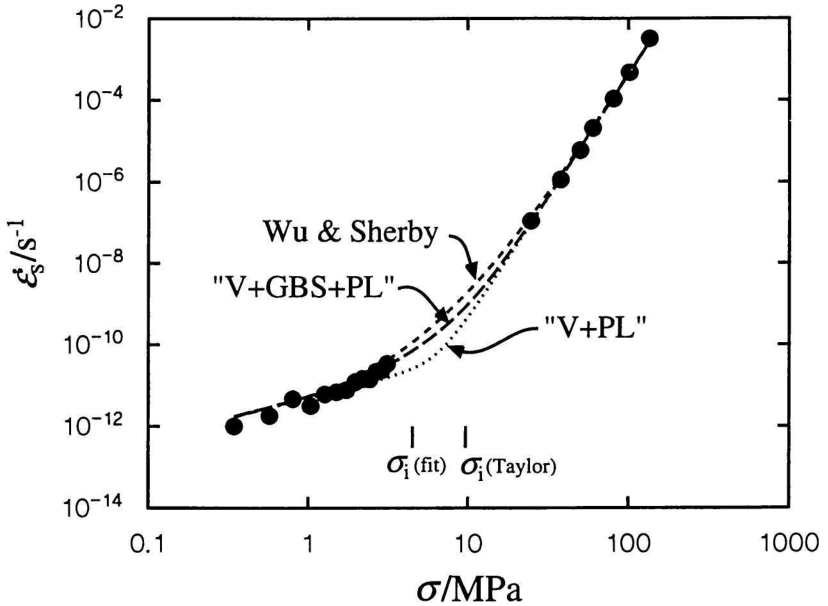

Fig. 8. The secondary creep rate dependence on stress for the Cu—6.5 % Al solid solution at 773 K. Data points in power-law region are taken from the paper of *Pahutová et al.* [31]. See the text for explanation of different curves and σ_i values.

the same way as secondary creep rates, which indicates similar deformation mechanisms in both stages. On the other hand, the above-mentioned mechanisms cannot fully explain primary creep. The following specific processes may be responsible for the primary creep: stress-induced grain growth [23], generation of local internal stresses in grains [24], emission and absorption of vacancies by subgrain boundaries [25], flexing of links of the three-dimensional dislocation network [26], and growth of characteristic dimension of a three-dimensional dislocation network associated with lattice dislocation density decrease in time [27].

Primary creep observed would require such increase in grain size, which exceeds many times the inaccuracy in measurement of grain size. Thus the grain growth during creep can be excluded as a source of primary creep deformation.

On the other hand, processes of generation of internal stresses and dislocation links bowing contribute without any doubt to the primary strain. Nevertheless, the normalized strain $\varepsilon_p/\varepsilon_e$ (where ε_e is an elastic strain) due to these processes is limited to the value

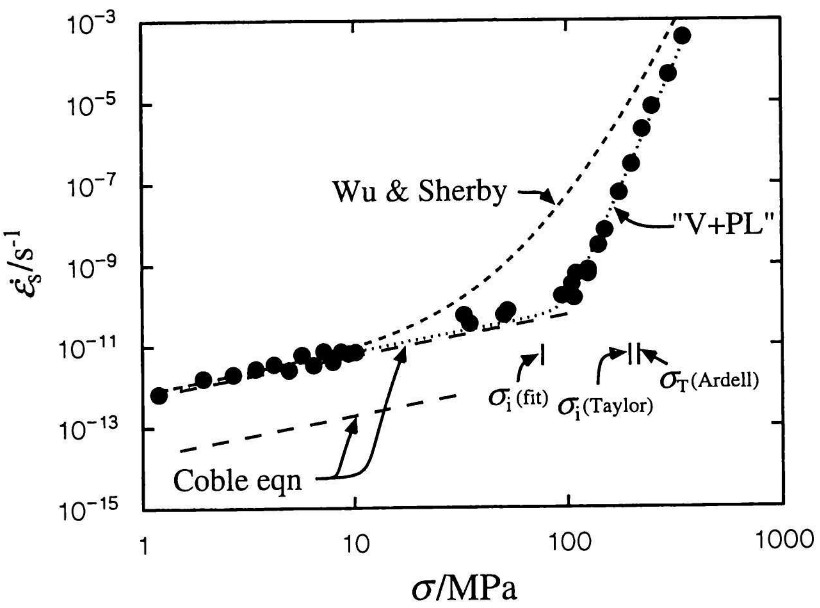

Fig. 9. The secondary creep rate dependence on stress for P-91 heat-resistant steel at 873 K. Data points in power-law region are taken from the paper of *Skleničkа et al.* [20]. See the text for explanation of different curves and σ_i, σ_T values.

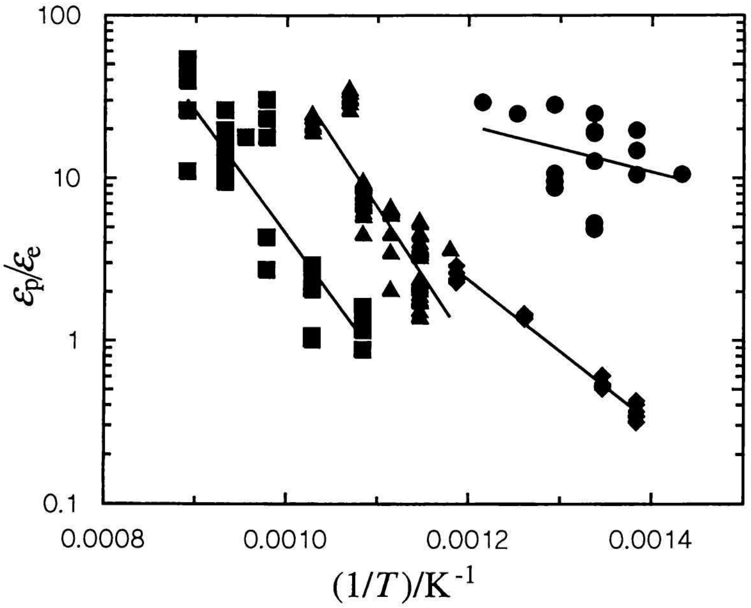

Fig. 10. Elastic strain normalized primary strain $\varepsilon_p/\varepsilon_e$ dependence on reciprocal temperature. ▲ P-91, ■ NiCr, ● CuAl, ◆ α-Fe.

of about 1.2. Since primary strains observed exceed many times this value (Fig. 10), the primary creep cannot be explained exclusively by these two mechanisms.

Subboundaries were observed rather rarely in pure metals and solid solutions. They disappeared during the deformation process. Their contribution to primary creep should be taken into consideration. However, the process of matter transport among subboundaries cannot explain the extent of primary creep observed in stated materials. On the other hand, the mechanism is probably responsible for the large primary strains in the P-91 heat-resistant steel. Nevertheless, it does not provide any explanation for the observed strong dependence of the $\dot{\varepsilon}_i/\dot{\varepsilon}_s$ ratio on temperature.

In spite of this fact no growth of the characteristic dimension of dislocation network can be fully excluded. The decrease in dislocation density needed for the strains observed can be below the TEM technique detection limits. If the lattice dislocations activity is responsible for the primary strain, the differences in crystal structure and in stacking fault energy can explain differences in the primary creep extent.

The main difficulty lies in interpretation of temperature dependence of primary creep strain measured (Fig. 10). On the contrary to this observation, all the above-mentioned models propose the primary strain to be independent of temperature. The only explanation may consist in a combination of some processes with different temperature dependence, if none of them is clearly dominant.

Secondary Creep

Grain Size Dependence

Viscous creep rate depends on reciprocal of the third power of grain size for the Coble diffusional creep (as can be seen from eqn (*1*)) or is independent of grain size for the Harper—Dorn dislocation creep (by its definition). Nevertheless, some indirect dependence of Harper—Dorn creep rate on grain size may occur when some other structural parameter is changed along with the grain size (for instance the dislocation density).

As it is obvious from Fig. 5, the grain size dependence of creep rate of the Cu—6.5 % Al solid solution is in a very good agreement with the Coble eqn (*1*). The creep rates measured are about two times higher than that predicted by the Coble theory. This difference may be accounted to the "grain size" problem mentioned above. Since no differences in dislocation density correlating with the grain size were found there is no reason to consider whether the viscous creep mechanism in the Cu—6.5 % Al solid solution is the Coble diffusional creep. It should be noted that in this particular case D_B coefficient was measured independently using the sample of the same material as was used for the creep tests. The usual problem of sensitivity of D_B to segregating impurities is then excluded.

Behaviour of both the α-Fe and Ni—15 % Cr solid solutions is not so simple and clear. Large scatter of creep rate values for coarser grain sizes replaced grain size dependence close to that required by the Coble creep theory and observed for fine grain sizes (up to about 120 μm). This behaviour was also observed for α-Zr [13], α-Ti [28], and β-Co [29]. It used to be interpreted as a transition from the diffusional creep to the Harper—Dorn creep, but attempts to identify the structural source of the scatter failed. Differences observed in dislocation density were not significant and did not correlate to the creep rate.

Though Figs. 6 and 7 look similar, there are two important differences, which should be noted. While the NiCr experimental creep rates for finer grain size region are in excellent agreement with those predicted by the Coble theory, for α-iron the experimental creep rates are more than two orders higher than the predicted ones. This difference is rather obvious for pure metals, but no explanation has been published until now. The other difference is that for NiCr the curve (labelled as "C + HD" in figures) represents measured data relatively well, and this is not true for α-iron. The curve was drawn under assumption that the diffusion and the Harper—Dorn creep mechanisms contribute independently to the rate.

Transition from Viscous to Power-Law Creep

Models of the Harper—Dorn creep by *Wu* and *Sherby* [9] and by *Ardell* [10] tried to unify the Harper—Dorn and the power-law creep as two sides of one deformation process. Thus, their main aim was to predict the transition stress or even the shape of $\dot{\varepsilon}_\mathrm{s}$ *vs.* σ curve. In the model of Wu and Sherby, the transition is given by the value of internal stress σ_i, which they derive from Taylor's relation $\sigma_\mathrm{i}/E \approx 0.4b\sqrt{\rho}$, where E is the Young's modulus, b is the length of Burger's vector, and ρ is the dislocation density. In the Ardell's model [10], the transient stress σ_T is also derived from dislocation density using a little bit more complicated formula.

The stress dependences of creep rate showing the transition from the viscous to the power-law creep are drawn in Fig. 8 for Cu—6.5 % Al and in Fig. 9 for P-91, respectively. In figures, the fitted creep curves according to Wu and Sherby are drawn along with the curves based on the assumption of independent contributions of viscous and power-law creep mechanisms. The latter curves are labelled "V + PL" For the Cu—6.5 % Al solid solution the curve according to Wu and Sherby represents experimental data better than that of independent creep mechanisms. If the third creep process with the stress exponent $n = 2$ (grain boundary sliding) is accepted, then the agreement becomes comparable to the model of Wu and Sherby. This curve is labelled "V + GBS + PL" Internal stresses σ_i derived from the fitted curve and values calculated from the Taylor's relation can be seen in the same figure, too. The calculated value is about twice as high as that derived from the experimental data, which can be considered as acceptable approximation. Nevertheless, it is necessary to point out that the viscous creep mechanism in the Cu—6.5 % Al alloy is the Coble diffusional creep, as was demonstrated above. Thus the fact that the curve according to Wu and Sherby can be fitted successfully on the experimental data does not provide any information about the viscous creep mechanism.

The situation is quite different in Fig. 9 for P-91 steel. Transition stress is much higher and the transition is relatively sharp. The model of Wu and Sherby does not fit experimental values well, while the curve labelled "V + PL" does. Both the Taylor's relation and the Ardell's model are capable to predict high transition stresses.

Two lines for the Coble theory prediction are drawn in Fig. 9. The reason is that the literature values of $D_\mathrm{B}\delta_\mathrm{B}$ product vary in a wide range, so minimum and maximum values were used. If the maximum value is accepted, the agreement between the experimental and the calculated creep rates is excellent. Since the grain size dependence cannot be tested for this material, the agreement itself cannot be considered as an evidence for the Coble creep. It indicates only that the Coble diffusional mechanism is a possible deformation mode in the viscous creep region of the P-91 heat-resistant steel.

CONCLUSION

Primary creep was found in all tested materials except for the pure α-zirconium. The initial creep rate depends on test conditions in the same way as the secondary creep rate. The high dislocation density and small subgrain size is probably responsible for the large strain and high initial rate in the P-91 heat-resistant steel. Nevertheless, none of primary creep mechanisms under consideration is capable to explain all the features observed in primary stage. The temperature dependence of normalized primary strain was observed in all tested materials, which contradicts all the theories published until now.

From the presented data it can be concluded for secondary creep that viscous creep behaviour of various materials under similar conditions is not consistent. Its features can be summarized as follows:

Grain size dependence of creep rate fulfils the requirements of diffusional creep theories below some critical grain size $\overline{L}_{\mathrm{crit}} \approx 120\ \mu\mathrm{m}$.

Above critical grain size the dependence is replaced by data large scatter of about one order of magnitude for all materials except for the Cu—6.5 % Al solid solution. The source of this scattering has not been discovered yet.

The transition from viscous to power-law creep is smooth for pure metals and solid solutions and relatively sharp for the P-91 steel.

The transition stress is much higher in the P-91 steel than in pure metals and solid solutions. Both Wu and Sherby's and Ardell's models are able to explain this difference on the basis of different dislocation density.

Experimental creep rates are in good or acceptable agreement with those predicted by the Coble theory for solid solutions and steel, but they are much higher for pure metals.

The following important consequences can be derived for the evidences of particular viscous creep mechanisms:

The grain size dependence below the critical grain size seems to be strong evidence for the diffusional mechanism, but when the source of scatter remains unknown above critical grain size, it cannot be excluded from possible influence below the critical grain size. Thus the above evidence cannot be considered as conclusive.

The curve of transition from viscous to power-law creep can be fitted well on the data of the Cu—6.5 % Al solid solution, where the viscous creep is most probably the Coble diffusion creep. On the other hand, it fails for the P-91 steel, where the Harper—Dorn creep can be easily assumed. The fact that the curve can be fitted on the experimental data then provides no evidences for particular creep mechanism. This conclusion was published recently by *Wang* [30], too.

Similar conclusion as above can be drawn for the predictions of transient stress.

No current model is capable to explain all apparently contradictory phenomena of viscous creep, even under limited conditions. In author's opinion, both better knowledge of random grain boundaries structure and all processes running inside them is needed, as well as the knowledge of processes within the whole dislocation network.

Acknowledgements. One of the authors (L. K.) gratefully acknowledges financial support from the Grant Agency of the Academy of Sciences of the Czech Republic (# A 2041702).

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