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Constitutive description of creep in silicon iron at high temperatures

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Primary and steady state creep in a Fe–3 wt% Si solid solution was investigated in the temperature interval 823–973 K at applied stresses which resulted in minimum creep rates of 10^{-7}–$10^{-3}\,\mathrm{s}^{-1}$. The creep curves were described by the McVetty equation and the dependence of its parameters on external variables was discussed. For a constitutive description of the curves based on the choice of the internal stress as a single internal variable, a set of constitutive equations that include the experimentally found relation between the creep rate and the internal stress were suggested. Consequences resulting from this suggestion for the internal stress values which follow from the analysis of the creep curves were discussed.

1. Introduction

The equations that describe creep behaviour can be obtained by integrating the constitutive equations, which describe the kinetics of the process, and the evolution of internal variables [1]. The internal variables should reflect the structural state of the material and its influence in the deformation process. These demands are probably fulfilled by the internal stress, which is generated by the structure and opposes the process of straining. Internal stress resulting from contributions of strain hardening and recovery seems to be an appropriate internal variable of the primary creep [2]. A macroscopic mean value of the internal stress can be obtained experimentally in the conditions of creep, for example by the method of Ahlquist and Nix [3]. It has proven to be an adequate measure of the internal stress acting in the creep process [4] and corresponds to structure data [5]. The possibility of a direct measurement of the internal stress gives a chance to gain independent information on real values of this internal variable.

When the influence of the materials structure is described by internal stress as a single internal variable, only two equations are required for the constitutive description: a kinetic equation relating the strain rate to external variables and to the internal stress, and an evolution equation of the internal stress.

The aim of the present work is to discuss the constitutive description of high temperature creep leading to the McVetty equation [6] of the creep curve,

$$\varepsilon = \varepsilon_0 + \dot{\varepsilon}_s t + \varepsilon_T[1 - \exp(-t/\tau)] \qquad (1)$$

In Equation (1), ε is the total strain in the time t, ε_0 is the initial strain resulting from loading, ε_T the strain reached by the primary transient creep process, τ the relaxation time of the primary transient creep and $\dot{\varepsilon}_s$ the steady state creep rate. Equation 1 is often a rather good approximation of the creep curve in its primary and steady state stages [7]. As a single internal variable the internal stress is taken and the kinetic equation is chosen in a form respecting the real stress sensitivity of the creep rate observed in the experiment. A fulfilment of the last demand leads to a formulation of more general constitutive equations, admitting an arbitrary value of the stress sensitivity parameter of the creep rate.

The choice of silicon iron as the experimental material was motivated by a projected investigation of some microstructural aspects of creep and the ease of microstructural investigation of this material by etch pitting. Results of this investigation will be published in another paper. The present study on a constitutive description of creep in the alloy was performed to obtain an objective understanding of the creep curve characteristics.

2. Experimental procedure

The experimental material was Fe–3 wt% Si solid solution of technical purity, which was cold rolled and annealed in the initial state. A final annealing of 4 h at 1323 K in a hydrogen atmosphere lead to a homogeneous microstructure with grains elongated in the direction of rolling, the average size being 193 μm and the size aspect ratio was 1.92. The dislocation density in the grains did not exceed $1 \times 10^{12}\,\mathrm{m}^{-2}$.

The creep specimens, 50 mm in gauge length and $3.5 \times 3.0\,\mathrm{mm}^2$ in cross-section, with their longitudinal axis parallel to the rolling direction, were subjected to creep under a hydrogen atmosphere at temperatures of 823, 873, 923 and 973 K and applied stresses to which minimum creep rates within the interval from 10^{-7}–$10^{-3}\,\mathrm{s}^{-1}$ correspond. The creep curves were

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obtained in the form of a digital recording of experimental points $\varepsilon(t)$ equidistant in the true strain range up to 0.35 (limited by the range of the creep machine). About 70–350 data points were available for each curve. The reproducibility of the creep curves judged on the basis of a comparison of five curves obtained at $T = 873$ K and $\sigma = 100$ MPa was very good. In none of the creep tests performed over the range of creep conditions mentioned previously was a pronounced tertiary stage, or a fracture produced.

The creep curves were fitted to the McVetty equation by means of an optimization procedure using the least squared deviations of strain as a criterion.

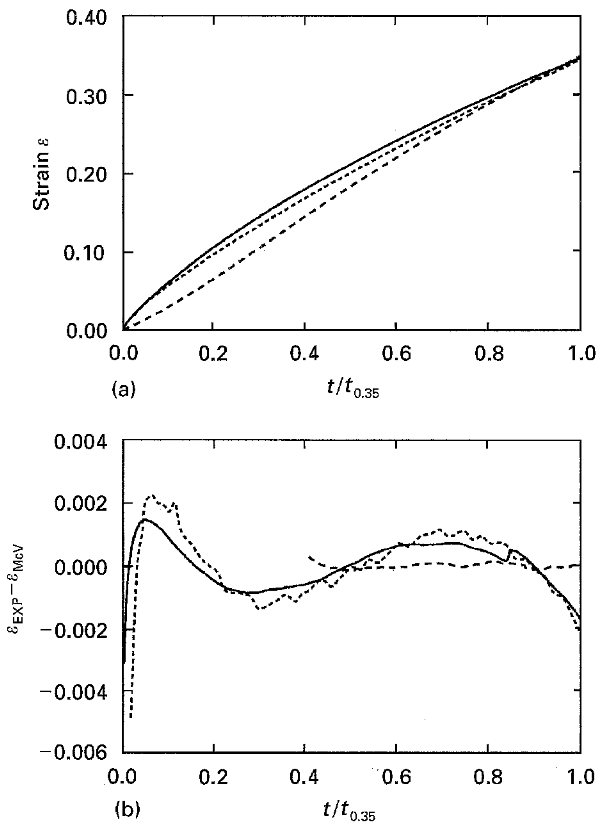

Figure 1 Typical creep curves of the Fe–3wt % Si solid solution taken at 923 K (a): a sigmoidal ("alloy") curve at low applied stress. ((– – –), $\sigma = 30$ MPa) and normal ("metal") primary creep curves at higher applied stresses ((—) $\sigma = 75$ and (- - - - -) $\sigma = 150$ MPa); (b): their deviation from the fitted McVetty curve.

3. Results and discussion

3.1. Creep curves and the McVetty equation

Typical creep curves of the material are shown in Fig. 1a in coordinates of true strain ε versus relative time of testing $t/t_{0.35}$ ($t_{0.35}$ is time to reaching the strain $\varepsilon = 0.35$, i.e., 5.64×10^5 s at the applied stress $\sigma = 30$ MPa, 5.10×10^3 s at $\sigma = 75$ MPa and 7.32×10^1 s at $\sigma = 150$ MPa). At the lowest σ at each temperature, the material shows as a rule "alloy" behaviour [8], i.e., the creep curves have a sigmoidal character, they start with an incubation period for the accelerated creep, which after some time is followed by a normal primary stage. At higher applied stresses the material behaves as a "metal" [8], i.e., the normal primary stage exists from the beginning of the creep process. A relatively small decrease of the creep rate indicates a small degree of hardening of the material during creep. The initial elongation is also very small over the whole range of the applied stress.

The normal primary creep curves and the normal primary parts of the sigmoidal creep curves could be fitted to the McVetty equation within standard deviations of the strain of about $\cong 0.001$, the worst case was 0.003. Fig. 1b shows typical "undulations" of the experimental curve $\varepsilon_{\mathrm{EXP}}(t/t_{0.35})$ around the fitted McVetty curve $\varepsilon_{\mathrm{MCV}}(t/t_{0.35})$. The McVetty equation overestimates the value of strain at the start of the primary stage by overestimating the initial elongation ε_0 and then letting the creep strain grow with a rate significantly lower in comparison with the experimental data. At the end of the tested interval of strain, a contribution of the tertiary stage process, which has not been included in the present analysis, can play a role. The creep curves obtained under high applied stresses show further small deviations across the whole strain range, corresponding to fluctuations of the strain rate during the rapid creep test.

We can conclude that a substantial part of the creep curves, at least in the interval between 0.1–0.9 $t/t_{0.35}$ can be approximated rather well (i.e., within an acceptable standard deviation) by the McVetty equation.

3.2. Parameters of the creep curves and constitutive equations

McVetty's equation, Equation 1, expresses the time dependence of the total strain ε in terms of four phenomenological parameters, which can be evaluated as independent characteristics of the curve. The corresponding equation for the creep rate may introduce alternative parameters, dependent on the above mentioned values: the maximum transient creep rate

$$(\dot{\varepsilon}_{\mathrm{T}})_{\max} = \varepsilon_{\mathrm{T}}/\tau \tag{2}$$

or a dimensionless parameter

$$A = \varepsilon_{\mathrm{T}}/(\dot{\varepsilon}_{\mathrm{s}}\tau) \tag{3}$$

The last expression is closely related to the internal stress in a constitutive description which uses the internal stress as a variable. The kinetic equation is then based on the back stress concept [8, 9], which supposes the strain rate to be controlled by the difference $(\sigma - \sigma_{\mathrm{i}})$, which expresses the point that the internal stress σ_{i} opposes the deformation process. The simplest form of the kinetic equation is based on a power relationship between $\dot{\varepsilon}$ and $(\sigma - \sigma_{\mathrm{i}})$ and can be written in the form

$$\dot{\varepsilon} = \dot{\varepsilon}_{\mathrm{s}}\left(\frac{\sigma - \sigma_{\mathrm{i}}}{\sigma - \sigma_{\mathrm{is}}}\right)^{m^*} \tag{4}$$

Note that with $m^* = 1$ Equation 4 includes also an approximation of the $\dot{\varepsilon} \propto \sinh[B(\sigma - \sigma_{\mathrm{i}})]$ proportionality (B is a parameter in MPa^{-1}), acceptable for a small value of the argument, $B(\sigma - \sigma_{\mathrm{i}}) \leq 1.5$. The evolution equation for the internal stress, which with Equation 4 and $m^* = 1$ leads to the McVetty equation, is [7]

$$\dot{\sigma}_{\mathrm{i}} = (\sigma_{\mathrm{is}} - \sigma_{\mathrm{i}})/\tau \tag{5}$$

The parameter σ_{is} is the steady state value of the internal stress reached by definition in the steady state stage of creep under an applied stress σ. The parameter A can then be expressed as

$$A = \frac{\sigma_{is} - \sigma_{i0}}{\sigma - \sigma_{is}} \tag{6}$$

where σ_{i0} is the initial value of σ_i corresponding to $t = 0$. Equation 5 combined with Equation 4 leads to [7]

$$\dot{\sigma}_i = h\dot{\varepsilon} - r \tag{7}$$

where h is the coefficient of strain hardening,

$$h = \left(\frac{\partial \sigma_i}{\partial \varepsilon}\right)_{t=\text{const.}} = (\sigma - \sigma_{is})/(\dot{\varepsilon}_s \tau) \tag{8}$$

and r is the recovery rate,

$$r = \left(\frac{\partial \sigma_i}{\partial t}\right)_{\varepsilon=\text{const.}} = (\sigma - \sigma_{is})/\tau \tag{9}$$

Evidently, in our procedure, both h and r are constant in the course of the creep curve.

Regarding the kinetic Equation 4, objections can be sometimes raised against the weak stress dependence of the creep rate given by the choice of the exponent $m^* = 1$. We can propose an alternative evolution equation for the internal stress that with Equation 4 will represent a set of more general constitutive equations leading to the McVetty equation and admitting an arbitrary value of m^*:

$$\dot{\sigma}_i = \frac{1}{m^*\tau}(\sigma - \sigma_i)\left[1 - \left(\frac{\sigma - \sigma'_{is}}{\sigma - \sigma_i}\right)^{m^*}\right] \tag{10}$$

By analogy to Equation 6, the equation relating the parameter A to the stress parameters has the form

$$A = \left(\frac{\sigma - \sigma'_{i0}}{\sigma - \sigma'_{is}}\right)^{m^*} - 1 \tag{11}$$

Equation 10 with $m^* \neq 1$ cannot be easily rewritten to the form of Equation 7 and the coefficient of strain hardening h' and the recovery rate r' cannot be derived uniquely. The closer approximation to reality of the kinetic equation created by admitting the experimental value of m^* in Equation 4 is paid for by a more complicated evolution equation for σ_i. The physical meaning of Equation 10 is probably more apparent in its form

$$\frac{\dot{\sigma}_i}{\sigma - \sigma_i} = \frac{1}{m^*\tau}\left(1 - \frac{\dot{\varepsilon}_s}{\dot{\varepsilon}}\right) \tag{12}$$

obtained by combining it with Equation 4: a relative change of the effective stress $\sigma^* = \sigma - \sigma_i$ with time is a linear function of the reciprocal creep rate $\dot{\varepsilon}$, scaled by $1/m^*$.

3.3. Creep data

Fitting of the creep curves to Equation 1 yielded values for the parameters in Equation 1 from which the other parameters defined by Equations 2–11 could be derived. We now summarize their relations to the external variables (applied stress σ and temperature T) and some relations between the parameters themselves.

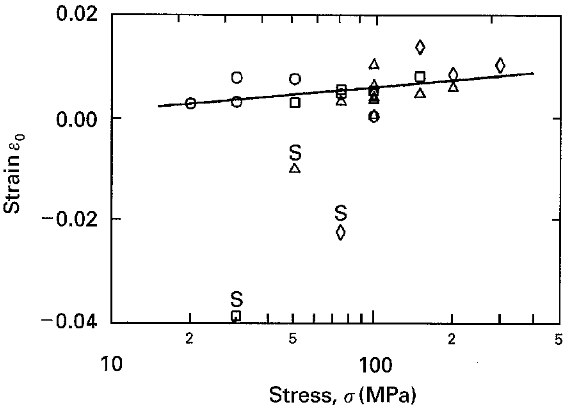

Figure 2 Initial elongation ε_0 plotted as function of the applied stress σ. The points S give hypothetical data of sigmoidal curves. The experimental data were recorded at, (◇) T = 823, (△) T = 873, (□) T = 923 and (○) T = 973 K.

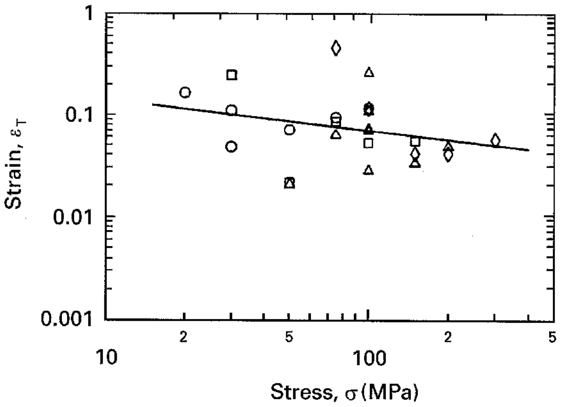

Figure 3 Applied stress dependence of the transient strain ε_T. The data were recorded at (◇) T = 823, (△) T = 873, (□) T = 923 and (○) T = 973 K.

The initial strain ε_0 resulting from initial elongation was found to grow very slightly with the applied stress, Fig. 2. To sigmoidal creep curves starting with an incubation period a negative value of ε_0 was formally ascribed by fitting their normal primary section to Equation 1.

The transient creep strain ε_T represents about 0.25–0.33 of the total creep strain ε reached within the test and slightly decreases with the applied stress, independent of temperature as is shown in Fig. 3.

The relaxation time τ of the transient process and/or the maximum transient strain rate $(\dot{\varepsilon}_T)_{MAX}$ and the rate of recovery r show similar dependences on the external parameters as the steady state strain rate $\dot{\varepsilon}_{s'}$ which can be generally expressed in the form

$$[\dot{\varepsilon}_{s'}(\dot{\varepsilon}_T)_{MAX}, 1/\tau, r] = C\sigma^n \exp\left(-\frac{Q}{RT}\right) \tag{13}$$

The exponent n and the activation energy Q, which for $\dot{\varepsilon}_s$, $1/\tau$ and r at least at the three higher temperatures and for $(\dot{\varepsilon}_T)_{MAX}$ in the whole temperature

TABLE I Values of the applied stress sensitivity parameter n and apparent activation energy Q in eq. (13)

Parameter of creep curve	n	Q [kJ mol^{-1}]
$\dot{\varepsilon}_s$	5.70 ± 0.08	373 ± 8
$(\dot{\varepsilon}_T)_{MAX}$	5.68 ± 0.17	382 ± 14
$1/\tau$	5.67 ± 0.24	390 ± 24
r	6.82 ± 0.26	371 ± 26

interval can be evaluated as constants independent of σ and T, have rather similar values - see the summary in Table 1. Values of the exponent n correspond to the value $n \approx 5$ typical for "metal" creep behaviour [8], the activation energy is higher than the value characterizing the lattice diffusion in alpha iron, $\Delta H_{SD} = 255$ kJ mol^{-1}, in the corresponding range of temperatures [10, 11]. A higher slope in the plots of Fig. 4(a–d) showing the stress dependences of data corresponding to the temperature 823 K - could possibly be understood as an expression of a different, applied stress dependent, activation energy Q in the temperature range 823–873 K. Alternatively it may indicate another temperature dependence influencing the factor C. Similar dependences of parameters of the transient and steady state creep component on external variables of the creep process may be understood as an indication that both the components are controlled by the same microstructural mechanism.

The dimensionless parameter A, plotted against the stress in Fig. 5, shows no pronounced temperature dependence and only a weak tendency to decrease with increasing applied stress. Values of A can be used to evaluate the steady state internal stress σ_{is} whenever the initial internal stress σ_{i0} is known. Unfortunately, the last value cannot be derived from any results of the present computations. Nor can it be easily determined experimentally. It is evident, that within the present constitutive description, the same creep behaviour is obtained for any combination of σ_{is} and σ_{i0} giving the same value of A. Thus, evolutions of internal stress in various intervals (σ_{i0}, σ_{is}), to which

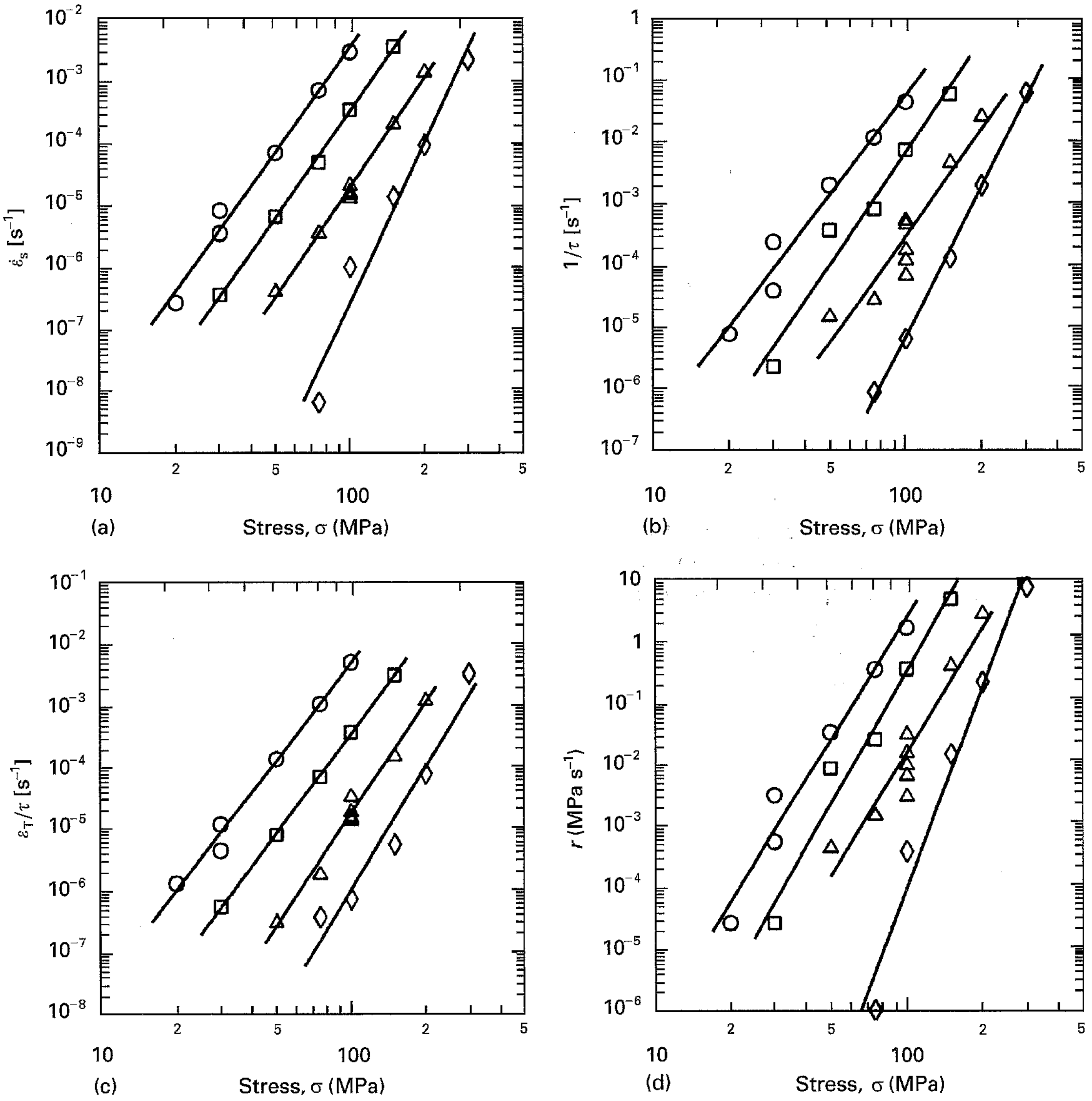

Figure 4 Dependences on applied stress and temperature of the creep curve parameters (a) steady state creep rate $\dot{\varepsilon}_{s'}$, (b) reciprocal value of the relaxation time τ of the transient creep, (c) maximum transient creep rate ε_T/τ, and (d) recovery rate r. The data were recorded at (◇) T = 823, (△) T = 873, (□) T = 923 and (○) T = 973 K and $m^* = 1$.

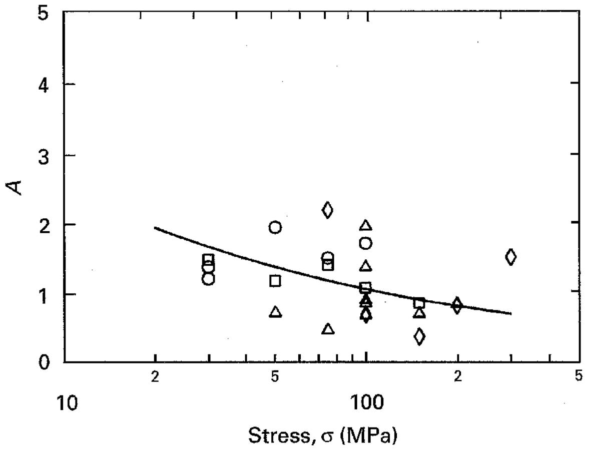

Figure 5 Parameter A of Equation 3 plotted versus applied stress at the temperatures T; (◇) 823, (△) 873, (□) 923 and (○) 973 K.

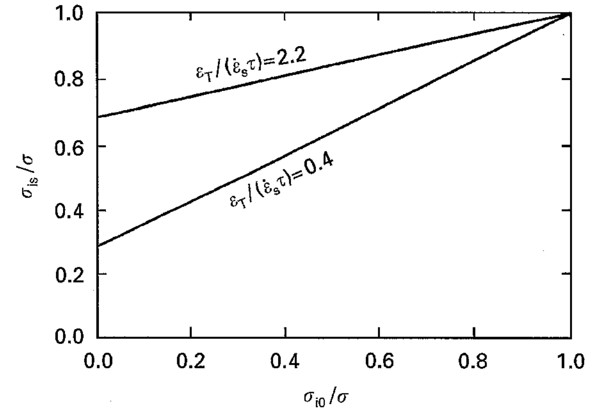

Figure 6 Influence of initial internal stress value σ_{i0} on the value of steady state internal stress σ_{is} in the steady state creep. The value of $m^* = 1$.

different ranges of structures should correspond, may result in the same creep curve. This fact may introduce some doubt on the instantaneous internal stress as a relevant internal variable. Another problem, important for the value of σ_{i0}, is introduced by the fact that the experimental creep curve deviates from the McVetty equation in the initial stage of creep, $t \leq 0.1t_{0.35}$. The values of σ_{i0} mentioned above are thus hypothetical values corresponding to the McVetty equation extrapolated to $t = 0$.

Fig. 6 illustrates (for $m^* = 1$) the effect of the value of σ_{i0} on the value of σ_{is}. In accord with Equations 6 and 11 the steady state internal stress σ_{is} is linearly dependent on σ_{i0} and the choice of $\sigma_{i0} = 0$ leads to minimum values of σ_{is}, i.e. $(\sigma_{is})_{MIN}$ representing the lower limit of this value in the present structure.

In accord with Equations 8 and 9 the recovery rate r and the strain hardening coefficient h represent upper limits of these parameters if $(\sigma_{is})_{MIN}$ has been used for their evaluation. The strain hardening coefficient seems to be independent of temperature as is shown in Fig. 7.

We will now compare the internal stress values calculated from a constitutive description with available experimental data.

For the evaluation of internal stress σ'_{is} from Equation 11, the value of m^* can be taken from experimental data of two creep curves, during which the evolution of the internal stress was measured by a special experimental technique [12]. Curve (a) gives values of $T = 823$ K and $\sigma = 175$ MPa, and curve (b) $T = 973$ K and $\sigma = 45$ MPa. Detailed data and the analysis of the evolution of the internal stress are discussed elsewhere [13]. The power law dependences of the creep rate $\dot{\varepsilon} \propto (\sigma - \sigma_i)^{m^*}$ give values of $m^* \approx 3$ and 1.8, respectively, for curves (a) and (b). These values can be compared with the result of Solomon and Nix [14], of $m^* = 2$, found for the stress dependence of both the primary and the steady state creep rate in the same alloy and internal stress measured by the strain transient dip test technique of Ahlquist and Nix [3].

In Fig. 8, values of the minimum internal stress in the steady state creep $(\sigma_{is})_{MIN}$ evaluated from Equation 11 for $m^* = 1$, 2 and 3 are plotted as functions of the applied stress σ. Apparently, the $(\sigma_{is})_{MIN}$ versus σ dependence can be approximated by a single straight line corresponding to a simple proportionality between $(\sigma_{is})_{MIN}$ and σ, independent of temperature. This result is in qualitative accord with the experimental data of Pahutová *et al.* [15]. By increasing the value of m^* we obtain lower values of $(\sigma_{is})_{MIN}$ and, consequently, a lower slope of the proportionality line, for values of $m^* = 1$, 2 and 3, the corresponding values of $(\sigma_{is})_{MIN}/\sigma$ are 0.50, 0.29 and 0.20 respectively.

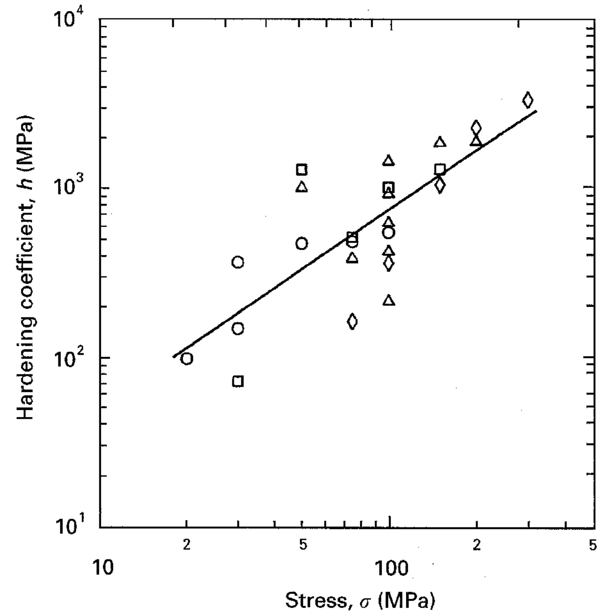

Figure 7 Applied stress and temperature dependence of the coefficient of strain hardening h. The data were recorded at (◇) T = 823 K, (△) T = 873 K, (□) T = 923 K and (○) T = 973 K.

The above ratios are lower than the experimental data on the ratio σ_{is}/σ measured by Orlová *et al.* [13] at the steady state stage of the creep curves (a) and (b), i.e. 0.58 and 0.62, respectively. If we extend this result as an assumption to the whole set of our curves by assuming $\sigma_{is}/\sigma = 0.60$, Equations 6 and 11 will yield non-zero average values of σ_{i0}, i.e. $\sigma_{i0}/\sigma = 0.21$, 0.44 and 0.50 for $m^* = 1$, 2 and 3, respectively. The latter two values are in rather good agreement with the

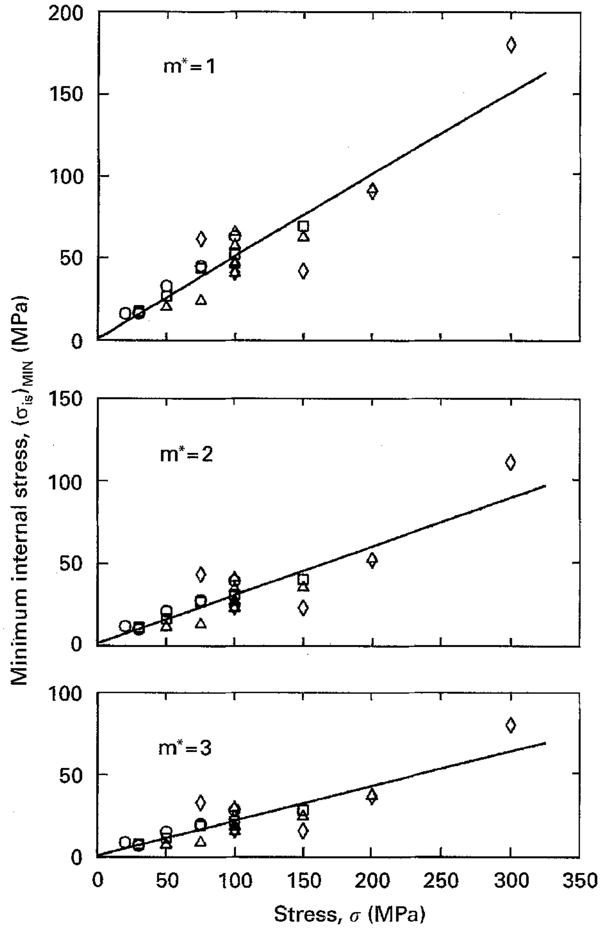

Figure 8 Applied stress dependence of the minimum internal stress $(\sigma_{is})_{MIN}$ evaluated from creep curve parameters for different values of exponent m^* in the kinetic equation 4. The data were recorded at (◇) T = 823, (△) T = 873, (□) T = 923 and (○) T = 973 K.

results of analysis of experimental data on the curves (a) and (b) of Orlová *et al.* They note however that there are alternative methods to Equations 5 and 10 to describe the evolution of the internal stress.

4. Conclusions

High temperature creep in a Fe–3 wt% Si solid solution can be described by means of the McVetty equation with parameters dependent on the applied stress and temperature. The manner of this dependence suggests that both the transient and the steady state component of the primary creep process are controlled by the same microstructural mechanism.

For a constitutive description taking the internal stress as internal variable, a set of equations admitting the power law effective stress dependence of the primary creep rate was suggested. The price for the fact that the kinetic equation can now be chosen to correspond to experimental data is paid for by a more complicated evolution equation for the internal stress. The minimum internal stress value in the steady state creep stage which is deduced from creep curve parameters is proportional to the applied stress and is independent of temperature. It decreases with increasing the exponent in the kinetic equation, i.e. for a higher effective stress sensitivity of the creep rate. The internal stress evolution probably starts from a nonzero initial stress, which should exist in the material at the beginning of creep.

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