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# EFFECT OF CREEP STRAIN ON CREEP RATE IN THE TEMPERATURE RANGE 550–640 °C

## VPLIV DEFORMACIJE NA HITROST LEZENJA PRI TEMPERATURAH OD 550 °C DO 640 °C

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Experimental and calculated creep rates were examined for a high-chromium, creep-resistant steel samples quenched and tempered for 2 h and 400 h at 800 °C. Creep testing and annealing were carried out at 550 °C to 640 °C. It was found that the difference in the creep rates due to the particle coarsening and dissolution was independent of the temperature, while the ratio of the experimental and calculated creep rates increased with the temperature of the creep tests. The results suggest that an increase in the creep rate is related to the process of vacancy climb in parallel with the increase in the diffusion rate of the iron in the ferrite.

Keywords: creep-resistant steel, $M_{23}C_6$ particle coarsening and dissolution, experimental and calculated creep rates, effect of temperature

Hitrost lezenja je bila eksperimentalno določena in izračunana za jeklo, odporno proti lezenju, z visoko vsebnostjo kroma pri vzorcih, ki so bili kaljeni in popuščani 2 h oz. 400 h pri 800 °C. Stourni preizkusi lezenja in žarjenja so bili izvršeni pri 550 °C do 640 °C. Razlika v hitrosti lezenja zaradi raztapljanja oziroma rasti izločkov je bila neodvisna od temperature, medtem ko je razlika med eksperimentalno in izračunano hitrostjo lezenja večja pri višji temperaturi. Rezultati meritev in izračunov kažejo, da je povečanje hitrosti lezenja povezano z večjo hitrostjo plezanja vrzeli, ki je vzporedno s povečanjem hitrosti difuzije železa v feritu.

Ključne besede: jeklo, odporno proti lezenju, rast in raztapljanje izločkov $M_{23}C_6$, eksperimentalna in izračunana hitrost lezenja, vpliv temperature

## 1 INTRODUCTION

The microstructure of creep-resistant steels consists of a ferrite matrix with different elements, mostly chromium in solid solution, and particles, mostly carbides and carbonitrides, with sizes in the range from a few nm to about $1 \cdot 10^3$ nm. Those particles are unshearable obstacles to the gliding of the matrix mobile dislocations. For a particle size ($d_p$) greater than the spacing (mutual distance) of the mobile dislocations, the particles intercept the mobile dislocations with a probability $p \geq 1$, while for smaller particles the intercept probability is $p \leq 1$.[1] So far, it has not been determined whether the unshearability is related to a minimum particle size, i.e., the number of carbide molecules in the particle. For example, the lattice parameter ($l_p$) of the cubic carbide $Cr_{23}C_6$ is $l_p = 1.016$ nm,[2] and the number of molecules in the particles is $N_{pm} \approx d_p^3/l_p$. The number increases rapidly with the particle size: it is $N_{pm} = 8$ for particles with $d_p = 2$ nm and $N_m = 125$ for $d_p = 5$ nm. With isothermal tempering, the average particle size increases and their number decreases, as a result of the duality of the particle-coarsening process: the growth of coarser particles, the shrinking of particles with an intermediate size and even the dissolution of smaller particles.[1] This means that by tempering, the average size of the particles increases, and it increases more rapidly during a creep test, as the iron self-diffusion and the chromium diffusion in ferrite are enhanced by tensile stress.[3,4]

For creep-resistant steels, the improvement of Hornbogen's creep equation[5,6] for a uniform distribution of particles was proposed, with the substitution of the particle spacing $\lambda$ by $(\lambda - d)$ as a measure of the dislocation climb length increase by overcoming of particles inclusion of a constant representing the increase of the stress exponent $n$ from $n = 2$ to $n = 3.65$:

$$\dot{\varepsilon} = \frac{k_\sigma \cdot b^2 \cdot \sigma^n \cdot \rho \cdot D \cdot (\lambda - d)}{k_b \cdot T \cdot G} \quad (1)$$

where $\sigma$ is the acting stress ($\sigma = 170$ MPa), $\rho$ is the density of the mobile dislocations ($\rho = 0.978\ 10^{14}\ m^{-2}$), $D$ is the diffusion coefficient, $d$ is the average particle size, $\lambda_p$ is the average particle spacing, $b$ is the Burgers vector ($b = 2.5 \cdot 10^{-10}$ m), $k_b$ is the Boltzmann constant ($k_b = 1.38 \cdot 10^{-23}\ J\ K^{-1}$), $T$ is the temperature, $G$ is the shear modulus[7] and $k_\sigma = \sigma^{3.65}/\sigma^2 = 4.78 \cdot 10^3$.

By substituting the molar content of carbide $M_{23}C_6$ with the molar content of the chromium in solution in ferrite as a parameter in the LSW (Livshitz-Slyozov-Wagner) equation, a reasonable agreement was obtained for the calculated (Eq. 2) and experimental coarsening rate of the $M_{23}C_6$ particles at 800 °C. Also, Eq. 3 was deduced for the calculation of the coarsening rate at lower temperature:[8]

$$d_t^3 - d_0^3 = \left( \frac{8 \cdot S \cdot \gamma \cdot \Omega \cdot D}{9 \cdot k_b \cdot T} \right) \cdot t \qquad (2)$$

$$k_{cT} = k_{c,1073} \left( \frac{D_{Cr,T} \cdot 1073}{D_{Cr,1073} \cdot T} \right) \qquad (3)$$

where $d_t$ is the particle size at the tempering time $t$, $d_0$ is the initial particles size, $D$ is the chromium diffusion rate, $T$ is the tempering temperature, $S$ is the atomic content of chromium in solid solution in the ferrite, $\gamma$ is the carbide particle matrix interfacial energy ($\gamma$ = 0.37 J m$^{-2}$), $\Omega$ is the volume of diffusing atoms, $D$ is the chromium diffusion rate ($D = D_0 \exp(-Q/RT)$) with $D_0$ = 3.7 · 10$^{-3}$ m$^2$ s$^{-1}$ and $Q$ is the activation energy ($Q$ = 267 kJ mol$^{-1}$),[9] $k_b$ is the Boltzmann constant and $k_{c1073}$ is the experimental coarsening rate at 800 °C ($k_{c1073}$ = 2.94 · 10$^{-27}$ m$^3$ s$^{-1}$).

In this work, the effect of the creep strain on the change of particle size and spacing is investigated with a constant creep stress and time.

## 2 CALCULATIONS

The creep rate and creep fracture of creep-resistant steels depend of a large number of variables,[10] because with the creep temperature, the initial microstructure of the ferrite and the uniform distribution of particles is changed due to particle coarsening. With this coarsening, several processes occur, i.e., the growth of the particles' average size and spacing, the dissolution of small particles, the decrease in the number of particles and of grain-boundary stringers.[10] The change in the particles size and spacing is calculated for 100 h of tempering at 550–640 °C in steps of 30 °C using Eqs. 2 and 3. Assuming the particles to be spheres, the number of particles $N_p$ was calculated by applying the series:[1]

$$f = \frac{\pi}{6} \cdot \sum_1^n d_1^3 + d_2^3 + d_3^3 + \ldots + d_n^3 = \frac{\pi}{6} N_p d_a^3$$

$$\text{and } N_p = \frac{6f}{\pi d_a^3} \qquad (4)$$

where $f$ is the volume of particles, $N_p$ is the number of particles, $d_1$, $d_2$, $d_3$,.... $d_n$ is the decreasing size of the particles and $d_a$ is the average particles size. The volume of particles in the investigated steel was $f$ = 0.047 [10] and it was determined that the particles were carbide $M_{23}C_6$ ($Cr_{18}Fe_3Mo_2C_6$).[11,12]

The particles dissolution velocity was deduced as:[1]

$$d_d^3 - d_0^3 = k_{td} \qquad t_d = \frac{d_d^3 - d_0^3}{k_c} \qquad v_d = \left( \frac{d_d^3}{t_d} \right)^{1/3} \qquad (5)$$

where $d_d$ is the size of the dissolving particles, $k_c$ is the isothermal coarsening rate (the term in parenthesis in Eq. 2), $t_d$ is the dissolution time, $d_0$ = 2 nm and, $v_d$ is the dissolution velocity.

The volume of the particles increases with $d_d^3$, and for this reason the parameter $d_0$ = 2 nm was omitted from the calculations of the dissolution velocity.

The initial average particle size $d_{ia}$ was deduced for the tempering of specimens quenched in oil as:[1]

$$(d_{ia})^3 = k_{cT}\, t \qquad (6)$$

where $k_c$ is the coarsening rate at 1073 K ($k_{c800\,°C}$ = 2.94 · 10$^{27}$ m$^3$ s$^{-1}$) and $t$ is the tempering time at 1073 K, and $d_{ia}$ = 148 nm was deduced.[8] The volume of carbide particles $Cr_{18}Fe_3Mo_2C_6$ $f$ = 0.047 was deduced from the content of chromium in the investigated steel.[13] The calculated average particle size agrees well with the average size $d$ = 140 nm assessed from micrographs.[13]

The increase of the particle size with a tempering time of 100 h and the creep test temperature is deduced as:

$$\Delta d_{aT} = (k_{cT}/k_{c1073})\, \Delta d_{1073} \qquad (7)$$

where $\Delta d_{aT}$ is the increase of the average particle size with 100 h of tempering at temperature $T$ = (823, 853, 883 and 913) K, $k_{cT}$ is the coarsening rate deduced from Eq. 3, $k_{c1073}$ is the coarsening rate at 1073 K ($k_{c1073}$ = 2.94 · 10$^{-27}$ m$^3$ s$^{-1}$) and $\Delta d_{1073}$ is the increase of particles with an initial size of 148 nm after 100 h tempering at 1073 K. The number of particles $N_{1p}$ = 2.11 · 10$^{19}$ m$^{-3}$ was then deduced from Eq. 4 for $f$ = 0.047.[14]

Then, the average particle spacing $\lambda$ for the particles coarsening at the test temperature was calculated as:[6]

$$\lambda = 4\, d/\pi f^{1/3} \qquad (8)$$

With tempering rsp. creep test temperature, a significant number of particles with a size in the lower part of size distribution is dissolved.[1] The size of the dissolved particles $d_{dm}$ was first deduced from Eq. 5, and then the part of particle with size $d_d \le d_{dm}$ determined for $M_{23}C_6$ of average size $d_a$ = 157 nm.[1] Following that the number of undissolved particles after 100 h of tempering at the creep temperature was deduced to be $N_{pT} = N_{ip}\,(10^{-2} N_{nd})$.

The changes of the average particle size $d_u$ and spacing $\lambda_u$ for the undissolved particles with 100 h at the creep test temperature were calculated as:

$$d_u = \left( \frac{6 \cdot 10^{27} \cdot f}{\pi \cdot N_{pT}} \right)^{1/3} \quad \text{and } \lambda_u = k_u \cdot \left( \frac{6 \cdot 10^{27}}{N_{pT}} \right)^{1/3} \qquad (9)$$

with $k_u$ = 1.585 constant characteristic for the stochastic distribution of cube particle. With an equal average particle size and $f$ = 0.047 the difference of in the particle spacing deduced from Eq. 8 and 9 is about 1 %.

With the change of the creep temperature in Eq. 1, the parameters $T$, $D_{Fe}$, $\lambda$, $d$ and $G$ are changed, and it is possible to calculate the change of creep rate with the known iron self-diffusion rate $D_{Fet}$ and chromium $D_{Crt}$ diffusion rate by tempering, particles size and spacing and shear modulus. In the calculation it should be kept in mind that by creep test iron self-diffusion rate is increased and the chromium diffusion rate, determining the change of particles size and spacing, changes as well. The iron self-diffusion and chromium diffusion rates were calculated from the data in[8] and the shear modulus deduced from.[9]

## 3 RESULTS

In[3,4] it is stated that the number of vacancies in ferrite is increased by tensile stress and the iron and chromium diffusion rate are increased. It is reasonable to presume that with an increased content of vacancies, the climb velocity is also increased. Eq. 1 indicates that with a constant creep stress, the increase of the creep rate is proportional to changes of the iron diffusion rate, of the parameter ($\lambda - d$) and of the shear modulus $G$.[7] Assuming that the effect of temperature and creep is equal for iron and chromium diffusion, the change of particle size and spacing was calculated.

The results of the calculations listed in **Table 1** indicate that the increase of coarsening rate due to the growth of the average particle size is much lower than that due to the dissolution of small particles. The greatest increase of creep rate due to particle growth was $\Delta\dot{\varepsilon} = 0.3 \cdot 10^{-8}$ s$^{-1}$, about 0.4 %, which was obtained at the highest creep test temperature of 913 K (640 °C). The increase of the creep rate due to the dissolution of small particles was significantly greater and it increased with the creep test temperature. At 913 K (640 °C) it was $\Delta\dot{\varepsilon} = 4.3 \cdot 10^{-8}$ s$^{-1}$ and by 6.1 %, about 15 times, greater than that due to the growth of the average particle size.

The difference of the experimental creep rate and that due to the particle dissolution increased with the test temperature and was $\Delta\dot{\varepsilon} = 0.47 \cdot 10^{-8}$ at 823 K and $\Delta\dot{\varepsilon} = 645.4 \cdot 10^{-8}$ at 913 K.

In **Figure 1** the experimental creep rate, as well as the iron diffusion rate by tempering and the apparent iron diffusion rate by creep are depicted as the dependence of creep test temperature. As expected for diffusion-controlled processes, the log value of the ordinate is proportional to the abscissa ($1/T$). However, the dependences

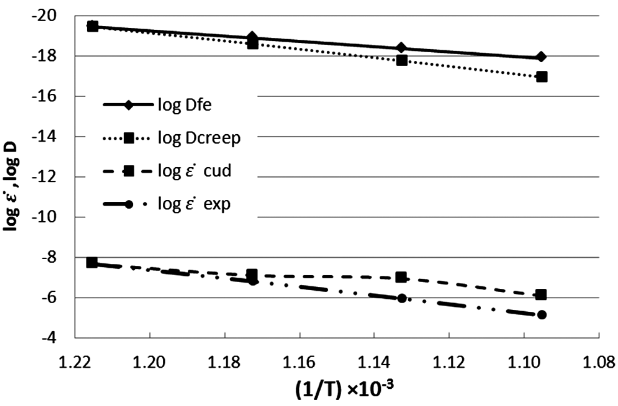


**Figure 1:** Creep rate calculated for undissolved particles ($\dot{\varepsilon}_{cud}$), experimental creep rate ($\dot{\varepsilon}_{crep}$), iron diffusion rate by tempering ($D_{Fetemp}$) and apparent iron diffusion rate by creep test ($D_{Fecreep}$) versus the inverse of the creep test temperature

**Slika 1:** Hitrost lezenja, izračunana za neraztopljene izločke ($\dot{\varepsilon}_{cud}$), eksperimentalna hitrost lezenja ($\dot{\varepsilon}_{crep}$), hitrost difuzije železa pri žarjenju ($D_{Fetemp}$) in navidezna hitrost difuzije železa pri lezenju ($D_{Fecreep}$) v odvisnosti od recipročne vrednosti temperature

are not parallel, as with creep tests the changes of the parameter ($\lambda - d$) and shear modulus $G$ in Eq. 1 are independent of the iron diffusion rate.

For $T \geq 823$ K and creep stress 170 MPa, the effects of an increase of the temperature on the experimental creep rate and the iron creep diffusion rate are:

$$\lg \dot{\varepsilon}_{exp} = -7.677 + 2.11\ 10^4\ 1/T \text{ and}$$
$$\lg D_{Fecreep} = -19.277 + 2.07\ 10^4\ 1/T \qquad (10)$$

In Eq. 1 the constant $k = 4.78 \cdot 10^3 = \sigma^{3.5}/\sigma^2 = 4.78 \cdot 10^3$. The equation can be written for the similar steel as well as coarsening and dissolution rate of carbide par-

**Table 1:** Basic parameters and results of the calculations for the temperature 823–913 K (550–640 °C)
**Tabela 1:** Osnovni parametri in rezultati izračunov za temperature 823–913 K (550–640 °C)

| | | | | |
|---|---|---|---|---|
| Temperature (K) | 823 | 853 | 883 | 913 |
| Temperature (°C) | 550 | 580 | 610 | 640 |
| Shear modulus (MPa · $10^3$) | 54.7 | 53.7 | 52.3 | 51.7 |
| Coars. part size, $d_c$/nm | 148 | 148 | 148.2 | 148.7 |
| Average spacing of $d_c$ particles ($\lambda_c$) | 523 | 523 | 524 | 526 |
| Particles coarsening rate (m$^3$ s$^{-1}$) | $2.52 \cdot 10^{-31}$ | $1.28 \cdot 10^{-30}$ | $4.42 \cdot 10^{-30}$ | $1.40 \cdot 10^{-29}$ |
| $D_{Fetemp}$ /(m$^2$ s$^{-1}$) | $3.68 \cdot 10^{-20}$ | $1.33 \cdot 10^{-19}$ | $4.41 \cdot 10^{-19}$ | $1.35 \cdot 10^{-18}$ |
| $D_{Crtemp}$/(m$^2$ s$^{-1}$) | $4.72 \cdot 10^{-20}$ | $1.85 \cdot 10^{-19}$ | $6.64 \cdot 10^{-19}$ | $2.18 \cdot 10^{-18}$ |
| $D_{Fecreep}$/(m$^2$ s$^{-1}$) | $3.74 \cdot 10^{-20}$ | $2.61 \cdot 10^{-19}$ | $1.80 \cdot 10^{-18}$ | $1.14 \cdot 10^{-17}$ |
| $K_{ccreep}$ /(m$^3$ s$^{-1}$) | $2.52 \cdot 10^{-31}$ | $3.29 \cdot 10^{-31}$ | $1.80 \cdot 10^{-29}$ | $5.65 \cdot 10^{-29}$ |
| Creep rate by $d_i$ and $\lambda_i$, $\varepsilon_i$/($10^{-8}$ s$^{-1}$) | 1.99 | 7.08 | 23.80 | 70.20 |
| Creep rate of growth of $d_a$, $\varepsilon_{at}$/($10^{-8}$ s$^{-1}$) | 1.99 | 7.09 | 24.00 | 70.50 |
| Size of dissolved particles, $d_d$/nm | 6.26 | 8.36 | 11.90 | 17.30 |
| Part of undissolved particles, $d_d$/($10^{-2}$ $N_i$) | 90.16 | 88.09 | 85.12 | 82.22 |
| Number of undissolved particles, $N_{pT}$/($10^{19}$ m$^{-3}$) | 1.28 | 1.25 | 1.21 | 1.17 |
| Average size of undissolved particles (nm) | 154.00 | 155.00 | 157.00 | 159.00 |
| Average spacing of undissolved particles, $\lambda_{ud}$/nm | 569.00 | 574.00 | 580.00 | 587.00 |
| Creep rate due to $\lambda_t$ increase, $\varepsilon_{\lambda t}$/($10^{-8}$ s$^{-1}$) | 2.07 | 7.44 | 25.30 | 74.60 |
| Experimental creep rate, $\varepsilon_{exp}$/($10^{-8}$ s$^{-1}$) | 2.50 | 15.50 | 110.00 | 720.00 |
| Ratio 19/18 | 1.21 | 2.08 | 4.34 | 9.65 |
| Ratio 9/7 | 1.02 | 2.04 | 4.27 | 8.44 |
| Ratio 18/11 | 1.05 | 1.05 | 1.05 | 1.05 |

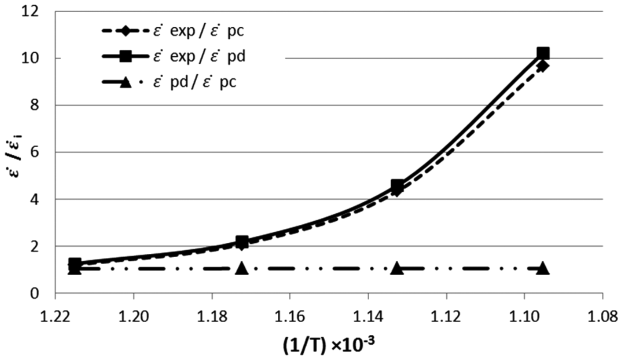


**Figure 2:** Effect of creep test temperature on the ratio of the experimental creep rate ($\dot{\varepsilon}_{exp}$) versus creep rates calculated considering the particles coarsening ($\dot{\varepsilon}_{pc}$) and dissolution ($\dot{\varepsilon}_{pd}$) by tempering at creep test temperature and ratio of creep rates calculated for dissolution and growth of average particles size ($\dot{\varepsilon}_{pd}/\dot{\varepsilon}_{pc}$)

**Slika 2:** Vpliv temperature preizkusa lezenja na razmerje eksperimentalna hitrost lezenja ($\dot{\varepsilon}_{exp}$) proti hitrosti lezenja, izračunani za rast ($\dot{\varepsilon}_{pc}$) in raztapljanje ($\dot{\varepsilon}_{pd}$) izločkov pri žarjenju pri temperaturi preizkusa, in razmerje izračunanih hitrosti lezenja za raztapljanje izločkov in rast povprečne velikosti izločkov($\dot{\varepsilon}_{pd}/\dot{\varepsilon}_{pc}$)

ticles as $\dot{\varepsilon} = k_1 \sigma^n D_{\mathrm{Fecreep}}$ with $k_1 = (b^2 \rho / k_{\mathrm{b}} TG)$ and $n$ = 3.65 and the effect of change of creep stress on iron diffusion and creep rate deduced.

The ratio of the experimental creep rate rsp. creep rate calculated considering the coarsening of particles and the dissolution of particles versus the creep temperature shown in **Figure 2** is independent of the creep temperature. It indicates that the increase of temperature has an equal effect on the dissolution and coarsening of the particles, as both depend on the diffusion rate of iron and chromium.

The growth of iron creep diffusion rate ($D_{\mathrm{Fetemp}}$) with increasing temperature is lower than the change of the creep rate and the difference is much higher than 1.15 due to the creep stress deduced from data in.[4] In the particle disjunctive matrix the gliding stress is decreased strongly, while the climbing stress is diminished much less. As creep strain consists of the glide and the climb of dislocations, it is reasonable to assume that the climb velocity is greater with the greater diffusion rate of vacancy in matrix. Based on available data, it seems reasonable to assume that $D_{\mathrm{Fecreep}}$ in **Table 1** and **Figure 1** represent[5] the increase of a parallel increase of climb velocity, rsp. creep rate related to the content of vacancies.

## 4 CONCLUSIONS

- The coarsening and dissolution rates for $M_{23}C_6$ particles in a high-chromium, creep-resistant steel was calculated for the temperature interval 550 °C to 640 °C;
- The creep rate was then calculated for the investigated range of temperature using the equation with particle size and spacing as parameters and determined experimentally with static tests 100 h by creep stress 170 MPa for equal temperatures;
- In the considered temperature interval, the creep rate was increased for above one order of magnitude stronger with small particles dissolution than with coarser particles growth;
- The iron apparent diffusion rate increased strongly by increasing creep tests temperature;
- Based on the obtained results, empirical relations are deduced for the increase of the experimental creep rate and the apparent iron diffusion rate in ferrite by creep tests with increased temperature.
- It is suggested that the increase of the experimental creep rate with creep temperature are related to a greater iron diffusion rate and a greater climb velocity due to the greater content of vacancies as well as the climb stress in the particles disjunctive matrix.

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