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EFFECT OF OPERATING TEMPERATURE ON MICROSTRUCTURE AND CREEP RESISTANCE OF 20CrMoV 121 STEEL

VPLIV OBRATOVALNE TEMPERATURE NA SPREMEMBO MIKROSTRUKTURE IN ODPORNOSTI PROTI LEZENJU JEKLA 20CrMoV 121

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The degradation process of an X20CrMoV 121 steel with an initial microstructure of tempered martensite was investigated. The effects of the change of microstructure type, and of the carbide particles' size and distribution were determined. Accelerated creep tests showed that the change of the mode of distribution of the carbide particles decreases more strongly due to creep resistance than the increase of the particle-to-particle distance. In the temperature range 550 °C to 800 °C, for annealing times from 2 h up to 1344 h, the hardness decreases faster at lower temperatures.

It is possible to determine with sufficient reliability whether the steel's resistance to creep deformation was reduced below a safe level with non-destructive verification of the steel's microstructure and hardness.

Key words: steel creep resistance, evolution of microstructure, kinetics of coarsening of carbide particles, isothermal change of hardness from 550 °C to 800 °C.

Opisan je proces degradacije jekla X20CrMoV121 z začetno mikrostrukturo iz popuščenega martenzita. Analiziran je vpliv spremembe mikrostrukture ter velikosti in porazdelitve karbidnih izločkov. S preizkusi pospešenega lezenja je bilo ugotovljeno, da sprememba porazdelitve karbidnih izločkov močneje vpliva na odpornost proti lezenju kot povečanje razdalje med izločki. V razponu temperature med 550 °C in 800 °C in za žarjenje do 1344 h se trdota po 2 h hitreje zmanjšuje pri nižji temperaturi žarjenja.

Z zadostno zanesljivostjo je mogoče na podlagi trdote in mikrostrukture oceniti, ali se je zaradi obratovanja zmanjšala odpornost proti lezenju pod varen prag.

Ključne besede: odpornost jekla proti lezenju, evolucija mikrostrukture, kinetika rasti izločkov, izotermno zmanjšanje trdote med 550 °C do 800 °C.

1 INTRODUCTION

Some of the essential equipment parts in thermal power stations operate at temperatures where processes take place which change the initial microstructure of the steel and decrease the steel's creep resistance. The reliability of these parts during operation depends – especially if the operation time approaches the projected life time – strongly on the reliability of the methods used to assess the residual life. Different methods are in use: control of microstructure evolution and assessment of creep damage[1,2]; lifetime prediction using data on scale thickness for thermally stressed parts[3]; assessment of the residual life using equations deduced from empirical data[4]; and micromechanical modelling based on long-term creep damage in steel[5,6]. A fast and inexpensive method of verifying the eventual changes of the steel's properties is the non-destructive checking of the microstructure using the replica method and hardness measurements. For a new method developed to determine the creep resistance of steel using very small specimens[7,8], it is claimed that small, 0.5-mm-thick specimens can be cut out from parts of sufficient thickness without decrease in the reliability. This method makes it possible, in principle, to test the properties of a specific microstructure in the heat-affected zone of welds[9]. On the other hand, considering that creep consists of a flow of dislocations, the question arises: Are creep processes reliably reproduced on specimens with a thickness close to the steel's grain size and with a high rate of resorption of dislocations on the specimen surface?

The aim of this paper is to show that careful tests of the microstructure and the hardness on the heat- and pressure-stressed parts of the equipment allow to evaluate, with sufficient reliability, whether the residual steel creep resistance is sufficiently above the critical value. The reliability of this assessment depends strongly on the reliability of the correlation between the microstructure and the creep resistance of the steel.

2 CHANGES OF THE STEEL'S MICROSTRUCTURE AND THE CREEP RESISTANCE IN THE CREEP-TEMPERATURE RANGE

In some essential parts of thermal power stations the steel temperature is above that which allows a significant rate of self-diffusion of iron atoms and gliding of dislocations, which generates creep deformation and vacancies[10]. The creep resistance in the secondary creep region depends on the steel's capacity to hinder the movement of dislocations. This hindering is obtained, generally, with a sufficient number of precipitates. According to theoretical predictions, the creep rate ($\dot{\varepsilon}$) for a matrix with precipitates is[11]

$$\dot{\varepsilon} = (b^2/kTG)\; L\; \varepsilon^2\; D\; N \qquad (1)$$

were b is the Burgers vector, k is the Boltzmann constant, T is the temperature in K, G is the shear modulus, L is the average particle-to-particle distance, σ is the stress, D is the coefficient of (auto)diffusion in α-iron, and N is the density of dislocations.

With other factors constant, the creep rate is proportional to the particle-to-particle distance, which grows by maintaining the steel in the creep-temperature region.

In a specimen of a tube cut out from an overheater after approximately 57000 h of operation the microstructure was very different on the chimney than on the flame side[12]. On the chimney side the initial habitus of martensite with stringers of carbide particles along martensite sub-boundaries was conserved virtually unchanged (**Figure 1**), while, on the flame side the microstructure consisted of an aleatory distribution of carbide particles in ferrite (**Figure 2**). This shows that on the flame side the steel was heated for a sufficient time above the temperature of internal recrystallisation in martensite grains.

Accelerated creep tests were performed on steel cut out from both sides of the tubes, and the results shown in **Figure 3** were obtained[12]. The accelerated steel creep rate was more than four times greater on the flame side than on the chimney side of the same tube. The microstructure in **Figure 2** differs from that in **Figure 1** in the mode of distribution and in the average size of the precipitates. In **Figure 1**, smaller particles are aligned in stringers, while in **Figure 2** the carbide particles are aleatory distributed, and being coarser by the same volume fraction of carbide phase, the average particle-to-particle distance is greater than in **Figure 1**. Equation (1) shows that the creep rate increases proportionally to the increasing particle-to-particle distance. At first view it could be concluded that the greater creep rate of the

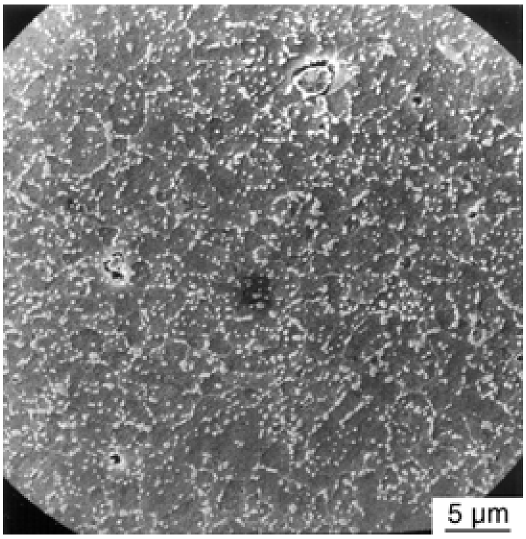

Figure 2: magn. 2000 x. Microstructure in the wall on the opposite side of the tube in **Figure 1**

Slika 2: pov. 2000-kratna. Mikrostruktura v steni cevi diametralno nasproti **sliki 1**

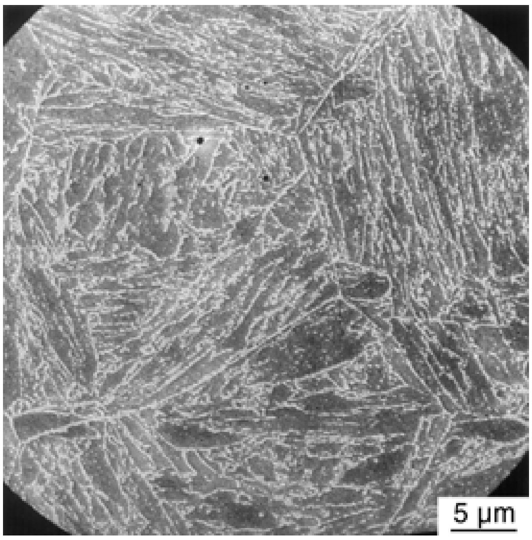

Figure 1: magn. 2000 x. Microstructure in the cooler wall side of an overheater tube 42 mm × 4 mm of steel X20CrMoV 121 after 57000 h of operation

Slika 1: pov. 2000-kratna. Mikrostruktura v hladnejšem delu stene cevi 42 mm × 4 mm iz jekla X20CrMoV 121 po 57000 h obratovanja pregrevalca pare

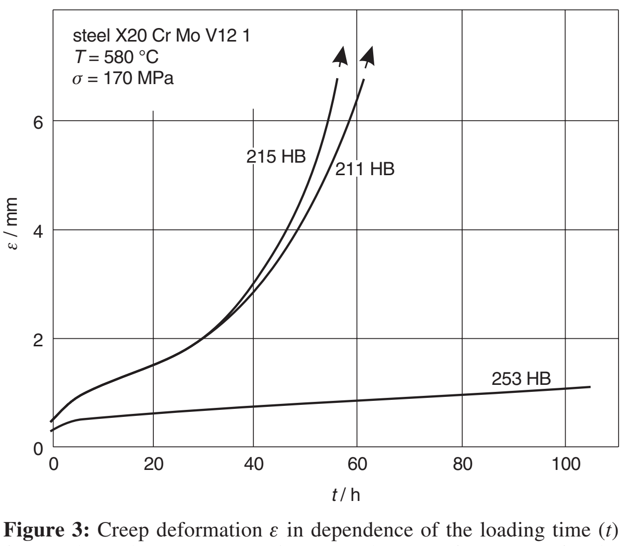

Figure 3: Creep deformation ε in dependence of the loading time (t) for accelerated creep tests at 580 °C and stress of 170 MPa for the steel with the microstructure in **Figure 1 and 2**

Slika 3: Deformacija z lezenjem ε v odvisnosti od trajanja obremenitve (t) pri preizkusu pospešenega lezenja pri 580 °C in napetosti 170 MPa za jeklo z mikrostrukturo na **slikah 1 in 2**

steel in **Figure 2** is due to the greater particle-to-particle distance.

On a microscopic scale the distribution of carbide particles in **Figure 1** is not uniform and the particle-to-particle distance in the same stringer is much smaller than the stringer-to-stringer distance or the particle-to-particle distance in **Figure 2**. In **Figure 1** the average distances for particle to particle in the stringers is approximately 0.05 µm, and the distance stringer to stringer is approximately 0.9 µm. In Figure 2 the average particle-to-particle distance is approximately 1 µm. According to equation 1, the creep rate is similar in areas between stringers and with the uniform distribution of precipitates, while it is much smaller with crossing the stringers of particles. In terms of creep rate and resistance to deformation the microstructure in **Figure 1** is heterogeneous, and that in **Figure 2** is homogenous. It is evident, that the difference in creep rate in **Figure 3** represents a combined effect of the difference in carbide particles' distance and of their distribution, and that the creep rate would be smaller with a smaller stringer-to-stringer distance.

The effect of the average particle-to-particle distance and of the particles distribution was investigated for a quenched steel X20CrMoV121 annealed up to 1344 h at 800 °C[13,14]. After a few hours of annealing, the microstructure consisted of only tempered martensite with carbide precipitates in stringers at the martensite sub-boundaries (**Figure 4**). After longer annealing two effects were observed, a selective coarsening of single precipitates and an increasing share of obliteration of the martensite habitus (**Figure 5**).

According to the Livšic–Wagner law[15,16], with a constant quantity of precipitated phase, the kinetics of the diffusion-controlled coarsening of particles is:

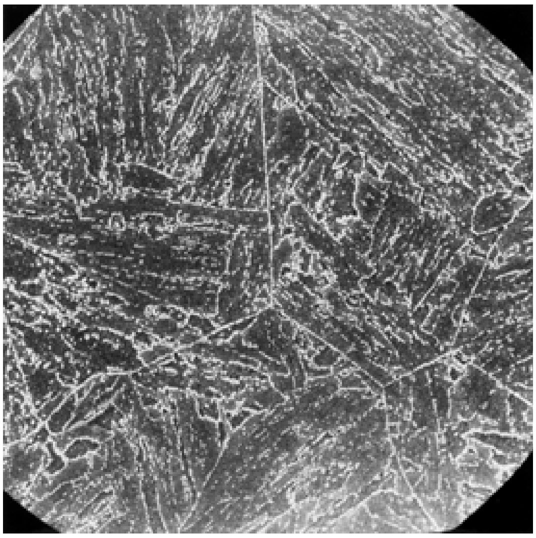

Figure 4: magn. 2000 x. Steel 20CrMoV121. Microstructure after oil quenching and annealing for 7 h at 800 °C

Slika 4: pov. 2000-kratna. Jeklo X20CrMoV 121. Mikrostruktura po kaljenju v olju in 7 h žarjenja pri 800 °C

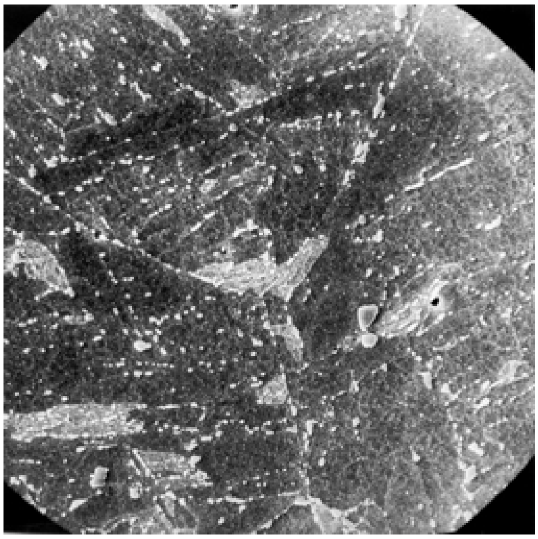

Figure 5: magn. 2000 x. Steel X20CrMoV121. Microstructure after quenching in oil and 672 h of annealing at 800 °C

Slika 5: pov. 2000-kratna. Jeklo X20CrMoV 121. Mikrostruktura po kaljenju v olju in 672 urah žarjenja pri 800 °C

$$d_t^3 - d_0^3 = (k/RT)\ V^2\ D\ \gamma\ t \quad (2)$$

where d_t is the size of precipitates at the time t, d_0 is the initial size of precipitates, R is the universal gas constant, k is the Boltzmann constant, V is the molar volume of the precipitated phase, y is the precipitate-matrix interface energy, D is the average diffusion coefficient, and t is the annealing time. In the coarsening process of carbide particles, carbon is transferred from less-stable and dissolving particles to more stable and growing particles.

The coarsening kinetics (Ck) is theoretically a function of the cubic root of the tempering time, thus $(Ck) = f\,(t^{1/3})$, rsp. $Ck = f\,(t^{0.33})$. For the annealing of the steel X20CrMoV121 at 800 °C, the empirically determined kinetics of coarsening of the carbide particles was[13,14]:

$$\lg d_{800} = -1.38 + 0.27 \lg t \quad (3)$$

The average particles' size increases as a function of $t^{0.27}$, and slower than that theoretically predicted. The difference between the theoretical and empirical coarsening kinetics of carbide particles could be related to different causes, e.g., the initial, on a microscopic scale, heterogeneous distribution of particles, the change of the mode of distribution of particles during the annealing, the change of chemical and phase compositions of particles during the annealing and the effect of the interfacial reaction matrix-carbide phase. With the same quantity of carbide phase (f) the average particle-to-particle distance (L) is proportional to the average particle size (d)[11]:

$$L = 4d/\pi f^{1/3} \quad (4)$$

The quantity of carbide phase is independent of the isothermal annealing time and the introduction of data relevant to the tested steel in equation (4) gives $L = 3.86\ d$. Accordingly, the average particle-to-particle distance grows approximately four times faster than the average particles size.

The quenched steel X20CrMoV 121, previously annealed for a different time at 800 °C, was submitted to 100 h accelerated creep tests under test conditions – temperature 580 °C and stress 170 MPa – found earlier to be sensitive to the effect of steel microstructure on creep rate[12]. In **Figures 6a and 6b** examples of the obtained curves creep rate rsp. creep deformation versus testing time are given with clearly distinguished periods of primary and secondary creep rate. In **Figure 7** the dependence of the average particle-to-particle distance versus secondary creep rate is shown[14,17,18]. The creep rate increases proportionally to the particle-to-particle distance, as expected from equation (1), up to the inflection point at a critical particle-to-particle distance. Off side the critical point, the creep rate is increased by approximately 4 times. The difference on and away from the critical point is similar to that in **Figure 3** for the steel from the flame and the chimney side of the same tube. Microstructural examinations have shown that the inflection point was achieved after the major part stringers of the carbide particles was obliterated.

The increased secondary creep rate of the side of the inflection point reflects both the growth of the particle-to-particle distance and the decreased share of particles in the stringers. It is evident that the effect of the distribution of precipitates on the creep rate is even greater than the effect of the particle-to-particle distance.

For the steel X20CrMoV 121 it was possible, on specimens cut out from a steam collector, to distinguish the change of microstructure due to the operation time and the temperature. In **Figure 8** the microstructure is shown for a steel with the martensite habitus virtually unchanged, while on a different place of the same collector, where the steel was heated in an operation to a higher temperature, aleatory distributed carbide particles and voids around non-metallic inclusions were found[19] (**Figure 9**).

On properly prepared replicas the microstructure of the tempered martensite is clearly shown (**Figure 10**). The presence of single cavities at the grain boundaries (**Figure 11**) and their coagulation to intergranular cracks indicates an accelerated creep rate and shows that the steel creep resistance is diminished below a safe level[1]. The cavities are produced with movements of jogs of screw dislocations[11] and are formed at grain boundaries because of the higher creep rate in the grain-boundary regions[20].

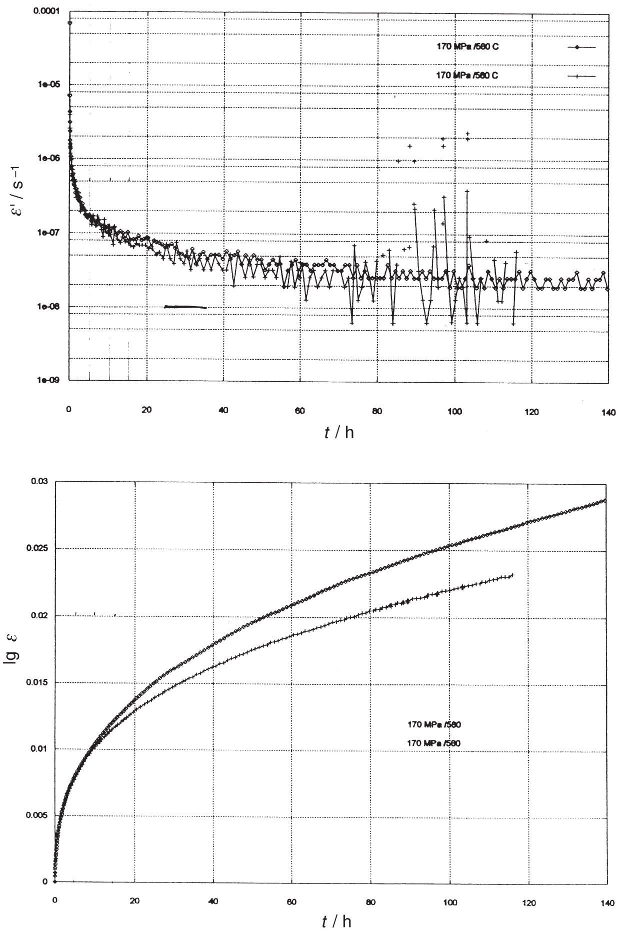

Figure 6: Curves for two parallel specimens recorded by accelerated creep tests at 580 °C and 170 MPa. Upper Figures: creep rate $\dot{\varepsilon}$ and lower Figures: creep deformation ε in dependence of the loading time *t*. Specimens of steel X20CrMoV 121 quenched in oil and annealed for 7 h at 800 °C before the creep test

Slika 6: Krivulje preizkusa pospešenega lezenja pri 580 °C in 170 MPa za dva paralelna preizkušanca. Zgoraj: hitrost lezenja $\dot{\varepsilon}$ in spodaj: skupna deformacija ε z lezenjem v odvisnosti od trajanja obremenitv *t*. Preizkušanca iz jekla X20CrMoV 121 sta bila kaljena v olju in žarjena 7 h pri 800 °C.

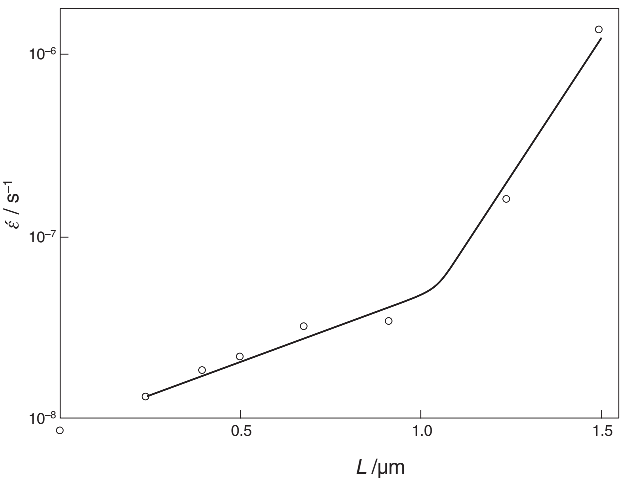

Figure 7: Dependence of accelerated creep rate $\dot{\varepsilon}$ at 580 °C and 170 MPa versus the average carbide particle-to-particle distance *L* for the steel X20CrMoV 121 quenched in oil and annealed for 7 h at 800 °C

Slika 7: Odvisnost med hitrostjo pospešenega lezenja $\dot{\varepsilon}$ pri 580 °C in 170 MPa ter razdaljo med karbidnimi izločki *L* za jeklo X20CrMoV 121 kaljeno in žarjeno 7 h pri 800 °C

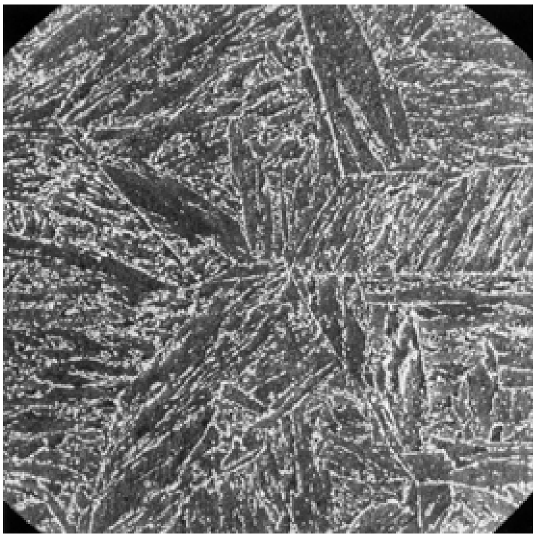

Figure 8: magn. 2000x. Microstructure of the steel X20CrMoV 121 in a specimen of steel cut out from a steam collector after long operation time
Slika 8: pov. 2000-kratna. Mikrostruktura jekla X20CrMoV 121 v vzorcu, ki je bil odrezan iz kolektorja pare po dolgotrajnem obratovanju

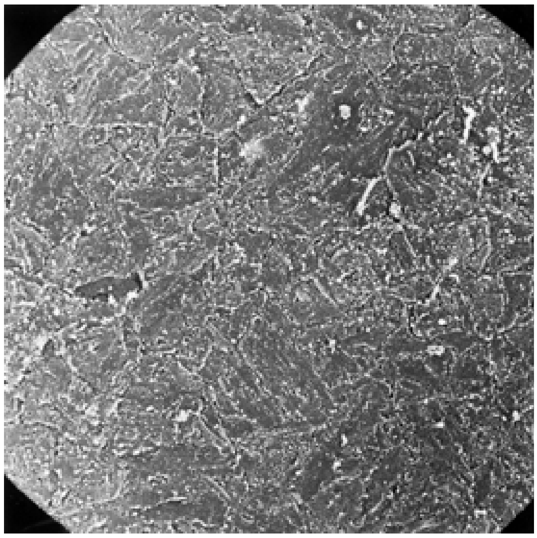

Figure 10: magn. 1000 x, replika. Tempered martensite on the surface of a steam collector
Slika 10: pov. 1000-kratna, replika. Popuščeni martenzit na površine kolektorja pare

3 HARDNESS

The increase of the yield stress $\Delta\tau$ related to the movement of a dislocation in a lattice with precipitates is[21]:

$$\Delta\tau = 0.85\ (3Gb/2\pi d)\ \ln\ (d/x) \qquad (5)$$

where d is the precipitates size, G is the shear modulus, b is the Burgers vector and x is the diameter of the dislocation core.

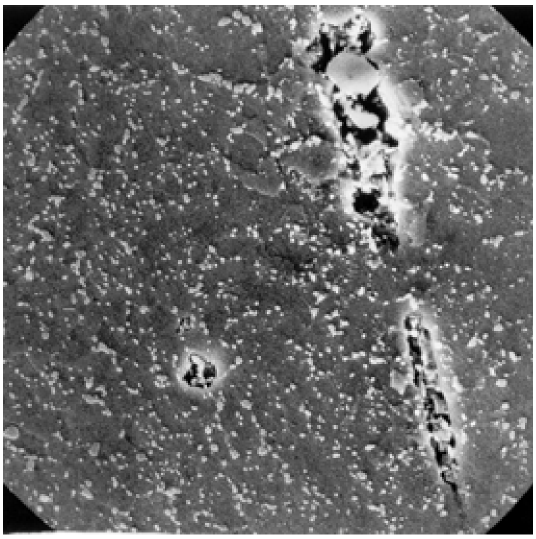

Figure 9: magn. 2000 x. Microstructure of the steel X20CrMoV 121 cut out from an overheated part of a steam collector
Slika 9: pov. 2000-kratna. Mikrostruktura jekla X20CrMoV 121 v vzorcu, odrezanem iz pregretega kolektorja pare

Neglecting the term ln(d/x), it can be assumed that the increase of the yield stress is inversely proportional to the average precipitates size, rsp. to the particle-to-particle distance. By hardness determination the metal is plastically deformed in the indented volume. For this reason, hardness is related also to the resistance of the metal-to-plastic deformation (yield stress) and the strain-hardening propensity. For structural steels the hardness is approximately proportional to their yield stress, and with increasing particle-to-particle distance both the steel creep resistance and the hardness are decreased. As indicated in **Figure 3**, hardness is significantly lower for the steel with lower creep resistance.

For the oil-quenched steel X20CrMoV 121 the effect of annealing time up to 1344 hours on hardness was determined in the temperature range from 550 to 800 °C in steps of 50 °C. The softening kinetics is shown in **Figure 12**. In a relatively short period of time after the start of isothermal tempering the hardness is rapidly and strongly decreased, mostly due to the relaxation of the internal stresses generated with the transformation of austenite to martensite. For an isothermal tempering of 7 h and longer at 600 °C and of 2 hours and longer for the temperature of 650 °C and higher, the hardness (H) decreases from the initial level (H_0) proportionally to the log value of the annealing time (t):

$$H = H_0 - k \lg t \qquad (6)$$

For the annealing at 800 °C the hardness at time t is:

$$H_{800,\,t} = (2.37 - 0.18 \lg t) \cdot 10^2 \qquad (6a)$$

As can be deduced from **Figure 12**, the value of the exponent k increases with the lowering temperature and shows that by longer annealing time the softening is the faster for 650 °C. It is possible that for annealing at 600 °C, part of the hardness is due to the precipitation of the Laves phase (FeCr)Mo_2[22,23]. The kinetics of softening is of a similar form as the kinetics of the growth of carbide particles and of the particle-to-particle distance, as would be expected from equation (5).

Assuming that the chemical composition of the precipitates is not changed during the tempering, carbon is the element transferred with solid-state diffusion to the coarsening carbide particles. According to equation (2) the isothermal coarsening kinetics is theoretically proportional to the coefficient of diffusion of the carbon in ferrite as $D^{1/3}$. This coefficient increases with the annealing temperature as $D_T = f\,[\exp{-(Q/RT)}]$, with Q as diffusion activation energy. Considering Q = 80.2kJ/mol, it can be deduced that the coarsening of particles, if it was diffusion dependent, should be, for example, 1.63 times faster at 750 °C than at 700 °C.

Empirically, the contrary was established and the softening is even faster at 700 °C than at 750 °C. The role of carbon diffusion is, thus, negligible in the process of the softening of the investigated steel. Several explanations are possible, e.g., the difference in the initial size and number of precipitates, the effect of the interface reaction solid solution of alloying elements in ferrite-carbide particles and the change of the chemical and phase composition of the carbide phase. It was established that the phase composition of the precipitates evolves in the sequence $M_3C \rightarrow M_{23}C_6 \rightarrow M_7C_3$ because carbide particles gather from the solid solution chromium and to smaller extent also molybdenum[14,17] and reject iron atoms in ferrite.

The hardness decrease for up to 2 h rsp. 7 h of annealing is approximately proportional to the increase of temperature from 600 °C to 800 °C. If the kinetics lines in Figure 12 are extrapolated to the zero annealing time, the decrease of hardness is theoretically independent on the annealing time obtained. This hardness difference increases linearly with the temperature from 600 °C to 800 °C[16] and it is probably related to the steel yield stress at every temperature.

After annealing for 1344 h in the temperature range from 550 °C to 750 °C, the microstructure remained typical for the tempered martensite with carbide particles in stringers (**Figure 13**). At 800 °C the start of the change of microstructure with obliteration of carbide stringers was observed after 24 h of annealing, after the longer annealing of 168 h the predominant part of stringers was decomposed and after longer tempering carbide stringers were very rare (**Figure 5**).

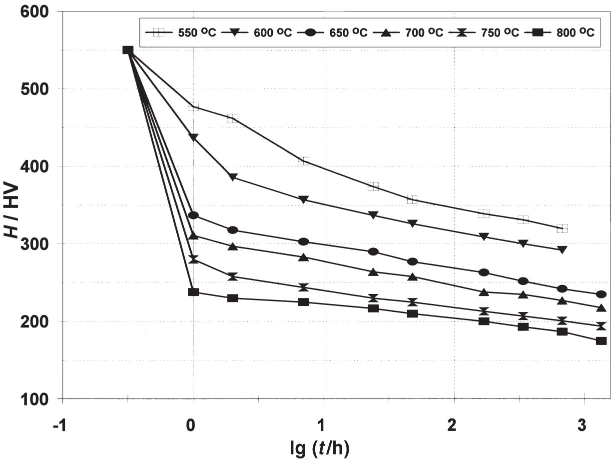

Figure 12: Dependence of Vickers hardness H versus annealing time t in temperature range from 550 °C to 800 °C for the oil-quenched steel X20CrMoV 121

Slika 12: Trdota po Vickersu H za jeklo X20CrMoV121, ki je bilo kaljeno v olju, v odvisnosti od trajanja žarjenja t pri temperaturah med 550 °C in 800 °C

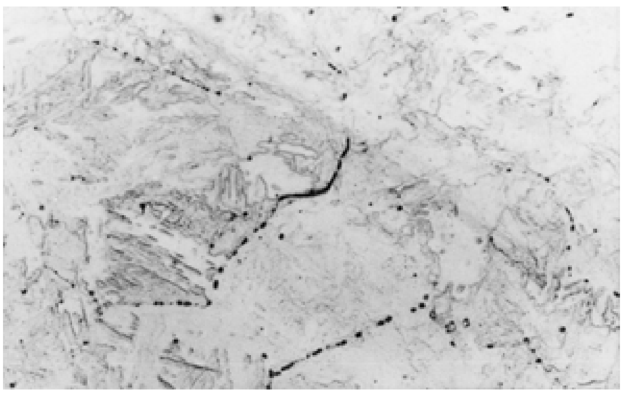

Figure 11: magn. 500 x, replica. Voids and microcracks at grain boundaries in the steam collector wall after a long time of operation. Steel X20CrMoV121

Slika 11: pov. 500-kratna. Pore in mikrorazpoke po mejah kristalnih zrn v jeklu X20CrMoV 121 v steni kolektorju pare, ki je bil dolgo časa v uporabi

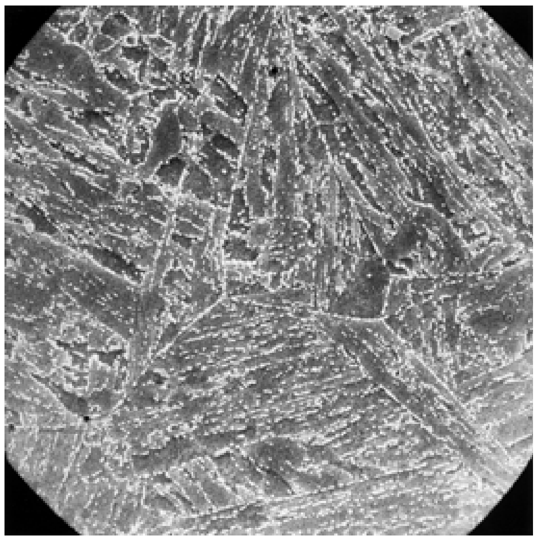

Figure 13: magn. 2000 x. Microstructure of the quenched steel X20CrMoV 121 annealed for 1344 h at 750 °C

Slika 13: pov. 2000-kratna. Mikrostruktura kaljenega jekla X20CrMoV 121, ki je bilo žarjeno 1344 h pri 750 °C

Considering the results of this investigation it is concluded that the verification of the microstructure and hardness permits a reliable assessment of the steel creep resistance on the basis of an empirically verified dependence creep rate – microstructure and hardness. In new steels the creep resistance is enhanced with the addition of niobium and tungsten [24,25,26,27]. Both elements form carbide rsp carbonitride precipitates of similar size to vanadium carbonitride and different chemical stability than precipitates of vanadium carbonitride in the steel investigated. For this reason, the effect of temperature and annealing time on hardness and creep rate must be verified empirically for each type of steel.

4 CONCLUSIONS

The steel X20CrMoV 121 was oil quenched and annealed for up to 1344 h in the temperature range from 550 °C to 800 °C. The evolution of microstructure and hardness were investigated and, for the steel tempered at 800 °C, also accelerated creep tests were performed. Some data obtained with examinations of specimens cut out from steam tubes and from a failed steam collector as well as examinations of microstructure using the replica method were considered also. The following conclusions are proposed, based on the empirical findings in this work and from the quoted references:

By annealing the quenched steel X20CrMoV 121 in the temperature range from 550 °C to 800 °C for a time longer than 2 h, the hardness decreases proportionally to the log value of the annealing time and with a softening rate which is greater at lower temperatures. The hardness after 2 h of annealing decreases proportionally to the increase of temperature in the range from 600 °C to 800 °C.

For the steel X20CrMoV121 quenched and annealed at 800 °C the accelerated creep rate increases proportionally to the particle-to-particle distance until carbide particles are found mostly in stringers, characteristic for the microstructure of tempered martensite. When in the major part of the microstructure carbide stringers are obliterated and the habitus of tempered martensite is changed to an aleatory distribution of carbide particles in ferrite, the creep rate increases by approximately four times.

It is possible to evaluate with sufficient reliability if the resistance to creep deformation for a selected steel in parts of thermal equipment operating at high temperature was diminished below the long-term safe level on the basis of the examination of microstructure and hardness measurements.

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