Evolution of Lath Substructure and Internal Stresses in a 9% Cr Steel during Creep

Valeriy DUDKO,* Andrey BELYAKOV and Rustam KAIBYSHEV
Belgorod State University, Pobeda 85, Belgorod, 308015 Russia.

(Received on June 4, 2016; accepted on November 10, 2016; J-STAGE Advance published date: December 2, 2016)

The changes in the dislocation substructure and internal stresses in a tempered 9% Cr heat resistant steel during creep at 923 K were studied. The mean lath size gradually increased from 330 nm for the initial tempered state to 740 nm once the specimen had crept to failure under a nominal stress of 118 MPa after 1,271 hours. Correspondingly, the dislocation density within the lath decreased from $6.2 \times 10^{14}$ m$^{-2}$ to $\sim 10^{14}$ m$^{-2}$. The tempered structure of the martensite lath was characterised by large lattice curvatures, which were attributed to a high density of dislocations with like signs and the long-range stress fields originating from the martensite lath boundaries. An internal stress of 49 MPa, as evaluated by measuring the lattice curvature within individual laths in specimens crept to 1% (just after transient creep stage), is comparable to the threshold stress of 51 MPa estimated from the creep rate and stress relationship. The improved creep resistance of advanced 9% Cr martensitic steel results from both dispersion strengthening and the internal stresses of the martensite lath substructure.

KEY WORDS: steel; creep; dislocation boundaries; misorientation; internal stress.

1. Introduction

In fossil power plants, 9% Cr martensitic steels, such as P92, are used for essential components of the boiler and steam turbine, which operate under ultra-super critical steam conditions.1) The superior creep strength of 9–11% Cr steels is achieved by preventing dislocation motion during plastic deformation due to independent action of such strengthening mechanisms as solute hardening, precipitation hardening and substructure hardening.1,2) The solute strengthening and precipitation strengthening may diminish during creep due to depletion of solid solution with W and Mo and coarsening of nanoscale MX carbonitrides, which are distributed homogeneously throughout the ferritic matrix.1–4) In general, these processes lead to remarkable strength degradation at tertiary creep stage,4–6) although the coarsening rate of MX carbonitrides, which provide superior creep resistance of high Cr steels, is quite low.

Stability of tempered martensite lath structure (TMLS), which provides substructure strengthening, under creep conditions plays a key role in the creep strength of modern high chromium heat resistant steels.1–9) These steels lose their superior creep resistance due to the replacement of lath structure by subgrain structure during creep.1–9) The transformation of TMLS to subgrain structure occurs through two sequential process. Firstly, the knitting reactions between free dislocations and lath boundaries result in the dislocation annihilation leading to a decrease in the lattice dislocation density and a gradual transformation of the lath boundaries to subgrain boundaries.3,4,10,11) The dispersed particles of M23C6 carbides and Laves phases precipitated on boundaries during tempering and creep, respectively, and the MX carbonitrides impede this knitting reactions.1,4,7,9,12) In addition, substitutional atoms such as W, Mo, Co and Cr hinder the dislocation slip within the ferritic matrix and the rearrangement of lattice dislocations by climb that also slows down the knitting reactions between dislocations and lath boundaries.1,4,12) Secondly, the migration of subboundaries leads to complete replacement of TMLS by well-defined subgrains.1,3–9) The acceleration of tertiary creep due to extensive migration of low-angle boundaries is considered to be the main reason for the creep strength breakdown, i.e., a sudden drop of the allowed stress in the range of long-term creep.1,4–6,13) In general, the evolution of TMLS during creep have been addressed in several studies.3,10,14,15) However, limited attention was paid to the evolution of lath boundaries with strain. As a result, the relationship between the lath boundary characteristics and the creep behaviour in heat resistant steels is still unclear due to the lack of experimental data although a detailed analysis of the internal stresses inherent to martensite laths might be useful for the structural design of advanced steels for high temperature applications.

The aim of the present work was to explore the evolution of lath structure in a P92-type steel during creep at a temperature of 650°C and an applied stress of 118 MPa, that is inflection point for transition from short-term to long-term creep.3–6) In particular, this study was focused on the relationship between the characteristics of TMLS and
the creep behaviour. The evolution of secondary phases and TMLS during creep and long-term aging was considered in companion work\(^3\) in some detail.

2. Experimental

A P92-type steel, Fe-0.1 C-0.17 Si-0.54 Mn-8.75 Cr-0.21 Ni-0.51 Mo-1.60 W-0.23 V-0.07 Nb (all in mass\%), was fabricated at the Chelyabinsk Metallurgical Plant, Russia.\(^3,16\) This steel was solution treated at 1 323 K and then tempered at 993 K for 3 hours. Tensile specimens with a cross section of 7 × 3 mm\(^2\) and gauge length of 25 mm were subjected to creep tests at 923 K under an initial stress from 118–200 MPa. Several creep tests at a stress of 118 MPa were interrupted at different strains to study the structural evolution.\(^3\)

The structures were characterized using a JEM-2100 transmission electron microscope (TEM) to image the longitudinal sections of the specimens. The specimens were prepared by electro-polishing with a solution of 10% perchloric acid in glacial acetic acid. The lath/subgrain sizes were measured from the TEM micrographs using the linear intercept method, which included all the clearly visible (sub)boundaries. The dislocation density was evaluated by counting the number of individual dislocations within the grain/subgrains in at least six arbitrarily selected TEM images for each data point. Misorientations at the lath boundaries were studied using conventional Kikuchi-line technique with TEM.\(^17\) At least 50 boundaries were analyzed for each specimen. The small lattice curvatures within the individual laths/subgrains were evaluated from the rotation vector component in the plane of the electron diffraction pattern.\(^18\) Also, the internal stresses were studied by X-ray diffraction\(^19,20\) using a Rigaku diffractometer with a Cu target.

3. Results

3.1. Tempered Microstructure

TEM micrograph of a tempered P92 steel structure is shown in Fig. 1(a). It is clear that a martensitic transformation followed by tempering at 993 K formed a microstructure consisting of martensite laths with high internal dislocation densities. Some individual dislocations and a dislocation wall inside a lath are indicated by arrows in Fig. 1(c). The lath thickness and the dislocation density within the lath are 330 nm and \(6.2 \times 10^{14} \text{ m}^{-2}\), respectively. The majority of lath boundaries have misorientations below 3° (Fig. 1(b)). The average lath boundary misorientation calculated by TEM Kikuchi-line method is 2.7°.

The lattice curvatures and associated internal stresses within the elongated laths were studied using the convergent beam Kikuchi diffraction technique. An example of such an analysis is shown in Fig. 1(c). The letters in the lath interior (i.e., A and B) indicate the local regions where the crystal lattice orientations were precisely determined. The Kikuchi diffraction patterns obtained from the lettered regions are schematically shown in the insert with A and B indicating the position of the central electron beam. The lattice curvature between these selected local regions can be roughly evaluated by measuring the distance between points A and B in the diffraction patterns.\(^18,21\) The residual shear stresses, \(\tau\), associated with the maximal elastic distortion can then be calculated as follows:\(^21\)

\[
\frac{\tau_{\text{TEM}}}{G} = \frac{0.35t\theta}{l},
\]

where \(G\) is the shear modulus, \(t\) is the foil thickness, \(\theta\) is the angular disorientation between any two points within the
lath, and \( l \) is the distance between the measured points. The internal stresses were evaluated for 10 different laths and the average value was \( 1.9 \times 10^{-3} \text{ G} \).

3.2. Creep Behavior

The creep rate – strain curves at 923 K for the initial stresses of 118, 140, 160, and 200 MPa are shown in Fig. 2(a). The transient creep regime occurs during creep to strains of 1–4%, when the creep rate becomes minimal for the different stresses. An apparent region of steady-state creep can be identified in the strain interval 1–4% for simplicity since it is difficult to accurately determine the strain for the onset of the tertiary stage.22) Further deformations at strains above approximately 4% correspond to the tertiary creep regime.

The creep rate can be approximated as \( \dot{\varepsilon} \sim \exp(b\varepsilon) \). At 118 MPa, the transient stage is relatively short and the steady-state appears at a strain of \( \varepsilon=1\% \) (Figs. 2(b) and 2(c)). The transient stage is characterized by the parameter \( d\ln\dot{\varepsilon}/d\varepsilon \sim -985 \) (Fig. 2(c)), which describes the kinetics of dislocation rearrangement into a stable configuration. The obtained parameter of \( -985 \) is ~2 times higher in the absolute value than that of \( -449 \) in the 3%Co modified P92 steel,23) suggesting the high rate of the dislocation rearrangement during transient creep in the present study. It is worth noting that the minimum strain rate, \( \dot{\varepsilon}_{\text{min}} \), is observed at an offset strain of 1% and further strain increase up to \( \varepsilon=4\% \) leads to insignificant increase in the strain rate at apparent steady-state creep. An acceleration of the creep rate is observed in tertiary creep at \( \varepsilon>4\% \) and the acceleration parameter, \( d\ln\dot{\varepsilon}/d\varepsilon \sim 46 \), is nearly independent on strain.4,23,24) The absolute value of the \( d\ln\dot{\varepsilon}/d\varepsilon \) parameter in the tertiary stage is much smaller than that in the transient stage (Fig. 2(c)) that is indicative of the significantly slow processes at tertiary creep.4,13,22–24)

The stress dependence of minimum strain rate is commonly expressed by a power law relationship of applied stress, \( \sigma \),

\[
\dot{\varepsilon}_{\text{min}} = A\sigma^{n^*}, \quad \text{................................ (2)}
\]

Where \( A \) is a constant, \( n^* \) is an apparent stress exponent. The variation of \( \dot{\varepsilon}_{\text{min}} \) with \( \sigma \) is plotted in Fig. 2(d) using a double logarithmic scale. The creep behaviour of the P92 steel at 923 K is characterized by relatively high apparent stress exponent. Moreover, the apparent stress exponent is stress dependent. Namely, \( n^* \) increases from 11 to 17 as \( \sigma \) decreasing over the range from 200–118 MPa.

3.3. Crept Microstructure

Typical lath substructures that evolved during creep at
923 K under an initial stress of 118 MPa are shown in Fig. 3. The creep is accompanied by a gradual increase in the lath thickness and a decrease in the dislocation density within the laths, although some dislocation substructure as shown by arrows in Fig. 3 remains in the lath interiors until creep fracture. The laths tend to acquire more or less rectangular shape, which look like ordinary hot worked substructures especially in the tertiary creep regime (Figs. 3(c) and 3(d)). The quantitative results for the mean lath size and the interior dislocation density are presented in Fig. 4. Under transient creep the lath thickness and average misorientation remains unchanged, while the lattice dislocation density decreases to $3.6 \times 10^{14} \text{ m}^{-2}$. Then, the lath thickness increases to 430 nm and the dislocation density further decreases to $2.2 \times 10^{14} \text{ m}^{-2}$ during the apparent steady-state. The most pronounced lath growth from 430 nm to 710 nm that is accompanied by almost twofold decrease in the lattice dislocation density occurs in the strain range of 4–8%. The obtained strain dependence of the lath/subgrain size, $D$, matches the empirical relationship between the subgrain size and strain, $\varepsilon$, derived in:27)

$$\log D = \log D_0 + \log (D_0 / D_0) \exp(-\varepsilon / 0.12) \ldots \ldots (3)$$

where $D_0=10bG/(\sigma_0(1+\varepsilon))$, $b$ is the Burgers vector, and $\sigma_0$ is the initial nominal stress. The dashed line in Fig. 4 indicates the change in the lath size during creep as calculated by Eq. (3). The lath growth correlates to the decrease in dislocation density. Namely, following a rapid drop during early deformation the dislocation density within the lath decreases almost fourfold for creep from 1 to 8%. It should be noted that the tertiary creep regime corresponding to the strain range from 8 to 18%, is not accompanied by a remarkable change in the lath size and dislocation density.

Most of the lath boundaries are characterized by small misorientations of approximately 1–3° (Fig. 3). A detailed analysis revealed small variations in the lath misorientation distributions during creep (Fig. 5). The lath boundary misorientation distribution is characterized by a sharp peak at around 1° after $\varepsilon=1\%$. The apparent steady-state stage, strains of 1–4%, is accompanied by an increase in the frac-

![Fig. 3. Typical lath substructures for a P92 steel after creep at 923 K under an initial stress of 118 MPa to various strains: (a) 1%, (b) 4%, (c) 8%, (d) 18%. The numbers indicate the lath boundary misorientations in degrees.](image-url)

![Fig. 4. Strain dependences of the transverse lath size ($D$) and dislocation density ($\rho$) in a P92 steel during creep at 650°C under an initial stress of 118 MPa. The dashed line corresponds to the lath size calculated by Eq. (3).](image-url)
tion of boundaries that have misorientations below 1°; the peak around 1° decreases and broadens. Increasing the creep strain further to 8% does not remarkably affect the lath misorientation distribution. The appearance of lath/subgrain boundaries with rather large misorientations above 3° was recorded for samples strained to 18%, i.e. the fractured specimens. Therefore, the average misorientation of lath boundaries insignificantly decreases during the transient creep followed by a decrease from 2.7° to 2.1° during the apparent steady-state creep. Upon tertiary creep the average misorientation gradually increases to 2.8° in the necking portion of the fractured specimen (Fig. 6). As a result, the average misorientations of low-angle boundaries after heat treatment and rupture are almost the same.

Despite the relatively low dislocation density within the laths during creep (~10^{14} m^{-2}), dislocation walls and separate dislocations are observed within the laths (Fig. 7) and the lath structure possesses significant internal distortion. The lattice curvatures within the individual laths shown in Fig. 7 suggest that the crystal lattices of the selected laths are bent to rather large angles, which are comparable to the lath boundary misorientations. The lath lattice curvatures revealed by the TEM analysis could be connected to the excess of dislocations with similar Burgers vectors. In this case, the density of these dislocations can be roughly evaluated as follows:

\[ \rho^* = \theta / (bl) \] .......................... (4)

The dislocation density estimated by Eq. (4) is shown in Fig. 4 for the sake of convenience. In contrast to the total dislocation density (as obtained by counting individual dislocations within the lath), the density of dislocations with like signs decreases rapidly during the transient creep. Then,
The $\rho^*$ value remains almost constant during further creep at $\varepsilon \geq 1\%$.

The huge lattice curvatures are indicative of the high internal stresses maintained by the lath structure. The average values calculated using Eq. (1) for these internal stresses are displayed in Fig. 8 with the results of the X-Ray analysis. The transient creep stage is characterized by a rapid release of internal stresses, which decrease from approximately 70 MPa in the tempered state to approximately 50 MPa after a 1% creep. The steady-state and tertiary creep is accompanied by a gradual decrease of the internal stresses to about 25 MPa after an 18% creep (fractured specimen). It should be noted, however, that the kinetics of the stress releases revealed by the TEM and X-Ray studies are different. Generally, the variation in the internal stresses revealed by X-Ray analysis is similar to the evolution of free dislocation density ($\rho$ in Fig. 4). Namely, following a rapid decrease during the transient creep, the internal stresses gradually approach 25 MPa for a creep strain above 8%. However, the internal stress release revealed by the TEM study is characterized by lower kinetics for creep strains of 1–8%. In these samples, the internal stresses remain above 40 MPa, though level of 25 MPa is attained for the fractured specimen.

4. Discussion

4.1. Evolution of Dislocation Substructure

The changes in the tempered martensite lath substructure for P92 steel follow a common evolutionary behaviour. Namely, the transverse lath size gradually increases with the creep strain and approaches a saturation value of 0.8 $\mu$m, which is quite close to the subgrain size of 1 $\mu$m expected as a function of the flow stress ($D = 10bG/\sigma$). Correspondingly, the dislocation density within the laths decreases during creep and approaches $\sim 10^{14}$ m$^{-2}$ at large strains. For the tempered state, the changes in the lath/subgrain size and dislocation density obey a well-known relationship, i.e., $D \sim \rho^{-0.5}$ (Fig. 9). This relationship is valid for all creep stages and,
therefore, describes the relationship between the lath thickness/subgrain dimensions and the free dislocation density under all creep conditions.4,28,32)

The fast kinetics of dislocation rearrangement during transient creep is attributed to annihilation of lattice dislocations with opposite signs, because the average misorientation of lath boundaries and their density remain almost unchanged (Figs. 4 and 6). An emission of new lattice dislocations by lath boundary sources does not compensate these annihilation events and the annihilation rate of free dislocations, $d\rho/dae \sim -2.6 \times 10^{16}$ m$^{-2}$, is 5 times higher than the annihilation rate of the excess dislocations, $d\rho^e/dae \sim -4.4 \times 10^{15}$ m$^{-2}$ (Fig. 4). The lath boundaries play a role of sinks of lattice dislocations.12) The number of annihilation events between lattice dislocations and lath boundaries is higher than the number of lattice dislocations trapped by lath boundaries. The lattice dislocations trapped by lath boundaries compensate the annihilation of intrinsic dislocation composing these boundaries, since the average misorientation does not change remarkably (Fig. 6).

Martensitic transformations in steels occur in combinations with accommodation processes and produce a high dislocation density.29,33) A tempering treatment of high Cr steels affects slightly their dislocation structure,29,34–36) which retains high dislocation density (Fig. 10(a)). During transient creep the annihilation of the dislocations with opposite signs is the main process of microstructural evolution that provides a release of internal elastic stress as illustrated in Fig. 10(b). Neighboring laths contain sets of dislocations with opposite signs. During transient creep, these dislocations start to glide towards the lath boundaries in both neighboring laths immediately upon loading.12) Next, these dislocations with opposite signs attain a critical annihilation distance of ~1.6 nm for edge dislocations by climb along a lath boundary and ~50 nm for screw dislocations in a lath boundary plane.30) It is worth noting that a cross-slip of screw dislocation along a lath boundary is not necessary for their mutual annihilation in most cases, taking into account that the distance between gliding dislocation is <100 nm.12) Thus, the annihilation of free dislocations with opposite signs occurs at lath boundaries (Fig. 10(b)) that leads to decreasing the $\rho^e$ value. At the onset of creep, the number of mobile dislocations is high and creep occurs with high rate. Then, the number of mobile dislocations decreases due to annihilation events during transient creep resulting in a rapid decrease in the creep rate. It is obvious that a very high $d\ln \dot{\varepsilon}/dae$ value of ~ −985 is attributed to the fast disappearance of these mobile dislocations.

It is known that lath boundaries play a role of dislocation sources and dislocations emitted by these sources glide inside the lath until the encounter with other lath boundary.12) The interaction of dislocations with opposite signs leads to their annihilation and decreases the lath boundary misorientation.6,30) Although lath boundaries exhibit misorientations of several degrees, the gliding dislocations...
do not cross them. During steady-state creep, the rate of dislocation release significantly decreases to \( \frac{\partial \rho}{\partial \varepsilon} \sim 4.7 \times 10^{15} \text{ m}^{-2} \) (Fig. 4). New lattice dislocations result from knitting out of lath boundaries (Fig. 10(c)). As a result, the misorientations of lath boundaries, the density of free lattice dislocations, and the internal elastic stresses decrease. However, there is no remarkable effect of the knitting reactions on the \( \rho^* \) value, because the rates of emission and annihilation of lattice dislocations with like signs are nearly the same. Concurrently, the dislocation glide along laths takes place and these dislocations arrange in transverse boundaries. The dislocation glide along the laths occurs very slowly since the gliding dislocations are connected to the lath boundaries, which play a role of effective pinning agents in addition to MX carbonitrides and boundary M23C6 carbides and Laves phase. Therefore, an apparent steady-state with a low creep rate occurs.

At tertiary creep, disappearance of some lath boundaries and increasing lath thickness due to the progressive knitting reactions highly facilitate dislocation glide. A decrease in the lattice dislocation density slows down to \( \frac{\partial \rho}{\partial \varepsilon} \sim 3 \times 10^{15} \text{ m}^{-2} \) in the strain interval 4–8%. The number of mobile dislocations entered to lath boundaries becomes higher than the number of annihilation events and, therefore, the average lath boundary misorientation slightly increases. At relatively large strains of \( \varepsilon \geq 8\% \), the densities of both the lattice dislocations (\( \rho \)) and the dislocations with like signs (\( \rho^* \)) do not change with strain until rupture.

4.2. Evolution of Internal Stress

The internal stresses (\( \tau_{\text{X-Ray}} \)) evaluated by the X-ray analysis are attributed to the dislocation density in the lath interior (\( \rho \)). The relationship between \( \tau_{\text{X-Ray}} \) and \( \sqrt{\rho} \) clearly shows that the internal stresses can be expressed using a well-known relationship:

\[
\tau_{\text{X-Ray}} = \alpha Gb \sqrt{\rho} \quad \text{........................(5)}
\]

for \( \alpha = 0.17 \) (Fig. 11). Decreasing the \( \rho \) value with strain leads to decreasing the internal stress. There is, however, a discrepancy between the \( \tau_{\text{X-Ray}} \) value and the internal stresses associated with the local lattice curvature as determined by Eq. (1) (\( \tau_{\text{TEM}} \) in Fig. 8) in the strain interval 4–8% of tertiary creep. Upon apparent steady-state and tertiary creep in the strain interval 4–8% the operation of lath boundary sources produces excess of dislocations with like signs and \( \tau_{\text{TEM}} > \tau_{\text{X-Ray}} \). As a result, the local internal stresses within one lath retain at a high level until relatively large strains. At \( \varepsilon > 8\% \), the tertiary creep is accompanied by a gradual annihilation of dislocation excess in rather coarse laths and \( \tau_{\text{TEM}} \sim \tau_{\text{X-Ray}} \). However, the lath structure does not completely replaced by subgrains with stress-free boundaries and the creep strength breakdown was not observed under the studied conditions.

A dispersion of secondary phase particles plays an important role in the recovery of the dislocation lath structure. A gradual detachment of the lath/subgrain boundaries from the boundary particles during creep promotes the knitting reaction and the release of internal stress and thus accelerates the creep rate.

4.3. Threshold Stresses

The presence of threshold stresses is suggested by the variation in the stress exponent, i.e., the increase in the stress exponent with decreasing strain rate, shown in Fig. 2(d). The steady-state strain rate of the material, during which the dislocation movement is impeded by the threshold stresses, is generally represented by a relationship with the following form:

\[
\dot{\varepsilon}_{\text{min}} = A(\sigma - \sigma_{\text{th}})^n \quad \text{..........................(6)}
\]

where \( \sigma_{\text{th}} \) is the threshold stress, \( n \) is the true stress exponent. To estimate the threshold stress, the experimental data corresponding to minimal creep rates were treated as follows. Several curves of \( \dot{\varepsilon}_{\text{min}} \) vs \( \sigma \) were plotted using different \( n \) (Fig. 12). It is found that a stress exponent of \( n = 5.5 \) yields the best linear fit for \( \dot{\varepsilon}_{\text{min}} \) and \( \sigma \). Extrapolating this plot to a zero strain rate yields a threshold stress of 88 MPa.

Threshold stresses are commonly related to dispersed particles. In P92-type steels, the M23C6 and Laves phase particles mostly precipitate at the various boundaries between the lath, block and prior austenite grains.
and may serve as obstacles to dislocations moving along laths with interparticle spacing equal to lath thickness. For dislocations moving across laths, the threshold stresses can originate from MX-type particles, which are homogeneously distributed throughout the martensitic structure.\textsuperscript{13–9,14,16,22,40) Several models of the dislocation-particle interactions have been developed to evaluate the threshold stress.\textsuperscript{23,41–43) The threshold stresses can be compared to the Orowan stress, $\tau_0$, which causes dislocation bowing between particles:\textsuperscript{41) $\tau_0 = 0.84Gb/\lambda$, \hspace{1cm} (7) 

where $\lambda$ is the interparticle distance, and $d$ is the particle size. Another model relates the threshold stress to the extra back stress, $\tau_b$, required to generate sufficient additional dislocation length to climb past an obstacle:\textsuperscript{41,42) $\tau_b = 0.3Gb/\lambda$, \hspace{1cm} (8) 

Another model uses the detachment stress, $\tau_d$, required to break the dislocation from the particle after the climb has finished: $\textsuperscript{41,43)}$ $\tau_d = Gb(1-K^2)^{1/2}/\lambda$, \hspace{1cm} (9) 

where $K$ is a relaxation parameter. The magnitudes of the theoretical threshold stresses corresponding to particle distribution at a creep strain of 1% are given in Table 1 along with the experimental values for the threshold and internal stresses. The following parameters were used to calculate the theoretical values of $\tau_0$, $\tau_b$, and $\tau_d$: $G = 60$ GPa at $650^\circ$C,\textsuperscript{44) $b = 2.48 \times 10^{-10}$ m.\textsuperscript{46) The planar spacing between particles is $\lambda = 0.5d$, where $d$ is the MX particle size.\textsuperscript{5,38) The $F_v$ is the volume fraction of the MX particles, i.e., $0.23\%$ and $d$ is the average MX particle size.\textsuperscript{5) The $\Lambda=230$ nm is lower than lath thickness and, therefore, contribution of boundary particles to threshold stress can be discarded. A relaxation parameter, $K$, was assumed to be 0.6.\textsuperscript{42,43)} It should be noted that the exact magnitude of the relaxation parameter cannot be estimated because of the numerous assumptions and approximations used during the treatments.\textsuperscript{43) A low relaxation parameter of 0.6 was chosen to provide the best match to the experimental data. However, numerous studies on detachment stresses have reported $K$ values of more than 0.8.\textsuperscript{1,41–44) Therefore, the real operating detachment stress should be less than those presented in Table 1.

The Orowan and internal stresses measured by the X-Ray and TEM methods are quite close to the experimental threshold stresses. It should be noted that the Orowan mechanism effectively operates at low temperatures and at rather high strain rates, whereas moving dislocations can overcome the obstacles via diffusion-controlled local climbing under creep conditions at elevated temperatures.\textsuperscript{2,41–45) It is unlikely that the Orowan mechanism is responsible for the creep behaviour under the studied conditions. The local climb model predicts a threshold stress approximately 40% of the experimental one and, therefore, can contribute to the creep resistance of the steel.

The various models presented above as well as the TEM and X-Ray analyses predict possible threshold stresses quite close to the experimentally determined value (Table 1). Probably, several dislocation restraining mechanisms operate concurrently and contribute to the overall creep resistance. Dispersed particles and forest dislocations can be overcome by any moving dislocation with the assistance of local energy fluctuations (thermal activation). The processes of thermally activated unpinning have been found to become significant at small particle size and high temperatures.\textsuperscript{46) However, the internal stress raised by dislocations with like signs and non-equilibrium lath boundaries should affect the dislocation motion irrespective of thermal activation. These stresses should provide an upper limit to the threshold stress. To clarify the effect of the various mechanisms retarding the dislocation motion and creep behaviour, the relationship between the creep rate and effective stress ($\sigma - \sigma_{th}$) is plotted in Fig. 13 using the different threshold stress models. The best agreement to the experimental results was obtained for the threshold stresses calculated from the local lattice curvatures. Therefore, the improved creep resistance in the P92-type steels results from the concurrent operation of several mechanisms that retard the dislocation motion. Namely, the threshold stresses of the dislocation climbing/detachment, i.e., those associated with dispersed particles, are enhanced by the internal stress from large lattice distortions inherent to the martensitic lath substructure.

The positive effect of the boundary carbides on the retardation of knitting reaction is attributed to hindering dislocation motion. In addition, matrix MX carbonitrides effectively pin lattice dislocations moving across and along

<table>
<thead>
<tr>
<th>Creep Strain, $%$</th>
<th>Orowan stress</th>
<th>Local climb</th>
<th>Detachment stress</th>
<th>Internal stresses (TEM)</th>
<th>Internal stresses (X-Ray)</th>
<th>Experimental threshold shear stress</th>
</tr>
</thead>
<tbody>
<tr>
<td>Eq. (7)</td>
<td>Eq. (8)</td>
<td>Eq. (9)</td>
<td>$\tau_{EM}$ (Eq. (1))</td>
<td>$\tau_{X-Ray}$</td>
<td>$\tau_a = \sigma_{th}/\sqrt{3}$ [26]</td>
<td></td>
</tr>
<tr>
<td>0</td>
<td>62</td>
<td>19</td>
<td>51</td>
<td>76</td>
<td>66</td>
<td>–</td>
</tr>
<tr>
<td>1</td>
<td>62</td>
<td>19</td>
<td>51</td>
<td>49</td>
<td>47</td>
<td>51</td>
</tr>
<tr>
<td>4</td>
<td>62</td>
<td>19</td>
<td>38</td>
<td>43</td>
<td>34</td>
<td>–</td>
</tr>
<tr>
<td>8</td>
<td>53</td>
<td>16</td>
<td>44</td>
<td>40</td>
<td>26</td>
<td>–</td>
</tr>
<tr>
<td>18</td>
<td>42</td>
<td>13</td>
<td>34</td>
<td>22</td>
<td>22</td>
<td>–</td>
</tr>
</tbody>
</table>

Fig. 13. Relationship between the creep rate and the effective stress, which clarifies the effect of various mechanisms retarding the dislocation motion.
laths. Synergetic effect of dispersoids and lath boundaries on dislocation motion provides slow rate of the knitting reaction and retards relaxation of internal stresses in advanced 9% Cr martensitic steels.

5. Conclusions

The evolution of the dislocation substructure in tempered P92-type martensite steel was studied during creep at 923 K. The main results are summarised as follows:

(1) An average lath size of 330 nm and dislocation density of $6.2 \times 10^{14} \text{ m}^{-2}$ within the lath were obtained for the steel after tempering at 993 K for 3 hours. The tempered structure was characterised by large curvatures in the lattice, which resulted in internal stresses of 75 MPa and were attributed to the high density of interior dislocations with like signs as well as the non-equilibrium state of martensite lath boundaries.

(2) The average misorientation of the lath boundaries was approximately $2.5^\circ$ and did not vary significantly during creep under 118 MPa, while the mean lath size increased to 740 nm and dislocation density decreased to $\sim 10^{14} \text{ m}^{-2}$. The lath growth and dislocation recovery were accompanied by a release of the internal stress. Following a rapid twofold decrease during the transient creep, the internal stresses associated with the large lattice curvatures barely changed upon creep during the accelerated creep stage at large strains.

(3) The stress dependence of the minimal creep rate obeyed a power law function with a stress exponent, which increased from 11 across the strain rate range from $10^{-6}$ – $10^{-5} \text{ s}^{-1}$ to 17 across the range from $10^{-8}$ – $10^{-7} \text{ s}^{-1}$, that suggested a threshold stress of $\sim 50 \text{ MPa}$.

(4) For a creep strain range of 1–8%, the lath interior retained large lattice curvatures and had internal stresses of 40–50 MPa, which are comparable to the experimentally estimated threshold stresses at a creep strain of 1% (roughly corresponding to the minimal creep rate). Both the dispersion strengthening and the high internal stresses inherent to the martensite lath substructure are responsible for the superior creep resistance of P92-type steels.

Acknowledgements

The study was financially supported by the Russian Science Foundation, under grant No. 14-29-00173. The authors are grateful to the staff of the Joint Research Center, Belgorod State University for their assistance with instrumental analysis.

REFERENCES