Recovery Processes in the Ferrite Phase in C–Mn Steel

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The static recovery kinetics in ferrite were investigated by means of the stress relaxation technique. Samples were deformed in compression for a variety of temperatures, strains and strain rates.

The internal stress was found to decrease as recovery proceeded. In addition for a given relaxation time, the internal stress was found to decrease with increasing temperature. The results were analysed using a model previously proposed in the literature, the main parameters being activation energy and activation volume. At temperatures between 150 and 300°C the activation energy for recovery was close to that for dislocation core diffusion, whilst between 450 and 600°C it was close to the value for lattice diffusion of α-Fe. The activation energy was found not to depend on strain and strain rate.

The activation volume for the lower temperature regime was approximately constant whilst for the higher temperature regime it decreased with increasing temperature. The activation volume was found not to depend on strain or strain rate.

Analysis of the activation energies and activation volumes suggests that above 450°C, the rate controlling recovery mechanism is due to thermally activated glide of jogged screw dislocations, which are decorated with carbon solute atoms.

KEY WORDS: stress relaxation; ferrite; recovery; activation energy; activation volume.

1. Introduction

Intercritical rolling is one of the new, very promising hot rolling strategies for making thin hot rolled steel strip for C–Mn steels,1–5) as it enables the production of thinner strip without requiring higher rolling forces and mill investments. However, the control of the sheet thickness and the final sheet properties is much more complex than in the standard austenitic rolling strategies. The origin of the increased complexity is in the concurrency of several processes: austenite recovery, austenite recrystallisation, deformation stimulated transformation, ferrite recovery and ferrite recrystallisation. Much research has been done on some of these processes, in particular the recrystallisation of the austenite6–8) and the deformation stimulated transformation,2,9,10) but so far recovery, in particular recovery in the ferritic state, has received only little attention.11) However, for the development of a reliable integral model for microstructure development during intercritical rolling a better understanding of recovery kinetics after deformation in the ferritic region is highly desirable.

The present paper investigates the static recovery kinetics in ferrite in a low alloy C–Mn steel. The approach has been to experimentally determine the kinetics of recovery using the stress relaxation technique for a variety of temperatures, strains and strain rates. The kinetic data has been analysed using a model previously reported in the literature.12) From this model the activation energy and activation volume for recovery has been determined for a range of temperatures, strains and strain rates.

Whilst there is little data on ferrite recovery kinetics, there is a wide range of literature on high temperature creep of metals. Experimental studies have revealed the activation energies and activation volumes for creep in e.g., pure BCC iron13) and niobium.14) At the same time, classical theories of creep in the literature (e.g., Barrett and Nix,15) Gibbs16) and Evans and Knowles17) have been developed, which can be used to explain the rate controlling mechanisms of dislocation motion during deformation. Therefore for the current investigation, this literature shall be used to discuss the rate controlling mechanisms for recovery in ferrite.

Finally the substructure after deformation is presented in the form of scanning electron micrographs and an electron backscattering diffraction orientation map, which were obtained from specimens quenched during stress relaxation tests.

2. Recovery in Metals

Recovery generally involves both the annihilation of dislocations and their reorganisation into lower energy configurations, e.g., cells or subgrains. These two processes can occur both during deformation (dynamic recovery), and on subsequent annealing, (static recovery). As stated earlier, this investigation is concerned only with static recovery.

Generally static recovery models in the literature can be grouped according to the detail in the dislocation structure
that they consider. In the most simple approach, recovery is modelled via the reduction of an overall dislocation density.\textsuperscript{12) In a more sophisticated model,\textsuperscript{19) a distinction is made between dislocations within subgrains and those that make up the subgrain boundaries. Recovery is then related to the dislocation density within subgrain interiors and to the subgrain size. However, when applying this model there are seven fitting parameters to consider. Whilst four parameters can be calculated, two of these parameters still require knowledge of dimensionless constants which need to be determined from experiments. Finally, one parameter can vary by several orders of magnitude, depending on the initial dislocation density within the cells.

Because of the above uncertainties, a model based on just the dislocation density is preferred for modelling the experimental results in the present study. The models that are known in the literature are now discussed.

The classical approach to modelling recovery has been developed by Kuhlmann (1948), Cottrell and Aytekin (1950) and Friedel (1964).\textsuperscript{19)

In the description of Friedel\textsuperscript{20) it is assumed that the recovery proceeds via thermally activated cross-slip of dislocations. The rate of decrease of the internal stress is given by:

\[
\frac{d\sigma}{dt} = -c \exp \left( -\frac{(Q_0 - V \sigma)}{kT} \right) \quad \text{...(1)}
\]

where \(\sigma\) is the stress due to dislocations, \(t\) is time, \(k\) is Boltzmann's constant, \(T\) is temperature, \(c\) is a factor having the dimensions of Pa s\(^{-1}\), \(Q_0\) is the activation energy for recovery and \(V\) is the activation volume. This volume can be written as:

\[
V = b^2 l_s \quad \text{...(2)}
\]

where \(b\) is the Burgers vector and \(l_s\) is an activation length.\textsuperscript{21) According to Nes,\textsuperscript{21) it is approximately equal to the spacing of the rate-controlling obstacles to dislocation glide, e.g. the spacing of forest dislocations or jogs, or the spacing between solute atoms.

The activation length is therefore expected to be of the order of nanometres. For an aluminium–2.5wt%Mg alloy Verdier \textit{et al.}\textsuperscript{12} found that the activation volume varied between 22b\(^3\) and 46b\(^3\). With \(b\) for aluminium being equal to 0.286 nm,\textsuperscript{29) this corresponds to activation lengths around 10 nm.

The model can be experimentally tested by fitting the model expression to the data using \(Q_0\), \(V\) and \(c\) as fitting parameters.

A more recent approach is due to Verdier \textit{et al.}\textsuperscript{12} The model assumes that the internal stress relaxation is due to thermally activated dislocation annihilation and reorganisation, and therefore to plastic relaxation. The plastic relaxation rate \(\dot{e}\) is related to the change in internal stress \(\sigma\) by:

\[
\frac{d\sigma}{dt} = -\dot{e} E \quad \text{...(3)}
\]

where \(E\) is Young's modulus. The relation between \(\dot{e}\) and the dislocation behaviour is given by:

\[
M \dot{e} = pb\dot{e} \quad \text{...(4)}
\]

where is the Taylor factor (\(M=2\) for BCC metals\textsuperscript{1}), \(p\) is the dislocation density, and \(v\) is the dislocation velocity. The dislocation velocity is thermally activated and influenced by \(\sigma\) according to:

\[
v = b\dot{e} \frac{Q_0}{kT} \exp \left( -\frac{\sigma}{kT} \right) \quad \text{...(5)}
\]

where \(v_0\) is the Debye frequency. This results in the following overall equation for the relaxation of the internal stress:

\[
\frac{d\sigma}{dt} = -\frac{E b^2 \dot{e} v_0}{M} \exp \left( -\frac{Q_0}{kT} \right) \sinh \left( \frac{\sigma v_0}{kT} \right) \quad \text{...(6)}
\]

The dislocation density is then converted to a stress due to dislocations only, using:

\[
\sigma = M \alpha G b \sqrt{\rho} \quad \text{...(7)}
\]

where \(G\) is the shear modulus and \(\alpha\) is a constant of the order of 0.3.\textsuperscript{15) The shear modulus dependency on temperature for ferrite is given by\textsuperscript{22}:

\[
G = 64 \times 0.0001 \times (1 - 0.00044(T - 300)) - 0.032(T - 573) \quad \text{...(8)}
\]

where \(G\) is in MPa and \(T\) is in Kelvin. Combining Eqs. (6) and (7) with \(G=3E/8\), the following is obtained:

\[
\frac{d\sigma}{dt} = -\frac{64 E s^2}{9 M \alpha^2 E} \exp \left( -\frac{Q_0}{kT} \right) \sinh \left( \frac{\sigma v_0}{kT} \right) \quad \text{...(9)}
\]

To test the model on experimental stress relaxation data requires \(Q_0\) and \(V\) to be used as fitting parameters.

Comparison of Eqs. (1) and (9) shows that the exponential term in Eq. (1) is replaced by a sinh term in Eq. (9), and secondly that \(c\) in Eq. (1) is replaced by \(4\sigma E/9 M \alpha^2\) in Eq. (9).

For modelling the experimental work, Eq. (9) was preferred over Eq. (1), since the approach of Verdier \textit{et al.} is both physically and mathematically more rigorous.

It should be noted that models previously developed to describe the high temperature steady state creep of metals\textsuperscript{13,16) are similar in form to Eqs. (1) and (9). According to Barrett and Nix\textsuperscript{13)} the steady state creep rate (strain rate) is given by:

\[
\dot{e} = 2\pi \rho \beta \frac{b}{a_0} D_0 \exp \left( -\frac{Q_0}{kT} \right) \sinh \left( \frac{\sigma v_0}{2kT} \right) \quad \text{...(10)}
\]

where \(\rho\) is the mobile screw dislocation density, \(\beta\) is the number of atoms per unit cell, \(a_0\) is the lattice parameter and \(D_0\) is a constant. The reason for the similarity is that in both processes the rate is thermally activated and influenced by an acting stress.

3. Experimental

The steel used for the present study has the composition 0.19 wt% C, 0.445 wt% Si, 1.46 wt% Mn and 0.033 wt% Al.

Cylindrical samples were machined from rolled plate, with diameter 10 mm and length 12 mm. The samples were machined with the axis parallel to the rolling direction.
To investigate the static recovery kinetics, a Gleeble® 3500 thermo-mechanical simulator was used. Each test comprised of three stages. Firstly a heat treatment schedule was applied. Samples were heated via electrical resistance heating (under vacuum) to 1100°C to austenitise for 3 min. Then samples were cooled to 679°C (in the two-phase region) and held for 10 min. (This was done for comparison with future intercritical deformation tests.) Next, the samples were cooled at a rate of 5°C/s to the desired test temperature and held for 5 min.

In the second stage the samples were deformed in compression. lubrication was provided via graphite paste. To protect against the possibility of carbon pick-up during the test, tantalum sheets were used as a protective layer between the sample and the lubricant.

Finally in the third stage, the recovery after deformation was monitored. This was achieved by measuring the stress required to maintain a constant strain in the sample. After 50 min, samples were water quenched at a quenching rate of around 20°C/s. In addition some samples were quenched after 120 min relaxation.

Samples quenched after relaxation provided material for examination via electron backscattering diffraction (EBSD) and scanning electron microscopy (SEM) techniques.

Three series of stress relaxation tests were carried out. In the first series the effect of temperature on the recovery kinetics was investigated. Test temperatures used were in the range 150–650°C. The strain for each test was 0.15, whilst the strain rate used was 0.1 s⁻¹.

In the second series of tests the effect of strain was investigated by carrying out tests with strains in the range 0.05–0.25. The strain rate was kept constant at 0.1 s⁻¹, whilst the temperature used for each test was 550°C.

In the third series of tests the effect of strain rate was investigated using rates of 0.01–0.6 s⁻¹. The strain was maintained at 0.15 for each test whilst the temperature used was 550°C. In addition, multiple measurements were performed to test the reproducibility.

Finally one relaxation test was carried out with a higher strain of 0.5, using a strain rate of 0.6 s⁻¹.

To compare the experimental data with the recovery model, the experimental flow stress values during relaxation are converted to stress values due to dislocations σ₀ according to:

\[ \sigma_0 = \sigma_f - \sigma_y \]  \hfill (11)

where \( \sigma_f \) is the experimental flow stress and \( \sigma_y \) is the yield stress of ferrite. The yield stress was estimated from the stress–strain curves for each test temperature. However, since the carbon concentration of this steel is 0.19 wt%, the yield stress obtained for each temperature is an upper estimate of the yield stress for ferrite at that temperature, due to the presence of pearlite or bainite. For the data at higher temperatures (550 and 600°C), the flow stress data begins to approach constant values at long recovery times. Thus for high temperature data the yield stresses of ferrite could be estimated better by evaluation of the plateaus.

4. Results and Discussion

4.1. Effect of Temperature on Recovery Kinetics

Example stress relaxation curves are shown in Fig. 1, for the series of tests concerning the effect of temperature.

Firstly, considering only the experimental curves, we can see that for every temperature the stress decreases with time. This is due only to recovery, since there is no distinctive three-stage curve as would be observed if recrystallisation had occurred.21)

The effect of increasing temperature on the recovery kinetics is twofold. Firstly the initial stress decreases. Secondly the stress relaxation rate increases.

The first effect is due to a larger degree of dynamic recovery during deformation. The second effect is explained by the model, i.e. the motion of dislocations can be considered to occur by thermally activated mechanisms.

4.2. Comparison of Model with Experiment

Figure 1 also shows the curves obtained using Eq. (9). The best fits to the data were obtained by allowing both \( Q_0 \) and \( V \) to vary for each separate curve. Attempts to fit the model to all the experimental data by using a single value for \( Q_0 \) and a temperature dependent value for \( V \) did not give satisfactory results. As can be seen, there is good agreement between experiment and model.

Figure 2 shows the dislocation density evolution with time for both experimental data and the model. Dislocation densities were calculated from experimental \( \sigma_0 \) values by

![Stress relaxation curves for test temperatures of: (A) 450°C, (B) 500°C, (C) 550°C and (D) 600°C. Strain is 0.15 and strain rate is 0.1 s⁻¹. Experimental data is indicated by markers and the best fit from Eq. (9) by solid lines.](image1)

![Evolution of dislocation density during recovery. (A) 450°C, (B) 500°C, (C) 550°C, (D) 600°C. Strain is 0.15 and strain rate is 0.1 s⁻¹. Experimental data is indicated by markers and the model fit by solid lines.](image2)
applying Eq. (7). The modelled dislocation densities were obtained by applying Eq. (7) to the $\sigma_s$ values resulting from the model calculations.

From Fig. 2 it can be seen that the dislocation density generally decreases with increasing temperature. The exception is for the temperature of 600°C, where during the early stages of recovery, the dislocation density is higher than for the lower temperatures. This is likely to be caused by the error in determining $\sigma_s$ and the fact that $G$ from Eq. (8) does not take into account the effect of composition.

4.3. Activation Energy for Recovery

The activation energies that gave the best fit for each temperature investigated are presented in Fig. 3.

As can be seen from Fig. 3, $Q_a$ appears to rise with increasing temperature until 450°C after which it remains approximately constant. The activation energy for temperatures between 150°C and 300°C appears to be close to that for dislocation core diffusion ($Q_a = 174$ kJ/mol$^{25}$) in $\alpha$-iron. For temperatures between 450 and 600°C, the experimental data is in reasonable agreement with the activation energy for lattice self diffusion ($Q_a = 251$ kJ/mol$^{25}$) in $\alpha$-iron. For the temperature range 300–450°C the data suggests a transition region between the two processes.

This result compares well with the work of Michalak and Paxton$^{24}$ who studied the recovery of zone-refined iron during annealing between 300 and 500°C. Their work suggested a stress independent activation energy (i.e. equivalent to $Q_a$) of around the value for lattice self-diffusion. In the present study however, at temperatures of 300, 335 and 375°C, the analysis indicates a contribution from dislocation core diffusion to the observed activation energy.

In addition, a very similar dependency of the activation energy on temperature shown in Fig. 3, has been observed for creep in aluminium, copper, tantalum and tungsten.$^{17}$ At low temperatures the creep activation energy is due to dislocation core diffusion whilst at higher temperatures the activation energy is due to lattice self-diffusion.

As regards the rate determining mechanism of thermally activated dislocation motion, there are four possibilities: first, climb of edge dislocations, second, glide of jogged screw dislocations, third, cross-slip of screw dislocations and fourth, solute drag.$^{18}$

Solute drag cannot be rate controlling in this case since the results suggest activation energies for self-diffusion and dislocation core diffusion in $\alpha$-iron. Furthermore, thermally activated cross-slip of screw dislocations is expected to result in a linear decay of stress with time.$^{18}$ Since such a decay was not observed, this leaves only the first two mechanisms which can occur under the current conditions. These mechanisms are now discussed:

The climb of edge dislocations involves the formation and migration of jogs. If it assumed that the jogs are pre-existing, or form without thermal nucleation, the activation energy for jog migration and therefore recovery is given by$^{25}$:

$$E_{cl} + E_{m} = E_d$$

where $E_{cl}$ is vacancy formation energy, $E_{m}$ is the vacancy migration energy and $E_d$ is the self-diffusion activation energy. The activation energy for vacancy migration, and therefore jog migration can be smaller than $E_d$, since vacancies can diffuse also along the dislocation lines, i.e. $E_d$ becomes equal to the value for core diffusion.$^{25}$

The glide of jogged screw dislocations also involves the migration of jogs controlled by vacancy diffusion. Thus, the same argument presented above for climb of edge dislocations, also applies for this case, i.e. the activation energy will be either equal to that for self-diffusion, or equal to the core diffusion value.

Since both the above processes can have the same activation energies and both are likely to occur at the same time, the experimental activation energy, i.e. close to the core diffusion value or the self-diffusion value, does not enable a distinction to be made between these two processes.

4.4. Activation Volume for Recovery

From Fig. 4 it can be seen that the activation volume appears to be approximately constant at about $3 \times 10^{-28}$ m$^3$ between 150 and 375°C. Above 375°C it appears to rise to a peak value at 450°C before decreasing with increasing temperature. Comparison of Figs. 3 and 4 suggests that when the activation energy is close to the value for dislocation core diffusion, the activation volume can be treated as constant. When the activation energy is close to that for lattice self diffusion, the activation volume is a decreasing function of temperature.

The activation volumes observed in the present experiments can be used to calculate the activation length from
Eq. (13) stems from the fact that during deformation (and also on annealing) there will be both annihilation of jogs via lateral motion and creation of new jogs due to interaction of dislocations. When the rate of jog annihilation equals the jog creation rate an equilibrium jog separation is established. The ratio of the jog velocity ($\dot{v}_j \exp(-Q_j/kT)$), to the velocity of the screw dislocation ($\dot{\gamma} b/\kappa_s$), determines the size of the separation.

The trend of increasing activation length with increasing temperature has been observed experimentally for pure BCC iron,\textsuperscript{13,27} magnesium\textsuperscript{28} and pure niobium.\textsuperscript{14} However, according to Arieta and Sellars,\textsuperscript{29} for niobium containing HSLA steel, the activation volume for austenite was found to generally decrease with increasing temperature.

This difference in behaviour can be due to the effect of solute atoms (i.e. niobium) which can retard the lateral drift of jogs.\textsuperscript{18} If the jog velocity is reduced, it follows that the ratio of the jog velocity to the screw velocity can decrease as the temperature is increased. From Eq. (13) this will cause the equilibrium separation to decrease with increasing temperature.

In the current steel, examination of quenched samples (Sec. 4.6) suggests that carbon segregates to dislocations during deformation or relaxation. Thus the carbon atoms can act to retard the lateral drift of jogs on screw dislocations causing the observed reduction in activation volume with increasing temperature.

For the case of recovery controlled by climb of edge dislocations, if it is assumed that their annihilation consists of a glide movement followed by a climb movement, the activation length is given by\textsuperscript{18}:

$$ l_e = l_g + \frac{l_c}{l_g}$$

**Table 1.** Effect of strain and strain rate on recovery activation energy and activation volume. Temperature of deformation and relaxation is 550°C.

| Strain rate ($s^{-1}$) | $Q_e$ (kJ/mol) | $V$ ($10^{-9} m^3$) |
|------------------------|---------------|-------------------|
| 0.05                   | 217           | 1.6               |
| 0.15                   | 231           | 2.6               |
| 0.25                   | 228           | 2.6               |
| 0.15                   | 223           | 2.4               |
| 0.1                    | 231           | 2.6               |
| 0.5                    | 226           | 2.7               |
| 0.5                    | 236           | 2.2               |

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4.6. Analysis of Deformation Substructures

The substructures obtained after deformation at 550°C to a strain of 0.5 with strain rate 0.6 s\(^{-1}\), and to a strain of 0.15 with strain rate 0.1 s\(^{-1}\), are shown in Figs. 5 and 6 respectively.

In Fig. 5(A) a clear subgrain structure is visible (thinnest black lines correspond to a misorientation between 0.5° and 2°), together with precipitates present on both subgrain boundaries and grain boundaries. Figure 5(B) confirms the subgrain structure suggested by the EBSD map. In addition there is evidence of precipitation on the grain boundary and also the subgrain boundaries. The precipitates were identified, both from the EBSD pattern and by energy dispersive spectroscopy (EDS), as cementite. In addition, it was found that the precipitates on the subgrain boundaries in Fig. 5(A) were present where the misorientation was more than 3°.

Figure 6 shows a boundary separating two ferrite grains. It can be seen that there is a small contrast difference within the ferrite grains but no clear subgrain structure is visible. In addition there is some evidence of precipitation of cementite on the grain boundary.

The precipitates are likely to have been formed in the following way. First during deformation and subsequent relaxation, the carbon atoms in interstitial sites in ferrite can move towards the dislocations. This occurs by interaction of their elastic strain fields. The carbon atoms then form atmospheres around the dislocations. On subsequent quenching of the microstructure the matrix becomes saturated with carbon and due to the fast diffusion of carbon in ferrite (even at room temperature), precipitation occurs.

5. Conclusions

It has been shown that the stress relaxation technique is able to reveal the recovery kinetics of ferrite deformed over a range of temperatures, strains and strain rates. The recovery model of Verdier et al. has been successfully applied to the experimental stress relaxation data. The activation energy and volume for recovery were found to vary with temperature, whilst they did not vary with strain and strain rate. At low temperatures (150–300°C) the activation energy was found to be close to that for dislocation core diffusion. At higher temperatures (450–600°C) the activation energy agreed with that found for lattice self diffusion.

In the temperature range 150–375°C the activation volume was approximately constant whilst between 450–600°C it decreased with increasing temperature. This is in contrast with a number of experimental data in the literature, where the activation volume increases with temperature. It is suggested that a decreasing activation volume can occur, for a rate controlling process involving thermally activated glide of jogged screw dislocations contaminated by solute atoms (in this case carbon).

Finally, SEM and EBSD techniques showed that there was no substructure visible after relaxation at a strain of 0.15, whilst at a higher strain of 0.5 a clear subgrain structure was observed. For both cases there was some precipitation of cementite which was attributed to occur during quenching.

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