The Static and Metadynamic Recrystallization Behaviour of an X60 Nb Microalloyed Steel

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A rapid method has been developed to determine recrystallization kinetics of Nb microalloyed steels by interrupted hot torsion test. The softening behaviour was achieved as a function of different processing parameters. The method clearly identified three regions, where the strain dependency of the recrystallization rate varied. Firstly, at large strains the rate of recrystallization was not a function of strain; this is generally ascribed to metadynamic recrystallization. At lower strains the time to 50% recrystallization showed a power law relationship with strain, characteristic of static recrystallization. A further break point exists on the time for 50% softening curve when strain induced precipitation occurs in the material. The experimental results were used to estimate the time for 50% softening and to anticipate the onset of the strain induced precipitation for the alloy of this study. Grain refinement of the recrystallized austenite continued to strains significantly beyond the peak stress and beyond the static to metadynamic recrystallization rate transition.

KEY WORDS: static recrystallization; metadynamic recrystallization; Nb steel; thermomechanical processing.

1. Introduction

The recrystallization behaviour of austenite plays a major role in determining the final microstructure in thermo-mechanically processed steels. Three dominant recrystallization processes have been proposed: dynamic, static and metadynamic recrystallization. Each of these can affect the microstructural evolution under different processing conditions. For most commercial hot deformation processes static recrystallization (SRX) is the dominant mechanism. This is particularly important in microalloyed steels, where small levels of Nb, or other elements, are added to deliberately retard, or stop, static recrystallization. This leads to strain accumulation in the austenite which produces a refined ferrite grain size on transformation.

Many studies have been undertaken to estimate the rate of recrystallization as a function of the process parameters such as strain (ε), strain rate (ε̇) and temperature (T). Most of these studies are based on the early model developed by Sellars where the fraction recrystallized, X, is given by

\[ X = 1 - \exp \left( -0.693 \left( \frac{t}{t_{0.5}} \right)^n \right) \] ..........................(1)

where, X is the volume fraction of the recrystallized grains, t is time (s), t_{0.5} is the time to 50% recrystallization (s) and n is the Avrami constant. The value for t_{0.5} is modelled by

\[ t_{0.5} = A \epsilon^{-m} d_0^n Z \exp \left( \frac{Q_{\text{rex}}}{RT} \right) \] ..........................(2)

where, Q_{\text{rex}} is the apparent activation energy for recrystallization (J/mol), ε is the strain, d_0 is the initial grain size (μm), R is the gas constant, T is the temperature (K), Z is the Zener–Hollomon parameter (s^{-1}) and A, m, p, q are constants. The value for Z is given by:

\[ Z = \epsilon^k \exp \left( \frac{Q_{\text{def}}}{RT} \right) \] ..........................(3)

where, Q_{\text{def}} is the activation energy for deformation (J/mol) and k is the strain rate (s^{-1}).

Hodgson earlier developed a new method to determine the model co-efficients for C–Mn steels. This method involved determining the time to 50% softening at large strains, beyond the strain to the peak (in the stress–strain curve). At these strains recrystallization occurs both during (dynamic) and after (metadynamic) deformation. Metadynamic recrystallization (MDRX) is essentially a nucleation free static recrystallization after dynamic recrystallization (DRX). Under these conditions it has been shown the 50% softening in respect to MDRX can be estimated from:

\[ t_{0.5} = B Z^p \exp \left( \frac{Q_{\text{rex}}}{RT} \right) \] ..........................(4)
where, \(B\) and \(r\) are constants. The constant \(r\) in Eq. (4) for C–Mn steels has been measured as \(-0.8\). For typical values of \(\dot{Q}_{\text{def}}\) (300 kJ/mol) and \(Q_{\text{ms}}\) (230 kJ/mol) this equation essentially becomes only a function of the strain rate:

\[
\dot{t}_{0.5} = C \dot{\varepsilon}^{-0.8} \quad \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdots \cdOTS 

2. Method

The recrystallisation behaviour of a commercial Nb microalloyed (X60) pipeline steel of composition (wt%) C 0.07, Mn 1.5, Si 0.2, S 0.01, P 0.014, Ni 0.09, Cr 0.01, Mo 0.01, Cu 0.17, V 0.04, Nb 0.03, Ti 0.01, Al 0.035 was investigated in the current work. Torsion samples with a gauge length of 20 mm and a gauge diameter of 6.7 mm were prepared from transverse direction of a hot rolled plate. All tests were conducted using a hot torsion machine and general procedure described elsewhere.\(^\text{28}\) The specimen surface was protected from oxidation using argon.

The samples were reheated to 1 200°C for 2 min prior to cooling to 1 000°C, where they were deformed to a strain of 0.3 at a strain rate of 3 s\(^{-1}\), held for 30 s to achieve full recrystallization and an equiaxed initial grain size of 36 \(\mu\)m, and then cooled to the test temperature. Each test involved deforming to the required strain then holding for a given period and then re-deforming to a strain of 0.3. Softening was determined using conventional methods.\(^\text{30}\)

One of the features of this method is that it is possible to re-use samples after each test. Here the specimen was cooled through the transformation and then reheated to the soaking temperature with the pre-deformation conditions as above. This leads to a similar starting grain size and state of the microalloying elements. This is a distinct advantage of the torsion test and leads to rapid and efficient generation of the data, described below.

Some specimens were held to give a full recrystallization and quenched using water sprays from the deformation temperature for further microstructural investigation. The time \((\approx 0.15 \text{ s})\) for the quenched specimens to reach to ambient temperature was considered to be sufficient to freeze the deformed structure. Samples were cut from the gauge area of the torsion specimens for metallography. A heated \((\approx 80°\text{C})\) solution of saturated picric acid and Teepol was used to reveal the prior austenite grain. The average linear intercept grain size was measured for at least 10 random fields using computer aided image analysis at \(\approx 100 \mu\)m below the specimen surface which was cut longitudinally.\(^\text{30}\)

3. Results

The general principle of this approach is best illustrated by considering the results for 1 000°C. The true stress versus true strain curve shows the strain to the peak stress, \(\varepsilon_p\), is 0.88 (Fig. 1). An analysis of the work hardening rate utilising precise determination of the inflection point in \(\theta-\sigma\) plot reveals that the critical stress \((\sigma_c)\) for initiation of dynamic recrystallization was 141 MPa (Fig. 2). Details of this analysis are explained elsewhere.\(^\text{10}\) According to this analysis the strain for the critical stress \((\varepsilon_c)\) was 0.46 \((\approx 50\%\) of \(\varepsilon_p\)).

Here Eq. (4) suggested that at 1 000°C and a strain of 2.0 the time for 50% softening by metadynamic recrystallisation would be 0.2 s. In fact, at a strain of 2.0 the time to 50% softening was 0.5 s, consistent with solute retardation
by Nb on the rate of recrystallization. With this information the strain of the first deformation (note that the terms first or second deformation refer to those deformations after the initial roughing pass used to refine the austenite) was decreased with the same inter deformation time of 0.5 s. This continued until there was less than 50% softening; in this case at a strain of 1 where only 20% softening occurred. Assuming an Avrami exponent of 1 in Eq. (1) that is an estimate for a range of steels,\textsuperscript{12,13} it was predicted that the time to 50% softening was 2 s. By confirming this value (in some cases we confirm the new value in others we use an approximated value for the next step) the time to 50% softening at the next strain of 0.6 was estimated to be between 5 and 10 s. This estimate was based on a guess that the strain exponent in Eq. (2) was between $\frac{1}{2}$ and $\frac{3}{11}$.

Once the new value was obtained a plot of $\log t_{0.5}$ versus $\log \varepsilon$ was used to join the two data points and a linear fit assumed to provide estimates to $t_{0.5}$ at lower strains.

This process was continued to obtain Fig. 3 for the entire range of conditions examined. The main features to note from this are: (i) a sharp break in recrystallization behaviour at a strain of 0.8, which is close to the strain for the peak stress ($\varepsilon_p$) but considerably above $\varepsilon_c$ and, (ii) a linear behaviour below this critical strain which corresponds with an exponent of $-2.3$ in Eq. (2). The temperature was then decreased to 850°C to examine the effect of strain induced precipitation. Exactly the same procedure was followed.

At 1000°C above the critical strain $\varepsilon^{*} = 0.8$ the time for softening (0.3 s) was almost independent of prior strain indicating that MDRX was the dominant softening mechanism (Fig. 3). At strains below $\varepsilon^{*}$ the rate of softening was strain dependent and it was possible to describe this region using an m value of 2.2 (Eq. (2)). The critical strain $\varepsilon^{*}$ increased from 1.8 to 5.2 with a decrease in temperature from 900 at 850°C and there was no strain independent regime (Fig. 4).

The first change in static recrystallization behaviour at 850°C occurred at a strain of 1.1 and a holding time of 30 s, which was close to the critical strain for the initiation of dynamic recrystallization (break point A in Fig. 4). At lower strains than this critical strain, $\varepsilon_{ppt}$, there was an increased strain dependence (Fig. 4). It was possible to describe this region using an m value of 11. However, this concept that there is another recrystallization process is misleading as holding for longer times at a strain of 0.7 did not give a significant increase in softening. In other words, it is not possible to develop an equation similar to (2) to be used in Eq. (1) as the overall softening process is more complicated and rarely goes to completion in this high strain region. Hence, at these lower strains there has been strain induced precipitation (SIP) prior to complete recrystallization.\textsuperscript{6} A similar break point was observed at 875°C at a strain of 0.7 (break point B in Fig. 4) while at 900°C the effect of strain on the rate of recrystallization was very similar to 1000°C (Fig. 4). The strain induced precipitation was not also observed at a lower strain rate of 0.3 s$^{-1}$ and...
900°C (Fig. 5), even though the time for recrystallization was longer at 3 s⁻¹.

Evolution of the average austenite grain size in the absence of strain induced precipitation at 900°C was investigated by quenching the samples at the time estimated for 95% softening using an Avrami equation of \( n = 1 \). The results reveal that grain refinement in the hot worked material continues well above the peak strain \( \varepsilon_p \) and the strain at which softening becomes independent of the applied strain, termed \( \varepsilon^* \) in the current work (Fig. 6). This has been observed in other studies of C–Mn steels.¹⁴)

4. Discussion

The approach used in the current work to rapidly identify the recrystallization kinetics has been shown to be very useful for microalloyed steels. The earlier approach was able to determine the effect of process and microstructure variables on the recrystallization behaviour of plain C steels, highlighting the transition from strain dependent to strain independent recrystallization.¹⁰) In the current work it has been shown that this method can also provide a rapid estimate for the critical strain below which strain induced precipitation becomes independent of the applied strain, termed \( \varepsilon_p \) in the current work (Fig. 6). This has been observed in other studies of C–Mn steels.¹⁴)

4.1. Kinetics of Recrystallization

The results revealed that metadynamic recrystallization, static recrystallization and strain induced precipitation occur after deformation. Complete MDRX occurs at strains above which the material saturates with DRX nuclei during hot deformation and therefore recrystallization occurs independent of the applied strain by a growth process.⁹) For instance, a complete strain independent MDRX was clearly observed at strain 1 for the conditions used at 1000°C (Fig. 4). A decrease in strain below the strain independent softening led to a classical SRX. The rate of softening decreased with a decrease in the applied strain due to a lower dislocation density which provides the driving force for SRX (that is a nucleation and growth process).¹⁵) The SRX, in agreement with previous studies ⁵,¹⁵) was observed in all annealing conditions for the alloy used in this work (Figs. 4 and 5). However, precipitation prevents the SRX at strains lower than the critical strain \( \varepsilon_{ppt} \) (Points A and B in Fig. 4) which leads to a significantly lower softening rate. Such break points were not observed in the rest of the softening \( t_{0.5} \) curves confirming that conditions for strain induced precipitation have not been met.

4.2. Estimation of the \( t_{0.5} \)

The model for estimation of the time of 50% MDRX shows a decrease in exponent of strain rate from \( n = 0.8 \) (for C–Mn steels) to \( n = 1.25 \) revealing a stronger effect of strain rate on MDRX for the present alloy (Eq. 9).

\[
t_{0.5(MDRX)} = 1.1 \varepsilon^{-1.25} 
\]

Equation (10) estimates the time for 50% SRX softening. The outcome of this model represents a better agreement with experimental data at all applied strains (Fig. 7) compared to the model proposed by Hodgson and Gibbs ⁵ (Eq. (7)). The apparent estimated activation energy for recrystallization is 60 kJ/mol higher than the activation energy reported in previous studies ⁵.

\[
t_{0.5(SRX)} = 2.9 \times 10^{-21} \varepsilon^{-2.5} \delta^{-0.3} \exp \left( \frac{390000}{RT} \right) 
\]
4.3. Initiation of Strain Independent Softening

MDRX should start at \( \varepsilon_c \) where the first dynamically recrystallized nuclei become available for the following post dynamic recrystallization (Fig. 3). However, strain independent softening, which is often termed MDRX in the literature, commences at a critical strain \( \varepsilon^* \) which can be larger than \( \varepsilon_c \). The conditions at which strain independent softening initiates has not been systematically studied. However, it seems that \( \varepsilon^* \) has a complicated relationship with \( \varepsilon_c, \varepsilon_p \) and strain to the steady state stress (\( \sigma_s \)).

For instance, Fig. 8 illustrates that an increase in \( Z \) value (with a \( Q_{SRX} = 360 \text{kJ/mol} \) for alloy of this study) leads to an increase in the ratio \( \varepsilon^*/\varepsilon_p \). This is in contrast to the \( \varepsilon_c/\varepsilon_p \) ratio which is almost constant (Fig. 9). A dramatic increase in \( \varepsilon^*/\varepsilon_p \) is observed when the \( Z \) value is between 2 \( \times \) 10^10 s\(^{-1}\) and 10^11 s\(^{-1}\). Without a detailed study of the complete microstructural evolution it is difficult to precisely ascertain the reason for this marked change. However, it is important to realise \( t_{0.5} \) is the time for a significant volume fraction of recrystallization. As proposed elsewhere,\(^{14} \) it is likely that there is a combination of true metadynamic (i.e. the growth of nuclei as grain formed dynamically) and static (i.e. nucleation and growth of new grain) recrystallization for strains between \( \varepsilon_c \) and \( \varepsilon^* \). Figure 8 shows that above the value 10^10 s\(^{-1}\) an increase in \( Z \) does not have a significant effect on \( \varepsilon^*/\varepsilon_c \). In fact the material failed before reaching to strain independent region in respect to these \( Z \) values (no data points available in Fig. 4).

The fact that the time for 50% recrystallization shows a similar strain relationship below and above \( \varepsilon_c \) suggests that \( t_{0.5} \) is being determined by conventional static recrystallization. However, it is also important to note that the volume fraction of dynamically recrystallized material is also a strong function of strain between \( \varepsilon_c \) and the steady state strain (which is difficult to accurately estimate due to the deformation heating, but for example at 1000° C and 3 s\(^{-1}\) (Fig. 1)) would appear to be in the range, 2.2–2.5) and that \( t_{0.5} \) may also be strongly affected by this until a critical volume fraction is reached. More work is required to understand this.

One of the critical factors to emerge from this, though, is that it is necessary to understand the evolution of the microstructure in these steels from the onset of dynamic recrystallization through to the steady state and then how this changes with holding time after deformation. A number of authors\(^{13,15} \) have even suggested discontinuities between conventional static and metadynamic recrystallization. This is clear from Fig. 3 if there was only data for softening before \( \varepsilon_c \) and for strain independent MDRX. At the critical strain the time would jump from approximately 0.8 to 0.3 s for 50% softening. With the full softening data, as here, it is clear there is no discontinuity.

In terms of grain refinement, it seems the critical strain \( \varepsilon^* = 0.1 \) in Fig. 6 does not correspond to the point at which maximum grain refinement is achieved in the recrystallized microstructure. Grain refinement to some extent was observed above the \( \varepsilon^* \) up to the strain 2.5 which was the strain for steady state dynamic recrystallization (Fig. 6). This seems surprising, as the lack of further increase in the rate of recrystallization at higher strains suggest some sort of steady state in the static process. This is an area that requires further clarification.

4.4 Estimation of the Precipitation Start Time

To be able to identify the time and strain at which SIP initiates the outcomes of the Eq. (1) and Eq. (6) are compared. Equation (1) with an \( n \) exponent of 1 estimates the time for 5% static recrystallization (\( t_{0.05} \)) 2 s at 850°C (break point A in Fig. 4). This time has a good agreement with the result of the Eq. (6) which approximates the time for 5% SIP 3.5 s. The same calculations for the break point at 875°C (point B in Fig. 4) estimate the time for SRX \( t_{0.5} = 3.7 \text{s} \) which again is close to the estimated time for 5% precipitation of 2.5 s. These outcomes show that at the break point (\( \varepsilon_{\text{ppt}} \)) the onset of the strain induced precipitation appears to overlap the time for initiation of SRX. Moreover, Fig. 4 illustrates that the value for \( \varepsilon_c \) and \( \varepsilon_{\text{ppt}} \) are very close at 850 and 875°C. The reason for this observation is not yet clear. However, these limited results suggest there may be a relationship between the \( \varepsilon_c \) and \( \varepsilon_{\text{ppt}} \) for the alloy of this study. These results suggest that the rapid method presented in this study may be utilised (i) to estimate the time for 5% precipitation due to SIP and (ii) to identify the strain for initiation of the SIP during hot deformation. Further investigation is required to identify the possible relationship between the SIP break point and the volume fraction of the recrystallized material in respect to the \( t_{0.5} \) curves.
5. Conclusions

A rapid hot torsion method was developed to follow the recrystallization behaviour of a Nb microalloyed steel over a wide range of process conditions. The method was capable of identifying regions where metadynamic and static recrystallization occurred. It also showed conditions where strain induced precipitation occurred prior to static recrystallization. It was possible to quickly develop accurate models for the ratio of static and metadynamic recrystallization in the absence of strain induced precipitation. The recrystallized grain size continued to be refined, even beyond the strain where the rate of recrystallization changed from strain dependent to strain independent.

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