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# Softening Kinetics of Plain Carbon Steels Containing Dilute Nb Additions

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**Abstract.** The recrystallisation and precipitation kinetics of a plain carbon steel with 0.017 % Nb were studied using the double-hit deformation technique for interpass holding of 5 and 20s. The present study focuses on the effect of prestrain and deformation temperature on recrystallisation behaviour of the investigated steel. The fractional softening was calculated based on the percentage difference between the areas under the interrupted and uninterrupted deformations flow curves. The $T_{5\%}$ and $T_{95\%}$, marking the beginning and end of recrystallisation, respectively, are determined as a function of strain. Quantitative microstructural studies validated the findings from the softening studies. The predicated results of recrystallisation regime are found to be in agreement with industrial observation and other experimental measurement for this steel. It can be seen that the dilute additions of Nb can influence the static recrystallisation of austenite under certain rolling condition which may lead to improved mechanical properties of steel.

## 1. Introduction

Microalloyed high strength steels containing single addition of niobium (Nb), titanium (Ti) or vanadium (V) or combination of these elements represents an important class of steels which have been subject to more scientific investigation than any other group of steel in last four decades. Thermo-mechanical controlled processing (TMCP), consisting of controlled hot rolling followed by controlled cooling immediately, have enabled the development of microalloyed steels with improved strength, toughness and weldability. The unique combination of properties arises from the precipitation of carbides, nitrides, or carbonitrides and the interaction of these precipitates with the process of recrystallisation and grain growth of austenite. In TMCP rolling practice, the austenite is heavily deformed at a temperature where there is minimum recovery and no recrystallisation of the deformed austenite. Subsequent cooling leads to the nucleation of the low temperature transformation products (i.e. ferrite, bainite) on the grain boundaries and sub boundaries of the deformed austenite, resulting in a fine and homogeneous microstructure after phase transformation [1, 2].

However, depending on the deformation conditions, the austenite may exhibit partial static recrystallisation behaviour within a certain temperature range. The upper and lower limits of partial recrystallisation region are commonly known as the recrystallisation-limit temperature ($T_{95\%}$) and the recrystallisation-stop temperature ($T_{5\%}$). Both of these temperatures are functions of several factors, including intrinsic parameters such as alloy chemistry and extrinsic processing parameters such as strain, strain rate per pass and interpass delay time [3, 4, 5]. It is essential to carry out finishing passes of TMCP at temperature below $T_{5\%}$ to get optimum benefits out of precipitation and recrystallisation interaction.

The work presented in this paper has been carried out on a plain carbon steel with 0.017 % Nb. The reason for choosing this composition is to study the influence of dilute additions of Nb on the static recrystallisation of austenite and thus final mechanical properties of such steels. Nb has the highest effect in raising the $T_{5\%}$ by means of solute drag and strain-induced precipitation in austenite under given deformation conditions in the finish rolling stage. Much of the published works on recrystallisation kinetics of low carbon microalloyed steels are for the Nb addition ranging from 0.03-

0.10 %. There is very less research work directed towards studying the effect of dilute additions of Nb during control rolling of low carbon steels.

The present study involves characterizing the recrystallisation kinetics using the “double-hit” deformation technique for interpass holding of 5 and 20s. A parameter called “fractional softening” used firstly by Kwon and DeArdo has been successfully employed as a method to determine any softening or hardening during deformation [6,7]. The effect of strain per pass and deformation temperature on the the lower ($T_{5\%}$) and upper limits ($T_{95\%}$) of recrystallisation has been evaluated based on softening criteria. Quantitative microstructure characterization of recrystallisation fraction and aspect ratios have further validated findings from the softening studies.

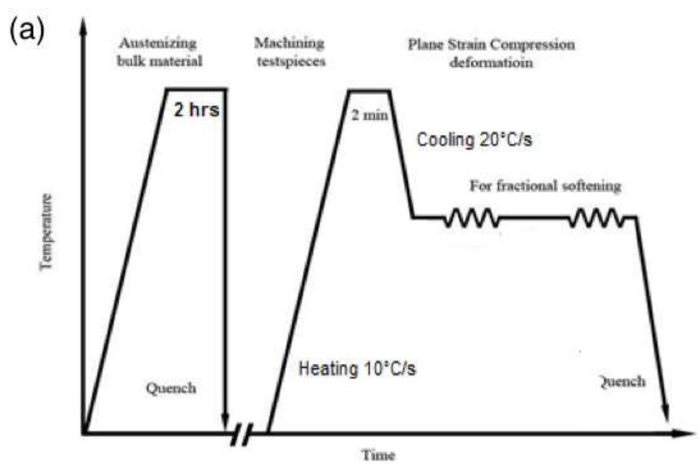
## 2. Experiment Details

The material used in the present study is a plain carbon steel microalloyed with dilute addition of Nb (designated “Nb200”) whose detailed chemical composition is given in Table 1. The steel melt was made through vacuum induction melting in a laboratory unit capable of producing 30 kg ingots at Tata Steel, UK facilities. The cast ingots were soaked at 1300°C for 2 h and hot rolled into thick plates of 25±3 mm by two passes in a 2-high experimental rolling mill. Prior to machining of test specimens, the as received plates were sectioned into equal lengths and soaked at 1250°C for 2 h in a box furnace filled with a $N_2$ atmosphere and subsequently water quenched to room temperature. The aim of this homogenization heat treatment was to dissolve the precipitates of Nb(C,N) back into solid solution and to restore a fully recrystallized equiaxed austenite structure prior to hot deformation studies.

Table 1. Chemical composition of the laboratory heat (wt %)

| Material ID | C | Si | Mn | P | S | N | Nb |
|---|---|---|---|---|---|---|---|
| Nb200 | 0.20 | 0.19 | 1.01 | 0.015 | 0.007 | 0.008 | 0.017 |

The plane strain compression (PSC) test specimens having a geometry of 60mm long x 30mm wide x 10mm high, were machined from the homogenized plates. The static recrystallisation studies were carried out using a “double-hit” test technique on a purpose-built servo-hydraulic thermomechanical compression (TMC) testing machine at The University of Sheffield. The specimens were reheated to 1250°C, held for 120 s to allow for equilibrium and then force-air cooled to respective deformation temperature. The hot deformation was undertaken in a double pass of equal magnitude for interpass holding time of 5 and 20 s. Following deformation, specimens were water quenched down to room temperature as shown in Figure 1 (a). A constant nominal strain rate of 15 $s^{-1}$ was used for all the experiments. Boron nitride spray was applied on all specimens as a lubricant to prevent any sticking between the specimen and the PSC tools during the high temperature deformation.



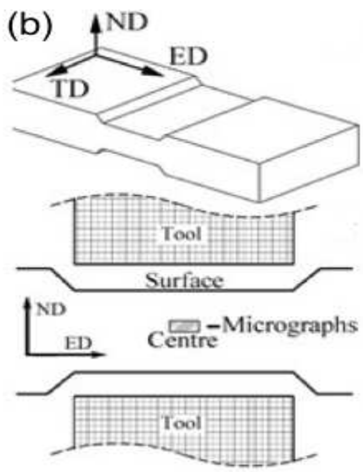




Figure 1. (a) Schematic representation of the TMCP cycles subjected to the PSC samples within the TMC machine; (b) position of the micrographs taken from deformed PSC test specimen

The "double-hit" deformation tests consisted of subjecting the specimen to a first pass of a given prestrain ($\varepsilon_1$), then unloaded, held for a predetermined interpass time, and then a second deformation of equal magnitude ($\varepsilon_2$=$\varepsilon_1$) was applied under isothermal condition. A total of three deformation temperatures, namely, 850°C, 950°C, and 1050°C, and four different levels of prestrain, 0.10, 0.20, 0.30, and 0.40, were used for the present study. For each deformation condition, an uninterrupted test was also carried out, which involved a monotonic deformation ($\varepsilon$) without any intermediate holding and of magnitude equal to the sum of the two deformations in the interrupted test ($\varepsilon_1$+$\varepsilon_2$).

The quenched specimens were sectioned along the rolling-normal direction (RD-ND) as shown in Figure 1 (b). The metallographic preparation and examination was carried out using a standard technique. The final polished specimens were etched with a solution consisting saturated aqueous picric acid plus wetting agent to reveal the prior austenite grain size. Quantitative microstructure characterisation was carried out using an optical microscope.

## 3. Results

### A. Fraction Softening/Hardening Studies

The load-displacement data of the TMC machine were recorded and later converted to equivalent stress-equivalent strain flow curves according to a standard procedure [8]. The fractional softening of austenite was then calculated based on the area under these flow curves. The details of this technique and the method of calculation are described elsewhere [6, 7, 9].

Figure 2 (a) shows the high temperature flow characteristics at different deformation temperatures obtained from the uninterrupted tests. The flow curves clearly show continuous strain hardening throughout the deformation, indicating no dynamic recrystallisation taking place during deformation at these temperatures. Hence, any softening observed during interpass holding in double-hit tests can be attributed to static restoration (i.e. recovery and recrystallisation) events. While Figure 2 (b) shows the flow stress curves obtained from both uninterrupted and interrupted tests at 950°C for a given prestrain of 0.40 ($\varepsilon_1$=$\varepsilon_2$=0.40) and interpass time of 5 s. The calculated softening fractions is negative i.e. hardening which can be seen from the equivalent stress-strain curve.

The the overall softening/hardening behaviour of austenite as measured from area under flow curves is presented in Table 2. The notable features are: (1) for any given prestrain, the amount of softening increases with increasing temperature; and (2) at any given deformation temperature, the amount of softening increases with an increase in prestrain. At 850°C, all the softening curves for both interpass time of 5 and 20s (except 0.10 prestrain) exhibit negative softening or in other words hardening.

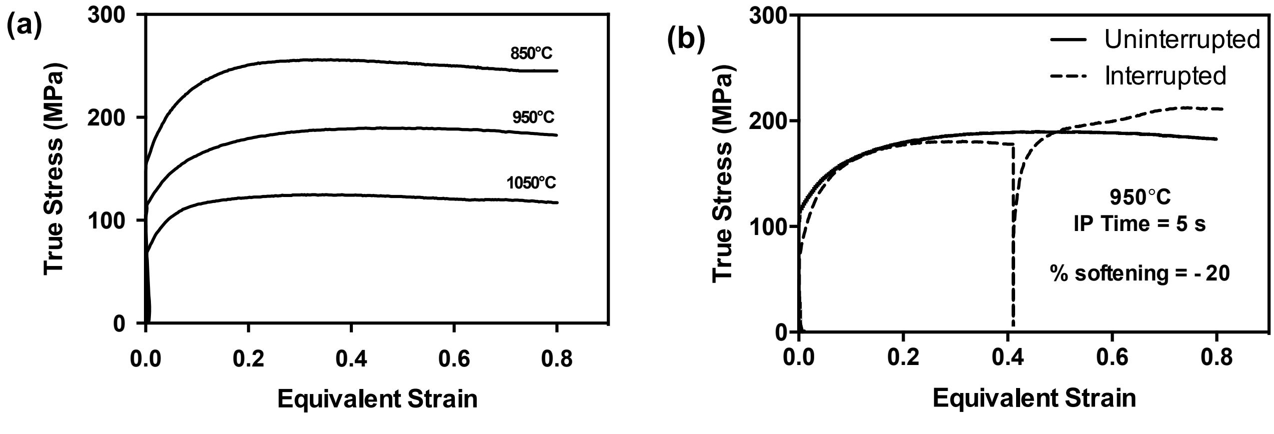



Figure 2 (a) Flow Stress curves at different deformation temperature during uninterrupted deformation; and (b) Flow Stress curves at deformation temperature of 950°C for a true strain of $\varepsilon$ = 0.40, interpass time of 5s. All tests were conducted at constant strain rate of 15 $s^{-1}$.

Table 2. % fractional softening as function of temperature for different prestrains

| Interpass Time (s) | Temperature (°C) | Prestrain | | | |
|---|---|---|---|---|---|
| | | 0.10 | 0.20 | 0.30 | 0.40 |
| 5 | 850 | 17 | - 8 | -3 | -20 |
| | 950 | 16 | 22 | 35 | -20 |
| | 1050 | 20 | 60 | 80 | 96 |
| 20 | 850 | 30 | -5 | -10 | -20 |
| | 950 | 26 | 32 | 47 | 20 |
| | 1050 | 30 | 60 | 80 | 90 |

**B. Microstructure Studies**

Figure 3 (a) shows the microstructure of a homogenized plate specimen, reheated at 1250°C for 2 h. An austenite microstructure of equiaxed grains and fairly uniform in size was observed. The mean austenite grain size was 230 ± 5 µm in the RD-ND direction. Figures 3 (b) through (d) shows microstructures of samples subjected to a true strain of 0.40 with interpass time of 5 s at deformation temperatures of 1050°C, 950°C, and 850°C, respectively. The specimens were quenched to room temperature immediately after the deformation. The micrograph shown in Figure 3 (b) exhibit a completely recrystallised microstructure consisting of small equiaxed grains distributed uniformly throughout the matrix. Figure 3 (c) exhibits deformed austenite microstructure but shows also beginning of recrystallisation while that shown in Figure 3 (d) exhibits a highly deformed microstructure with complete unrecrystallised grains. The grain size and an aspect ratio of recrystallized austenite were measured by the linear intercept method.

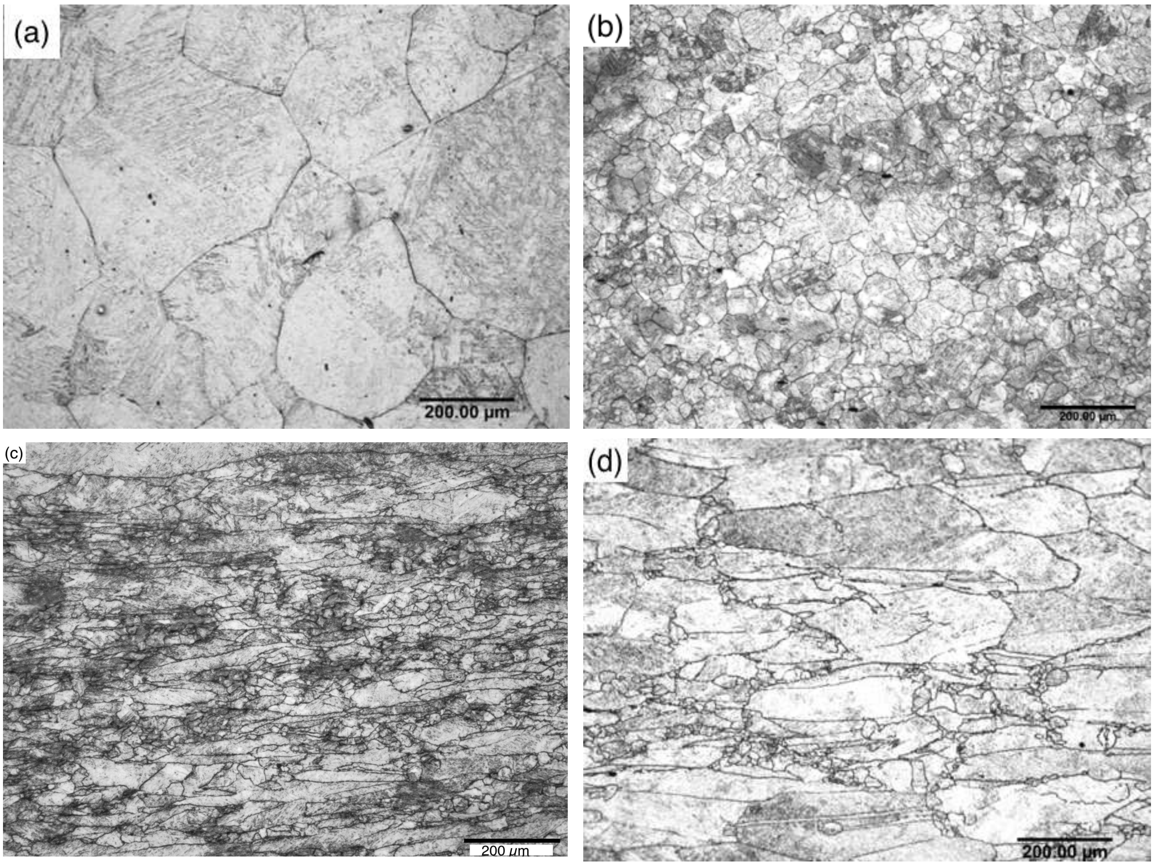



Figure 3. Light micrographs illustrating initial structure prior to deformation (a) 1250°C; deformed microstructure (b) 1050°C, (c) 950°C and (d) 850°C after double-hit test at a true strain (ℇ) of 0.40 and quenched.

## 4. Discussion

All of the softening curves at 850°C exhibited a negative softening (i.e. hardening) for both interpass time of 5 and 20s which is lowest deformation temperature in present study. As static restoration processes are slow at 850°C, the precipitation hardening due to strain-induced precipitation of Nb(C,N) dominates. This hardening of austenite is reported by earlier numerous microstructural investigation of low carbon microalloyed steels with higher Nb content.

A further increase in deformation temperature at 950°C resulted in softening up to 20% for interpass time of 5s up to prestrain of 0.30. This observed initial softening is attributed to recovery process of austenite alone. This results are in agreement with earlier investigators who had observed similar levels of softening due to recovery of austenite [3,10,11]. The softening fraction increased for longer interpass time of 20s for similar deformation conditions as shown in Table 2. It indicates that the time available in between two deformation passes is sufficient for static recrystallisation of austenite to occur. It is worth to note that negative softening (i.e. hardening) is observed for prestrain of 0.40 and interpass time of 5s. This could be due to strain-induced precipitation of Nb(C,N) which dominates over restoration processes for larger strain and recrystallisation is inhibited.

For low carbon microalloyed steels, the contribution from recovery to the overall softening is limited as a result of the medium stacking fault energy of the austenite. It was found that approximately 20% of the overall softening in such steels is due to recovery, which corresponds to the $T_{5\%}$ temperature, whereas about 60% of the overall softening can be correlated to the $T_{95\%}$ temperature [3,10,11]. The Figure 4 shows the regime of recrystallisation based on above softening criteria.

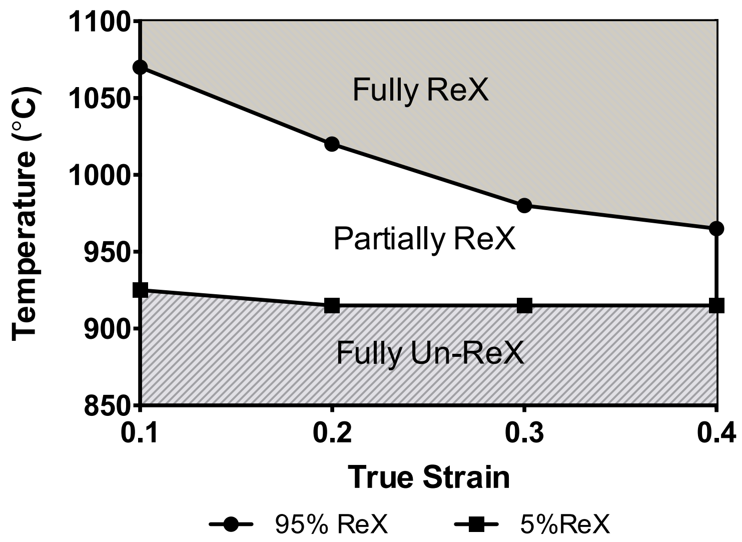



Figure 4. Effect of prestrain on the $T_{5\%}$ (corresponding to 5%Rxn) and $T_{95\%}$ (corresponding to 95% Rxn) temperatures

The interpretation of Figure 4 allows an important conclusion to be drawn: even a dilute addition of 0.017% Nb is sufficient under certain rolling condition to cause an appreciable inhibition of static recrystallisation of austenite. This means that when a steel being rolled in these conditions, when it reaches a deformation t emperature of 925°C, it no longer recrystallizes between successive rolling passes and will accumulate a residual strain.

It is well understood that recrystallisation is retarded only when the precipitation pinning force ($F_P$) becomes greater than recrystallisation driving force ($F_R$). In the present hot deformation study, the use of a well controlled PSC testing method enables the accurate determination of increase in flow stress and hence the stored energy can be measured effectively to calculate $F_R$. The $F_P$ will be estimated from the precipitate volume fraction data using TEM studies. The relationship between the recrystallisation-stop temperature ($T_{5\%}$) and net driving pressure ($F_R$ - $F_P$) will be established based on hot deformation studies and its results.

The ongoing research work involves the hot deformation studies of on similar carbon steel with dilute additions of Nb in 0.005 % (designated "Nb50") and 0.010 % (designated "Nb100") with identical experimental parameters. These will help to give fundamental understanding of effects of strain induced precipitation and solute drag effect of dilute Nb additions on static recrystallisation kinetics of austenite in low carbon steels.

## 5. Conclusions

1. The softening fraction analysis indicate that an increase in deformation temperature gives increase in softening fractions at constant strain rate and interpass time conditions.

2. An increase in prestrain leads to increase in softening for any given deformation temperature.

3. The fraction hardening observed at 850°C is attributed to the precipitation hardening caused by Nb(C,N) in deformed austenite and subsequent inhabitation of recrystallisation.

4. The $T_{5\%}$ and $T_{95\%}$ temperatures are determined by applying the softening criteria which are further confirmed by quantitative microstructural observations.

## 6. Acknowledgement

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