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# Dissolution and precipitation behaviour in steels microalloyed with niobium during thermomechanical processing

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ABSTRACT

The thermomechanical processing of high strength low allow (HSLA) steels during low-temperature roughing, followed by rapid reheating to higher temperatures was investigated to better understand the Nb dissolution kinetics in austenite, and the subsequent precipitation behaviour during the final finishing passes. For comparative purposes, two experimental 0.06 wt% C steels were studied, one containing 0.03 wt% Nb (Nb steel), and the second containing both 0.03 wt% Nb and 0.02 wt% Ti (Nb–Ti steel). Processing of these steels consisted of a simulated roughing schedule, with the final roughing pass taking place at 850 °C. The strain-induced precipitation intensity in the steels subsequently quenched where characterised using transmission electron microscopy. Following this, the steels were rapidly reheated at a rate of 10 °C/s to a temperature of 1200 °C, held at temperature for various times, and water quenched to room temperature so that both the precipitate dissolution kinetics, together with the austenite grain coarsening kinetics could be established.



## 1. Introduction

Thermomechanical controlled processes (TMCP), consisting of controlled hot rolling followed by controlled cooling, have been developed to improve strength, toughness and weldability of microalloyed steels [1,2]. The improvement in mechanical properties from TMCP is due to the refinement of the austenite microstructure, maximising the austenite boundary area and deformation band density, subsequently increasing the number of nucleation sites prior to the development of the transformation microstructure. The enhanced mechanical properties of low-carbon microalloyed steels thereby arises from a combination of the refined ferrite grain size and the dispersion hardening through the precipitation in ferrite [3–7].

Microalloying elements in steels, such Nb, facilitate grain refinement through precipitation of carbides/carbonitrides in austenite thereby inhibiting the static recrystallisation of austenite, resulting in a fine final microstructure. In addition, titanium has frequently been added to HSLA steels to enhance the control of the austenite and transformed ferrite grain sizes during both the deformation and subsequent heat treatment process [8]. Therefore, during TMCP, these microalloying elements can precipitate as carbides or carbonitrides to increase the nucleation sites for obtaining a fine ferrite grain size [9,10].

The consequence of a low finishing temperature is high energy consumption through high mill loads [11]. To improve the production efficiency and reduce energy consumption, a new rolling process is explored in this work, which involves the addition of a reheating process between rough and finish rolling. The effect of this reheating on the deformed austenite, and particularly the precipitate dissolution kinetics, is the focus of this study.

Much of the existing published work is concerned with the type and distribution of precipitates based on single microalloy additions with idealised solution treated and ageing experiments after deformation [9,11–15]. Less work has been directed towards the effects of multiple microalloy additions during the thermomechanical processing. Therefore, in this work, two types of Nb and Nb–Ti steels were selected for comparative reasons. These two steels were rough rolled at 850 °C, followed by the immediate reheating to 1200 °C. This low roughing temperature of 850 °C was below the recrystallisation-stop temperature ($T_{5\%}$) for each steel. The state of Nb (Nb in solution or Nb(CN) as a precipitate) was analysed as a function of hold time at this isothermal reheat temperature, to establish the precipitate dissolution and austenite grain coarsening kinetics. Through a comparison of the two steels, the effect of Ti on the NbC precipitates and dissolution kinetics has been be clarified.

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## 2. Experimental procedure

### 2.1. Compositions and processing conditions

The materials used in the research consisted of two laboratory cast microalloyed steels containing Nb (designated "Nb steel"), and Nb and Ti (designated "Nb–Ti steel"), which were made by vacuum induction melting and poured into ingots having dimensions 220 mm × 65 mm × 28 mm at ArcelorMittal. The compositions of these steels were analysed by Sheffield Testing Laboratories and are listed in Table 1. The ingots were soaked at 1300 °C for 2 h and hot-rolled from 28 mm to 12 mm thick plates by two passes in a 2-high experimental 50 tonne Hille rolling mill with a finishing temperature of 1100 °C followed by an ice water quench. Plane strain compression (PSC) specimens, having a geometry 60 mm long × 30 mm wide × 10 mm high, were machined from the hot-rolled plate. PSC testing was performed using the thermomechanical compression (TMC) machine at The University of Sheffield [16]. Samples were reheated to 1100 °C, held for 30 s and then force-air cooled to the deformation temperature of 850 °C. Deformation was undertaken in a single pass using a strain of 0.3 with a constant true strain rate of 10 $s^{-1}$. Immediately following this, the steels were rapidly reheated at a rate of 10 °C/s to a temperature of 1200 °C, held for various times at this temperature, and subsequently water quenched to room temperature, as shown in Fig. 1.

### 2.2. Grain size and microstructure

Longitudinal specimens were prepared for metallographic examination using standard techniques. The polished specimens were etched by picric acid at 60 °C to reveal the prior-austenite grain boundaries. The average austenite grain size was measured using the linear intercept method (ASTM E-112) by optical microscopy.

### 2.3. Micro-hardness testing

Micro-hardness testing was conducted using a Durascan 70 micro-hardness tester on the polished Nb and Nb–Ti steels. A Vickers diamond indenter tip was used with load of 1 N and a hold time of 15 s for each micro-hardness tests. An array of micro-hardness tests were performed at distances of 0.5 mm with forty test measurements on each sample.

### 2.4. Precipitates calculation

Transmission electron microscopy (TEM) was carried out to identify the strain-induced precipitation. Carbon extraction replica specimens were prepared in the standard manner using a light 2% Nital etch. The extraction replica samples were examined in FEI Tecnai 20 and JEOL 2010F TEM to observe the precipitates details. The particle diameter distribution of precipitates was measured with quantitative image software Image J. In addition, the chemical analysis for the precipitates was conducted using an Oxford instruments energy dispersive X-ray spectroscopy (EDX) detector (Oxford Instruments, Oxford, UK) and electron energy loss spectroscopy (EELS) analysis. The measurement of the sample thickness

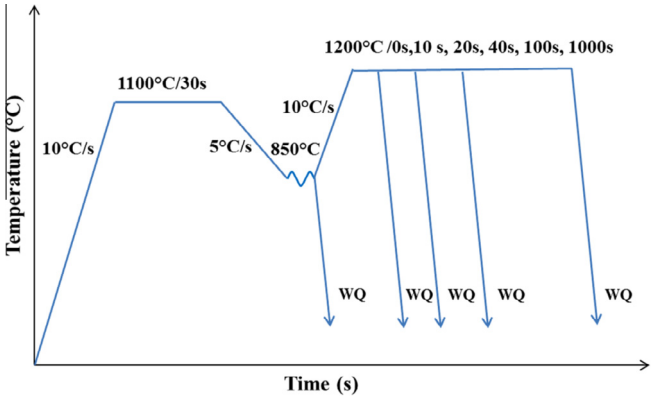


**Fig. 1.** Schematic representation of rough rolling and reheating process within the TMC machine, indicating the conditions where specimens were quenched to enable for microstructural characterisation.

was carried out using EELS in Gatan GIF. This was estimated from the low energy region of the spectrum, given by Eq. (1) [17,18]:

$$t = \lambda \ln (I_t/I_0) \quad (1)$$

where $t$ represents the thickness of material; $\lambda$ is the mean free path of specimen; $I_t$ is the total number of electrons in the EEL spectrum and $I_0$ is the number of electrons having lost no energy.

## 3. Results

### 3.1. Microstructures

Specimens were examined following rough rolling at 850 °C and reheating at 1200 °C for various holding times. Optical micrographs, Figs. 2 and 3, reveal different prior-austenite grain morphologies as a function of process conditions and composition. As the roughing temperature was below $T_{5\%}$, the deformed prior-austenite grains had been elongated and measured to be 18.7 ± 1.6 μm in the Nb steel and 16.9 ± 0.7 μm in the Nb–Ti steel (Figs. 2(a) and 3(a)). The deformed and reheated optical microstructures are presented in Figs. 2(b–f) and 3(b–f) for both the Nb and Nb–Ti steels, respectively. Figs. 2(b) and 3(b) show the samples reheated to 1200 °C followed by an immediate quench, which exhibited a fully recrystallised prior-austenite grain structure with an average size of 73.1 ± 3.9 μm in the Nb steel and 47.8 ± 3.6 μm in the Nb–Ti steel. When the steels were held for 10 s at 1200 °C, the recrystallised structure had coarsened slightly (Figs. 2(c) and 3(c)) with 86.4 ± 3.5 μm in the Nb steel and 50 ± 5.5 μm in the Nb–Ti steel. The prior-austenite grain size increased to 109.7 ± 2.9 μm at 20 s and 120.5 ± 3.9 μm at 40 s in the Nb steel, and 64.5 ± 2.5 μm at 40 s in the Nb–Ti steel. More coarsening occurred with an increase in holding time from 100 s to 1000 s, while the measurement of the average prior-austenite grain rapidly increased from 141.3 ± 5.3 μm to 260.7 ± 9.8 μm in the Nb steel, and from 91.2 ± 4.7 μm to 112.4 ± 5.4 μm in the Nb–Ti steel (Figs. 2(f), (g), and 3(e), (f)). Clearly the prior-austenite grain size of Nb steel grew more rapidly during the reheating process compared to the Nb–Ti steel.

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

| | C | Si | Mn | Cr | Ni | Nb | Ti | N | P |
|---|---|---|---|---|---|---|---|---|---|
| Nb steel | 0.067 | 0.11 | 0.77 | 0.01 | 0.02 | 0.03 | <0.01 | 0.0058 | 0.016 |
| Nb–Ti steel | 0.065 | 0.11 | 0.77 | 0.01 | 0.02 | 0.03 | 0.02 | 0.0062 | 0.016 |