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Influence of deformation temperature on the ferrite grain refinement in a low carbon Nb–Ti microalloyed steel

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Abstract

~~Grain refinement is one of the effective methods to develop new generation low carbon microalloyed steels possessing excellent combination of mechanical properties. In the present work, the microstructural evolution and ferrite grain refinement at various deformation temperatures were investigated using single pass isothermal hot compression experiments for a low carbon Nb–Ti microalloyed steel. The physical processes that occurred during deformation were discussed by observing the optical microstructure and analyzing the stress–strain responses. The results show that there is a close relation between the microstructural evolution and true stress–true strain responses during the deformation. Microstructural observation indicates that very fine ferrite grains of about 1.8–3 μm are obtained by deformation at 830–845 °C, about A_r , ± 10 °C. The obtained stress–strain curves suggest the occurrence of strain-induced dynamic transformation (SIDT) of γ to α at this deformation temperature range. © 2006 Elsevier B.V. All rights reserved.~~

~~Keywords: Low carbon microalloyed steel; Hot compression; Grain refinement; Strain-induced transformation~~

1. Introduction

The thermomechanical controlled processing (TMCP) of microalloyed steels has been employed for some times in the production of plates and sheet material in order to optimize mechanical properties. The central feature of thermomechanically processed steel is the ultrafine grain size in the final product. Therefore, the ferrite grain refinement of structural steels has attracted considerable interest from engineering scientists due to its unique role of increasing both strength and toughness. Demand for steels possessing good combination of these properties, and weldability has led to the development of low carbon microalloyed steels.

There are several TMCP routs to obtain the fine ferrite grain microstructure. Controlled rolling is one of the most important operations for producing low-cost, high strength steels with yield strength as high as 600 MPa [1]. A limiting ferrite grain size of around 5 μm appears to exist using commercial controlled rolling. In recent years, several groups have reported

achieving ferrite grain size below the nominal 5 μm limit of controlled rolling steels, using laboratory scale TMCP methods for low carbon microalloyed and plain carbon steels [2–5]. For producing ultra fine ferrite grain size (<2 μm), there are potentially three mechanisms [6–9]: (1) strain-induced transformation (SIT), (2) transformation from dynamically recrystallized austenite, and (3) dynamic recrystallization of ferrite. In recent years, several research groups have reported achieving very fine ferrite grain sizes via strain-induced transformation in plain carbon steels, which has been confirmed by optical microstructural observations [10–15]. However, there is very little information concerning ferrite grain refinement by SIT mechanism in the case of microalloyed steels. Furthermore, it is well known that hot deformation has important effect not only on the microstructural changes but also on the hot flow stress–strain curves. Therefore, in addition to optical microstructural observation, hot flow stress–strain curves can be used as another evidence to determine if SIT has been occurred during the deformation.

In the present work, the isothermal hot compression tests were conducted on a low carbon Nb–Ti microalloyed steel to study the effects of deformation temperature on the evolution of ferrite grain refinement. In particular, attention is paid to ferrite grain refinement through SIT by both microstructural observation and stress–strain curves.

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Table 1
 Chemical composition of experimental steel (wt.%)

C	0.032
Si	0.15
Mn	0.74
P	0.009
S	0.007
Nb	0.014
Ti	0.013
Al	0.028
N	0.0031

2. Experimental procedure

The material used in the experimental work was a low carbon Nb–Ti microalloyed steel with the chemical composition shown in Table 1. The steel was prepared as 35 kg ingot in an induction furnace operating under argon atmosphere, and then refined by electro-slag remelting (ESR) in a laboratory unit. The ingot was reheated to 1250 °C for 1 h and hot rolled in 6 passes to 25 mm thick plate. The differential scanning calorimetry (DSC) technique was applied to measure the critical transformation temperatures. The Ar₁ and Ar₃ temperatures were found to be 751 and 837 °C, respectively. The cylindrical compression samples were machined out from hot rolled plate. The deformation tests were carried out according to the test schedule in Fig. 1. The samples were 18 mm in length and 12 mm in diameter, with the axis aligned in the rolling direction of the plate. Care was exercised to minimize friction between the test dies and the sample surface by machining flat-bottomed grooves on the end faces of samples. Graphite powders and thin pieces of mica sheet were used as lubricants in compression, resulting in fairly uniform deformation with negligibly small barreling.

The uniaxial compression tests were performed on a servo-hydraulic 600 KN computerized Materials Testing System (MTS, Model 8500) equipped with a resistant furnace. Prior to deformation, the samples were solutionized at 1150 °C for 5 min. The solution temperature was selected according to the solubility product of Ti and Nb precipitates [16,17]. At this temperature, Nb is completely dissolved and Ti is partially precipitated in the form of nitrides; very high temperatures, even in excess of melting point, being necessary for its total dissolution [18,19]. After solutionizing, the samples were cooled at a rate of 5 °C s⁻¹ to the

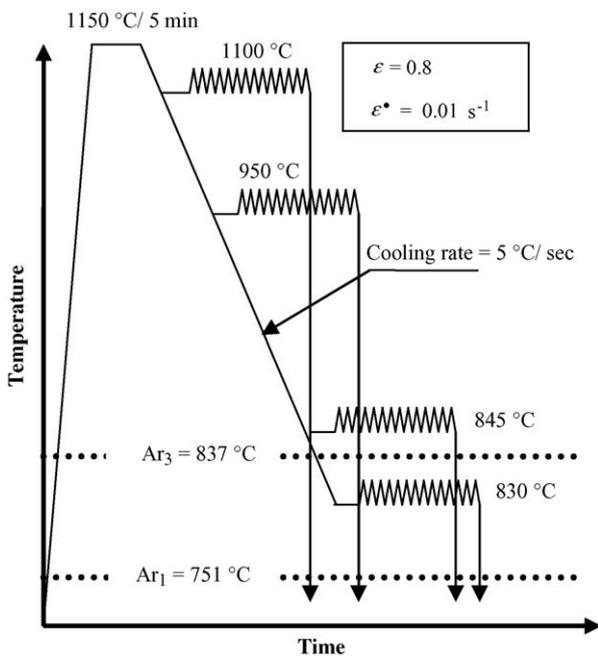


Fig. 1. Schematic representation of the TMCP conditions.

different desired deformation temperatures (1100, 950, 845, and 830 °C), and held for 20 s to homogenize the temperature throughout the samples. Then samples were isothermally deformed with single pass strain of 0.8 and at constant strain rate of 0.01 s⁻¹. All specimens were water quenched in 2 s after deformation. Optical microscopy was conducted on mid-plane sections containing the axis of compression, in order to study the microstructural changes.

3. Results and discussion

3.1. Stress–strain curves and related dynamic softening processes

Fig. 2 shows the stress–strain curves up to the strain of 0.8 at different deformation temperatures (830–1100 °C) and constant strain rate of 0.01 s⁻¹. As it can be seen in this figure, at the deformation temperature of 1100 °C, the flow curve exhibits the DRC type without any evidence of DRX. With decreasing the deformation temperature to 950 °C, the flow curve indicates the work hardening behaviour. When the deformation temperature decreases to 845 °C, just above Ar₃, the flow curve does not exhibit work hardening after the strain of about 0.32, and flow stress level remains constant during the deformation. This type of dynamic softening, non-work hardening, is attributed to the SIT of austenite to ferrite [20,21]. In other words, during the deformation at this temperature, further transformation of austenite to ferrite would also lead to apparent dynamic softening, as produced ferrite is much softer than austenite under these deformation conditions [14]. At the deformation temperature of 830 °C, flow curve would not show work hardening behaviour after the strain of about 0.32. This non-work hardening behaviour is also due to the SIT of austenite to ferrite as discussed above. Further investigation is needed to study the influence of precipitation on SIT dynamic softening kinetics.

Fig. 3 shows the maximum stresses at strain of 0.8 taken from the true stress–true strain curves in Fig. 2. It is shown that at the deformation temperature of 1100 °C the maximum stress is lowest compared to other temperatures. This is due to the DRC of austenite. However, with decreasing the deformation temperature to 950 °C, the maximum stresses increase due to work hardening of austenite. With decreasing the deformation

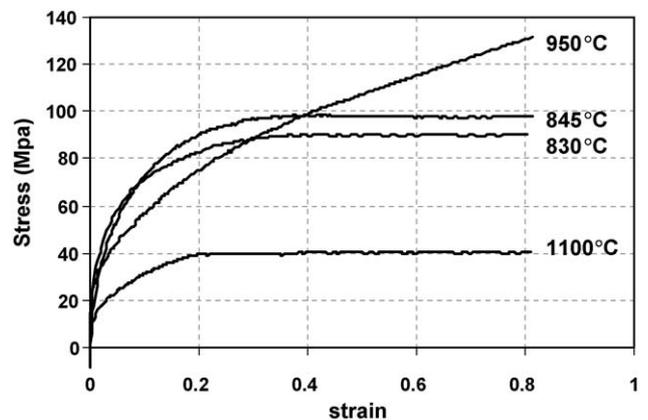


Fig. 2. Representative flow curves for the low carbon Nb–Ti steel obtained under various deformation temperatures and strain rate of 0.01 s⁻¹.

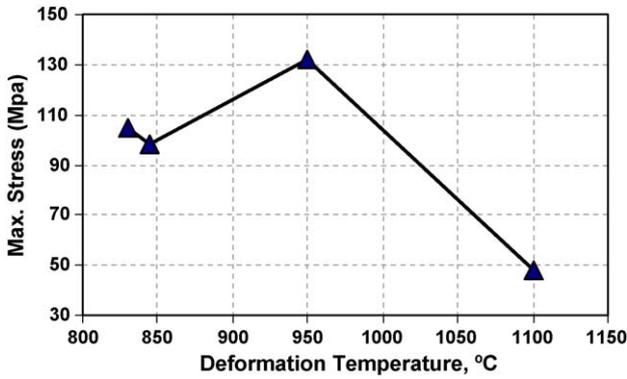


Fig. 3. Effects of deformation temperature on the maximum value of true stress at strain of 0.8 and strain rate of 0.01 s^{-1} , taken from the flow curves in Fig. 2.

temperature from 950 to 845 °C, the maximum stress decreases due to the occurrence of SIT of hard austenite to soft ferrite.

3.2. Microstructural evolutions

Fig. 4 represents the evolution of the microstructure and ferrite grain refinement at different deformation temperatures up to the strain of 0.8 with constant strain rate of 0.01 s^{-1} . As it can be seen, at 1100 °C acicular ferrite is dominant with polygonal ferrite as the second phase. The acicular ferrite matrix is characterized by the coarse non-equiaxed ferrite grains with an average size of about 11 μm . A remarkable characteristic of this type of microstructure is that it possesses a unique and irregular configuration, which has various size grains distributed in a chaotic manner with random orientations [22].

At 950 °C, the microstructures are fully equiaxed ferrite grains with the size of about 4 μm . The flow curves in Fig. 2 shows the work hardening of austenite at this deformation temperature. This indicates that the ferrite grains shown in Fig. 4

are produced from work hardened (pancaked) austenite through austenite to ferrite transformation. The sizes of these ferrite grains are smaller than that of produced from dynamically recovered austenite at 1100 °C. It is well known that ferrite tends to nucleate at the austenite grain boundaries. Thus, the ferrite grain size developed after the transformation strongly depends upon the austenite grain structure that appears just before the start of the transformation [23]. During straining austenite in non-recrystallization temperature, deformation bands, and twinning boundaries will form and the dislocation density inside austenite grains will be greatly increased which provided favorable nucleation sites and enhanced nucleation rate [24]. The grain refinement effect of deformation in the unrecrystallization temperature range is greater than that in the recovery and recrystallization temperature range [25]. In other words, the ferrite grain size produced by the austenite deformation at 950 °C is finer than that produced by austenite deformation at 1100 °C. The higher austenite decomposition kinetics causes the faster nucleation rate of ferrite. Unrecrystallized austenite will transform to ferrite at a faster rate than recovered or recrystallized austenite owing to two effects: (1) the higher internal energy of the deformed and thus less stable austenite and (2) the larger number of nucleation sites provided by defects [26].

At 845 °C, just above A_{r3} , it can be seen that the dominant phase is equiaxed ferrite grains with small amounts of deformed pro-eutectoid ferrite. The average of ferrite grain size is about 3 μm . The equiaxed ferrite grains are developed along the austenite boundaries. The presence of somewhat elongated pro-eutectoid ferrite after deformation explains that phase transformation occurs during deformation [27]. In general, deformation of austenite accelerates the transformation, raising the A_{r3} temperature. Considering the related flow curves shown in Fig. 2, these microstructures are produced during the SIT. This

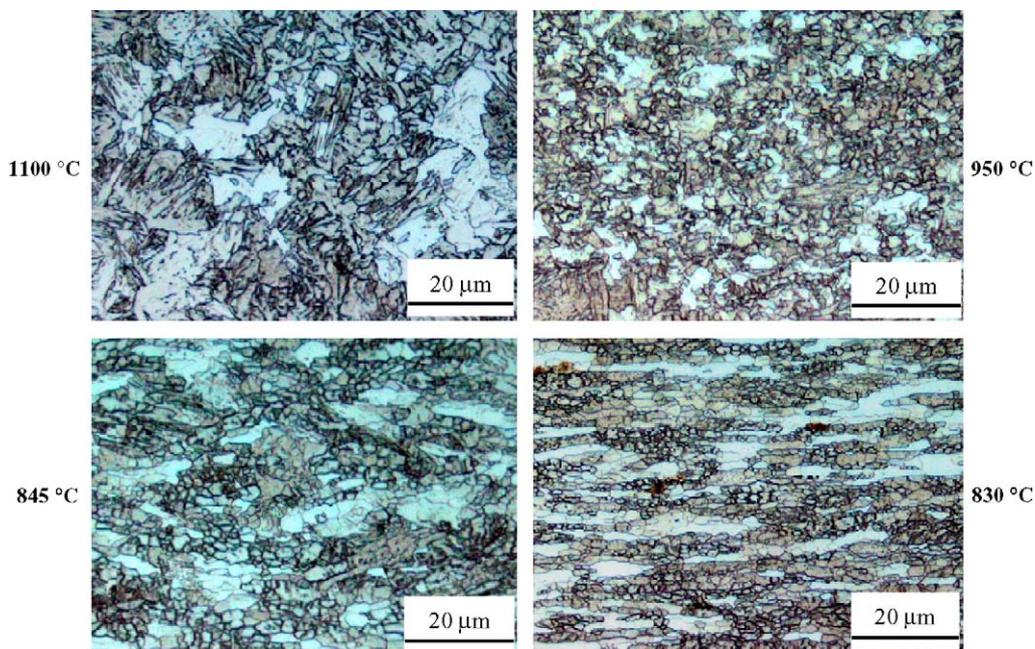


Fig. 4. Evolution of the microstructure and ferrite grains refinement at different deformation temperatures with the strain of 0.8, and the strain rate of 0.01 s^{-1} .

means that the austenite to ferrite transformation occurs dynamically during deformation. Producing of these fine ferrite grains in dynamic transformation can be attributed to two factors: one is high nucleation rate of ferrite and the other is random distribution of ferrite grain orientations [9]. There are somewhat coarse ferrite grains in microstructure achieved at 845 °C. This can be explained by that when the strain rate is low, the strain-induced ferrite grains will have enough time to grow [28].

At 830 °C, just below A_{r3} , as it can be seen the equiaxed ferrite is a dominant phase and the deformed pro-eutectoid ferrite is observed within this phase. If phase transformation is occurred during deformation, the grains would be somewhat elongated after deformation. According to the corresponding flow curve shown in Fig. 2, this microstructure is produced from SIT. It is of interest to note that the ferrite grain sizes achieved at this deformation temperature are smaller than those obtained from deformation temperature just above A_{r3} , 845 °C. It is due to the amount of undercooling which has been taken with respect to A_{r3} . The austenite to ferrite transformation depends on two factors [13]: first, the stored energy produced by deformation leading to the destabilization of austenite and, the second, the driving force caused by the degree of undercooling, which depends on the difference in the critical temperature A_{r3} and deformation temperature. By deformation at the temperature range of 845–830 °C, both above factors accelerate the austenite to ferrite transformation. The probability of nucleation largely increases in this case and leads to ultra fine-grained ferrite. The undercooling level could be determined by the following equation:

$$\Delta T = A_{r3} - T_d$$

where ΔT is the level of undercooling, T_d the deformation temperature, and A_{r3} is the transformation temperature which is 830 °C. The key to ultra-refinement is deforming austenite at deeply undercooled state [29]. Deformation of austenite just under A_{r3} enables the austenite to store much more deformation energy in SIT processing. The high stored energy significantly increases the driving force for austenite to ferrite transformation and lead to the formation of very fine ferrite grains [29]. Very fine ferrite grain size of 1.8 μm is produced in the sample undercooled to 830 °C and then deformed. In this condition, grain boundary of ferrite would be deformed and then, an inhomogeneous microstructure is generated near the deformed ferrite grains. The evolution of inhomogeneous microstructure could be explained by strain-induced grain boundary migration [30]. While the pre-eutectoid ferrite and remaining austenite coexist, the microstructure is inhomogeneous, because the coarse pre-eutectoid ferrite cannot recrystallize easily. This has been confirmed by the fact that the volume fraction of elongated ferrite increases with increasing the volume fraction of pro-eutectoid ferrite [13].

Fig. 5 shows the effect of deformation temperature on the ferrite grain size. As can be seen in this figure, the smallest ferrite grains were obtained at deformation temperature just below the A_{r3} , i.e. 830 °C. Therefore it can be concluded that the low deformation temperature with strain rate of 0.01 s^{-1} , leads to much more stored deformation energy in austenite. The high stored

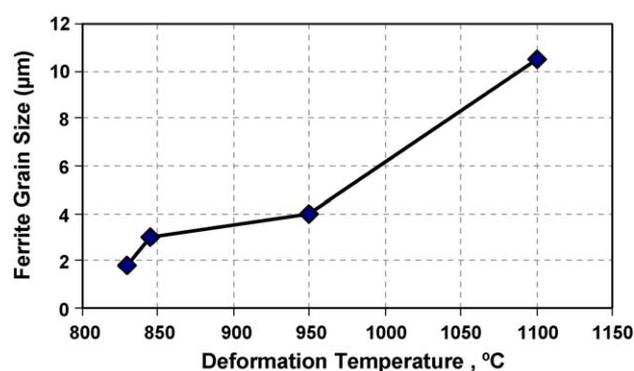


Fig. 5. Effects of deformation temperature on the ferrite grain size at strain rate of 0.01 s^{-1} and strain of 0.8.

energy significantly increases the driving force for austenite to ferrite transformation and leads to the formation of very fine ferrite grains [11].

4. Conclusions

The microstructural evolution and ferrite grain refinement of a low carbon Nb–Ti steel were investigated under various deformation temperatures at constant strain of 0.8 and strain rate of 0.01 s^{-1} through TMCP using hot compression tests. The following main conclusions can be drawn:

- (1) The ferrite grain refinement effect of single pass hot compression in the strain-induced transformation (SIT) temperature range is much stronger than that of deformation in the recovery or unrecrystallization temperature range.
- (2) The ultra-fine and equiaxed ferrite with a grain size of 1.8–3 μm can be obtained by applying deformation in the temperature range of 830–845 °C (about $A_{r3} \pm 10$ °C). The SIT mechanism is responsible for the ferrite refinement in this temperature range.
- (3) There is a close relation between the microstructural evolution and true stress–true strain responses during the deformation. The occurrence of SIT of austenite to ferrite can be monitored by analysis of hot flow stress–strain curves. At deformation temperatures close to A_{r3} , SIT leads to the dynamic flow softening in flow curve.
- (4) The softening behaviour during deformation at the temperature of 830 °C, just below A_{r3} , results from the contrary effects of softening of SIT and work hardening of austenite and ferrite. At this deformation temperature, the deformed and SIT ferrite grains coexisted together in final microstructure.
- (5) Deformation at temperatures close to A_{r3} is very effective on ferrite grain refinement.

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