Effect of Thermomechanical Processing on the Hot Ductility of a Nb–Ti Microalloyed Steel

Siamak AKHLAGHI and Steve YUE

Formerly Ph.D. Student at the Department of Mining and Metallurgical Engineering, McGill University. Now at Microlynx Inc., 1911-94 Street, Edmonton, Alberta, T6N 1E6, Canada. 1) Department of Mining and Metallurgical Engineering, McGill University, 3610 University St., Montreal, Quebec, H3A 2B2, Canada.

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Many attempts have been made to understand the problem of transverse cracking in continuous casting process. Much of this research has involved the study of hot ductility using ‘conventional’ isothermal hot ductility testing. In these tests, the specimens were isothermally tensile tested to fracture, at temperatures achieved by cooling from a solutionizing temperature, close to the solidus, or above the liquidus. These studies showed that hot ductility at the test temperature is highly depended on the thermal path followed by the specimens. The thermal histories experienced by strands during continuous casting were found to be quite complex and invariably involve rapid cooling and heating cycles. This may therefore lead to high thermal gradients, which, in turn, can generate strains in the surface of the solidifying strand. This then may alter the microstructural evolution of the strand surface and the corresponding hot ductility, a possibility that has not been addressed in any previous studies. Thus, the purpose of this study was to consider the effect of the thermomechanical history on the hot ductility of steel.

After in-situ melting and solidification, Nb–Ti microalloyed tensile specimens were subjected to a thermal history typical of a continuously cast billet. Different degrees of deformation were imposed on the specimens at selected stages of this thermal history, before tensile testing to fracture point at the time and temperature corresponding to the unbending stage of the billet casting. It was found that the hot ductility varied from 1 to 98%, depending on the stage in the thermal history at which deformation was executed. The microstructural evolution during the thermomechanical profile was followed to study the effect of thermomechanical history on the hot ductility.

KEY WORDS: hot ductility; deformation; thermal history; Nb–Ti microalloyed steel.

1. Introduction

Transverse cracking can occur during the continuous casting of steels and there is considerable evidence that suggests this problem takes place in the final stages of the continuous casting process.1–3) This correlates quite well with the observation that steels prone to transverse cracking exhibit very low ductilities (<20% reduction in area at fracture) at high temperatures.3–7) With regard to transverse cracking, hot ductilities are usually determined by isothermal tensile test that includes melting and then cooling to the desired test temperature. These “conventional” isothermal tensile tests reveal that most steels possess a temperature range over which the steel is embrittled. In general, the unbending operation, which takes place in the final stage of continuous casting process, coincides with this thermal region.1–7) It is frequently reported that performing the unbending operation at the temperatures below or above the aforementioned thermal region has been moderately successful in improving the incidence of cracking in some problem grades because the temperature range of embrittlement has been avoided.8) However, recent laboratory studies have shown that the thermal history itself has a pronounced effect on the hot ductility.9) In these new tests, tensile specimens were subjected to thermal histories typical of the surface of a billet during continuous casting. The hot ductility was found to be very sensitive to the minimum temperature attained in these thermal schedules. In particular, when the temperature of specimen dropped below the austenite-to-ferrite transformation temperature, the hot ductility at the unbending temperature was reduced, considerably.

Stress analysis studies of the surface of the strands produced by continuous casting process have demonstrated that stresses can be generated throughout the casting process. These could be due to ferrostatic pressure of the unsolidified melt against the solidified shell, friction between the mold and the solidified shell, mechanical misalignment, and thermal gradients at the surface of the strand.3,10,11) Thus, a metallurgically appropriate simulation of the conditions of continuous casting would incorporate a deformation history as well as a thermal history.

The theme of the present study is thus to investigate the hot ductility behavior of a Nb–Ti microalloyed steel, addressing the simultaneous influence of thermal and mechanical histories on hot ductility.
2. Experimental Materials and Procedure

The basic chemical composition of the steel, which was provided in the form of continuously cast billet by IPSCO, Regina, Saskatchewan, Canada, is given in Table 1. Tensile test specimens of 9.5 mm in diameter and 125 mm length were machined with the specimen axes parallel to the casting direction. Tensile testing was used to study the effect of thermomechanical history on the hot ductility of this steel. The experimental set up is shown schematically in Fig. 1.

In preparation for testing, the effective gauge length of each specimen is sheathed in a heat insulator (fiber-frax). This contained the liquid steel produced during reheating, which was the first step of the thermal history. The tests were conducted in an inert atmosphere of mixture of Ar/1%H₂ gas. A programmer and controller applied and controlled the desired temperature profile. Flowing He gas or water spraying was used for fast cooling or quenching of the samples, respectively. The temperature was measured by a pyrometer sighted between turns of the induction coil used to heat the specimen. A computerized control system, hydraulic power supply, closed-loop servo hydraulic, universal automated testing machine (model 510 MTS) is used for applying all mechanical strains, including the tensile strain to fracture (which simulates the unbending stage of continuous casting).

After melting, the specimens were subjected to a thermal history similar to that experienced by the surface of billet during continuous casting (Fig. 2). This thermal history, which is based on the temperature predictions of a commercial model (CrackExpert), was chosen because the resulting hot ductility at unbending was known to be poor. As can be seen, the specimens were heated at 2°C/s up to 10°C above the melting point (1 450°C), held for 5 s, brought down at the rate of 10°C/s to 780°C, heated again up to 1 200°C and finally cooled at a relatively slow rate down to the tensile test temperature of 1 020°C. At this temperature, (corresponding to the unbending temperature), the hot ductility was measured by pulling at a constant actuator displacement rate of 1.74 mm/min, which is roughly equivalent to a strain rate of \(5 \times 10^{-3} \text{s}^{-1}\). The effective gauge length was about 6 mm at center of specimen.

The effect of prior deformation, applied during this basic thermal history, on hot ductility was investigated by applying plastic strain at different stages of the thermal history. Deformation was applied in three different thermal regions during the cooling down segment after solidification (region A in Fig. 2). These were defined as i) temperatures in the proximity of the melting point, ii) intermediate temperatures, and iii) low temperatures. After applying the desired plastic strain, the specimens were strained to fracture at the unbending stage, as per the basic thermal history.

Microstructural studies were performed by conducting conventional metallographical examinations on samples quenched at the different portions of the thermal history. Microstructures were etched by 2% Nital; warm saturated picric acid was also used for revealing the primary austenite grain sizes. The grain size was measured using a linear intercept technique. Carbon extraction replicas were also made to examine the precipitate size and distribution using a transmission electron microscope (Jeol 2000FX) operating at an accelerating voltage of 160 kV. Surface of specimens after fracture were studied by a scanning electron microscope (JEOL JSM-840A) operated in secondary electron mode at 20 kV accelerating voltage.

3. Results

3.1. Hot Ductility Due to the Thermal History Alone

The reduction of area at the unbending point after following the basic thermal history shown in Fig. 2, was 10%. The fracture surface of the sample is presented in Fig. 3, which mainly shows brittle features mainly at grain boundaries but some small areas, indicated by arrows, show the occurrence of ductile deformation. The general appearance of fracture surface suggests an intergranular fracture. In addition the fracture surface was perpendicular to direction of loading.

3.2. Effect of Prior Deformation on the Hot Ductility at Unbending

(i) First Region: Temperatures in the Proximity of the Melting Point

A prior compressive strain of either 10 or 20% at a strain
rate of $8 \times 10^{-3} \text{s}^{-1}$ was superimposed onto the basic thermal history at start temperatures of 1440 or 1450°C. The resulting hot ductilities at the unbending stage are given in Table 2. It can be seen that deformation at temperatures close to the melting point improves the hot ductility markedly. The fracture surface of the sample after applying a 10% prior deformation at 1450°C, is shown in Fig. 4, and exhibits the classical ductile behavior. Decreasing the deformation start temperature from 1450 to 1440°C decreases the extent to which the hot ductility improves, but it is still a significant improvement over the hot ductility due to the thermal history alone.

(ii) Second Region: Intermediate Temperatures
Prior compressive strains of 1 and 15%, at the strain rate of $8 \times 10^{-3} \text{s}^{-1}$ were initiated at 1420 to 1100°C. The hot ductility results obtained from these conditions are shown in Table 3. It can be seen that deformation in this temperature range has no significant effect on the hot ductility, regardless of the deformation variables.

(iii) Third Region: Low Temperatures (900°C)
At this point in the thermal history, the thermal strains that may develop in the solidified shell of the strand in the continuous casting are in tension. Also, the highest levels of strain due to thermal gradients probably occur towards the end of the cooling segment, which corresponds to the largest thermal gradient. Thus, prior tensile strains of 1, 14 and 20%, at a strain rate of $8 \times 10^{-3} \text{s}^{-1}$ and at start temperature of 900°C were implemented. Hot ductility results corresponding to these conditions are given in Table 4. Results show that prior tensile deformation applied at 900°C is extremely deleterious to hot ductility for this thermal history. There is no strong change in hot ductility with change in level of strains applied.

3.3. Microstructural Examinations
3.3.1. Optical Microscopy Examinations
In order to comprehend the metallurgical reasons for the pronounced effect of thermomechanical history on the hot ductility, the microstructural evolution during different thermomechanical histories was followed.

3.3.1.1. Evolution of Microstructure during Thermal History Alone
The evolution of the microstructure during the thermal history alone was observed by quenching the specimens at the temperatures indicated in Fig. 5.

After quenching a specimen at 900°C, $T_1$ in Fig. 5, the microstructure showed only martensite, denoting a fully austenitic structure prior to quenching. The microstructure after quenching at $T_{\text{min}}$ (780°C) is shown in Fig. 6. As can be seen, it is composed of martensite and a thin layer of ferrite that has formed along the grain boundaries of austenite. The microstructure quenched at 900°C, corresponding to $T_2$ in Fig. 5, is shown in Fig. 7. Since the microstructure is entirely martensite, the ferrite phase has retransformed to austenite at this temperature.

The same microstructure with a somewhat larger austenite...
The size of the primary austenite grains at different points of the thermal history is given in Table 5. As can be seen, the austenite grain size generally increases with increasing time.

Table 5. Austenite grain size at different points of the thermal history.

| Temperature (°C) | Grain Size (μm) |
|------------------|-----------------|
| $T_1$            | 118             |
| $T_{\text{unbending}}$ | 120             |
| $T_2$            | 120             |
| $T_{\text{minimum}}$ | 131             |

3.3.1.2 Effect of Prior Deformation on Microstructural Evolution

The microstructure of the sample quenched at $T_{\text{min}}$ following a 10% prior compressive deformation in the proximity of the melting point in the thermal history is shown in Fig. 8. Comparing this micrograph with what was observed due to the thermal history alone, Fig. 6, indicates that imposing such a deformation leads to a finer parent austenite grain size and more ferrite at $T_{\text{min}}$.

The microstructure of a sample also quenched at $T_{\text{min}}$ but subjected to 10% prior compressive deformation at 900°C during the thermal history is illustrated in Fig. 9. This micrograph shows no significant difference compared to Fig. 6, suggesting that deformation at temperatures close to 900°C does not change the optical microstructure at $T_{\text{min}}$.

The austenite grain size of samples subjected to the above thermomechanical histories is given in Table 6. These results indicate that applying deformation at temperatures close to the melting point refines the austenite grain size at the unbending point, whereas imposing deformation at 900°C, during cooling after melting in the thermal histo-
ry, has no effect on the grain size at the unbending point.

3.3.2. Electron Microscopy Examinations

Figure 10 shows precipitate characteristics at $T_{\text{min}}$ in samples subjected to different thermomechanical histories. As can be seen, a few precipitates are formed at $T_{\text{min}}$ in the absence of deformation, Fig. 10(a).

Superimposing 10% deformation in the proximity of the melting point in the thermal history, leads to fewer precipitates at $T_{\text{min}}$, Fig. 10(b). Imposing the same amount of deformation at 900°C, in cooling after melting in the thermal history, leads to a remarkable increase in the numbers of precipitates at $T_{\text{min}}$, Fig. 10(c).

Figure 11 shows the precipitation distribution and size at $T_{\text{unbending}}$. A comparison with the corresponding micrographs in Fig. 10 shows that a higher volume fraction of precipitates has been formed with increasing time. At both temperatures, $T_{\text{min}}$ and $T_{\text{unbending}}$, fewer precipitates are formed by imposing 10% prior deformation in the proximity of the melting point, while copious precipitation occurs at $T_{\text{min}}$ and $T_{\text{unbending}}$ in samples exposed to 10% prior deformation at 900°C.

The relative frequency of precipitates formed under different thermomechanical histories is shown in Fig. 12. As can be seen, the frequency of precipitates, particularly the fine ones, formed at $T_{\text{unbending}}$ after 10% prior deformation at 900°C is much higher than the case where no deformation is applied, and both values are higher than for the case when prior deformation is applied at 1450°C. The same trend is appreciable at $T_{\text{min}}$ but the differences are less pronounced. This suggests that increasing time exaggerates any difference between volume fractions of precipitates formed at different points of the thermal history.

The energy dispersive spectrum of these precipitates revealed that precipitates are basically Nb–Ti carbonitrides.

4. Discussion

4.1. Prior Deformation in the Proximity of the Melting Point

10 or 20% prior compressive deformation in the proximity of the melting point, increases the ductility at the unbending stage to 98%, Table 2. However, lowering the prior deformation start temperature to 1440°C only improves the
hot ductility to 59%. It was also shown that prior compressive deformation at 1450°C leads to a decrease in the grain size at both $T_{\text{min}}$ and $T_{\text{unbending}}$, as well as a decrease in the numbers of precipitates, particularly at $T_{\text{min}}$.

The variation in flow stress with decreasing temperature in the proximity of the melting point is shown in Fig. 13. As the temperature decreases, the flow stress increases, as expected, but then decreases to a minimum, and then increases. This behaviour is due to substantial changes in microstructure taking place during deformation. Zhou et al. have reported that in a steel similar to this composition, the transformation of $\delta$-ferrite to $\gamma$-austenite takes place over this thermal range.12) Thus, deformation at this temperature range causes an accumulation of strain in the $\delta$ phase and, consequently, deformation induced transformation. This, in turn, can be responsible for the finer austenite grains observed in such specimens. The finer austenite grains accordingly provide more preferential sites for the nucleation of $\alpha$-ferrite, subsequently causing a higher volume fraction of ferrite at $T_{\text{min}}$. Fig. 8. The reason why there are fewer precipitates may be due to increased homogeneous precipitation at this temperature regime with respect to segregation of Nb and C to the austenite grain boundaries. A decreasing grain size would reduce the level of segregation at per unit area of grain boundary, since the grain boundary area per unit volume would increase. Hence fewer precipitates would be present at the unbending temperature.13)

In addition, a finer grain size also causes improvements in ductility via the following mechanisms.14)

i) it is more difficult to propagate cracks formed by grain boundary sliding through triple points.

ii) the crack aspect ratio, which controls the stress concentration at the crack tips, is reduced thus discouraging crack propagation.

iii) the critical strain for dynamic recrystallization is decreased, which increases the possibility of ductility improvement via grain boundary migration.

Prior deformation at 1440°C only improves the ductility to 59%, as opposed to the 98% ductility observed due to prior deformation at 1450°C. The reason for this may be due to the prior deformation occurring in the two phase region of $\delta$-ferrite and $\gamma$-austenite rather than in the single phase $\delta$-ferrite region. If, as suggested by Fig. 13, $\gamma$-austenite is softer than work-hardened $\delta$-ferrite, any applied strain would tend to concentrate in the austenite network that has formed at the prior $\delta$-ferrite grain boundaries. Thus, there would be reduced effect of deformation on the transformation of $\delta$-ferrite to $\gamma$-austenite, and a corresponding reduction in the impact of this prior deformation on the hot ductility at the unbending.

4.2. Second Region: Intermediate Temperatures during Cooling

Results obtained due to prior deformation in this temperature regime showed that regardless of the deformation temperature, extent of deformation or deformation mode, hot ductility at the tensile test temperature is not affected. This correlates with the observation that there is no change in precipitate characteristics in the microstructure. It is clear that such deformation will have no affect on transformation of $\delta$-ferrite to $\gamma$-austenite, because this transformation has already terminated.

4.3. Third Region: Low Temperatures (900°C)

Hot ductility measurements made on this thermal region revealed that ductility is severely impaired by implementing prior tensile deformation at 900°C. Also it was found that imposing deformation at 900°C causes copious precipitation at $T_{\text{min}}$ and $T_{\text{unbending}}$ temperatures. This extensive precipitation is likely due to deformation induced precipitation process.15)

It has frequently been observed that deformation can significantly increase the precipitation rate by up to two orders of magnitude.16) Some experimental studies have also
demonstrated that deformation not only accelerates but also increases the precipitation frequency. The effect of deformation on precipitation kinetics is usually attributed to the increase of dislocation density, which increases both the nucleation site density and diffusivities of precipitate forming elements in the material. The increase in volume fraction of precipitates always leads to the embrittlement of microstructure and thus decreasing of ductility at fracture point. This is the reason why reduction of area is decreased when prior deformation was applied at low temperatures (900°C).

5. Conclusion

The effect of thermomechanical history on the hot ductility of a Nb-Ti microalloyed steel was investigated. The following observations were made:

1. Applying prior strain in the proximity of the melting point improves hot ductility by an order of magnitude when the specimen is subjected to an embrittling thermal history.

2. This improved ductility was related to austenite grain refinement via a strain induced transformation of the δ-ferrite to austenite, which in turn correlated to a considerable decrease in the number of formed precipitates.

3. Implementing prior deformation at low temperatures (900°C) greatly embritles the microstructure by increasing the numbers of precipitates due to strain induced precipitation.

4. Imposing strains in the temperatures intermediate to the above temperatures does not affect hot ductility. Apparently, the temperature is too low to influence the δ-ferrite to austenite transformation, and too high to lead to any extensive strain induced precipitation.

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