

Tempering Response of Low-Carbon Microalloyed Steels



Tempering is a well-known process which is usually used to improve the ductility and toughness of steel produced by a quenching process. Similarly, through tempering, the mechanical properties of as-rolled products can be altered. However, there is limited information available regarding the tempering response of as-rolled low-carbon microalloyed steels. In the present study, the effects of tempering on three low-carbon microalloyed steels were examined. The benefits of tempering for these steels are presented.

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Introduction

As a major heat treatment process, quenching and tempering has been widely used for the production of many steel products. Quenching is employed to obtain the strength/hardness required for a given steel product, while tempering is used to produce a good balance between strength and toughness. This process has been well studied, and a wealth of knowledge related to the process is readily available in the literature. Aging, tempering and stress-relieving are also heat treatment processes utilized for the production of some steel products. In comparison, the studies related to these processes employed for as-rolled steel products are not readily available. There were some studies conducted in the past decades to assess how temper/aging affects the mechanical properties of steels; for instance, age hardening of Cu-alloyed steels.^{1–5} Based on the Cu-precipitation strengthening concept, high-strength weldable steel grades, such as ASTM A710 or HSLA-80 and HSLA-100, have been developed. In-line tempering of as-rolled steels was also studied by Bai et al.⁶ Their studies showed that the yield strength of a weathering steel grade (HPS-70W) can be significantly improved by hot stacking plates. The steel is produced by employing a controlled rolling + accelerated cooling practice. The microstructure

of the final product is a mixture of polygonal ferrite + acicular ferrite + martensite/retained austenite (M/A). When the temperature of the plate for stack cooling is sufficiently high (>572°F or 300°C), a temper/aging effect on the mechanical properties of the steel can be realized. The aging process introduces additional fine precipitates, which increases the strength of the steel through the precipitation strengthening mechanism. The tempering process softens the martensite phase in the M/A constituent and releases the residual stress associated with the formation of the M/A constituent, thereby resulting in improved toughness for the steel. Nonquench tempering or tempering of as-rolled steels has also been studied by Zhao and Chen et al.^{7, 8} Zhao et al. studied the tempering response of a line pipe steel with an acicular ferrite microstructure.⁷ The results obtained from that study indicate that an off-line tempering treatment at 600°C for 10 hours could increase the yield strength of the steel by almost 140 MPa (20 ksi) without negatively impacting its toughness. The study conducted by Chen et al. also demonstrated that off-line tempering of an as-rolled low-carbon Mo-Nb structural steel can increase the strength of the steel significantly without losing the ductility of the steel. Both studies concluded that additional fine precipitates introduced

by the tempering treatment were responsible for the increase in the strength of the steels. Bai et al. studied how a predeformation + tempering treatment affects the microstructure, and hence, the mechanical properties of a low-carbon Nb-bearing steel.⁹ Their study showed that a small predeformation enhanced the formation of fine NbC precipitates during the subsequent tempering treatment, thereby resulting in a significant increase in the yield strength of the steel. A similar conclusion was drawn from the study carried out by Yuan et al., i.e., predeformation enhances NbC precipitation, thereby increasing the strength of the steel.¹⁰ Based on these studies, it is expected that the mechanical properties of some steels could be further improved by an in-line or off-line tempering treatment. To shed more light on this topic, the tempering response of three low-carbon structural steels with different strength levels was studied and the results are presented in this article.

Materials and Tempering Experiments

Materials and Tempering Experiments

The steels used in this study are one plain C-Mn steel and two low-carbon microalloyed steels. The contents of Nb, V, and Ti and carbon equivalent (CE) in these steels are shown in Table 1. The carbon equivalent of the steels was calculated using Eq. 1. All these steels were produced from a scrap-based electric arc furnace (EAF) steelmaking process and rolled on a Steckel mill at SSAB Iowa. Steel A is a plain C-Mn steel and serves as a baseline for comparison. No Nb and V were intentionally added to this steel. Only a small amount of Ti (0.007 wt. %) was added for grain size control during slab reheating. Steel B is a low-carbon V-bearing steel, and Steel C is microalloyed with Nb, V and Ti. Ti is added for grain size control during slab reheating, Nb is added for austenite pancake rolling, and V is added for precipitation strengthening. Steel A is for a steel grade with a minimum yield strength of 40 ksi. Steel B is used for a steel grade with a minimum yield strength of 50 ksi, while Steel C is for a steel grade with a minimum yield strength of 100 ksi. Steels A and B are 0.250-inch-thick discrete plates and

were produced by employing recrystallization-controlled rolling practices with an aim finish rolling temperature of 1,550°F (843°C). The steels were air-cooled after rolling. Steel C is a 0.250-inch-thick cut-to-length (CTL) product produced using a controlled rolling + accelerated cooling + coil cooling practice. The coil was processed at a CTL line with a temper reduction of 1.6%. The as-received samples (in the as-rolled or CTL condition) were tempered according to the parameters given in Table 2. Three temperatures (525, 625 and 675°C) were used for the tempering treatment. The soaking time at the given temperatures was 15 minutes. The assumption was that for the two discrete plates (Steels A and B), the relatively high cooling rates due to the thin gauge of these plates may have prevented Ti or V from fully precipitating out during air cooling. Therefore, tempering may introduce more precipitates in the steels and change the mechanical properties of the steels in this way. For Steel C, although coil cooling may have the majority of Nb and V precipitated out during coiling, there may still be some Nb and/or V solutes available due to the high amount of these elements added to the steel. Therefore, tempering could introduce more fine precipitates in the microstructures of these steels and change the mechanical properties of the steels accordingly.

$$CE = C + \frac{Mn}{6} + \frac{(Cu + Ni)}{15} + \frac{(Cr + Mo + V)}{5} \quad (\text{Eq. 1})$$

Mechanical Properties

Tensile Properties

Sheet-type tensile specimens were prepared from the as-rolled and tempered samples. The tensile properties obtained from these samples are summarized in Table 3. The tensile stress-strain curves are shown in Figs. 1–3. Due to the similarity of the yielding behavior of the tempered samples, only those tempered at 525°C are shown. It should be noted that for these tests, the extensometer

Table 1

Chemical Compositions of Steels, wt. %

Steel	C	Mn	Cu + Ni + Cr + Mo	Nb	V	Ti	CE
A	0.058	1.11	0.61	0.002	0.004	0.007	0.309
B	0.056	1.32	0.70	0.003	0.082	0.003	0.368
C	0.055	1.81	0.97	0.076	0.087	0.013	0.506

Table 2

Tempering Parameters

Steel	Gauge, in.	Tempering temperature (T), °C			Time, min.
		525	625	675	
A	0.250	✓	✓	✓	15
B	0.250	✓	✓	✓	15
C	0.250	✓	✓	✓	15

was removed at a strain of 0.006, so only the early portion of the stress-strain curves are shown in these figures. For Steel A, the stress-strain curves in both as-rolled and tempered conditions exhibited discontinuous yielding behavior, as shown in Fig. 1. This yielding behavior is typical for steels with a microstructure consisting of polygonal ferrite and pearlite. For Steel B, the stress-strain curves

in both as-rolled and tempered conditions also exhibited discontinuous yielding behavior, as shown in Fig. 2. However, unlike the stress-strain curve of the tempered sample, the stress-strain curve of the as-rolled sample appears to be round in the early stage of yielding, as indicated by the arrows in the figure (as-rolled). This may indicate that the material has already started yielding at this point by a continuous yielding behavior, then transitioned to discontinuous yielding at point B. This also suggests that the microstructure of this steel may consist of not only polygonal ferrite and pearlite but also a third phase, such as M/A. This will be clarified by the microstructures presented later. For Steel C, the stress-strain curve of the as-rolled sample exhibited continuous yielding behavior, while that of the tempered sample exhibited discontinuous yielding behavior, as shown in Fig. 3.

The effect of tempering on the strength of the steels is further illustrated by the plots shown in Fig. 4. It can be seen that for Steel A, the yield strength of the steel was not affected significantly by tempering, while the ultimate tensile strength (UTS) of the steel was decreased slightly for the samples tempered at 625 and 675°C. For Steel B, the yield strength was increased by the tempering treatments, while the tensile strength was not affected by these

Table 3

Tensile Properties of Steel

Steel	Gauge, in.	0.2% yield strength, psi	Ultimate tensile strength, psi	Tensile elongation in 2 in., %	Y/T ratio	Condition
A	0.250	53,110	66,780	37.7	0.79	As-rolled
		52,500	67,920	38.2	0.77	525°C × 15 min.
		53,460	65,750	36.6	0.80	625°C × 15 min.
		53,705	64,786	36.6	0.82	675°C × 15 min.
B	0.250	56,160	76,590	34.4	0.74	As-rolled
		65,290	76,530	31.1	0.85	525°C × 15 min.
		66,500	75,790	30.8	0.89	625°C × 15 min.
		67,446	76,668	32.0	0.87	675°C × 15 min.
C	0.250	103,620	112,015	24.0	0.92	As-rolled
		104,525	112,380	17.4	0.93	525°C × 15 min.
		105,335	112,140	19.3	0.94	625°C × 15 min.
		102,434	111,282	20.1	0.92	675°C × 15 min.

Figure 1

Stress-strain curves of Steel A.

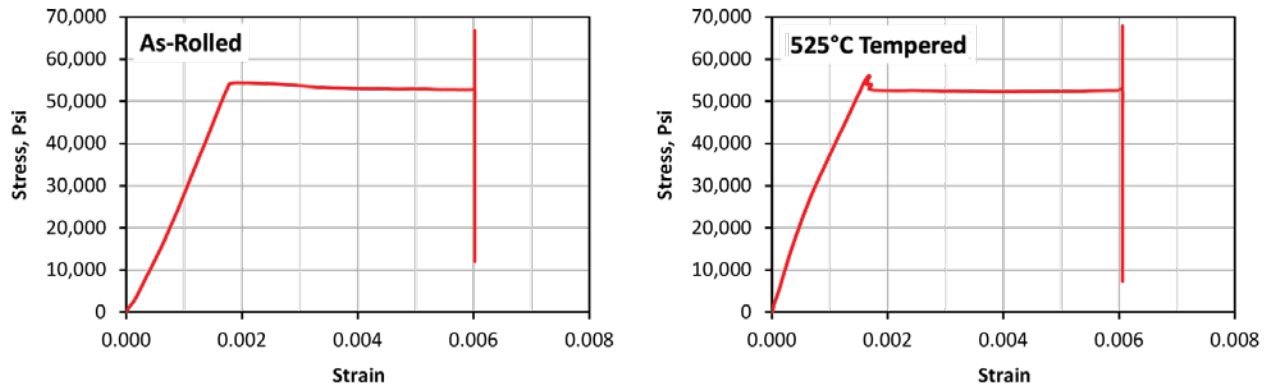


Figure 2

Stress-strain curves of Steel B.

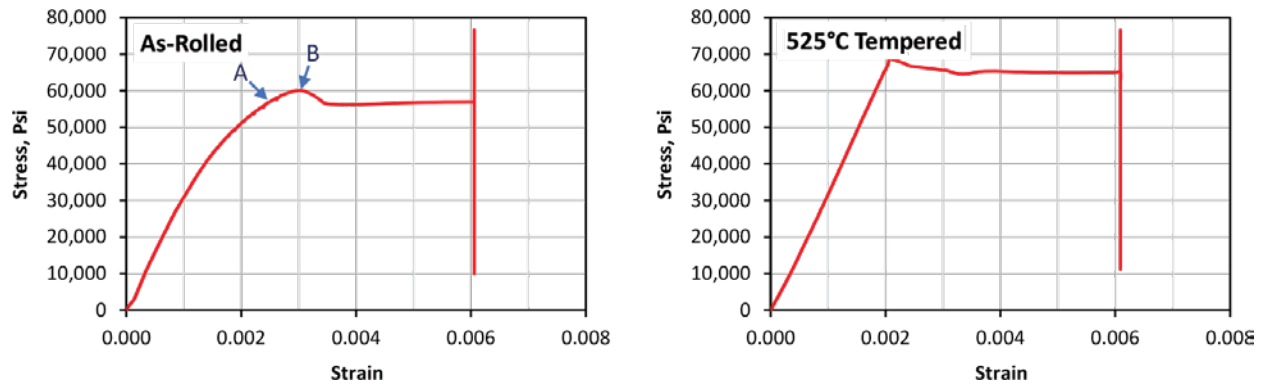


Figure 3

Stress-strain curves of Steel C.

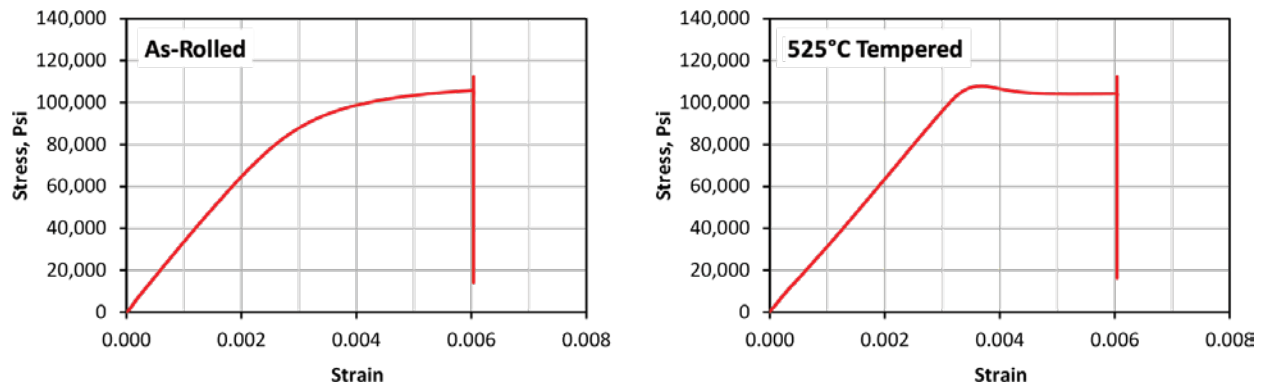


Figure 4

Effect of tempering on strength of steels.

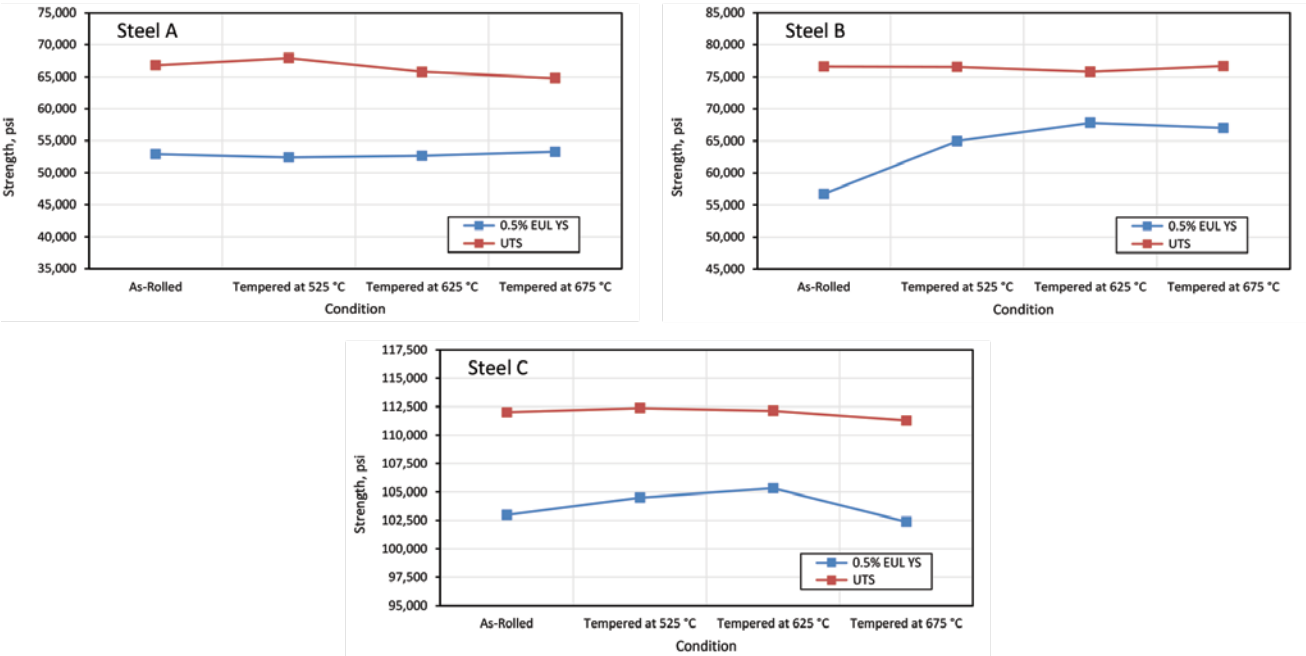


Table 4

Impact Toughness of Steels

TCVN at -40°C, ft-lbs								
Steel	Gauge, in.	Size, mm	E1	E2	E3	Avg E	FSE	Condition
A	0.250	5	56	64	63	61	122	As-rolled
		5	60	58	57	58	117	525°C × 15 min.
		5	65	56	69	63	127	625°C × 15 min.
		5	85	77	87	83	166	675°C × 15 min.
B	0.250	5	48	46	48	47	95	As-rolled
		5	74	86	75	78	157	525°C × 15 min.
		5	79	63	75	72	145	625°C × 15 min.
		5	77	82	86	82	163	675°C × 15 min.
C	0.250	5	43	41	37	40	81	As-rolled
		5	42	49	47	46	92	525°C × 15 min.
		5	41	41	43	42	83	625°C × 15 min.
		5	45	46	44	45	90	675°C × 15 min.

tempering treatments. For Steel C, the yield strength of the steel was increased by tempering at 525 and 625°C, but dropped back to the same level as that of the as-rolled condition for the sample tempered at 675°C. The tensile strength dropped slightly for the sample tempered at 675°C, but remained almost unchanged for the samples tempered at 525 and 625°C.

Impact Toughness

The toughness of the steels was evaluated by performing impact testing of Charpy V-notch (CVN) specimens in the transverse-to-rolling direction on a Instron 400 Charpy tester. Due to the light gauges of the plates, subsize CVN specimens were used for testing. All tests were performed at -40°C . The CVN results are summarized in Table 4. Full-Size-Equivalent (FSE) values were also calculated and shown in Table 4. The effect of tempering on the toughness of the steels is shown in Fig. 5. It can be seen from the figure that for Steel A, the CVN impact energy was not changed much by tempering at 525°C. However, the impact energy of the steel was increased by tempering at 625 and 675°C. Especially for the sample tempered at 675°C, the impact energy increased from 122 ft-lbs in the as-rolled condition to 166 ft-lbs in the tempered condition. For Steel B, the impact energy was increased by tempering at all three temperatures. It can be seen that for the sample tempered at 675°C, the impact energy increased from 95 ft-lbs to 163 ft-lbs. For Steel C, the impact energy did not change much for the tempered samples. Nevertheless, the toughness of the steel was not negatively impacted by the tempering treatments. For the given strength level and testing temperature, these values are considered to be good for the steel.

Microstructures

Samples for microstructure examination were prepared according to the standard metallography procedures. The polished samples were etched using 2% Nital and examined with a Nikon light optical microscope (LOM). The ferrite grain sizes were estimated using a Nikon microscope equipped with Clemex software. The samples were also examined with a Zeiss Ultra 55 scanning electron microscope (SEM) equipped with an energy-dispersive x-ray spectroscopy (EDS). The LOM photomicrographs of Steel A in both as-rolled and tempered conditions are shown in

Figure 5

Effect of tempering on impact toughness of steels.

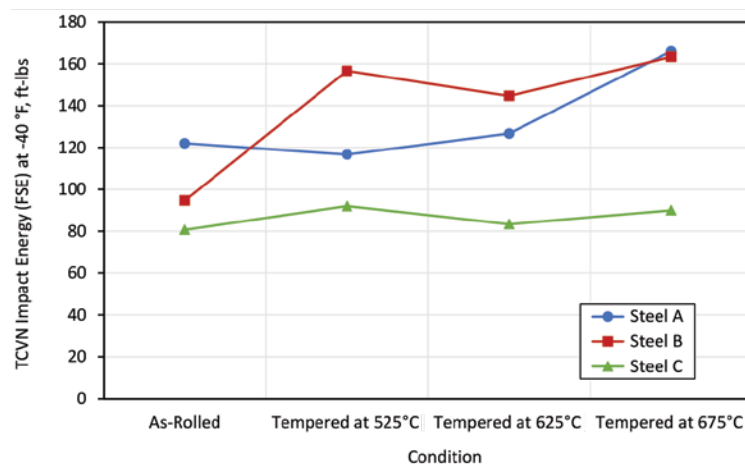


Fig. 6. The microstructures of the as-rolled and tempered samples consist of polygonal ferrite and pearlite. The ferrite grain sizes of these samples appear to be similar. To further examine the structure of pearlite in the microstructures, these samples were examined with SEM, and the micrographs are shown in Fig. 7. It can be seen that the microstructures of the as-rolled sample and the samples tempered at 525 and 625°C consist of polygonal ferrite and pearlite with cementite being a lamellar structure. For the sample tempered at 675°C, however, cementite was fully spheroidized. It is believed that this

Figure 6

Microstructures of Steel A (light optical microscope (LOM) photomicrographs).

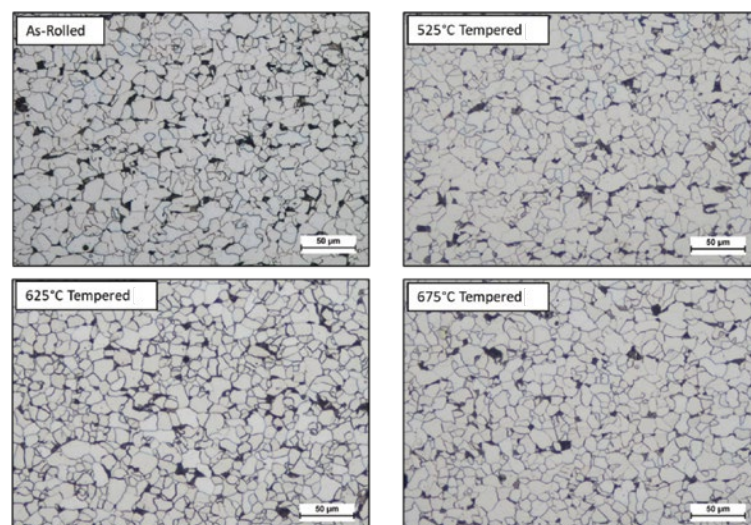


Figure 7

Microstructures of Steel A (scanning electron microscope (SEM) micrographs).

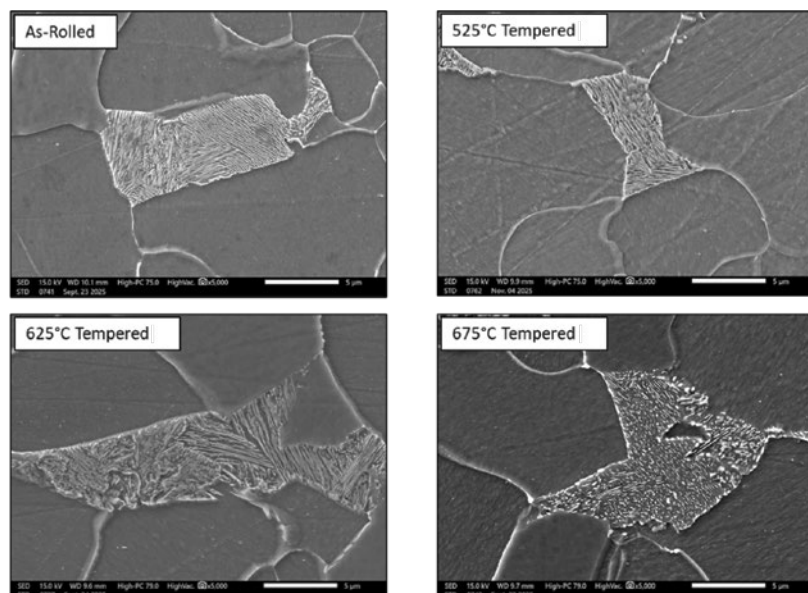
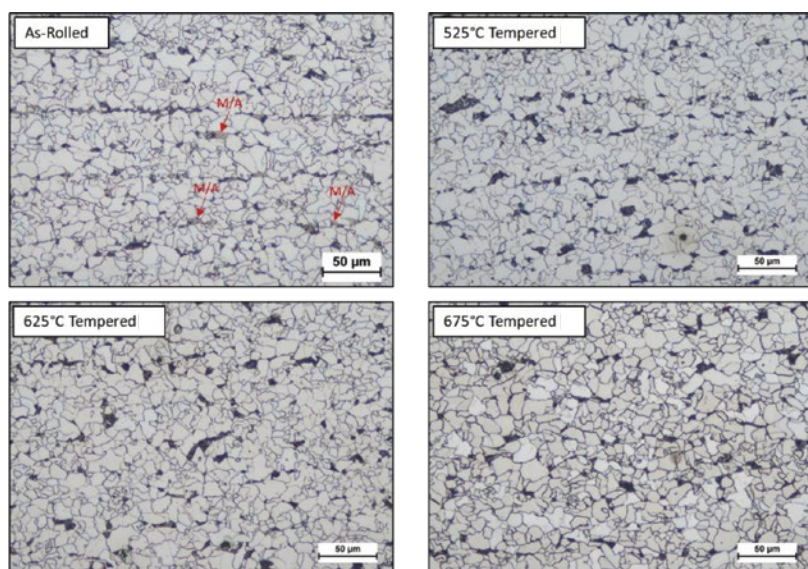


Figure 8

Microstructures of Steel B (LOM photomicrographs).



change in the morphology of cementite is attributable to the slight drop in the UTS and a noticeable improvement in the toughness of the steel. The microstructures of Steel B are shown by the LOM photomicrographs in Fig. 8. In general, the microstructures of these samples consist of polygonal ferrite and pearlite. However, there

are some brownish islands seen in the microstructure of the as-rolled sample, as indicated by the arrows in the micrograph. To show the details of the microstructures, these samples were examined with SEM and the micrographs of the samples are shown in Fig. 9. In the as-rolled sample, three microstructure constituents were observed, i.e., polygonal ferrite, pearlite and M/A. In the tempered conditions, no M/A was observed. The M/A constituent may have been tempered or decomposed to carbides and ferrite. However, lamellar cementite was still seen in the samples tempered at 525 and 625°C. In the sample tempered at 675°C, M/A was tempered and pearlite was spheroidized. As mentioned earlier, the presence of M/A in the microstructure of the as-rolled sample contributed to the round house shape of the stress-strain curve in the early stage of yielding. The noticeable improvement in the toughness of this steel in the tempered conditions is mainly attributable to the tempering of the M/A constituent in the microstructure.

The microstructures of Steel C are shown in Fig. 10. This is a CTL plate processed from a coil which was produced by employing a controlled rolling + accelerated cooling practice. The microstructure of the steel in the as-rolled condition consists of granular bainite with fine dispersed M/A islands. At the LOM level, the microstructures of the samples in the tempered conditions are similar to that of the sample in the as-rolled condition. To analyze the details of the microstructures, these samples were also examined using SEM. The SEM images of these samples taken in the secondary electron mode are shown in Fig. 11. In these SEM images, the bright phase indicated by arrows is M/A. M/A was seen in the as-rolled sample and the samples tempered at 525 and 625°C. In the sample tempered at 675°C, however, no M/A was observed. It suggests that the M/A phase has been largely tempered or decomposed to carbide and ferrite at this temperature.

Discussion

In general, the major contributors to the strength of steel include solid solution strengthening ($\Delta\sigma_{ss}$), precipitation strengthening ($\Delta\sigma_{ppt}$), dislocation strengthening ($\Delta\sigma_{dis}$), strengthening from texture ($\Delta\sigma_{tex}$) and strengthening from grain refinement ($\Delta\sigma_d$), such as that shown by Eq. 2:¹¹

$$\sigma_{YS} = \sigma_0 + \Delta\sigma_{ss} + \Delta\sigma_{ppt} + \Delta\sigma_{dis} + \Delta\sigma_{tex} + \Delta\sigma_d \quad (\text{Eq. 2})$$

For the steels used in this study, the strengthening mechanisms involved are different. Although strengthening from grain refinement is a common strengthening mechanism for all three steels, there was no precipitation strengthening for Steel A since no microalloying elements, such as Nb and V, were added to this steel. The ferrite grain sizes for the steel in the as-rolled and tempered conditions are shown in Fig. 12 (the blue curve). It can be seen that the ferrite grain size of this steel was not significantly affected by the tempering treatments. The grain sizes are almost identical for all four conditions, at around 11.5 μm . This explains why the yield strength of this steel did not vary much among the four conditions. The slight drop in the UTS observed for the sample tempered at 675°C was mainly caused by pearlite spheroidization. For Steel B, the ferrite grain size was also not affected by the tempering treatments, as shown by the red curve in Fig. 12. The grain sizes for this steel, however, are finer compared to those for Steel A. This is one of the contributors to the higher strength obtained for this steel. The rolling practice employed for this steel is similar to that used for Steel A. However, this steel contains 0.082 wt. % V. V(C,N) could have precipitated out in austenite in the late stage of rolling and produced a favorable austenite condition for the austenite-to-ferrite transformation. This may have resulted in a finer ferrite grain structure after the phase transformation. In addition, further VN and/or VC precipitation in ferrite during cooling retarded grain growth, resulting in the

Figure 9

Microstructures of Steel B (SEM micrographs).

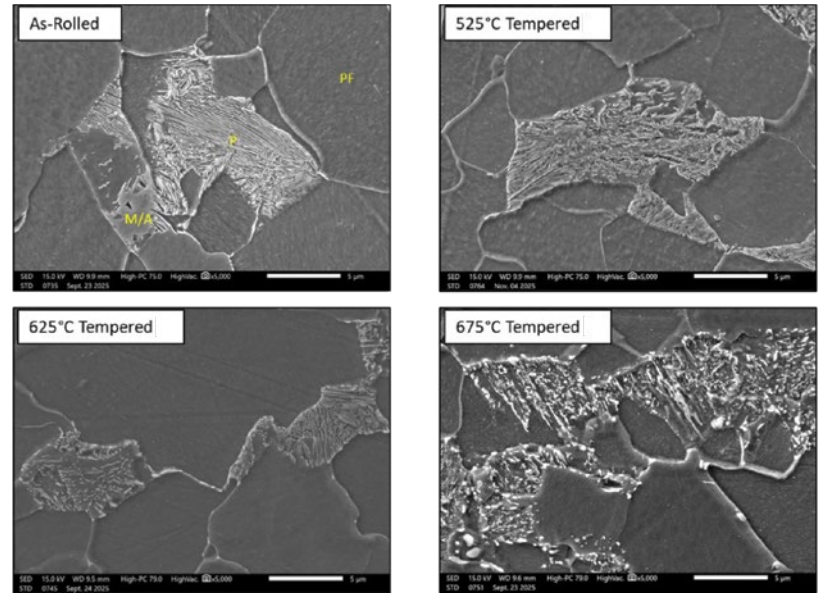
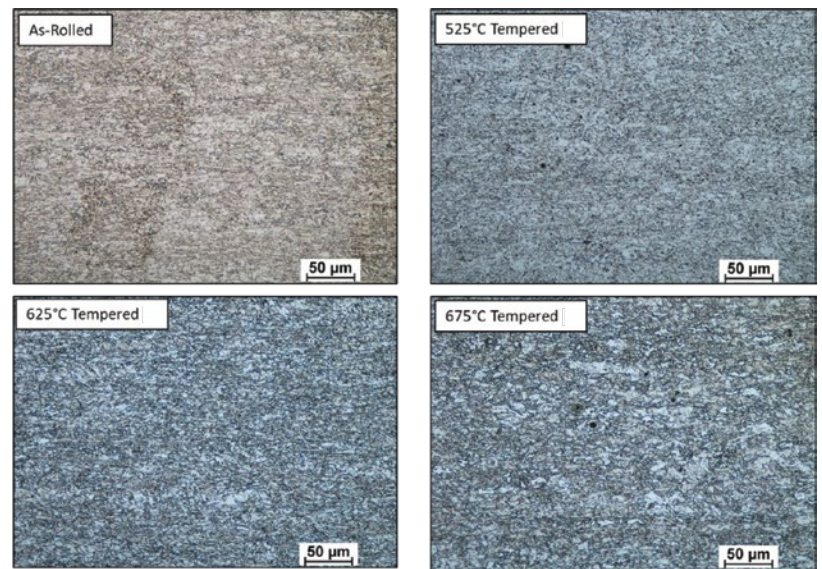


Figure 10

Microstructures of Steel C (LOM photomicrographs).



finer grain structure for this steel. As shown in Fig. 13, fine V-rich precipitates were observed in Steel B. These nanosized precipitates not only prevented ferrite grains from coarsening but also provided additional strength from the precipitation strengthening mechanism. It is possible that there were some V solutes that stayed in solution after cooling. These V solutes precipitated out

Figure 11

Microstructures of Steel C (SEM micrographs).

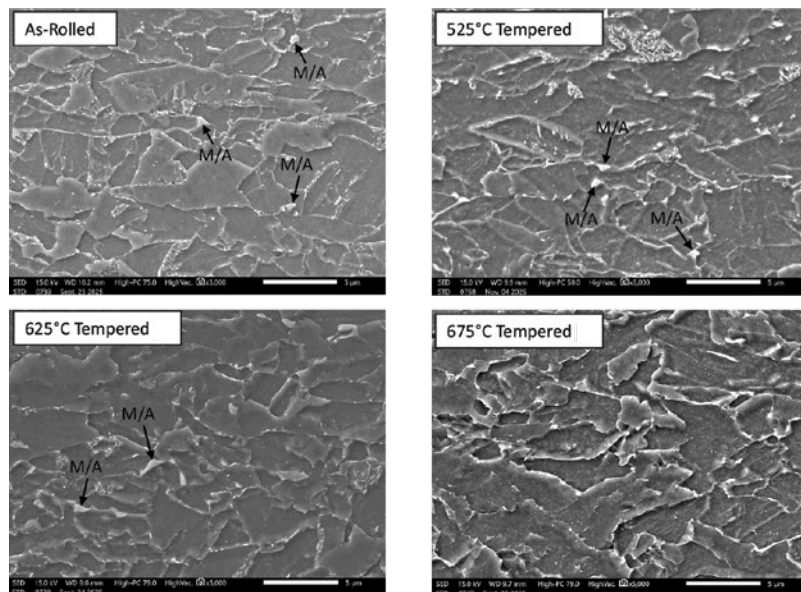
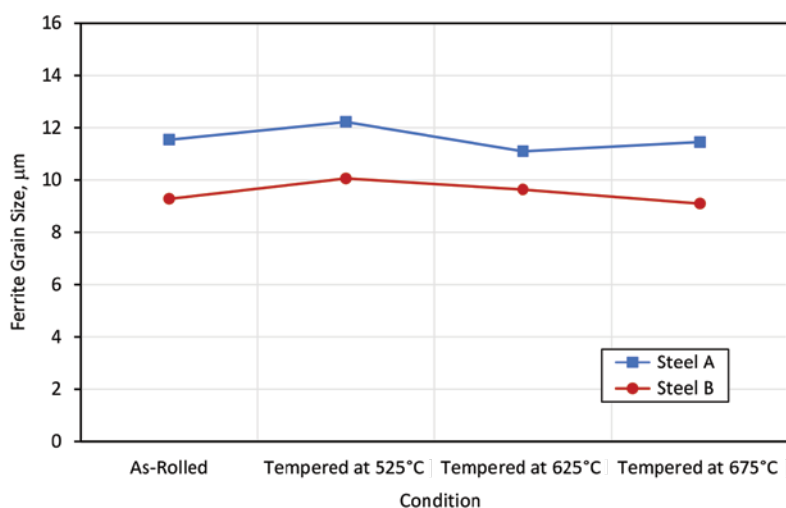


Figure 12

Ferrite grain sizes of Steels A and B.



during the tempering treatments, resulting in an increase in the yield strength of the steel. In addition, tempering of the M/A phase in the microstructure of Steel B may have also contributed to the higher yield strengths obtained in the tempered conditions. Tempering of M/A is usually accompanied by an increase in the yield strength and a slight decrease in the UTS of a steel with M/A in its microstructure.^{6,9} This negative effect of tempering M/A on UTS may have counterbalanced the positive effect of

precipitation on UTS, resulting in a net effect of no change in the UTS of the steel compared to that obtained in the as-rolled condition. However, the positive effects from tempering M/A and precipitation increased the yield strength of the steel by almost 11 ksi. From the tensile results shown in Fig. 4, it appears that the strength of the steel may have peaked out in the tempering condition of 675°C x 15 minutes. Either increasing the tempering temperature or time could result in precipitate coarsening, which will lower the strength of the steel.

Steel C contains large amounts of Nb and V, which was designed to utilize the strengthening mechanisms of grain refinement and precipitation. This steel was produced to a coil at the SSAB Iowa mill, then processed to CTL plates at a SSAB CTL line. A controlled rolling + accelerated cooling + coiling practice was employed for the production of this steel. Austenite conditioning was done through rolling below the T_R temperature (recrystallization stop temperature). The pancaked austenite grain structure provided many nucleation sites for the austenite-to-ferrite transformation, resulting in a fine grain structure after cooling. The in-line accelerated cooling with a moderate cooling rate produced a granular bainite microstructure. The slow coil cooling allowed precipitation to take place. As shown in Figs. 14 and 15, numerous fine Nb and/or Nb-V-rich precipitates were observed in this steel. It appears that the number density of precipitates in the tempered sample (Fig. 15) is higher than that in the as-rolled sample (Fig. 14), indicating that tempering introduced more fine precipitates in the steel. As shown in Fig. 11, the microstructure of Steel C consists of granular bainite with fine dispersed M/A islands. The strength-

ening mechanisms and tempering responses of this steel are very complicated. Similar to Steel B, tempering of M/A would increase the yield strength of the steel. However, tempering of bainite would decrease the yield strength of the steel due to the loss of dislocations. Meanwhile, introduction of new fine precipitates in the microstructure by tempering would increase the yield strength of the steel. Overall, the net effect of these multiple contributors to the

strength is an increase in the yield strength for the samples tempered at 525 and 625°C. For the sample tempered at 675°C, the yield strength dropped back to the level of the as-rolled sample. This suggests that coarsening of precipitates may have taken place at this temperature. Precipitate coarsening together with a more drastic loss of dislocations at this temperature may have outweighed the contribution from tempering the M/A phase. Nevertheless, the tensile properties and toughness of the steel tempered at this temperature are comparable to those in the as-rolled condition.

Summary

Tempering response of three low-carbon microalloyed steels was studied. The results obtained from these steels are summarized as follows:

- For Steel A, the yield strength of the steel was not affected by the tempering treatment. However, the UTS of the steel was decreased by tempering at higher temperatures (625 and 675°C). For Steel B, the yield strength was increased by the tempering treatment significantly, while the UTS of the steel was not affected by the tempering treatment. For Steel C, it appears that the yield strength of the steel increased slightly by tempering at 525 and 625°C, then dropped back to the same level as that of the as-rolled when the steel was tempered at 675°C. The UTS of the steel was not significantly affected by the tempering treatment.
- For Steel A, the toughness of the steel was not altered by tempering at 525 and 625°C. However, the toughness of the steel improved significantly by

Figure 13

Precipitation in Steel B (as-rolled).

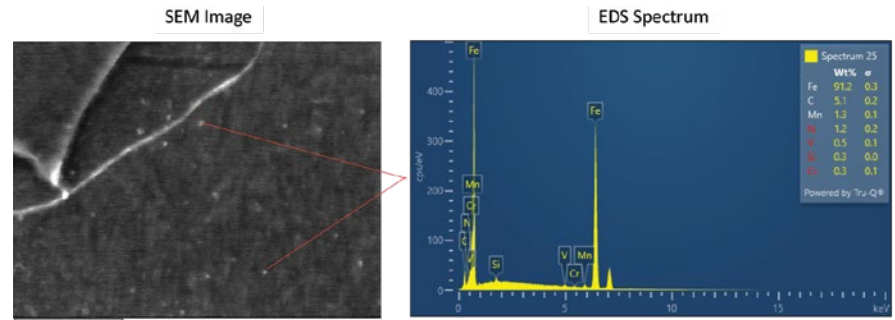


Figure 14

Precipitation in Steel C (as-rolled).

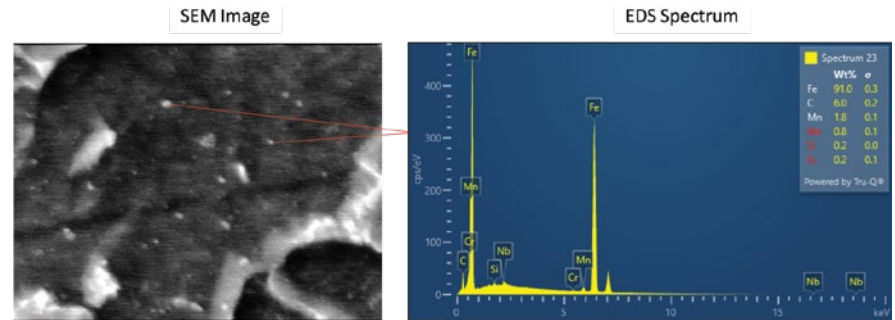
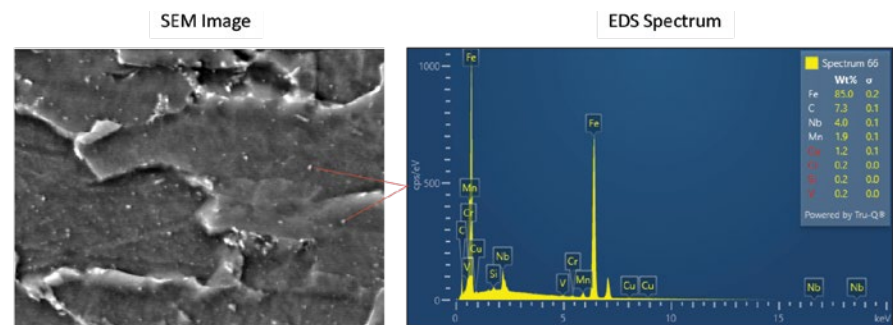


Figure 15

Precipitation in Steel C (tempered at 625°C).



tempering at 675°C. For Steel B, the toughness of the steel improved by tempering at all three temperatures (525, 625 and 675°C). It appears that the toughness of Steel C was not significantly affected by the tempering treatments.

- The microstructures of Steel A consist of polygonal ferrite and pearlite in all four conditions, although pearlite in the sample tempered at 675°C was fully spheroidized. This change in microstructure caused a drop in the tensile strength of the steel but improved the toughness performance of the steel significantly. In the as-rolled condition, the microstructure of Steel B consists of polygonal ferrite and pearlite with a small amount of M/A constituent. In the tempered conditions, the M/A phase in the microstructures of this steel was tempered. Pearlite in the microstructure of the sample tempered at 675°C appears to be spheroidized. For Steel C, the microstructures of the as-rolled samples, tempered at 525 and 625°C, consist primarily of granular bainite with some uniformly dispersed M/A islands. It appears that M/A in the 675°C tempered sample mostly decomposed to ferrite and carbide.
- V-rich particles were observed in Steel B. Although V-rich particles are present in the as-rolled sample, the tempering treatments may have introduced additional VN and/or VC precipitates, which increased the yield strength of the steel. Similarly, additional NbC and/or (Nb-V)C precipitates may have also been introduced in Steel C by tempering, resulting in the higher yield strengths obtained from the samples tempered at 525 and 625°C.

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