HIGH TEMPERATURE TEMPERING BEHAVIOR OF MARTENSITE DURING AHSS MANUFACTURING

Evgueni Poliak¹, Olga Girina¹, Pavan Venkatasurya¹, Damon Panahi¹

¹ArcelorMittal Global Research and Development, 3001 E. Columbus Dr., East Chicago, 46312USA

During manufacturing of highly hardenable AHSS undesirable martensite readily forms after hot rolling, welding, etc., causing poor cold rollability, thickness inconsistency, deteriorated strip shape, weld breaks, etc. To soften the material prior to downstream processing, high temperature tempering with short heating and cooling cycles is often needed. With rapid heating and cooling, martensite from fine grain austenite has mixed lath and plate morphology while coarse grain austenite produces predominantly lath martensite. In latter case the precipitation during tempering occurs at low angle lath boundaries through fast nucleation and sluggish growth and recovery of martensite is faster. With plate martensite the precipitation occurs at high angle boundaries proceeding through coarsening of carbides. Rapid heating and soaking can lead to partial reaustenitization and formation of fresh martensite during cooling causing abnormal tempering and negating softening.

Keywords: rapid heating, quenching, tempering, austenite grains, martensite morphology

Introduction

Current automotive designs drive the development of advanced high strength steels (AHSS) that combine high strength and excellent ductility. One way to achieve unique properties is to utilize quenching and partitioning (Q&P) process [1, 2] for steels with C and Si concentrations higher than in other automotive grades, also in combination with other hardenability enhancing elements. High carbon (> 0.3 wt. %) significantly affects weldability during manufacturing of both steel products and automobile parts. Moreover, because of chemistry specifics martensite readily forms in HAZ causing embrittlement. Detrimental martensite can also form during steel processing (e.g., in hot rolling, continuous annealing, etc.). This calls for necessity of various tempering operations, most importantly, post-welding heat treatment, that have to be short to prevent interruption of processing streamline and productivity loss. This implies rapid heating and cooling. In the present paper, the relationship between microstructure and properties after high temperature tempering with rapid reheating of fully martensitic steel with high C and Si contents are evaluated.

Experimental

The present study employed 0.4 C-2.0 Si steel with Mn, Cr and Mo additions. Laboratory cast 50 kg ingot was reheated at 1250°C and hot rolled in laboratory mill to 4 mm thick plates in two stages with the finishing temperature of about 860°C. Coiling was simulated by placing the plates into the furnace preheated to 650°C and slow cooling. Hot rolled plates were cut to 20 x 20 mm blanks that were austenitized in salt pot at 900°C for 30 sec, 90 min and 270 min. Heating rate in salt pot was estimated to be ~ 40°C/sec. After annealing the blanks were quenched in water. The asquenched blanks were tempered in salt pot at 600 – 750°C for 30 – 600 sec followed by water quenching to simulate fast cooling in welding. Heating rate in tempering was estimated to be also ~ 40°C/sec. Even after shortest soaking at 900°C, fully martensitic microstructure was observed in

quenched blanks indicating complete austenitization. Dilatometry of 4 x 4 x10 mm specimens cut from austenitized and quenched blanks was carried out with Bähr DIL 805L dilatometer with nitrogen as a cooling medium. The A_{c1} and A_{c3} temperatures in heating at ~ 40°C/sec were found to be around 785 and 850°C, respectively, and were practically the same for both hot rolled and quenched specimens. Microstructure was analyzed with scanning electron microscope JEOL-JSM-6060 after polishing and etching in 2% Nital solution and with transmission electron microscope JEOL 2100 TEM at 200 kV using thin foils. EBSD analysis was carried out with JEOL-JSM-7600F scanning electron microscope. Standard hardness tests were done on processed specimens.

Results and Discussion

The microstructure of hot rolled steel is illustrated in Fig. 1. As expected, the hot rolled material possessed ferrite-pearlite microstructure.



Fig. 1. Microstructure of hot rolled steel

Initial quenched martensitic microstructure

The maximum temperature attainable in the salt pot employed in this work is 900°C. This temperature can be much lower than that in a welding cycle. Long annealing times at 900°C were used to account for possible austenite grain growth during welding with rapid heating to higher temperatures and short soaking. Increase in soaking time from 30 sec, which is a typical duration of welding cycles, to 270 min produces significant growth of austenite grains (Fig. 2) equivalent to 30 sec annealing at ~ 1200°C. This temperature is a reasonable estimate of HAZ temperature in welding of 4 mm thick alloyed steel sheets. Grain growth could be slowed down by possible segregations of Mn and Mo at austenite grain boundaries.



Fig. 2. Martensite in samples quenched after austenitization for 30 sec (*a*), 90 min (*b*), 270 min (*c*) Martensite after quenching of austenitized blanks was sensitive to prior austenite grain size, i.e., to the time of austenitization at 900°C. In general, martensite in steels is known to be of two major

morphological types, lath and plate, distinguished by the shape of individual martensitic crystals. At higher carbon content (> 0.6 wt.%), the amount of plate martensite increases at the expense of lath martensite accompanied with decreasing M_s temperature. At lower (< 0.6 wt.%) carbon martensite is mostly of the lath type [3 – 8]. High alloyed martensite in high carbon steels can exhibit mixed morphology largely due to non-homogeneity of austenite [7]. In this work, martensite obtained had mixed morphology, mostly of the lath type with islands of plate martensite, Figs. 3 and 4. Martensite laths of about 1 µm wide and with the same or close habit planes were grouped into packets and blocks. Each packet can be considered as a group of laths with same crystallographic orientation [9].



Fig.3. Martensite morphology: austenitization at 900°C for 30 sec and 270 min, water quench

A THE AND A	Boundaries: Rotation Angle			n Angle	900 °C. 30s	
		Min	Max	Fraction	Number	Length
3 Chiller The The		2°	5*	0.000	35	1.01 microns
the second second second		6°	10°	0.005	507	14.64 microns
EAR man Aller Aller		11°	15°	0.004	441	12.73 microns
CHAR . PROVIDE T		16°	25°	0.006	558	16.11 microns
A PARTY AL		26°	35°	0.028	2875	82.99 microns
NA AND		36°	45°	0.017	1733	50.03 microns
All and Million and		46°	55°	0.033	3313	95.64 microns
and the first for the second		56°	65°	0.048	4848	139.95 microns
A PHERONE TOPS AND STOR		66°	75°	0.028	2860	82.56 microns
AND SAME STORE SAME	_	76°	85°	0.047	4794	138.39 microns
LA A CANADA COMMENT AND LA		86°	95°	0.751	75986	2.19 mm
Bum Bum But		96°	180°	0.001	121	3.49 microns
		101.001V	122-2121		-900	°C 270min
A THE HALL	Boun	daries	Rotatio	n Angle	900	°C. 270min
	Bound	daries Min	Rotatio	n Angle Fraction	900 Number	°C. 270min
	Bound	daries Min 2°	Rotatio	Fraction	900 Number 56	°C. 270min Length 1.62 microns
	Bound	daries Min 2° 6°	Rotatio Max 5° 10°	n Angle Fraction 0.000 0.007	900 Number 56 908	C. 270min Length 1.62 microns 26.21 microns 19.20 microns
	Bound	daries: <u>Min</u> 2° 6° 11°	Rotatio Max 5° 10° 15°	n Angle Fraction 0.000 0.007 0.005	900 Number 56 908 634 917	C. 270min Length 1.62 microns 26.21 microns 18.30 microns 23 59 microns
	Bound	daries Min 2° 6° 11° 16° 26°	Rotatio Max 5° 10° 15° 25°	n Angle Fraction 0.000 0.007 0.005 0.006 0.017	900 Number 56 908 634 817 2092	OC. 270min Length 1.62 microns 26.21 microns 18.30 microns 23.58 microns 0.10 microns
	Bound	daries <u>Min</u> 2° 6° 11° 16° 26° 36°	Rotatio Max 5° 10° 15° 25° 35° 45°	n Angle Fraction 0.000 0.007 0.005 0.006 0.017 0.016	900 Number 56 908 634 817 2082 2022	C. 270min Length 1.62 microns 26.21 microns 18.30 microns 23.58 microns 60.10 microns 58.37 microns
	Bound	daries: <u>Min</u> 2° 6° 11° 16° 26° 36° 46°	Rotatio <u>Max</u> 5° 10° 15° 25° 35° 45° 55°	n Angle <u>Fraction</u> 0.000 0.007 0.005 0.006 0.017 0.016 0.031	900 Number 56 908 634 817 2082 2022 3907	•C. 270min Length 1.62 microns 26.21 microns 23.58 microns 60.10 microns 58.37 microns 58.37 microns
	Bound	daries <u>Min</u> 2° 6° 11° 16° 26° 36° 46° 56°	Rotatio Max 5° 10° 15° 25° 35° 45° 55° 65°	n Angle <u>Fraction</u> 0.000 0.007 0.005 0.006 0.017 0.016 0.031 0.103	9000 Number 56 908 634 817 2082 2022 3907 13033	°C. 270min Length 1.62 microns 26.21 microns 18.30 microns 23.58 microns 50.10 microns 58.37 microns 112.79 microns 376.23 microns
	Bound	daries: <u>Min</u> 2° 6° 11° 16° 26° 36° 46° 56° 66°	Rotatio Max 5° 10° 15° 25° 35° 45° 55° 65° 75°	n Angle Fraction 0.000 0.007 0.005 0.006 0.017 0.016 0.031 0.103 0.036	900 56 908 634 817 2082 2022 3907 13033 4538	°C. 270min Length 1.62 microns 26.21 microns 28.30 microns 58.37 microns 58.37 microns 376.23 microns 376.23 microns
	Bound	daries: <u>Min</u> 2° 6° 11° 16° 26° 36° 46° 56° 66° 76°	Rotatio Max 5° 10° 15° 25° 35° 45° 55° 65° 75° 85°	n Angle Fraction 0.000 0.007 0.005 0.006 0.017 0.016 0.031 0.103 0.036 0.036	900 56 908 634 817 2082 2022 3907 13033 4538 4493	°C. 270min Length 1.62 microns 26.21 microns 23.58 microns 58.37 microns 58.37 microns 376.23 microns 131.00 microns 130.00 microns
	Bound	daries: <u>Min</u> 2° 6° 11° 16° 26° 36° 46° 56° 66° 76° 86°	Rotation Max 5° 10° 15° 25° 35° 45° 55° 65° 75° 85° 95°	n Angle Fraction 0.000 0.007 0.005 0.005 0.017 0.016 0.031 0.103 0.036 0.036 0.704	9000 Number 56 908 634 817 2082 2022 3907 13033 4538 4493 88749	•C. 270min Length 1.62 microns 26.21 microns 23.58 microns 23.58 microns 58.37 microns 112.79 microns 376.23 microns 13.100 microns 129.70 microns 2.56 mm

Fig.4. EBSD map of martensite morphology after austenitization for 30 sec and 270 min

With longer austenitization and hence with coarser austenite grains the volume fraction of plate martensite decreased, Fig. 2. TEM and EBSD (Figs. 3 and 4) confirm predominantly lath martensite morphology in case of coarse austenite grains, while fine grain austenite transformed into martensite with higher volume fraction of plate type. Moreover, the M_s temperature detected by dilatometry increases from 250°C for fine grain austenite to 320°C for coarse grain austenite, which is consistent with prevailing martensite morphology. This may seem counterintuitive as it is known that austenite grain coarsening can lower the M_s temperature [10]. However, since carbon content in all specimens remained the same, the "abnormal" variations in martensite morphology and M_s with prior austenite grain size can be attributed to non-homogeneous distribution of carbon between austenite grain boundaries and grain bulk during rapid heating. At short austenitization,

carbon is primarily in austenite grain interior due to insufficient time for diffusion to grain boundaries. At long austenitization carbon diffuses to austenite grain boundaries; this is enhanced by possible grain boundary segregations of Mn and Mo that have substantial affinities to carbon. As the result, the bulk average volume concentration of carbon in fine grain austenite is higher than that in coarse grain austenite which is reflected by predominantly plate martensite morphology and lower M_s temperature in the former case.

Hardness of martensite varied with austenitization time, i.e., with prior austenite grain size. Extending the duration of austenitization from 30 sec to 270 min and austenite grain growth reduced hardness of quenched steel from 53 to 40 HRc. This is consistent with M_s variations: lower hardness reflects higher extent of self-tempering during quenching and also indirectly confirms carbon redistribution between austenite grain boundaries and grain bulk at longer austenitization.

Effect of Tempering on Microstructure and Hardness

Tempering of iron-carbon martensite during relatively slow heating is quite complex and is generally known to progress in several consecutive and overlapping stages [11], all of which are controlled by diffusion enhanced in the presence of transformation induced lattice defects. Rapid heating suppresses diffusion. When quenched steel is quickly reheated to high tempering temperatures some of the low temperature tempering stages could be either impeded and shortened or accelerated, e.g., clustering of carbon at dislocations, precipitation of transient carbides and decomposition of retained austenite. Prior austenite grain size that largely determines carbon distribution between grain boundaries and grain bulk can also play a role different from that in tempering with slow heating The overall tempering kinetics, microstructure and resultant mechanical properties (hardness) that often serve to judge the efficiency of heat treatment [12] should differ from those in low heating rate tempering.

Upon tempering at 600°C (Fig. 5) the precipitation of carbides (alloyed cementite) slightly decorating prior austenite grain boundaries was observed already after short tempering. This can be linked to relatively slow growth of austenite grains during fast austenitization and to variations in martensite morphology due to redistribution of carbon between grain boundaries and bulk: migration of austenite grain boundaries was slowed down by segregated Mn and Mo, which were subsequently consumed by carbides precipitating during tempering predominantly at prior austenite grain boundaries. With coarser austenite grains fine precipitates were seen mostly along the boundaries of martensite laths and packets which could indicate higher bulk homogeneity of austenite in carbon. Longer tempering at 600°C led to coarsening of precipitates mostly located along prior austenite grain boundaries in fine grain austenite while in coarse grain austenite further precipitation of small cementite particles along numerous lath grain boundaries was observed.

This effect of austenite grain size and martensite morphology on cementite precipitation was also visible after tempering at higher temperatures and longer times. Cementite first precipitated along the boundaries of prior austenite grains, then at the boundaries of blocks, laths and plates, and then, with increasing tempering time and temperature, precipitation was forced inside martensite blocks, lath and plates. With smaller austenite grains and higher volume fraction of plate martensite the recovery of martensite proceeded through growth and coarsening of carbides while with coarser austenite grains and predominantly lath martensite morphology the recovery occurred by progressing nucleation of precipitates at laths boundaries. With fine austenite grains higher level of hardness was observed after tempering at temperatures of 600 and 650°C. Hardness decreases with longer tempering regardless of austenite grain size. Specimens with coarse prior austenite grains consistently had lowest hardness weakly dependent on tempering time. (Figs. 5 and 6).



Fig.5. Effect of prior austenite grain size and time of tempering at 600°C on microstructure and hardness. Captions in micrographs indicate time of austenitization at 900°C / time of tempering



Fig.6. Effect of austenitization conditions and time of tempering at 650°C on microstructure and hardness

Raising the tempering temperature accelerated precipitation and weakened the effect of martensite morphology regardless of tempering time (Fig. 7). These observations show that precipitation of carbides at low angle boundaries of martensite laths followed the precipitation at prior austenite grain boundaries as the tempering temperature or time were increased. Size of precipitates is finer within laths and at lath boundaries than at high angle austenite grain boundaries. With finer austenite grains and predominantly plate martensite morphology the precipitation occurred mostly

at austenite grain boundaries as course particles due to their fast growth. Some small particles precipitate inside martensite plates with a lower extent of recovery (Fig. 8). With coarser austenite grains and predominantly lath martensite morphology the precipitation was initiated at lath boundaries mostly through progressive nucleation of new particles and their sluggish growth which resulted in higher extent of martensite recovery. With intermediate austenite grain size and mixed martensite morphology the two precipitation mechanisms operate concurrently.



Fig.7. Effect of austenitization conditions and time of tempering at 700°C on microstructure and hardness



Fig. 8. Precipitates in martensite with lath (*a*: austenitization for 30 sec) and mixed (*b*: austenitization for 270 min) morphology after tempering at 700°C for 10min

Microstructure after long tempering at 750°C revealed recovered martensite with large precipitates in case of coarse austenite grains, while the case of fine austenite grains the martensite was less recovered and precipitates were smaller. This difference in the extent of martensite recovery can be related to the difference in morphology of the initial martensite. Plate martensite recovered later due to the delayed carbide precipitation compared to lath martensite, Fig. 9.



Fig. 9. Effect of austenitization conditions and time of tempering at 750°C on microstructure and hardness

After tempering above 700°C for more than 30 sec the presence of newly formed martensite was detected indicating that the tempering temperature had exceeded the actual A_{cl} temperature. This is schematically explained in Fig. 10. Rapid heating to 750°C followed by holding at this temperature is equivalent to lowering the heating rate. The longer the holding the lower the effective heating rate and hence the lower the A_{cl} temperature. At certain time of tempering at 750°C the actual A_{cl} temperature can therefore drop below 750°C causing re-austenitization and formation of "new" martensite in subsequent quenching.



Fig.10. Variation of A_{cl} temperature in tempering thermal cycle

After tempering at higher temperatures (700 and 750°C) the effect of austenite grain size on tempering kinetics and variation of hardness becomes somewhat more complicated. During tempering at 700°C the decrease in hardness with time is more pronounced in case of the smallest austenite grain size. This trend is seen upon raising the tempering temperature to 750°C but the relationship between hardness and tempering time changes: as the tempering time is increased beyond 300 sec the hardness increases as the consequence of re-austenitization during long soaking

at 750°C and subsequent formation of new martensite during quenching. Obviously, such tempering cannot be viable for any processing.

Conclusions

1. With rapid reheating, an increase in austenite grain size in high C-Si steel results in the different morphology of martensite. Fine prior austenite grains can be characterized by a mixture of lath and plate types of martensite while in case of coarse austenite grains the martensite is predominantly of the lath morphology.

2. Precipitation kinetics during tempering of fully martensitic structure at 600 – 700°C depends on martensite morphology. With predominantly lath type martensite the precipitation occurs at low angle lath boundaries mostly through fast nucleation and sluggish growth. With predominantly plate type morphology of martensite the precipitation takes place at high angle prior austenite grain boundaries and plates proceeding through coarsening of carbides with increasing tempering time and temperature.

3. Recovery of martensite is more complete in case of coarse austenite grains due to numerous precipitates of smaller size at lath boundaries which in turn results in a lower hardness.

References

1. J. Speer, D.K. Matlock, B.C. De Cooman, and J.G. Schroth, "Carbon partitioning into austenite after martensite transformation". *Acta Materialia*, 51, (2003), p. 2611

2. D.V. Edmond, K. He, F.C. Rizzo, and B.C. De Cooman, "Quenching and partitioning martensite — A novel steel heat treatment", *Material Science and Engineering A*, (2006), p. 25.

 V.H. Baltazar Hernandez, S.S. Nayak, and Y. Zhou, "Tempering of martensite in dual-phase steels and its effects on softening behavior", *Metallurgical and Materials Transactions A*, (2011), p. 3115
A.M. Sherman, G.T. Eldis, and M.Cohen, "The Aging and Tempering of Iron-Nickel-Carbon

Martensites", Metallurgical Transactions A, 14A, (1983), p. 995.

5. G.B. Olson and W.S. Owen, *Martensite* (ASM, 1999), p. 331.

6. G. Krauss, Steels: Processing, Structure, and Performance (ASM Int., 2015), p.682

7. G. Krauss and A.R. Marder, "The morphology of martensite in iron alloys", *Trans.ASM* 62, (1971), p.651

8. S. Morita, H. Tanaka, R. Konishi, and T. Furuhara, "The morphology and crystallography of lath martensite in alloy steels", *Acta Materialia*, 51, (2003), p.1789.

9. S. Morito, X. Huang, T. Furuhara, and T. Maki "The morphology and crystallography of lath martensite in alloy steels", *Acta Materialia*, 54, (2006), p.5323

10. Hong-Seok Yang and H.K.D.H. Bhadeshia, "Austenite grain size and the martensite-start temperature", *Scripta Materialia* 60, (2009), p.493.

11. G.R. Speich and W.C. Leslie, "Tempering of steel", *Metallurgical Transactions*, (1972), p.1043. 12. G. Gao, H. Zhang, X. Gui, and Z. Tan, "Tempering behavior of ductile 1700 MPa Mn-Si-Cr-C steel treated by Q&P process incorporating bainite formation", *Material Science and Technology*, (2014), p.1.